

Process Guide

for

ORMECON™ CSN FF

ORMECON™ CSN FF-W

Immersion Tin

Version 3.0

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Process Guide for ORMECON™ CSN FF / ORMECON™ CSN FF-W

1.1 Introduction

ORMECON™ CSN FF and ORMECON™ CSN FF-W are immersion tin surface finish processes for copper surfaces. They meet all requirements for a modern and environmentally friendly lead-free PCB surface finish:

- absolutely planar surface for SMD technology
- long shelf-life of coated bare boards (> 12 months with proper thickness)
- multiple soldering operations, also with intermediate storage
- easy to run and to monitor
- suitable for lead-free technology (lead-free solders are fully compatible)

Compared to other immersion tin processes ORMECON™ CSN FF and CSN FF-W also offer: Significant reduction of diffusion of the tin deposit:

- Advanced oxidation protection
- Higher temperature resistance for lead-free finishing
- Pure tin deposit, even with high copper load of the working solution
- Whisker-reducing effect due to Organic Metal Pre-Dip (further advanced for ORMECON™ CSN FF-W)

ORMECON™ CSN FF and ORMECON™ CSN FF-W increase the process safety and broaden the operating window, both for PCB manufacturing and assembling.

The aim of this Process Guide is to make the responsible engineers and operators familiar with the processes, the relevant parameters and actions necessary, to keep the processes and the equipment in excellent operating conditions.

The current Process Guide edition will be continuously adjusted for your benefit, so suggestions and recommendations for improvements are always appreciated.

Remark:

ORMECON™ CSN FF and ORMECON™ CSN FF-W are only suitable for pure copper surfaces. Exposure of other metals to the process solutions could lead to irreversible contamination of the baths and /or interfere with the tin deposit, leading to color changes and performance issues.

ORMECON™ CSN FF and ORMECON™ CSN FF-W are exclusively suitable for FR-4, PTFE and PD base materials. The compatibility with other base materials should be thoroughly checked prior to use, preferably with separate process chemicals, e.g. in a beaker.

CEM-1 material should not be used under any circumstances, because it leads to irreversible destruction of the tin bath and a dark, thinner tin deposit with poor solderability. Please turn to your local Ormecon International representative, if CEM-1 material should be processed.



Make yourself familiar with this Process Guide and the features of the processes, before using them.



1.2 Process Description and Control Parameter Survey

ORMECON™ CSN FF and FF-W represent the straightforward development of the world-wide successfully used ORMECON™ CSN, UNICRON™ and OMIKRON™ immersion tin processes. They meet increased demands and requirements of PCB manufacturers, assemblers and OEMs for a high quality surface finish with a high reliability, multiple solderability, perfect compatibility with lead-free technologies, long shelf-life and reduced tin whisker formation.

Compared to the above mentioned, well known processes of the Ormecon International group, ORMECON™ CSN FF and ORMECON™ CSN FF-W offer

- higher deposition speed
- improved solderability
- improved solder mask compatibility
- improved rinsing ability due to lower density and viscosity of the tin solution
- improved bath stability

ORMECON™ CSN FF Process

Product	Function	Process Parameter Temp. / Process Time	Control Parameter
ACL 7001	Acid Cleaner (General removal of grease, oxides, residues, etc); optional	40°C – 50°C approx. 1 – 3 min.	Specific Gravity, Acidity
Rinse		RT* / approx. 1 min.	Conductivity
MET 7000	Micro Etch (Copper oxide removal and copper activation)	28°C – 42°C; 1 – 2 min.	Specific Gravity, Hydrogen Peroxide content (H ₂ O ₂), Acidity (H ₂ SO ₄), copper content, etch rate, color
Rinse		RT* / approx. 1 min.	Conductivity
OMP 7000	Organic Metal Pre-Dip (Copper preparation for tin deposition)	15°C – 30°C; approx. 1 min.	Org. Metal content, pH, color, optical appearance
CSN 7004	Immersion Tin	Vertical max. 65°C, Horizontal max. 73°C (min. 40°C) 7 – 27 minutes	Specific Gravity, Acidity, Tin content, Copper content,
Warm Rinse	Quick and complete removal of all residues	50°C – 60°C 1.5 – 5 min.	Conductivity
DI Water Rinse	For minimizing ionic contamination	50°C – 60°C 3 – 5 min.	Conductivity
Dryer			

ORMECON™ CSN FF-W Process

Product	Function	Process Parameter Temp. / Process Time	Control Parameter
ACL 7001	Acid Cleaner (General removal of grease, oxides, residues, etc); optional	40°C – 50°C approx. 1 – 3 min.	Specific Gravity, Acidity
Rinse		RT* / approx. 1 min.	Conductivity
MET 7000	Micro Etch (Copper oxide removal and copper activation)	28°C – 42°C; 1 – 2 min.	Specific Gravity, Hydrogen Peroxide content (H ₂ O ₂), Acidity (H ₂ SO ₄), copper content, etch rate, color
Rinse		RT* / approx. 1 min.	Conductivity
OMP 7001	Whisker-reducing, Organic Metal Pre-Dip (silver deposition prior to tin application)	35°C – 45°C; approx. 1 min.	Ag content, Org. Metal content, pH, color, optical appearance
CSN 7004	Immersion Tin	Vertical max. 65°C, Horizontal max. 73°C (min. 40°C) 7 – 27 minutes	Specific Gravity, Acidity, Tin content, Copper content
Warm Rinse	Quick and complete removal of all residues	25°C – 35°C 1.5 – 5 min.	Conductivity
DI Water Rinse	For minimizing ionic contamination	25°C – 35°C 3 – 5 min.	Conductivity
Dryer			

*room temperature

Acid Cleaner 7001 ACL 7001

ACL 7001 is an acidic copper cleaner with special degreasing properties, and is designed for pre-treatment use in the ORMECON™ CSN FF and FF-W processes. It cleans the circuit board surface from grease and oils, developer residues after solder mask application and other impurities. ACL 7001 is made from 12.5% ACL 7001 C (concentrate) in DI water.

Micro Etch MET 7000

The second pre-treatment step MET 7000 is an acidic copper cleaner, based on hydrogen peroxide and sulfuric acid, stabilized with MET 7000 S. It is perfectly suitable for use prior to the tinning process, because it creates a smooth copper surface, which is important for the topography of the final tin finish. The average etch rate should be 0.8 – 2.4 µm within 1 – 2 minutes.

Organic Metal Pre-Dip OMP 7000

This acidic, aqueous Organic Metal containing dispersion is the key pre-treatment step in the ORMECON™ immersion tin technology. It deposits a very thin (80 nm) Organic Metal layer selectively on the copper surface that prevents copper tarnishing and therefore ensures a homogeneous, spot and stain-free surface finish. It also acts as a catalyst in the immersion tin deposition and leads to a dense, large grain size morphology of the tin deposit. The OMP 7000 bath is made up from 5% OMP 7000 C (concentrate) and 2.5% OMP 7000 B (buffer) in DI water.

Organic Metal Stabilizer OMP 7025

This is a product which is only occasionally used in case of strong foam formation during Pre-Dip operation. It helps to re-disperse settled / agglomerated Organic Metal residues, which have been formed as a result of foaming. It can also be used for tank / module cleaning, because it helps to remove OMP residues from equipment parts and tank walls.

Whisker-reducing Organic Metal Pre-Dip OMP 7001

In addition to the Organic Metal this product contains silver in an aqueous dispersion. Stabilized and catalyzed by the Organic Metal a nano layer of silver (15 – 45 nm) is deposited on the exposed copper surface which provides efficient whisker suppression. OMP 7001 is a 100% ready-to-use product.

Replenishment solution for whisker-reducing Pre-Dip OMP 7001 R

OMP 7001 R is necessary to replace components removed from the OMP 7001 Pre-Dip during operation (by drag out and plating). It is a concentrate containing silver, the Organic Metal and other necessary components. Replenishment depends on the area of PCB's processed. For details see description of replenishment on page 193 OMP 7001 Process Bath.

Immersion Tin solution CSN 7004

The tinning solution CSN 7004 is used to deposit a 0.8 – 1.2 µm thick metal layer on the pre-treated copper surface. The solution is lower in viscosity than other immersion tin baths, and therefore more suitable for fine line and µm vias / blind vias. The CSN 7004 working solution is made up from 9 vol.% CSN 7004-1 + 1 vol.% CSN 7004-2. Mix in drum before using, until all solids are dissolved!

Replenishing solution CSN 7004 R

In order to replace the used components of the tinning bath CSN 7004 R has to be added to the CSN 7004 working solution. CSN 7004 R also consists of two components and has to be mixed in a 9 : 1 vol. ratio prior to use (9 vol.% CSN 7004-R1 + 1 vol.% CSN 7004-R2). The replenishment amount of CSN 7004 R depends on the throughput of the line and replenishes removed tin and other required components. For proper replenishment CSN 7004 R is used in connection with CSN 7004 or CSN 7004 RG. For details see description of replenishment on pages 243 and 244.

Replenishing solution CSN 7004 RG

This is ready-to-use replenishment product that is only necessary after copper has been removed from the CSN 7004 working solution by cooling. Copper precipitates at low temperatures as a copper complex and removes an essential amount of complexing agent and other components from the bath. In order to replace these necessary components CSN 7004 RG is used, in addition to CSN 7004 R and CSN 7004. For details see description of replenishment after copper removal on page 245.

Catalyst for repair of CSN 7004 after local overheating CSN 7004 CAT

CSN 7004 occasionally suffers from local overheating effects, resulting in a loss of performance. Normally a tin bath suffering from degradation because of heat damage has to be dumped and remade. Ormecon International offers a product that can "cure" CSN 7004 working solutions and helps to keep the damaged bath. CSN 7004 CAT is made-up in a 1 : 1 volume ratio from CSN 7004-CAT1 and CSN 7004-CAT2. CSN 7004 CAT is added at 3% to the CSN 7004 tin bath, after mixing. For details see technical data on page 251.

Rinse Aid RAD 7000 C for improved rinsing

RAD 7000 is a rinse made from the biodegradable, basic (pH <7), organic concentrate **RAD 7000 C**. This rinse assists in the removal of residues following the CSN 7004 immersion tin bath and prior to soldering and fusing processes using fluxes. **RAD 7000 C** works without the use of silicone de-foamers and is used in a concentration between 1 and 25%, depending on desired grade of cleanliness. For details see technical data on pages 254 - 261.

For further information please refer to the technical data sheets and/or MSDS of the products.

1.2.1 Preparation of the Line

All line parts in contact with the ORMECON™ CSN FF / ORMECON™ FF-W solutions have to be thoroughly cleaned prior to bath set-up.

Cleaning procedure of new lines

New lines should be cleaned for several hours using the following sequence:

- Rinse all modules / tanks with water
- Fill up all modules / tanks with a 5% sodium hydroxide solution (NaOH) and heat the solution up to 50 – 60°C
- Keep the heated solution circulating in the modules for 12 – 24 h
- Drain the NaOH solution
- Rinse thoroughly with water
- Fill up the modules with 2 – 3 % sulfuric acid (H₂SO₄) and heat the solution to 50 – 60 °C as well
- Keep the heated solution circulating in the modules for 12 – 24 h
- Drain the H₂SO₄ solution
- Rinse thoroughly with water until the pH of the rinsing waters have turned neutral (pH=7)
- Start filling the tanks according to the sequence recommended on page 26.

Cleaning procedure of used lines

Contaminated old lines, that have already contained other solutions, have to be cleaned thoroughly with suitable cleaning chemicals. Etching solutions to remove residues, sludge and solvents are recommended, as well as caustic soda, hydrochloric acid or sulfuric acid solutions in suitable concentrations.

The following procedures are recommended for cleaning prior to using ORMECON™ CSN FF and / or ORMECON™ CSN FF-W, in case

- the line has been used for another process, not immersion tin
- the line has been used for another immersion tin process
- the cleaning is necessary for the maintenance of a line running with ORMECON™ CSN FF / ORMECON™ CSN FF-W

Cleaning of rinsing cascades after pre-treatment steps:

Since the quality and efficiency of the rinses is of major importance for the result of the ORMECON™ CSN FF and ORMECON™ CSN FF-W deposition, their cleaning has to be carried out with utmost care. If residues or sediments are visible in the rinsing modules, a chemical treatment is necessary:

- If the cascades are still operated prior to cleaning and used for an ORMECON™ process, close the water inlet and keep the bypass pipe between the cascades open.
- Drain the rinse waters and clean the sieves and dirt traps.
- Rinse the cascades with a water hose for first cleaning
- Fill up the cascades with water and add sodium hydroxide in order to reach a 5-10% solution.
- Start the circulation of the pumps and the flooding of the immersion trays.
- This first cleaning procedure should be carried out for at least 60 minutes. Dirty rollers have to be cleaned manually.
- Finally drain the sodium hydroxide solution and rinse the cascades until the pH of the rinsing waters has turned neutral (pH=7)
- Fill the cascades with fresh water according to the recommended rinse water quality.
- Adjust the water input with the flow meter, where available.
- Now the rinse is ready for production.

Cleaning of the working bath modules / tanks

(White, green) residues remaining in the line, have to be removed completely prior to starting up the line with a new bath. To achieve this, a 5 % NaOH solution should be circulated at a temperature of 50 °C until all visible residues are dissolved. After discharge of this solution, the module has to be rinsed thoroughly before the line is oxidatively cleaned with a mixture of oxidants, like sodium persulfate (5 kg to 100 l bath volume) or sulfuric acid. After this cleaning step, another rinsing is necessary until pH neutrality is reached. The filter cartridges have to be changed with each new set-up. The entire pump and filter system must be free of any remaining residue or cleaning solution.

For a complete cleaning process a time frame of 24 - 48 hours should be planned.

!	<p>OMP 7000 and OMP 7001 contain the Organic Metal in an aqueous dispersion. It is sensitive to pH and foreign particles, so their modules have to be thoroughly cleaned. The operation of OMP 7000 will result in Organic Metal residues on walls, rollers and other parts of equipment. They need to be removed as well as possible, prior to using a new Pre-Dip bath.</p>	!
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!	<p>CSN 7004 is formulated completely differently from other available immersion tin baths, including CSN 7004 of ORMECON™ CSN and OA8 of OMIKRON. So the tin modules have to be cleaned extremely thoroughly, to avoid contamination and irreversible damage to the CSN 7004.</p>	!
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The same applies when changing a used CSN 7004 tin solution because of a new make-up.

Recommended cleaning sequence for modules operated with OMP 7000, OMP 7001 and other process chemicals except for immersion tin (special cleaning procedure):

- If OMP 7000 has been operated it is recommended to add 0.1 – 0.5% of OMP 7075 STAB to the working bath 1 – 2 weeks prior to cleaning. This product will help to re-disperse settled Organic Metal residues and makes cleaning easier.
- If the line still contains chemicals, shut off the machine and cool down the chemicals in the tanks to room temperature, if applicable.
- Take out the chemicals and store them for re-use or waste treatment.
- Rinse the modules with a water hose for first cleaning
- Take the filtration cartridges out of the filtration units, if applicable.
- Fill up the modules with water. Add sodium hydroxide in order to reach a 5-10% solution and fill up the modules to cleaning level.
- Start the circulation of the pumps, the flooding of the immersion trays and the heating. Cleaning temperature should be between 50-60°C
- This first cleaning procedure should be carried out for at least 12 h in order to completely dissolve the encrustation precipitated in the modules.
- Switch off the heating elements 20-30 min prior to draining the cleaning solution to the waste water treatment.



After the alkaline chemical cleaning, the line may look very dirty, brownish; this can be caused by the reaction of some encrustation and the alkaline components.



- Rinse the modules with a water hose and clean the modules manually where it seems to be necessary.
- Clean the module mechanically, if applicable.
- If Organic Metal residues remain, high pressure water jet cleaning is possible.
- Close the outlet valves and refill the module with water and let the water circulate for at least 30 min. while all pumps are running.
- Drain the rinsing water and use the water hose for rinsing out any further residues. Check corners and hidden edges carefully. In horizontal line, lift the rollers and have a look into the modules if there are any residues left. Clean the sieves and dirt traps.
- If there is any sodium hydroxide solution left in the piping a second water rinsing step is necessary.
- When everything is clean and free from sodium hydroxide solution, the second chemical cleaning step can be started. Therefore the outlet valves have to be closed. Afterwards the modules have to be filled up with water and heated up to 40°C.
- Add 2 – 3 % sulfuric acid carefully into the modules and fill up with water to cleaning level.
- Start the circulation of the pumps, the flooding of the immersion tray (frequency controlled pumps on highest frequency) and the heating. Cleaning temperature should be between 50-60°C.
- An OMP 7000 module should be rinsed with sulfuric acid for only 1 –2 hours. For other tanks/modules this cleaning sequence should be carried out for at least 12 h in order to oxidize all residues on the polypropylene walls completely. OMP 7025 STAB (0.1 - 0.5%) could be added to the sulfuric acid to make OMP 7000 tank cleaning easier. OMP 7025 STAB should not be used for OMP 7001 tanks!
- Switch off the heating elements 20-30 min before the cleaning solution is drained to the waste water treatment.
- Rinse the modules with a water hose and clean the modules manually where it seems to be necessary.
- Close the outlet valves and refill the module with water and let the water circulate for at least 30 min with all pumps running.

- Repeat these rinsing steps at least 3-4 times, until the rinsing water reaches a pH value of approx. 6.0 - 7.0.
- The cleaning should be complete now.
- Finally the filtration units have to be equipped with new filtration cartridges, if applicable (not for OMP 7000 and OMP 7001). The entire pump and filter system must be free of any remaining residue or cleaning solution.
- Double check that no filter cartridges are used for the OMP 7000 / OMP 7001 Pre-Dip.
- Before the working solutions can be pumped (back) into the modules, some control and maintenance work should be carried out on the empty modules. Nozzles need to be checked and cleaned, rollers need to be checked, etc.

For cleaning procedures for nozzles please refer to page 20.

Recommended cleaning sequence for modules operated with tin prior to ORMECON™ CSN FF / ORMECON™ CSN FF-W installation:

!	<p>CSN 7004 is formulated completely differently from other available immersion tin baths, including CSN 7001 (and CSN 7001 V2) of ORMECON™ CSN and OA8 of OMIKRON. So the tin modules have to be cleaned extremely thoroughly, to avoid contamination and irreversible damage to the CSN 7004.</p>	!
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- If the line still contains chemicals, shut off the machine and cool the chemicals in the tanks to room temperature.
- Take out the chemicals and store them for re-use or waste treatment.
- Rinse the modules with a water hose for first cleaning
- Take the filtration cartridges out of the filtration units.
- Fill up the modules with water. Add sodium hydroxide in order to reach a 5-10% solution and continue filling the modules to cleaning level.
- Start the circulation of the pumps, the flooding of the immersion trays and the heating. Cleaning temperature should be between 50-60°C
- This first cleaning procedure should be carried out for at least 12 h in order to completely dissolve the encrustation precipitated in the modules.
- Switch off the heating elements 20-30 min prior to draining the cleaning solution to the waste water treatment.

!	<p>After the alkaline chemical cleaning, the line will look very dirty, brownish; this is caused by the reaction of the encrustation especially from the immersion tin solution and the alkaline components.</p>	!
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- Rinse the modules with a water hose and clean the modules manually where it seems to be necessary.
- Close the outlet valves and refill the module with water and let the water circulate for at least 30 min while all pumps are in function.
- Drain the rinsing water and use the water hose for rinsing out any further residues. Check corners and hidden edges carefully. In horizontal lines, lift the rollers and have a look into the modules if there are any residues left. Clean the sieves and dirt traps.
- If there is any sodium hydroxide solution left in the piping a second water rinsing step is necessary.

- If everything is clean and free from sodium hydroxide solution, the second chemical cleaning step can be started. The outlet valves need to be closed. Afterwards modules need to be filled with water and heated to 40°C.
- Add sodium persulfate salt carefully into the modules. As a rule of thumb take 5 kg of sodium persulfate salt for 100 l of water (50 g/L). Finally fill the modules to cleaning level.
- Start the circulation of the pumps, the flooding of the immersion tray (frequency controlled pumps on highest frequency) and the heating. Cleaning temperature should be between 50-60°C.
- This cleaning sequence should be carried out for at least 12 h in order to oxidize the dark residues on the polypropylene walls completely.
- Switch off the heating elements 20-30 min before the cleaning solution is drained to the waste water treatment.
- Rinse the modules with a water hose and clean the modules manually where it seems to be necessary.
- Close the outlet valves and refill the module with water and let the water circulate for at least 30 min with all pumps running.
- Repeat these rinsing steps at least 3-4 times, until the rinsing water reaches a pH value of approx. 6.0 - 7.0.
- The cleaning should be complete now.
- Finally the filtration units have to be equipped with new filtration cartridges. The entire pump and filter system must be free of any remaining residue or cleaning solution.
- Before the working solutions can be pumped (back) into the modules, some control and maintenance work should be carried out on the empty modules. Nozzles need to be checked on blockages from residues, rollers need to be observed, etc.

Changing of filter cartridges in the immersion tin modules

The function of the filtration, especially of the immersion tin working solution, is essential to continuously remove precipitates, e.g. which are formed by the oxidation of the tin (Sn(IV)).

The precipitates have no influence on the plating process, but will cause a sludge formation in the module and other equipment parts. Therefore a regular change of the filter cartridges is essential in order to increase the chemical cleaning intervals of the line.

As a rule of thumb, change the filter cartridges twice a week (based on a 5 day 6 hours operation or 300 - 400 m² / week).

!	<p>If the filter cartridges have to be changed more often, it is very likely that there is an increased air entrapment in the immersion tin solution. The reason for this needs to be determined and located.</p>	!
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A change of the filter cartridges is necessary, if the flow through the filtration unit is significantly reduced or zero. This is an important parameter, which has to be inspected by the operator in every shift. In case the filter cartridges need to be exchanged, please follow the recommended procedure:

- Vertical mode: Switch off pumps connected to the filtering system
- Horizontal mode: Switch the line into operation mode "OFF".
- Before opening the filtration units, depressurize the system by opening the de-aerating valves. No process solution should flow through the piping.
- Exchange filter cartridges.

Nozzle cleaning in the immersion tin modules of horizontal lines

A nozzle cleaning is necessary, if the openings of the pipes get partly blocked with white sludge (Sn(IV) precipitates) and/or other residues. This blocking will be indicated through a lower level in the immersion tray.

The consequences for the process will be a further increase of sludge formation due to local turbulence at the nozzles and an inhomogeneous temperature distribution in the immersion tray because of an insufficient liquid exchange in some areas.

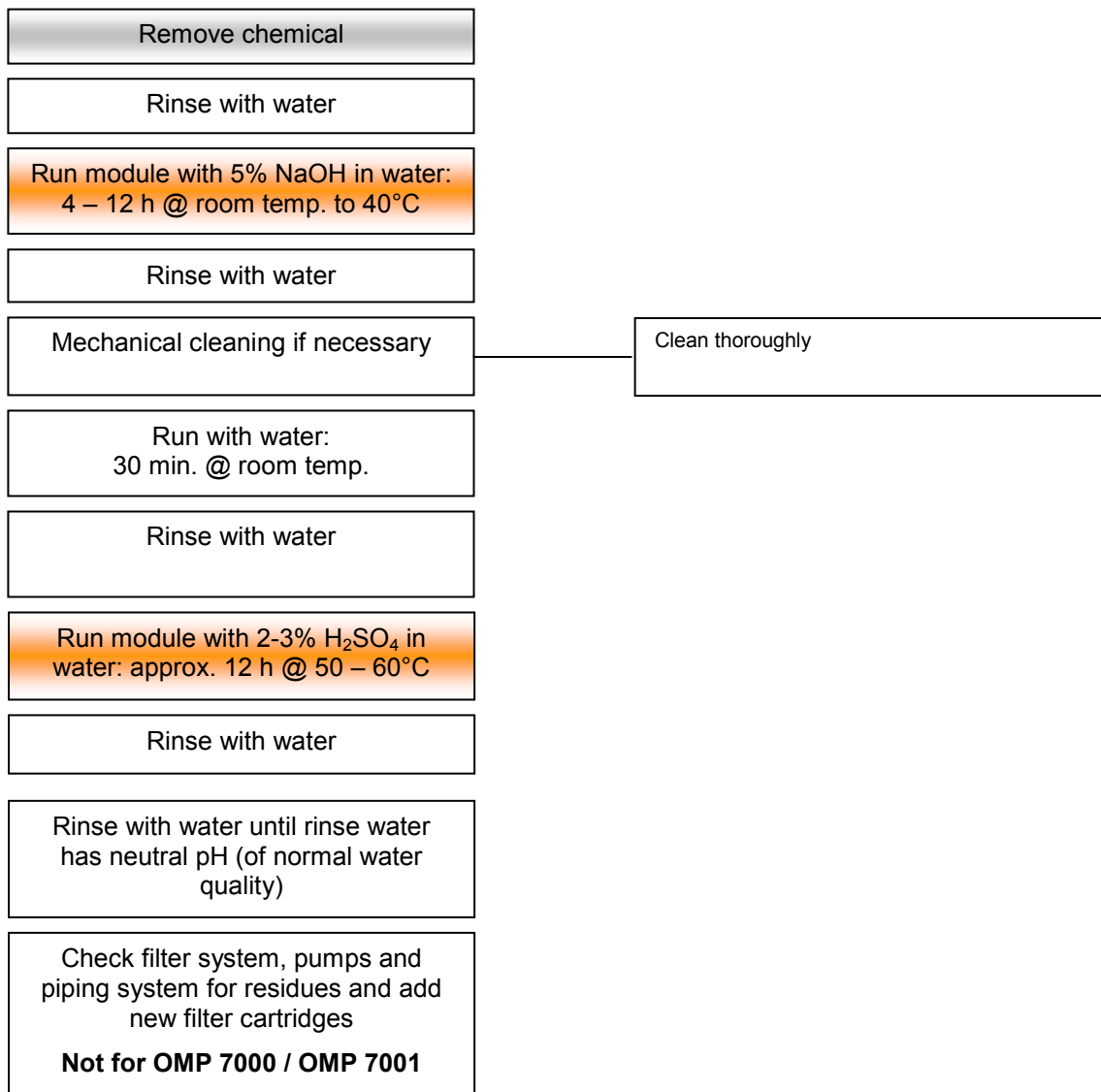
Cleaning procedure for nozzles

In order to change the upper and lower nozzles the line has to be switched into the operation mode "OFF". Remove the pipes and clean in a 5 - 15% sodium hydroxide solution. First soak them for at least 12 h in order to remove the serious encrustation. Rinse the pipes with water until the pH-value of the rinsing water is approx. 6.0 – 7.0. Now they are ready to be reinstalled. A second set of nozzle pipes can be used to replace the pipes immediately after removal.

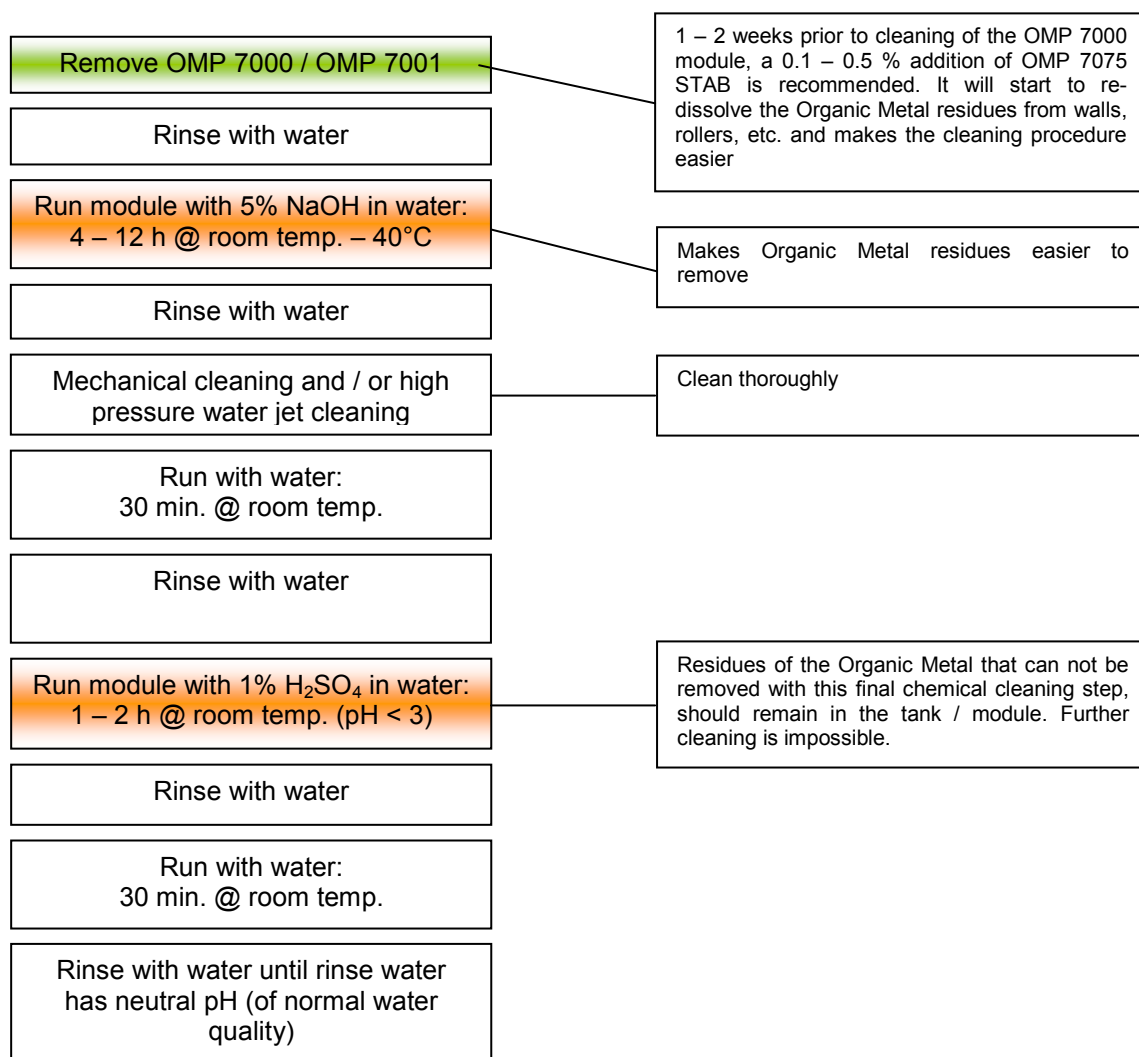
Cleaning Procedure – QUICK GUIDE Cascade rinses

Close water inlet and leave bypass pipe between cascades open
Drain old rinse water and clean sieves and dirt traps
Rinse with water
Fill up the cascade with 5 – 10 % NaOH in water
Start circulation pumps and flooding of immersion trays and run for at least 60 min.
Clean rollers and other parts manually
Drain NaOH solution
Rinse with water until pH has turned neutral (pH of your normal rinse water quality)
Fill cascades with fresh water, according to recommended rinse water quality
Adjust water input with flow meter, where available

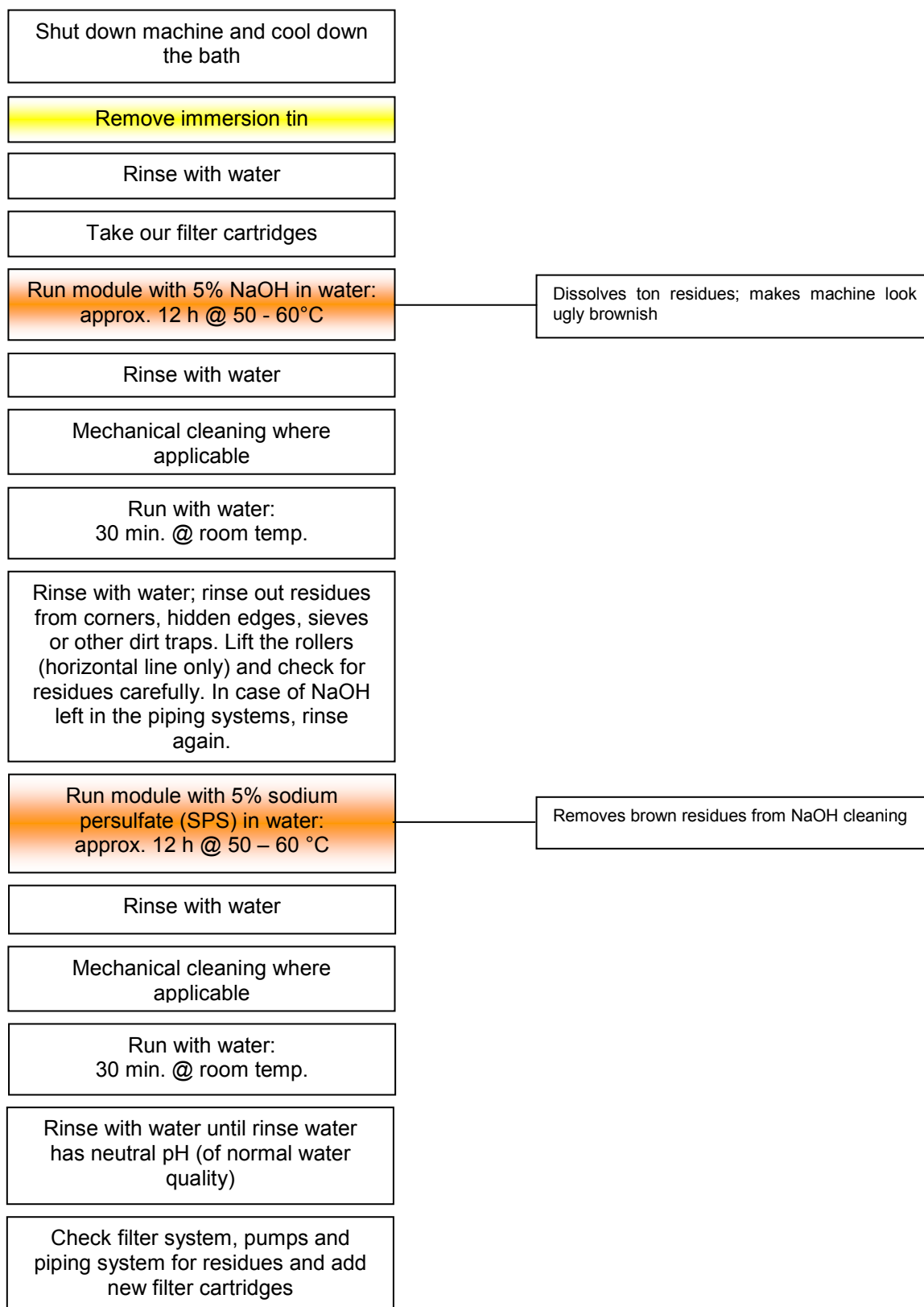
Cleaning Procedure – QUICK GUIDE / Acid Cleaner and Micro Etch



Cleaning Procedure – QUICK GUIDE / OMP 7000 / OMP 7001 Pre-Dip



Cleaning Procedure – QUICK GUIDE / Tin bath



Completion of the chemical cleaning and (re)start of the line

- If there are no visible defects, the modules can be filled with the process solutions. When the tanks are filled with the desired chemicals, switch on the heating, circulation and filtering systems. For a horizontal line get back into the operation mode "Production ". The solutions will now be mixed and heated up till they reach their nominal temperature. During the heat up procedure the filtration units have to be de-aerated.
- Inspect the line for leakage.
- Samples have to be taken from the working baths in order to control if all the analytical parameters are within the limit values. If not, a manual dosing of chemicals will be necessary.
- After the adjustment of the liquid level in the tanks / modules and before running the first production boards, some copper clad base material has to be processed through the line under production conditions. Besides showing the quality of the deposit, this material will help in the horizontal lines to clean the rollers and rolls from impurities and residues.
- If the tin surface is dull and cloudy, or if there are any streaks left on the surface check for possible reasons (also with the help of the Trouble Shooting Guide on page 73 - 76 and the FMEA on page 82) and repeat the procedure.



Production should not be started until the quality of the test material is perfect.



- Finally the test boards for measuring the tin thickness and the solderability tests should be processed. A layout for the solderability test panel can be provided by Ormecon. For details please refer to section *Test run after initial make-up* on page 29.

1.2.2 Bath Set-up and make-up procedures

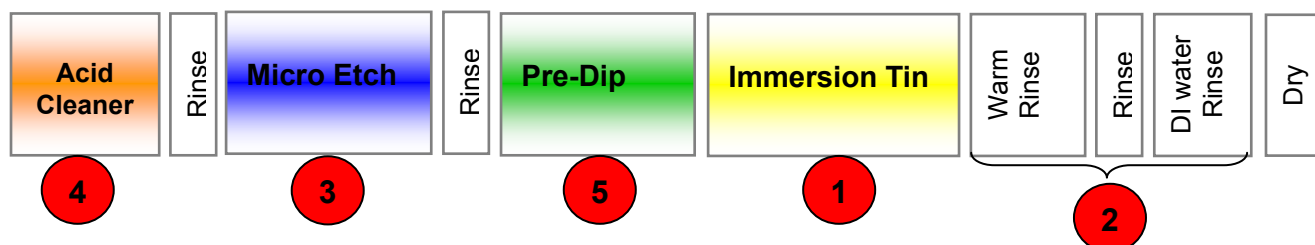
Since the plating solution CSN 7004 requires the longest time for heating up to its operating temperature, it is recommended to start the installation with the plating solution first. Next the final rinsing section tanks, which also require a temperature of 50 – 60°C, should be filled.

While these baths are heating up, the other chemicals can be prepared and installed.

The MET 7000 Micro Etch should be second, because the make-up causes an exothermic reaction by which the bath could be heated up to over 40°C. It needs time to cool down otherwise the etching rate will be too high.

The following sequence for make-up is recommended:

1. CSN 7004
2. Final rinses
3. MET 7000
4. ACL 7001
5. OMP 7000 / OMP 7001



Make-up survey

Product	Make-up		Remarks:
Acid Cleaner ACL 7001	DI water ACL 7001 C	87.5 vol% 12.5 vol%	ACL 7001 C concentration should not exceed 15%.
Micro Etch MET 7000	DI water Sulfuric Acid (H ₂ SO ₄ , 96%) MET 7000 S Hydrogen Peroxide (H ₂ O ₂ , 35%)	81.5 vol.% 10.0 vol.% 2.0 vol.% 6.5 vol.%	If you make up the solution by weight, consider the product's specific gravities.
OMP 7000	DI water OMP 7000 B OMP 7000 C	92.5 vol.% 2.5 vol.% 5.0 vol.%	If you make up the solution by weight, consider the product's specific gravities. Watch for green sediments and re-disperse before use (shake, stir well).

Product	Make-up	Remarks:
OMP 7001	100%	
CSN 7004	CSN 7004-1 90 vol. % CSN 7004-2 10 vol. %	Watch for yellowish white sediments and re-dissolve before use (heating up or rinse with warm working solution).

Operating temperatures:

Process bath	Temp. range	Recommended starting temp. for installation
Acid Cleaner ACL 7001	40 – 50 °C	40 °C
Micro Etch MET 7000	28 – 42 °C	35°C
OMP 7000	15 – 30 °C	room temperature
OMP 7001	35 – 45 °C	35°C
CSN 7004	Vertical: max. 65 °C Horizontal: max. 73 °C	Vertical: 64 °C Horizontal: 68°C

ACL 7001 make-up:

- Thoroughly clean tank before adding components and check tank for cleanliness, cracks and integrity of any tank linings used. Follow the make up recommendations given on page 105.
- Fill the tank $\frac{3}{4}$ full with DI water
- Add the required amount of ACL 7001 C to tank while stirring.
- Fill the tank to its operating level with de-ionized water.
- Set the bath to temperature.

MET 7000 make-up

- Thoroughly clean tank before adding components and check tank for cleanliness, cracks and integrity of any tank linings used. Follow the make up recommendations given on pages 114.
- Add $\frac{3}{4}$ of the DI water
- Slowly add sulfuric acid while stirring. **Caution: Exothermic reaction causes temperature increase (up to 40°C+ are possible!).**
- Add **MET 7000 S** stabilizer while stirring
- Finally add hydrogen peroxide.
- Mix properly and fill the tank to its operating level with the remaining DI water quantity.

Attention: Please follow precaution measures for handling corrosive substances. Ensure proper mixing during and after blending steps.

Caution: Do not store or move readily blended etching solution in tightly sealed containers.

OMP 7000 make-up

1. Thoroughly clean tank before adding components and check tank for cleanliness, cracks and integrity of any tank linings used. Follow the make up recommendations given on page 139.
2. Add $\frac{3}{4}$ of the DI water.
3. Slowly add **OMP 7000 B** buffer while stirring.
4. Add **OMP 7000 C** concentrate while stirring.
5. Mix properly and fill the tank to its operating level with the remaining DI water quantity.

Attention:

It is essential to submit water first and mix it with the buffer solution prior to the concentrate addition. Otherwise the Organic Metal will irreversibly flocculate and precipitate.

OMP 7000 C and OMP 7000 B are not compatible with each other in their concentrated form and should therefore never be mixed prior to being dissolved in water.

Remark:

OMP 7000 C contains small particles that tend to settle during storage. Before taking any OMP 7000 C out of the original container, please check for sediments and make sure all precipitation is re-dispersed by powerful shaking or stirring. If not, errors will occur in bath make-up and/or replenishment due to varying Organic Metal concentrations in the quantity taken out.

OMP 7001 make-up

1. Thoroughly clean tank before adding components and check tank for cleanliness, cracks and integrity of any tank linings used. Follow the make up recommendations given on page 163.
2. Fill tank with **OMP 7001**.
3. After filling the tank to desired level switch on circulation and heating system and set bath to temperature.

CSN 7004 make-up

1. Thoroughly clean tank before adding components and check tank for cleanliness, cracks and integrity of any tank linings used. Follow the make up recommendations given on page 195.
2. Add **CSN 7004-1** first.
3. Slowly add **CSN 7004-2** while stirring.
4. The made-up working solution has to be mixed well to level out the concentration prior to use.
5. Set the bath to temperature.
6. Condition the tin bath for 24 hours by keeping 65°C and running circulation / filtration system
7. (no boards should be run during conditioning period). For details about conditioning see page 29.

When emptying CSN 7004-1 and -2 containers that were stored and/or transported at lower temperatures, a yellowish crystalline sediment could remain on the bottom of the barrels. These are not decomposition products, but necessary bath components which have precipitated when exceeding the solubility limit at low temperatures. To ensure the defined bath composition, these sediments / crystals need to be dissolved prior to using the solution. To achieve this, the barrels used for make-up can be rinsed with plating solution taken from the bath, that has a temperature of >40 °C. For taking out partial quantities from precipitated containers, they should be heated up until all crystals are completely re-dissolved.

The process can be started after all process baths have been made-up properly and have reached their specific process temperature.

Detailed make-up procedures for all products are also given in the corresponding technical data sheets and in this Process Guide.

1.2.3 Conditioning Procedure of immersion tin after fresh make-up

Immersion tin baths are complexing chemicals, with structures only fully developing during operation and which naturally evaporate gases at elevated temperatures ($> 30^{\circ}\text{C}$). This also happens during transportation and storage in sealed containers that prevent these gases from leaving the solution and force them to stay "dissolved" in the solution.

A not fully developed plating structure of the tin bath chemical in combination with dissolved gases in solution (e.g. H_2S) can lead to a gray tin deposition directly after make-up. A conditioning of the tin bath is essentially necessary to develop its proper plating properties and let the dissolved gas evaporate from the fresh solution and develop the tin bath's full plating properties.

Conditioning procedure at operating conditions:

This method does not require any additional chemicals and is easy to execute:

1. Thoroughly clean tank / module before adding CSN 7004, following recommendations given on page 15. Check tank for cleanliness, cracks and integrity of any tank linings used.
2. Fill CSN 7004 into the tank with pumps / filters operating
3. Set the bath to 65°C
4. **Conditioning: Keep the temperature of 65°C stable for a period of 24 hours with pumps / filtration running.**
5. Do not run boards during conditioning period
6. After 24 h of operation, check acidity and / or specific gravity and adjust accordingly prior to continuing with first test boards.

The effectivity of the conditioning procedure should be checked with beta $[\beta]$ determination of the CSN 7004 and with test boards run as described in the next section (for details on beta determination please refer to page 33 and 238).

A conditioning is only necessary for fresh make-ups in a line (vertical and horizontal). CSN chemicals used for replenishment do not need to be conditioned prior to use, as they are added to a running system and only small quantities are used at a time.

1.2.4 Test run after initial make-up

After all process baths and rinses are filled and have reached their operating temperatures it is recommended to make a test run with both copper clad laminate and test panels in order to check plating quality, speed and determine the specific operating conditions within the operating window.

Copper clad laminate is used to determine the optical appearance of the tin deposit.

OC3 Test Panels, provided by Ormecon, are used to determine plating thicknesses by GCM (coloumetric measurement):

- Immersion tin in case of ORMECON™ CSN FF
- Immersion tin and silver in case of ORMECON™ CSN FF-W

It is also recommended to run a few representative customer boards through the line to check quality and look for issues such as e.g. solder mask attack.

1.2.5 Thickness measurements

All thickness measurements are usually made on Ormecon's OC3 test panels and are measured with a coulometric instrument. Based on these results a correlation with the customer's internal measuring methods is made (X-RF or coulometric).

Silver (only for ORMECON CSN FF-W)

For checking silver thickness after OMP 7001 take out the OC3 sample boards after the Pre-Dip. If the silver thickness is out of spec, the following actions are possible:

Silver layer thickness below 15 nm:	Keep immersion time / speed of line and increase bath temperature (max. 45°C).
	Keep temperature and increase immersion time / decrease speed of line.
Silver layer thickness above 45 nm:	Keep immersion time / speed of line and drop bath temperature (min. 35°C).
	Keep temperature and decrease immersion time / increase speed of line.

Remark for horizontal operations:

A change in the line speed has an impact on the immersion times in all modules and could lead to an undesired drop / increase of etch rate and immersion tin deposit thickness and further adjustments may become necessary for these process baths too. A temperature drop / increase of only the Pre-Dip is a more selected measure and should be preferred.

Tin

Tin thickness is measured on OC3 boards that have been run through the full process. If the tin thickness is out of spec, the following actions are possible:

Tin layer thickness too low:	Keep immersion time / speed of line and increase bath temperature (max. 73°C).
	Keep temperature and increase immersion time / speed of line.
Tin layer thickness too high:	Keep immersion time / speed of line and drop bath temperature.
	Keep temperature and decrease immersion time / speed of line.

Remark for horizontal operations:

A change in the line speed has an impact on the immersion times in all modules and could lead to an undesired drop / increase of etch rate and, in case of ORMECON™ CSN FF-W, of the silver layer deposition and further adjustments may become necessary for these process baths too. A temperature drop / increase of only the CSN 7004 working bath is a more selected measure and should be preferred.

Immersion time / line speed and process bath temperatures should be balanced in order to achieve the specified metal deposit thickness.

In case of quality problems with the immersion tin deposit, apart from thickness, please contact Ormecon International or a local representative for technical support.

1.3 Bath characteristics and important quality issues for the OMP 7000 / OMP 7001 and CSN 7004 working baths

1.3.1 Service Life

The ORMECON™ CSN FF and ORMECON™ CSN FF-W products, as described in this Process Guide, are designed for a continuous and sufficiently high material throughput.

The OMP 7000 Pre-Dip solution should be changed when the treated copper surface starts to become inhomogeneous or spotty. To prevent misinterpretation of any cosmetic issues, the user should contact Ormecon International prior to changing the bath.

Regarding the CSN 7004 plating bath, a minimum throughput of 5 m² circuit board material per 100 liter and day is recommended. At irregular working times and/or longer stand-by times, it can be necessary to adjust the bath chemistry in order to reach a continuously high layer quality.

With proper handling and correct bath maintenance approx. 5 to 6 m² double sided circuit boards with approx. 15 % copper per liter CSN 7004 can be put through.

1.3.2 Acid Cleaner ACL 7001

The Acid Cleaner removes grease and oils, developer residues after solder mask application and other impurities from the PCB surface. This is essential to prevent a disturbance of the tin deposition due to these residues. A drop in the cleaning properties of the Acid Cleaner bath could result in a stained, irregular tin finish. Even solderability problems are possible due to low tin deposit thickness. For replenishment details, see pages 112.

1.3.3 Micro Etch MET 7000

MET 7000 S concentration

This product stabilizes the Micro Etch working solution and keeps the green color of the bath. Dark blue solutions usually indicate an excessive hydrogen peroxide concentration or a low Stabilizer concentration. In case of a color change from green to blue, MET 7000 S needs to be added. For replenishment details, see pages 132 - 135.

Copper concentration

Due to the etching process, copper concentration increases in the working bath. The Cu concentration should be kept below 50 g/L at all times. Exceeding this limit could increase the risk for copper crystal formation on the PCB surface.

1.3.4 Pre-Pip bath OMP 7000

Organic Metal concentration

The Organic Metal dispersed in the Pre-Dip bath has to be determined depending on material throughput and has to be replenished regularly with the help of OMP 7000 C and OMP 7000 B. A low content of the Organic Metal will result in different grain structure, lower deposition speed and thickness and a stained surface finish. For replenishment details, see pages 155 - 157.

Contamination with other process baths

- Contamination of the OMP 7000 process bath with chemicals from the pre-treatment section should be prevented, because they would lead to an irreversible destruction of the Pre-Dip bath. A good rinsing of boards after ACL 7001 and MET 7000 is therefore highly recommended.
- Contamination of the Pre-Dip solution with tin has to be prevented as well. If too much CSN 7004 plating solution is carried in, for example due to a tilting circuit board or inadequate line construction, there is a danger of increased tin concentrations in the Pre-Dip, which subsequently increases the danger of chemical instability and an uncontrolled pre-plating. **OMP 7000 can only stand a CSN 7004 contamination of up to 1g/L (equal to 20 mg/L of tin and 100 mg/L of complexing agent) before irreversible flocculating occurs.**
- Tin contamination in the Pre-Dip could also lead to pre-plating, which hinders the tin layer growth in the plating bath, leading to uneven and spotty tin deposits with poor quality features. In this case the Pre-Dip solution has to be replaced.

1.3.5 Pre-Pip bath OMP 7001

Silver concentration

Silver acts as a whisker suppressing agent in the tin deposit. In order to ensure a proper silver plating thickness from the Pre-Dip, the concentration has to be thoroughly observed. A low silver content in the working bath will result in a low deposit thickness and whisker-reduction is no longer guaranteed. For replenishment details, see pages 167 and 168.

Organic Metal concentration

The Organic Metal dispersed in the Pre-Dip preserves and stabilizes the deposition of silver. A low content of the Organic Metal will affect the silver deposition and grain structure and reduce deposition speed and thickness of the surface finish. So it needs to be kept in spec. For replenishment details, see page 190.

Contamination with other process baths

- Contamination of the OMP 7001 process bath with chemicals from the pre-treatment section should be prevented, because they would lead to an irreversible destruction of the Pre-Dip bath. A good rinsing of boards after ACL 7001 and MET 7000 is therefore highly recommended.
- Contamination of the Pre-Dip solution with tin has to be prevented as well. If too much CSN 7004 plating solution is carried in, for example due to a tilting circuit board or inadequate line construction, there is a danger of increased tin concentrations in the Pre-Dip, which subsequently increases the danger of chemical instability and an uncontrolled pre-plating. **OMP 7001 can only stand a CSN 7004 contamination of up to 0.5 g/L (equal to 10 mg/L of tin and 50 mg/L of complexing agent) before irreversible flocculating occurs.**
- Tin contamination in the Pre-Dip could also lead to pre-plating, which hinders the silver plating and leads to an insufficient whisker suppression. If pre-plating occurs, the Pre-Dip has to be replaced.

1.3.6 Immersion Tin bath CSN 7004:

Beta value [β]

The beta value is a quality value invented by Ormecon GmbH to specifically determine the plating properties of a CSN 7004 process bath. β is a function of the deposition of a specific pure tin thickness at a defined tin bath temperature and a defined dwell time.

The best possible beta value of a CSN 7004 plating solution is $\beta = 1.1$

This is usually only achieved by very fresh solutions shortly after production. During transportation, storage and with operating a bath, the CSN 7004 solution naturally ages, which makes the β drop.

A range between $\beta = 0.7 - 0.9$ is normal and recommended as the operating window.

A drop of the beta value below 0.7 usually indicates that the bath has suffered from damage. Possible reasons for a beta drop could be overheating, contamination by organics or foreign metals, excessive Sn(IV) formation, etc. This is normally seen in a decreasing quality of the deposit. In case of a problem with the plated tin layer, it is recommended to check the beta value of the working solution. During and after the corrective action to eliminate the source of the damage, the beta should be checked again, in order to proof the efficiency of the correction. The beta should increase to the specified range of 0.7 - 0.9.

The beta should also be used as the benchmark for CSN 7004 conditioning after process installation. Storage and transportation can temporarily affect the plating conditions of the fresh CSN 7004 solution, resulting in a low beta value. However conditioning is required after initial make-up and the beta should be back in specification within 24 hours.

In order to determine the beta correctly, tin thickness, tin bath temperature and dwell time need to be measured with utmost care. Using wrong data for the beta calculation could lead to false results and wrong interpretations. Provisions for an accurate beta determination are:

- Check exact temperature of the plating bath manually with a thermometer. Do not rely on temperature readings from the equipment.
- Control dwell times precisely with manual stop watch
- Measure tin deposit with calibrated coulometric instrument (preferably GCM).
- Since the calculation of the beta is not trivial, a Beta Table is given in the analysis section of this Process Guide (page 238). This is set for a bath temperature of 65°C and 20 min. plating time and can be easily used to determine the beta value of your CSN 7004 plating solution (keep to these conditions thoroughly to get reliably results).

Stannous Tin concentration (Sn^{2+})

The tin dissolved in the plating bath is consumed depending on the throughput and has to be added back on a regular basis with the CSN 7004 R Replenisher solution, a mixture of the components CSN 7004-R1 and CSN 7004-R2. They too have to be mixed in a volume ratio of 9 : 1 prior to use. The correct mixing ratio of the components is of high importance. A low tin concentration causes a reduced deposition rate and an irregular grain structure, which will result in a spotty looking surface. For replenishment details, see pages 241.

Sn(IV) content

During use, the CSN 7004 plating solution tends to be cloudy with a white/yellowish precipitate of insoluble Sn(IV)-oxide. The precipitation reduces the tin content of the bath in proportion, and is removed out of the bath by filtration. This precipitation does not have a negative effect on the grain structure and deposition rate, as long as the suspended particles are filtered out of the bath continuously and the recommended tin concentration is kept constant. However Sn(IV)-oxide formation leads to a loss of tin

in the plating solution and requires tin replenishment. As a matter of fact Sn(IV) oxidation should be limited.

Copper content

Based on the replacement reaction, Cu ions are dissolved in the plating bath, depending on the amount of copper surface treated. With an increasing copper concentration, the risk of copper being co-deposited in the tin layer rises, and issues with solderability could occur, resp. multiple solderability of the final surface could become worse. It is therefore necessary to observe the copper content of the plating solution. For analysis details see page 240.

The plating bath should be exchanged if the copper content in the solution has reached 8.5 g/L. If the copper content exceeds the limit, take out min. 5% - 15% of the process solution and substitute the removed quantity with fresh, readily mixed CSN 7004 solution. This will drop the copper content. The described procedure can of course be executed at any copper concentration level and should be repeated whenever copper has reached the limit again.

The removed, copper-rich working solution could be regenerated if the CSN Regenerator is available. It should be dumped properly, if regeneration is not possible.

The copper content in the tin bath should be controlled, either by constant dilution of the plating solution with fresh CSN 7004 (regular replenishment or partial exchanges) or the use of a CSN Regenerator (see pages 66 - 71 for details)

Overheating of CSN 7004

The operating temperature of the plating bath must remain within narrow tolerances and may not deviate from the nominal value of max. $\pm 2^{\circ}\text{C}$, because the deposition rate is very much dependant on temperature conditions and local as well as total over-heating must be prevented.

Overheating of the CSN 7004 solution is one of the most common problems in operating the process. As a result of overheating, degradation products form in the plating bath and can lead to discoloration of the tin deposit (gray to black appearance). It is therefore essential to adhere to the heater capacity recommendation given in this Process Guide and to ensure good circulation of the bath around the heater. It is also recommended to avoid frequent heating and cooling. If possible keep tin bath at elevated temperature (35 - 40°C), even in times of no operation.

In case of the occurrence of quality problems related to overheating, CSN 7004 CAT can be used for bath recovery. Please refer to CSN 7004 CAT data sheet for details.

Density

The density of the bath must be kept within the tight tolerances. A deviation of the density can be caused by either insufficient or excessive water supply. The density is important to monitor, because an excessive dilution usually causes a drop of the deposition rate, while excessive loss of water due to evaporation, usually leads to an increase of viscosity followed by an increased risk of overheating and worse rinsability.

!	<p>The density of the bath should be monitored and kept within the specified range with the help of precise density controllers. Based on their results an automatic dosing system should level out the solution. DI water should be added in case of an increase of density due to excessive evaporation losses. In case of a decrease in density due to excessive drag-in of water, it is recommended to open the tank lids to ensure evaporation of water until the bath's density is back to normal.</p>	!
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After density adjustment the bath level should be adjusted with CSN 7004 solution.

Acidity

The acidity of the tin bath is directly correlated with its density. When the acid content in the plating bath drops below 4.0 mol/L, due to rinse water drag-in or hydrolysis of the dissolved tin, the deposition rate of the bath decreases significantly with the results explained above. When treated according to recommendations and when density is monitored and kept within specification, the acidity adjusts automatically.

Foreign Metal contamination

The tin bath is very sensitive to foreign metal contamination. Any metal contamination, other than Sn, Cu and Ag, usually leads to serious quality issues of the tin deposit, because of co-plating. Especially iron and antimony are crucial, but also aluminum and nickel can cause problems.

Metal contamination has to be avoided under all circumstances, because it affects the plating bath and therefore the tin deposit. Gray to black deposits usually occur with solderability problems and “yellowing” during reflow. The tin bath could also be irreversibly affected and needs to be exchanged.

It is therefore essential to avoid any metal contact with the tin bath solutions. Do not use metal baskets / racks without proper, pore-free coating (black or green HALAR or PP), do not use metal screws or fasteners, take care of proper heater choice and regularly check for signs of destruction, etc.

Iron

Iron contamination is very critical, especially the continuous emission of fresh iron to the CSN 7004 plating solution. Possible sources for iron contamination are metal parts, that are in contact with the immersion tin solutions, e.g. from baskets/racks (stainless steel baskets, or pores in plastic coat of metal racks due to insufficient resistance of plastic coating), screws or other fasteners, heaters, etc.

The CSN 7004 solution comes with a max. Fe concentration of ≤ 5 ppm. Should this value be exceeded during operation of the ORMECON™ CSN FF or ORMECON™ CSN FF-W process, a constant emitting iron source is present, bleeding fresh iron into the tin bath. It is essential for keeping a high tin deposit quality to identify the source of the constant iron contamination and eliminate it. A max. Fe content of 15 ppm is allowed before the process has to be stopped for iron source elimination. It depends on tin deposit performance, if the CSN 7004 solution can be further used or should be (partially) exchanged, after elimination of the iron source. See process specifications for details.

Antimony

The most common source for antimony is CEM-1 base material (antimony is used as flame retardant and tends to bleed-out during board processing). CEM-1 material should not be processed without prior testing by Ormecon GmbH for full compatibility.

!	<p>Ormecon International does not take over any guarantee for an affected CSN 7004 plating solution nor for a tin layer deposited from it, if</p> <ul style="list-style-type: none"> a. the iron content exceeds 15 ppm. b. any CEM-1 material had been run through it 	!
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Organic contamination

The tin bath is a very acidic medium and may be aggressive / corrosive to parts and components without proper resistance. Organic contamination of the immersion tin plating bath could occur because of drag-in of other process bath chemicals or contaminated rinse waters. But it could also occur because of a chemical attack of e.g. solder mask, coverlay (adhesive), legend ink, etc. Such contaminants could accumulate in the solution and seriously affect the tin plating quality. Typical effects of organic contamination are low deposition rate (= low tin thickness), color change of deposit, solderability problems, yellowing after reflow, etc.

The reason for such failures are residues of organic contamination which

- deposit on the bare copper surface when the board is immersed in the tin bath. They may hinder the copper dissolution in the tin bath and therefore significantly reduce the plating rate.
- are left on the tin surface, where they could affect the wetting of the tin and hence cause solderability problems. These residues could also degrade under reflow conditions and form a yellow or brownish layer on top of the tin.

Due to the unlimited variety of organic contamination possibilities, there is no suitable analysis method available. Should a discoloration, drop of deposition rate or yellowing after reflow occur, the process needs to be checked for organic contamination sources.

Under some circumstances, organic contamination can be removed from the tin surface with improved rinsing or the use of a rinse aid bath (see RAD 7000 technical data sheet) and solderability and yellowing issue can be improved. In any case it is recommended to find the source for the contamination and eliminate it.

Filtration

A continuous and efficient filtration of the plating solution is a pre-requisite for a nonporous layer formation and a minimum particle co-deposition. As a filter medium polypropylene units have proven to be suitable. A pre-filtering with 50 µm pore size should be followed by a fine filter with maximum 20 µm pore size.

Remark:

The OMP 7000 / OMP 7001 Pre-Dips should not be filtered to keep the Organic Metal particles in the solution. A circulation pump is recommended however to prevent sedimentation.

Bath circulation

The bath circulation should be controlled, in combination with a suitable line layout, to ensure lowest possible drag in of oxygen into the Pre-Dip and plating bath solutions. Turbulence in the liquids should be prevented. The balance/adjustment of temperature and concentration gradients has to be ensured.

!	<p>In horizontal lines the oncoming flow to the circuit boards is ensured by upper and lower nozzles (nozzle bars / flow bars). The hole diameter of these nozzles should not be too small to prevent high velocity of flow and resulting turbulence and foam formation. They also bear the risk of being blocked easily by precipitated Sn(IV) residues.</p> <p>In immersion lines the bath movement from the bottom to the top is of advantage. Air injection has to be prevented. It has to be ensured that the total casting, including the filter unit, is airtight.</p>	!
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Final rinsing

Remark: The first rinse is the most important step in the final rinsing section. The cleaning quality of the final rinse determines about a good or bad optical appearance of the tin plating (fresh or during aging/processing). A bad rinsing result from the first rinse can not be compensated by subsequent rinsing / cleaning.

After completion of the plating process, the circuit boards must be completely free from electrolyte residues. This can be best achieved by a **3 to 4 step cascade rinsing**, whereas the first rinsing stage immediately following the plating module, is operating at a temperature of either 50 °C or 30°C, depending on the process run:

- a. 50 °C for ORMECON™ CSN FF
- b. 30°C for ORMECON™ CSN FF-W

As an additional option RAD 7000 can be used in the final rinsing section.

The last cascade must be operated with DI-water, also with elevated temperature, to prevent salt residues drying on the circuit board surface. With sufficient spray pressure and spray angle, the total rinsing time should be between 1.5 to 5.0 min, where at least 0.5 min should be allowed for the DI-water rinsing stage.

Sufficient cleaning of the tin finish can only be achieved with clean rinse water. So the recommendation for rinse water quality given in this Process Guide on page 64, needs to be adhered to. Recommendations for proper equipment design are given in the *Equipment* chapter of this Process Guide (pages 41 - 71).

1.4 Operating ORMECON™ CSN FF and ORMECON™ CSN FF-W

1.4.1 New make-up of process baths

During regular operation of the ORMECON™ CSN FF and ORMECON™ CSN FF-W processes, the pre-treatment solutions have to be renewed. Together with this new make-up, some maintenance work could be carried out.

Any new make-up of the process baths has to follow this sequence of actions:

- Empty the tanks and drain the removed chemicals to the waste disposal system for proper treatment.
- Take the old filter cartridges out of the filtration unit
- Rinse the modules with a water hose and manually clean all equipment parts of the tanks. If the tanks / modules are very dirty, follow cleaning procedures given on pages 15 - 25. Completion of the chemical cleaning and (re)start of the line.
- Make sure the modules are completely empty and clean prior to refill
- Make-up new process baths according to the recommendations given in the Technical Data Sheets or in this Process Guide.

Pre-Dip and CSN 7004 plating baths:

It is not necessary to discharge and remake the baths over time. Generally the amount of replenishing chemicals added to the working baths is sufficient to renew the process baths continuously.

1.4.2 General Correction Instructions CSN 7004 working bath – Replenishment Sequence

When adding water and/or replenishing solutions, do not exceed the maximum bath volume. This is especially critical for the CSN 7004 plating solution, because a variety of ingredients and properties need to be checked and various additions may be necessary. The following method helps to prevent an overload of the CSN 7004 tanks:

1. Adjust specific gravity

The working range for the tin bath's specific gravity is 1.18 – 1.25 mol/L @ 20°C or 1.16 – 1.23 mol/L @ 60 – 70°C.

If the specific gravity is lower than specified, the bath has probably suffered from excessive dilution. In this case evaporation of water should be allowed by opening the lid of the tank. Check density during the evaporation process regularly, in order not to miss the right point to close the tank again.

If the density is higher than specified, the bath suffered from excessive evaporation. In this case DI water should be added to adjust the density.

If the density is ok, continue with 2).

2. Measure the copper content

If the copper concentration is below 8.5 g/L, continue with 3).

If the copper content exceeds the limit, take out 5 - 15% of the process solution (the missing amount will be added with fresh CSN 7004 solution later). The removed quantity could be regenerated if the CSN Regenerator is available. It should be disposed of properly, if regeneration is not possible.

3. Measure the tin concentration and replenish with CSN 7004 R

If density and copper concentration are within the given working range, the replenishment can continue with adding tin. The tin(II) concentration has to be increased with the CSN 7004 R replenishment solution until the nominal value within the working range is reached. For adding 1 g tin per liter active solution, 25 mL/L of CSN 7004 R need to be added.

4. Fill up to original level

With density, copper content and tin concentration being within the specified working ranges, it may still be necessary to fill up the tank volume to the original level. This should be done with fresh CSN 7004 process solution (readily mixed in a 9:1 volume ratio from CSN 700-1 and CSN 7004-2)

All replenishment additions should be executed slowly and with constant stirring. This applies especially for the addition of water, because the dilution effect could lead to a local hydrolysis of dissolved tin.

1.4.3 Maintenance Work

Sampling

The major pre-requisite for a secure process control is a regular sampling of all process solutions. While one sampling each day for the pre-treatment steps (acid Cleaner and Micro Etch) are sufficient, we recommend to take samples of the OMP 7000 / OMP 7001 Pre-Dip and CSN 7004 working baths at the beginning of every working shift. This is a recommended control measure.

Replenishments are usually made daily from general experience quantities, following Ormecon International's recommendations for process control. A full replenishment procedure based on an extensive analysis is usually made for ACL 7001, MET 7000 and OMP 7000 after approx. 7 m² / L and for OMP 7001 and CSN 7004 after 1 m² / L.

Recommended process parameters with their average and limit values are given in this Process Guide, as well as analysis and replenishment information tools.

The prerequisites for taking analysis samples are:

- The liquid level in the tanks / modules should be close to the nominal level, For ACL 7001, MET 700 and OMP 7000 DI water should be added to compensate evaporation and drag-out losses. For the CSN 7004 working solution, fresh, readily mixed CSN 7004 solution should be used. OMP 7001 samples can be taken without any prior additions.
- Filtration, re-circulation and flooding should be active for at least 30 minutes in order to ensure an optimum mixing of the process solutions
- Rinse the beakers for sample removal with process solutions at least twice. Only then is the removed sample ready for analysis.
- Follow the analysis procedures given in the *Analysis Guide* section of this Process Guide.

Determination of the temperature in the tanks / modules

To control the temperature in the process module, a manual measurement should be carried out using a thermometer. The measured value, with a tolerance of $\pm 2^{\circ}\text{C}$, should be compared with the reading on the PLC of the line. Adjustments should be made if necessary.

Determination of the temperature in the rinsing cascades

Although the temperatures in the rinsing cascades following the Acid Cleaner and Micro Etch are not fixed, it should be controlled periodically. Before measuring, the line should be in operation for at least 30 minutes. The measuring points are in the inlet and outlet areas of the cascades. The rinse water temperatures should be within the following ranges:

Rinse after ACL 7001	Average: 15 °C (5 – 35°C)
Rinse after MET 7000	Average: 15 °C (5 – 35°C)

With respect to the final rinse, the temperature standards are far higher. In order to achieve a rinsing result with excellent values for ionic contamination, the rinsing temperature is of major importance. Therefore the rinsing cascades of the final rinse, must fall within the following temperature ranges:

Final Rinse – cascade step 1	Average for FF process: 55 °C (50 – 60°C) Average for FF-W process: 30°C (25 – 35°C)
Final Rinse – cascade step 3 or 4 (DI water)	Average for FF process: 55 °C (50 – 60°C) Average for FF-W process: 30°C (25 – 35°C)

Determination of the conductivity in the rinsing cascades

Apart from the temperature, the efficiency of the rinses is very much dependent on the quality and cleanliness of the rinsing water. A parameter, which indicates the pollution of the rinse water is its conductivity. The determination of this parameter has to be carried out **at least once a day and/or at the beginning of each working shift**.

Further information on rinses can be found in the section *Rinse water quality* on page 64.

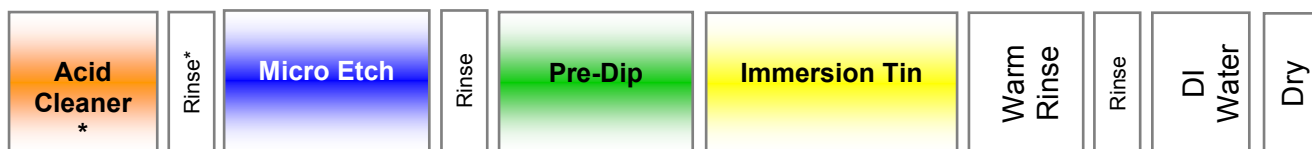
1.5 Equipment

ORMECON™ CSN FF / ORMECON™ FF-W can be operated in either vertical or horizontal technique. Vertical mode is usually suitable for small volumes of boards, especially for starting up with Immersion Tin surface finish technology. It involves a manageable initial investment for the equipment, even though chemical costs may be slightly higher compared to horizontal processing. Horizontal processing is preferred for mass production. It provides reliable and fully automated process conditions with the best possible surface finish quality.

The choice of operating mode basically depends on the expected volume to be processed and the willingness / ability for investment.

1.5.1 Specification for vertical ORMECON™ CSN FF and ORMECON™ CSN FF-W lines

ORMECON™ CSN FF / ORMECON™ CSN FF-W process sequence:



* The use of ACL 7001 and the following rinse is optional and depends on the quality of the boards prior to micro etching.

General information about equipment materials

The housing of all tanks should be welded PP with a temperature stability of $\geq 80^{\circ}\text{C}$. Screwed structures are not suitable.

Tanks and racks/baskets should not contain any metal parts that could be in contact with the process bath solutions, because of the risk of irreversible contamination of the chemistry.

Racks/baskets should be metal structures coated with pore-free black or green HALAR. Do not use blue HALAR. If plastic racks are used, PP is the preferred material (however plastic racks are not as stable as metal racks and are therefore not suitable for a high board load).

Plumbing should also be made from PP. Seals should be made from Viton or Teflon. No metal parts at all.

ORMECON™ CSN FF / ORMECON™ CSN FF-W vertical line specification details
Pre-treatment:

Process Step	Acid Cleaner ACL 7001	Double / Triple rinse ¹	Micro Etch MET 7000	Double / Triple rinse ¹
Tank material	PP / PVC	PP / PVC	PP / PVC	PP / PVC
Temperature [°C]	40 - 50	RT	28 – 42	RT
Dwell time [min.]	1 - 3	1 / 1 / 1	1 - 2	1 / 1 / 1
Heater required	Yes	No	Yes	No
Heater material	PTFE / Porcelain	--	PTFE / Porcelain	--
Filtration required	Yes	No	Yes	No
Cartridge size	10 µm	--	10 µm	--
Circulation of bath volume	2 - 4 times/ h	--	2 – 4 times / h	--
Air agitation	No	Yes	Yes	Yes
Exhaust	Yes	No	Yes	No
Level control	Yes	No	Yes	No
Dosing	Yes ²	No	Yes ²	No
Flow meter	No	Yes	No	Yes
Flow rate	--	7 – 10 l / m ²	--	7 – 10 l / m ²
Water quality	DI	city / soft water ³	DI	DI ⁴
Dump / Re-make	See data sheet	--	See data sheet	--
Vibration	No	No	No	Recommended

¹ These rinses could also be single rinses, if the recommended water quality is maintained. Water consumption of a single rinse would therefore be higher.

² for dosing details see Product Data Sheets and /or Process Guide

³ for water quality details please refer to pages 62 – 63 in this Process Guide

⁴ observe chloride content carefully when using OMP 7001 after this rinse.

Pre-Dips:

Process Step	Pre-Dip OMP 7000 (CSN FF process)	Pre-Dip OMP 7001 (CSN FF-W process)
Tank material	PP	PP
Temperature [°C]	15 - 30	35 – 45
Dwell time [min.]	1	45 – 60 sec
Heater required	no	Yes
Heater material	--	PTFE / Porcelain
Filtration required	No	no
Cartridge size	--	--
Circulation pump	Recommended	Yes
Circulation of bath volume	2 - 4 times/ h	2 – 4 times / h
Air agitation	No	No
Exhaust	Yes	Yes
Level control	Yes	Yes
Dosing	Yes ²	Yes ²
Flow meter	No	No
Flow rate	--	--
Water quality	DI	DI
Dump / Re-make	See data sheet	See data sheet
Vibration	No	No

² for dosing details see Product Data Sheets and /or Process Guide

Tank Design for OMP 7000 and OMP 7001

The Pre-Dip tank should be equipped with a circulation pump, but no filtration unit. The pump should circulate the entire bath volume 2 – 4 times every hour. It is necessary to adjust the pump's capacity carefully to avoid any turbulence of the bath and / or foam formation, because air (oxygen) that gets sucked into the solution could affect the bath's stability.

The continuous operation of a circulation pump is necessary to keep the small Organic Metal particles in the bath in a constant state of suspense. This helps to maintain the ideal Organic Metal working concentration, due to precipitation and sedimentation prevention.

A heater is usually not required for the OMP 7000 bath, however heating may be useful in some areas during the winter time to prevent this aqueous product from freezing. Cooling (coil) may be required in hot areas.

OMP 7001 should be heated to an operating temperature of 35 – 45°C to ensure proper deposition of the whisker-reducing layer.

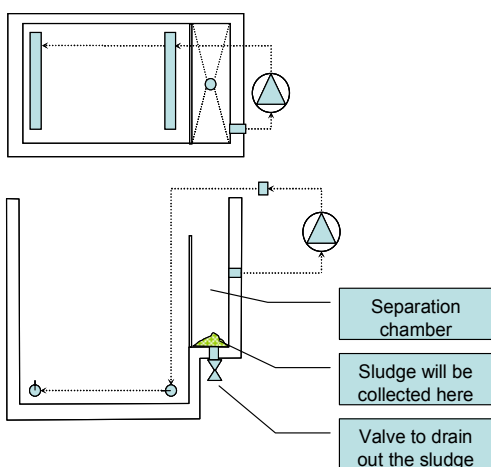
OMP 7000 and OMP 7001 should not be filtered at any time, because this would remove the small Organic Metal particles and the bath would lose its functionality. It would have to be replaced by a fresh bath.

Remark:

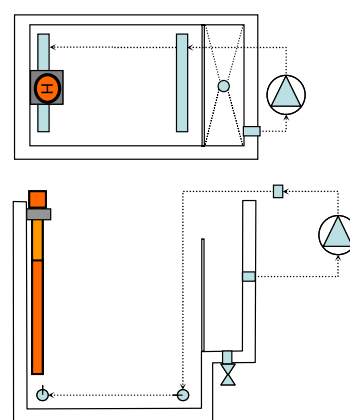
Should it be necessary to change the sequence of the tanks in a line, it is necessary to carefully cover the tanks of OMP 7000 and OMP 7001 to avoid any contamination with other chemicals, e.g. from dropping boards, as they are moved over the OMP 7000 / OMP 7001 tank. Any contamination of the two Pre-Dips with other process chemicals or contaminated rinse water could irreversibly destroy the Pre-Dip process baths.

Example:

Circulation with separator



Heater



Electrical heater placed in the circulation.
 $P = < 2 \text{ W/cm}^2$

Immersion Tin:

Process Step	Immersion Tin CSN 7004	Triple rinse CSN FF Process	Dryer
Tank material	PP	PP	Stainless steel
Temperature [°C]	Max. 65 Min. 40	CSN FF process: 50 / 50 / 50-60 ⁴ CSN FF-W process: 30 / 30 / 30-40 ⁴	80
Dwell time [min.]	5 - 30 ⁵	1.5 - 2 / 3 - 5 / 3 - 5	min. 15 – 20
Heater required	Yes	Yes	Yes
Heater material	PTFE / Porcelain	PTFE / Porcelain	
Filtration required	Yes	No	
Cartridge size	50 µm + 20 µm (double) or 10 µm (single)	--	
Pump	Yes ~ 1 W/l	--	
Circulation of bath volume	2 - 10 times/ h	--	
Air agitation	Could be useful	Yes	
Exhaust	Yes	No	
Level control	Yes	No	
Dosing	Yes	No	
Flow meter	No	- / - / Yes	
Flow rate	--	14 l / m ²	1380 l air / min.
Water quality	DI	DI < 20 µS	
Dump / Re-make	See data sheet	--	
Vibration	recommended	recommended	

⁴ Heating system required in the 3rd chamber or the cascade rinse (warm/warm/hot)

⁵ depending on desired tin thickness

Remark: For lead-free processing OEMs usually specify a minimum tin thickness of around 1.2 µm (1.07 µm measured with GCM) In order to achieve this, the dwell times in the tin baths should be adjusted:

! The recommended total exposure times in the Immersion Tin for standard soldering conditions are:
 → 9 – 15 min. for SnPb soldering (12 min.)
 → 16 - 27 min for lead-free soldering (21 min.) **!**

Tank Design for CSN 7004

The tank for the immersion tin bath is the most critical. It requires a special design to make it perfectly suitable for the chemical, to ensure the best possible and most reliable result and to keep consumption of chemistry low.

CSN 7004 is operated at temperatures of max. 65 °C in vertical mode. At such temperatures the bath usually suffers from degradation, especially due to local overheating, because the solution temperature is usually much higher around the heater. The formation of degradation products can not be avoided, but should be minimized with the help of a proper design:

- !**
- The heater surface temperature should not exceed 75°C
 - Outlet of the filtration cycle should be close to the heater, so that a continuous solution flow around the heater is guaranteed. This helps to minimize local overheating.
 - Frequent heating and cooling cycles damage the bath, because local overheating usually occurs during heating cycles. Leave the bath at operating temperatures if possible, even in times of no operation, or do not cool it down to room temp. completely, if another use of the process is expected shortly.
- !**

Despite the above recommended instructions, every tin bath naturally suffers from the accumulation of degradation products over time, called „aging“. The degradation products usually accumulate in the surface spheres of a bath and create a gray, sometimes almost black, deposit on the tin surface when pulling the boards out of the plating solution. This affects optical appearance and also solderability and stability of the tin deposit.

A proper bath movement usually prevents the degradation product from surface sphere accumulation. At the same time it enables gaseous degradation products to evaporate more easily.

- !**
- Ensure a proper bath agitation. The solution should be visibly streaming, while turbulence or foam formation should be avoided under any circumstances.
- !**

The formation of foam is an indicator for turbulence or air incorporation into the tin solution of any other kind. This is another sensitive and important issue with regards to Immersion Tin. Air (oxygen) leads to an oxidation of the Sn(II) ions to Sn(IV). Sn(IV) precipitates as a white residue and is not available for the surface finish deposition. So oxidation actually removes tin from the solution and makes replenishment necessary more frequently (= increase of process costs).

Furthermore Sn(IV) residues are soft in the beginning, but turn into a hard white crust over time. The accumulation of such precipitates could lead to damages in the piping and filter system, of pumps, rollers etc. They also significantly shorten the maintenance and cleaning intervals of the line. So oxidation should be definitely avoided.

This should be taken into account for the line design and process operation:



- Bath agitation should be ensured, but without turbulence (indicated by foaming)
- All inlets and outlets of drains, pipes, etc. should be below solution surface level.
- Ensure constant filtration of the CSN 7004 process bath during heat-up and operation. Make sure filter cartridges are changed regularly (for details see description below)



Filter system:

The CSN 7004 solution should be filtered with a two step filtration system to remove as much Sn(IV) precipitates as possible. Starting with a 50 µm PP filter followed by a 20 µm PP fine filter cartridge. The filtration system should be running with every start-up (heating up) of the baths and during operation. Make sure the filters are changed in regular intervals, otherwise the solution will become hazy and tin deposit quality could drop.

Rinses:

The deposition quality significantly depends on the purity and cleanliness of the boards. Several rinses should ensure a good cleaning of chemical and other residues between process steps. The rinse water should therefore be kept extremely clean, especially in static rinses. Dirty or contaminated rinse waters, e.g. loaded with process chemicals or other substances, could affect the tin deposit quality. A constant and easy water exchange should be possible.

For details on rinse water quality refer to chapter *Rinse water quality* on page 64.

Further information on equipment:

Mechanical agitation	0.1 – 0.2 m/min (stroke length 20 – 30 mm)
Rinses	all rinses are cascade rinses
Rinse water quality	according to specifications given on pages 64; conductivity meter measurement required
Tank design	A cover is recommended for all tanks to reduce evaporation losses and avoid contamination. The cover must allow air circulation above solution surface level
Dimension of tanks	the tank should have a minimum distance of 20 mm on each side of tank length and height, and 50 mm on each side of tank width to the immersed basket (see drawing)
Maximum loading	0.03 m ² / Liter of tin chemical
CSN 7004 tank	Ensure good bath agitation around the heater to avoid local overheating; agitation of surface must be visible (no turbulence)
Heater surface temperature should not exceed 75°C Indirect Ormecon heating system is preferred	

Basket

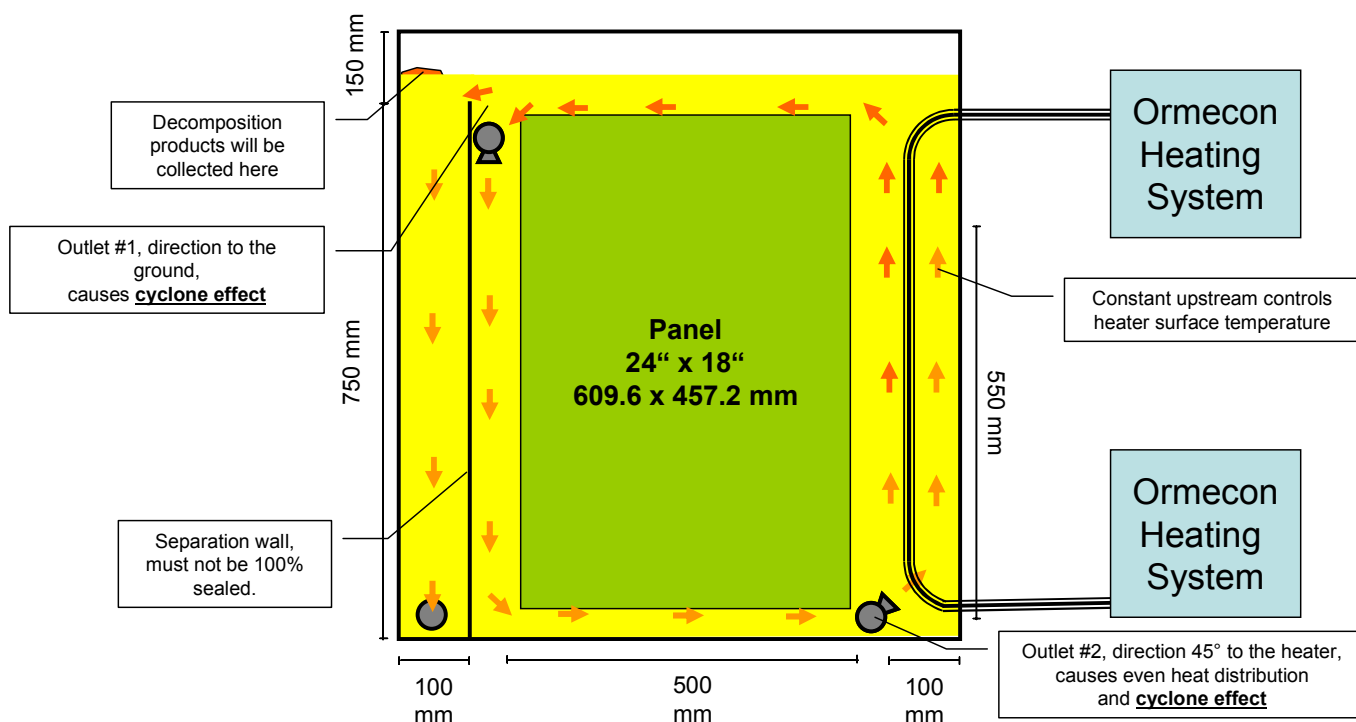
- minimum distance between the panels in the basket must be 15 mm (20 mm for backpanels);
- baskets could be metal structures, but need to be coated with either pore-free black or green HALAR (do not use blue HALAR!) or pore-free PP.
- if stability is not an issue, baskets could also be made from PP

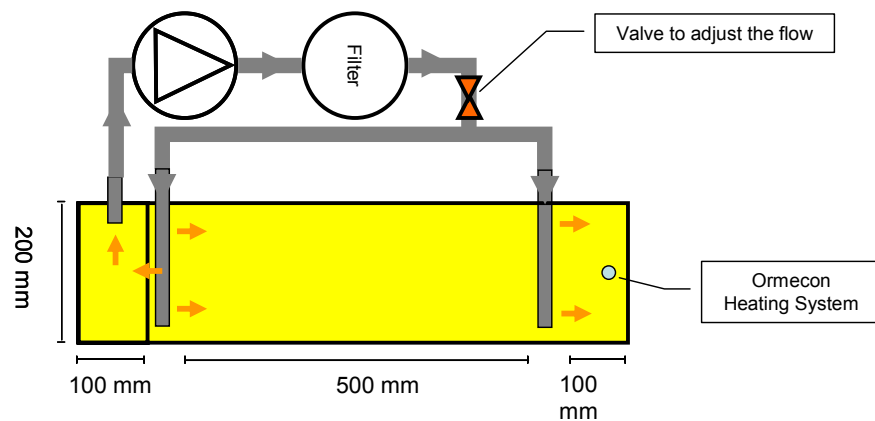
Plumbing

- no metal is allowed in contact with the solution! must also be made from PP. Seals should be made from Viton or Teflon. No metal parts at all.

!	<p>Important:</p> <ul style="list-style-type: none"> → Do not use any kind of metal for parts in direct contact to the process solutions → Foaming should be avoided under any circumstances, because it may lead to irreversible damage to the process solutions. 	!
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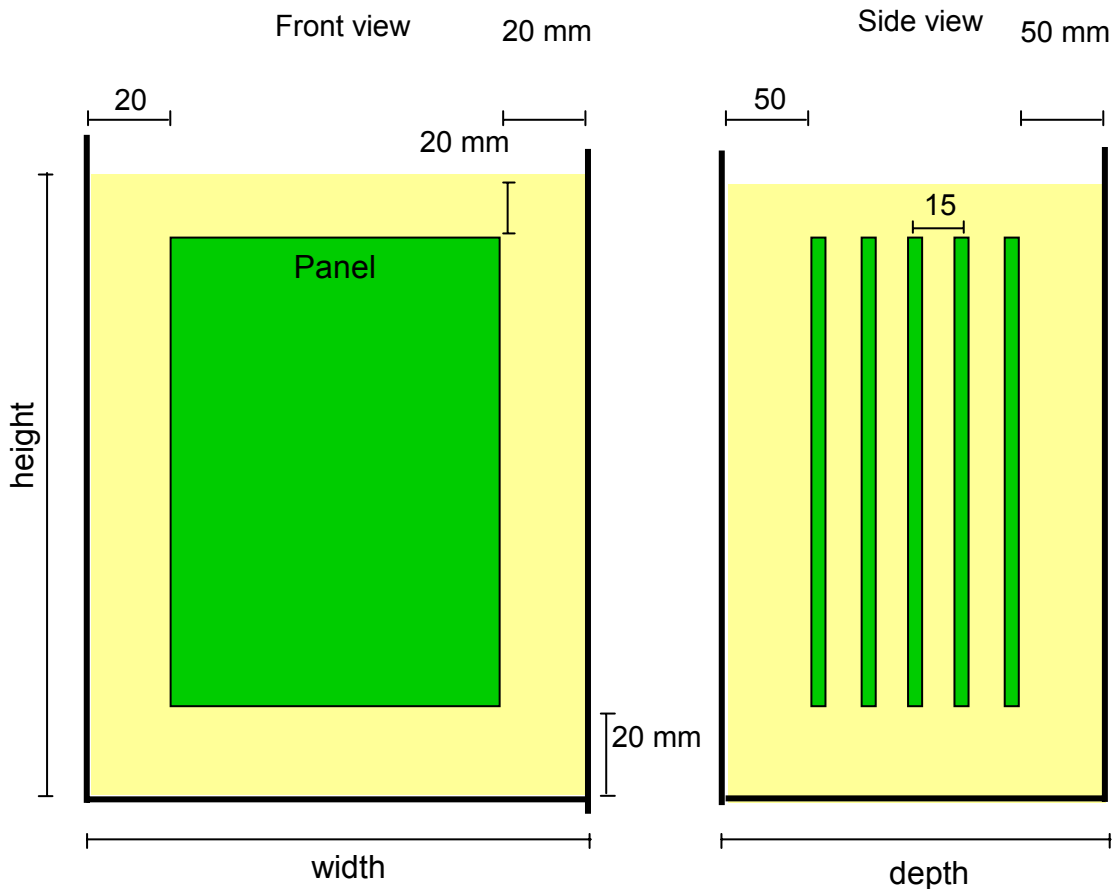
Example:



Top view

$$\text{Volume} = 900\text{mm} \times 800\text{mm} \times 200\text{mm} = 144 \text{ Ltr.}$$

This is an example, correct sizes must be defined by sizes of panels and basket.

Minimum distances of boards in racks:

Calculation of suitable tank volume for ORMECON™ CSN FF and ORMECON™ CSN FF-W
a. Calculation of basket load:
Example:

Panel size: 455 x 609 mm No. of panels = 10

→ total m² panel / basket = 10 x 0.455 x 0.609 = 2.77 m² panel

b. Calculation of tank volume based on recommended maximum loading:

The recommended max. loading is 0.03 m² / L

Tank volume = total m² panel / max. loading = 2.77 m² / 0.03 m²/L = 92.3 Liter

c. Calculation of size and volume of tank based on minimum distances

Min height = 20 + 609 + 20 mm = 649 mm = 6.49 dm

Min length = 20 + 455 + 20 mm = 495 mm = 4.95 dm

Min width = 50 + (9 x 15) + 50 mm = 235 mm = 2.35 dm

Tank volume = height x length x width = 6.49 x 4.95 x 2.35 = 75.5 Liter (dm³)

Conclusion:

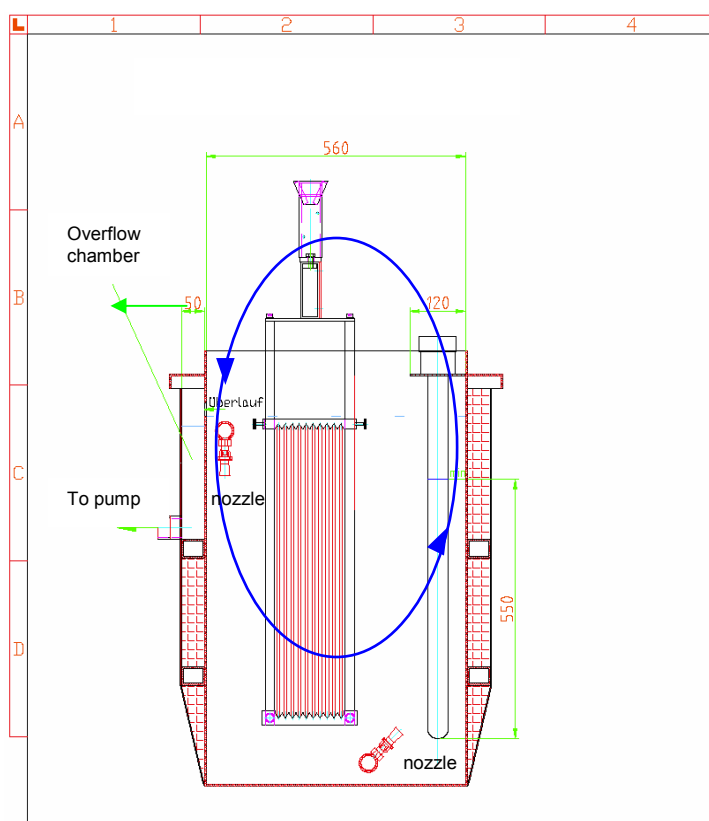
The bigger value resulting from equation #2 and #3 is recommended as the minimum tank volume. In this case the recommended volume is 92.3 Liter.

Immersion tin baths are always subject to the risk of overheating (see page 34). As a result, decomposition products are formed, which usually accumulate in the surface spheres of the CSN 7004 working solution. Pulling boards through these surface phases can affect the tin deposition quality. Proper bath agitation can minimize the risk of decomposition product formation; however the tank design can further support the prevention of accumulation.

A separated chamber at one side of the tank which collects the surface phase of CSN 7004 and two nozzles that ensure circulation of the bath in the tank have proven to be very efficient in the prevention of overheating effects. The advantages of such a design are:

- The module heater is homogeneously washed around by the plating bath. This prevents local overheating of the tin solution at the surface of the heater.
- Excellent mixing is achieved, which ensures a homogeneous temperature and concentration distribution.
- The tin solution is kept clean from the accumulation of degradation products, because the circulation forces the flow of these products into one corner of the tank. In addition the surface spheres are collected in the chamber which leads them to the filter units for cleaning.

In case of excessive formation of decomposition products it may become necessary to decant the surface of the plating solution in the tank and / or in the chamber though.

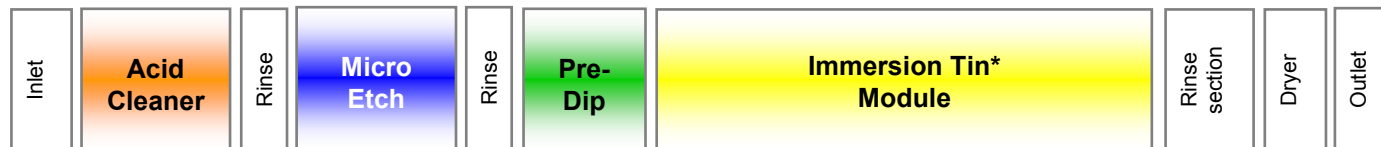


Circulation initiated by two nozzles:

- one with vertical direction,
- the other leading the solution upwards towards the heater in a ~ 45° angle

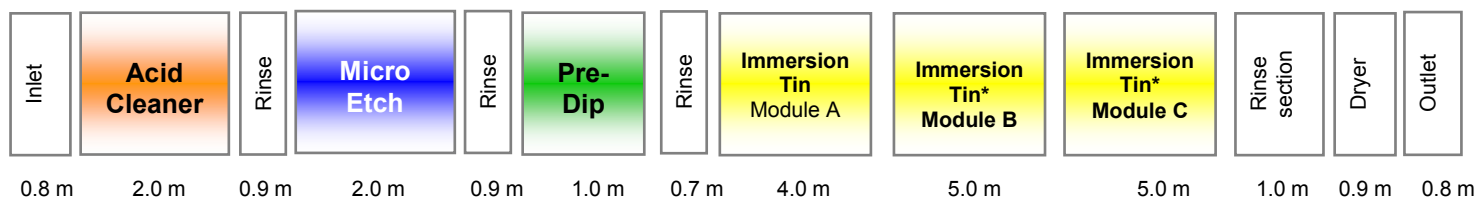
1.5.2 Specification for horizontal ORMECON™ CSN FF and ORMECON™ CSN FF-W lines

Horizontal ORMECON™ CSN FF / ORMECON™ CSN FF-W process sequence



* depending on the line configuration more immersion tin modules are also possible. Due to the operating temperatures of the CSN 7004 bath, heat expansion of the modules should be carefully considered for adequate design. An excessive length of the CSN 7004 modules could lead to problems.

Example of dimensions of a 1.0 m/min. line



Total length of the line: approx. 25.0 m

!	<p>Remark: For lead-free processing OEMs usually specify a minimum tin thickness of around 1.2 μm. In order to achieve this, approx. 14 minutes immersion time in the tin bath is required.</p>	!
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Capacity of horizontal lines

Capacity for one shift working condition						
8 hours/day for 20 days/month						
Line speed [m/min]	Panel 18" x 24"			m ²		
	per h	per day [8 h]	per month [20 d]	per h	per day [8 h]	per month [20 d]
0.25	30	237	4732	8	66	1319
0.30	35	284	5678	10	79	1583
0.50	59	473	9464	16	132	2638
0.80	95	757	15142	26	211	4220
1.00	118	946	18927	33	264	5275
1.50	177	1420	28391	49	396	7913
2.00	237	1893	37855	66	528	10551

Capacity for three shift working condition						
20 hours/day for 26 days/month						
Line speed [m/min]	Panel 18" x 24"			m ²		
	per h	per day [8 h]	per month [20 d]	per h	per day [8 h]	per month [20 d]
0.25	30	591	15379	8	165	4286
0.30	35	710	18545	10	198	5143
0.50	59	1183	30757	16	330	8572
0.80	95	1893	49211	26	528	13716
1.00	118	2366	61514	33	659	17145
1.50	177	3549	92291	49	989	25717
2.00	237	4732	123028	66	1319	34289

Parameter			
	width	length	m ² panel
Panel 18" x 24"	457.2 mm	609.6 mm	0.28 m ²
Panel gap	50 mm		
Panel width + panel gap	507.2 mm		

Space requirement for horizontal lines

Line speed [m/min]	Length [m]
0.25	approx. 11 – 14 m
0.50	approx. 18 – 23 m
1.00	approx. 24 – 30 m
1.50	approx. 33 – 38 m

In order to run a horizontal line with the best possible chemical consumption ratio, it is essential to ensure operation every day with the line running at a minimum of 60% + of its maximum capacity.

So for cost saving purposes it is essential to properly adjust the line's size to the estimated number of boards processed with immersion tin.

Process data for ORMECON™ CSN FF / ORMECON™ CSN FF-W processes

ORMECON CSN FF times and temperatures

Processing speed: recommended (0,25 - 1,5 m/min)

Treatment parameter		Time*	Temperature*
Module 1	Inlet		
Module 2	Acid Cleaner ACL 7001 (optional)	2 min. (1 - 3)	45 °C (40 – 50)
Module 3	2 – 3 stage rinse cascade	~ 1 min.	room temp.
Module 4	Micro Etch MET 7000	~ 2 min.	35 °C (30 - 40)
Module 5	2 – 3 stage rinse cascade	~ 1min.	room temp.
Module 6	Pre-Dip OMP 7000	45 sec. (30 -60)	room temp. (15 - 30)
Module 7	Immersion Tin CSN 7004	≥ 14 min. ¹	68 °C (max. 73)
Module 8	Expansion (optional)		
Module 9	Warm rinse as 3 stage cascade		50 - 60°C
Module 10	DI water rinse	~ 0,5 min	55 °C (50 - 60)
Module 11	Dryer		70 - 80°C
Module 12	Outlet		

¹ Immersion times for lead-free era. For SnPb soldering a minimum of **8 min.** (7 – 11) is sufficient

* Recommended parameter marked in bold, operating range given in brackets

!	<p>The recommended total exposure time in the Immersion Tin is:</p> <p>→ ≥ 8 min. (7 - 11 min.) for SnPb soldering</p> <p>→ ≥ 14 min. (12 - 18 min.) for lead-free soldering</p>	!
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ORMECON CSN FF-W times and temperatures

Processing speed: recommended (0,25 - 1,5 m/min)

Treatment parameter		Time*	Temperature*
Module 1	Inlet		
Module 2	Acid Cleaner ACL 7001 (optional)	2 min. (1 - 3)	45 °C (40 – 50)
Module 3	2 – 3 stage rinse cascade	~ 1 min.	room temp.
Module 4	Micro Etch MET 7000	~ 2 min.	35 °C (30 - 40)
Module 5	2 – 3 stage rinse cascade	~ 1min.	room temp.
Module 6	Pre-Dip OMP 7001	45 sec. (30 -60)	40 °C (35 - 40)
Module 7	Immersion Tin B CSN 7004	≥ 14 min. ¹	68 °C (66 – 70)
Module 8	Expansion (optional)		
Module 9	Warm rinse as 3 stage cascade		25 - 35°C
Module 10	DI water rinse	~ 0,5 min	30 °C (25 - 35)
Module 11	Dryer		70 - 80°C
Module 12	Outlet		

¹ Immersion times for lead-free era. For SnPb soldering a minimum of **8 min.** (7 – 11) is sufficient

* Recommended parameter marked in bold, operating range given in brackets

!	<p>The recommended total exposure time in the Immersion Tin is:</p> <p>→ ≥ 8 min. (7 - 11 min.) for SnPb soldering</p> <p>→ ≥ 14 min. (12 - 18 min.) for lead-free soldering</p>	!
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General technical data horizontal equipment

- Housing material: PP
- Every chamber should supply good agitation of chemicals along the surface and through the holes of PCB's
- **No metal parts in contact with the chemistry**

Pre-Dip OMP 7000 / OMP 7001

- no filter, but circulation pump (product tends to precipitate)
- absolutely no foaming (product is sensitive to oxidation)
- Heater <2 W/cm² (necessary for OMP 7001, heater optional for OMP 7000)
- Strong agitation around the heater at any time of heating

Immersion Tin CSN 7004

- recommended immersion time: 7 - 11 min. for SnPb soldering 12 - 18 min. for lead-free soldering
- absolutely no foaming (product is very sensitive to oxidation)
- Heater surface temperature should not exceed 75°C
- strong agitation around the heater at any time of heating
- two step filtration required with 50 µm followed by 20 µm PP filter
- Exhaust Volume: >500 m³/h

Horizontal machine configuration recommendations

1. Inlet Module	
Material:	PP-S/S
2. Process Module for Acid Cleaner ACL 7001	
Material	PP-S/S
Temperature	40 – 50°C
Process Time	120 sec. (60 – 180 sec)
Squeegee Rollers	EPDM
Filter	10 µm
Heaters	Glass, PTFE, porcelain
Design	Immersion tray with flow system to ensure good agitation through holes
3. Cascade Rinse Module	
Material:	PP-S/S
Temperature	Room temperature
Number of chambers	3
Design	3 chambers: 1. chamber with spray bar (upper and lower conveyor) 2. chamber with immersion tray 3. chamber with spray bar (upper and lower conveyor)
Fresh water supply	to the last chamber
Flow rate	60 - 600 l/h
Cascade overflow	to proceeding chamber
Squeegee Rollers	EPDM
4. Process Module for Micro Etch MET 7000	
Material	PP-S/S
Temperature	35°C (28 – 42°C)
Process Time	120 sec. (45 – 150 sec)

Squeegee Rollers	EPDM
Filter	10 µm
Heaters	Glass, PFTE, porcelain
Design	Immersion tray with flow system to ensure good agitation through holes
5. Cascade Rinse Module	
Material:	PP-S/S
Temperature	Room temperature
Number of chambers	3
Design	3 chambers: 1. chamber with spray bar (upper and lower conveyor) 2. chamber with immersion tray 3. chamber with <u>immersion tray</u> (spray bar is not allowed)
Fresh water supply	to the last chamber
Flow rate	60 - 600 l/h
Cascade overflow	to proceeding chamber
Squeegee Rollers	EPDM
6. Immersion Module for Pre-Dip OMP 7000 / OMP 7001	
Material	PP-S/S
Temperature OMP 7000	15 – 35 °C
Temperature OMP 7001	40°C (35 – 45°C)
Process Time	45 sec. (30 – 60 sec)
Squeegee Rollers	EPDM
Shafts	Carbon fiber
Filter	No filters
Circulation	Necessary, to prevent sedimentation
Heater material (only for OMP 7001)	Glass, PFTE, porcelain
Heater capacity (only for OMP 7001)	< 2 W / cm ²
Cooling	Optional for OMP 7000 / Necessary for OMP 7001
Design	Immersion tray with flow system to ensure good agitation through holes
Important:	<ul style="list-style-type: none"> - No metal parts in contact with the chemistry - Avoid foaming (product is sensitive to oxidation) - Strong agitation around the heater at any time of heating to prevent local overheating

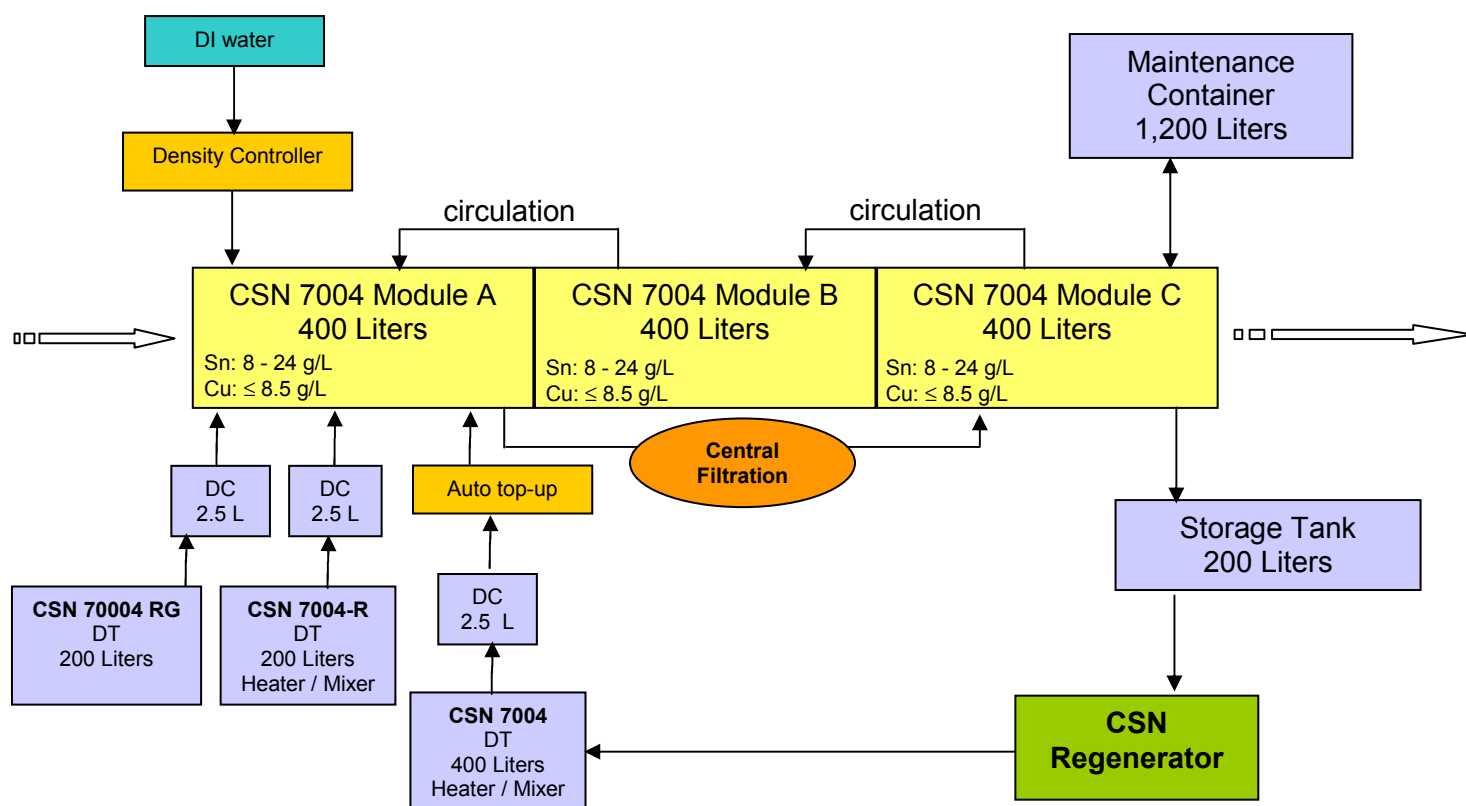
7 – 9 (10) Immersion Modules for Immersion Tin CSN 7004	
Material	PP, suitably coated metal parts
Temperature	68°C (max. 73°C)
Process Time total	12 - 18 min. (14 min.) ¹
Depending on the line speed, it may be necessary to use 3 tin modules, Module C should have the same size as Module B,	
Squeegee Rollers	PP-PVDF or EPDM
Shafts	Carbon fiber
Filter	Two step filtration with PP filters, 50µm followed by 20 µm
Exhaust minimum	500 m ³ / h
Heater material	Glass, PFTE, porcelain
Heater capacity	Heater surface temperature should not exceed 75°C
Strong agitation around the heater at any time of heating (product is very sensitive to partial (local) overheating).	
Design	Option 1: 2 – 3 independent tin modules; Option 2: One tin module only In any case: Immersion tray with adequate agitation along the surface and good agitation through holes (e.g. flow bars)
Density control	Automatic adjustment with DI water
Level control	Automatic with CSN 7004 process solution (readily mixed)
Dosing Containers	Only recommended for automated dosing If one sump is used, transfer pumps to sump only
CSN Regenerator	Recommended when average exposed Cu surface exceeds 20%

Important:	<ul style="list-style-type: none"> - No metal parts in contact with the chemistry - Avoid foaming (product is sensitive to oxidation) - Ormecon heating system preferred - Strong agitation around the heater at any time of heating to prevent local overheating - Stronger chemical agitation in the beginning of the immersion process - Volumes of the chemicals should be kept as small as possible
10 (11). Cascade Rinse Module	
Material:	PP-S/S
Temperature	For CSN FF process: 55°C (50 – 60°C) For CSN FF-W process: 30°C (25 – 35°C)
Number of chambers	3
Design	3 chambers: 1. chamber with spray bar (upper and lower conveyor) 2. chamber with immersion tray 3. chamber with spray bar (upper and lower conveyor)
Fresh water supply	to the last chamber
Flow rate	40 - 400 l/h
Cascade overflow	to proceeding chamber
Squeegee Rollers	EPDM
11 (12). Optional additional rinses (single chamber):	
Ultrasonic rinse	1 - 3 min.
High Pressure rinse	1 - 3 min
High flow rate rinse	1 - 3 min.
RAD 7000 rinse	1 - 3 min. (pH control required)
12 (13). DI Water Rinse Module	
Material:	PP-S/S
Temperature	55°C (50 – 60°C)
Number of chambers	1
Design	1 chamber with spray bars (upper and lower conveyor) or alternative (recommended): 1 Chamber with immersion tray
Flow rate	40 - 400 l/h
Squeegee Rollers	EPDM

12 (13). Dryer Module	
Temperature	70 – 80°C
13 (14). Outlet Module	
Material	PP-S/S

Machine Configuration Details

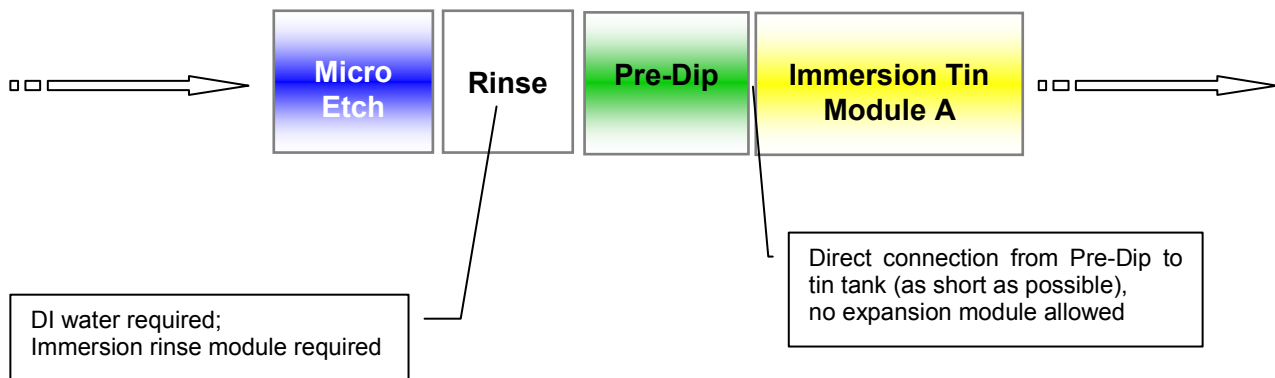
1. Example for Automated Replenishment:



DT = Dosing Tank
DC = Dosing Container

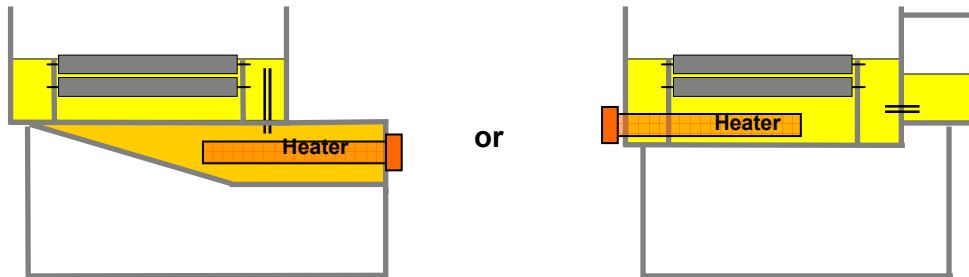
In case of using one sump for the supply of various modules, density control and density dependent DI water addition, as well as CSN 7004, CSN 7004 R and CSN 7004 RG additions are made to the tin bath in the sump. Central filtration will also be installed for sump chemicals only.

2. Detail for OMP 7000 / OMP 7001 section

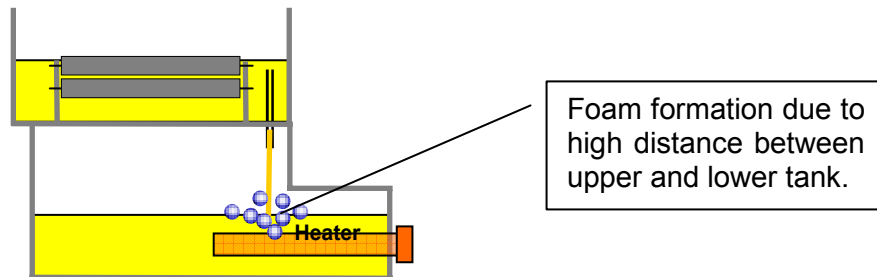


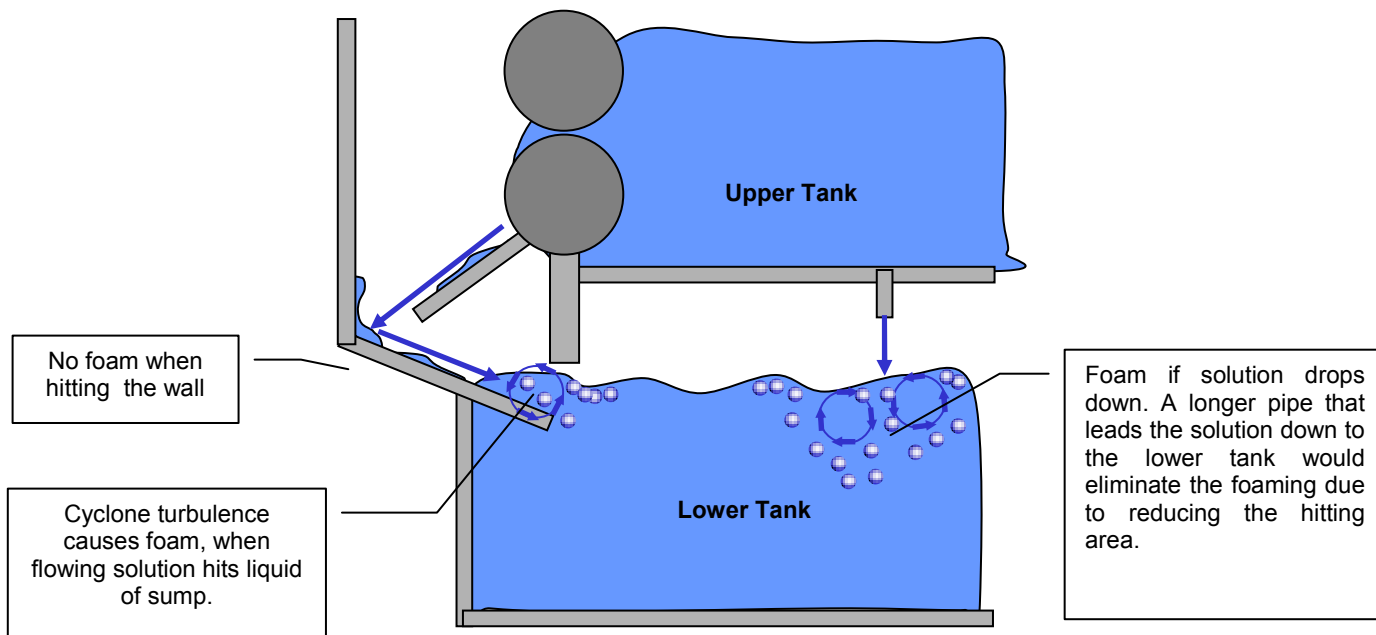
3. OMP 7000 / OMP 7001 / CSN 7004 tank design for foam formation prevention

Suitable Designs:



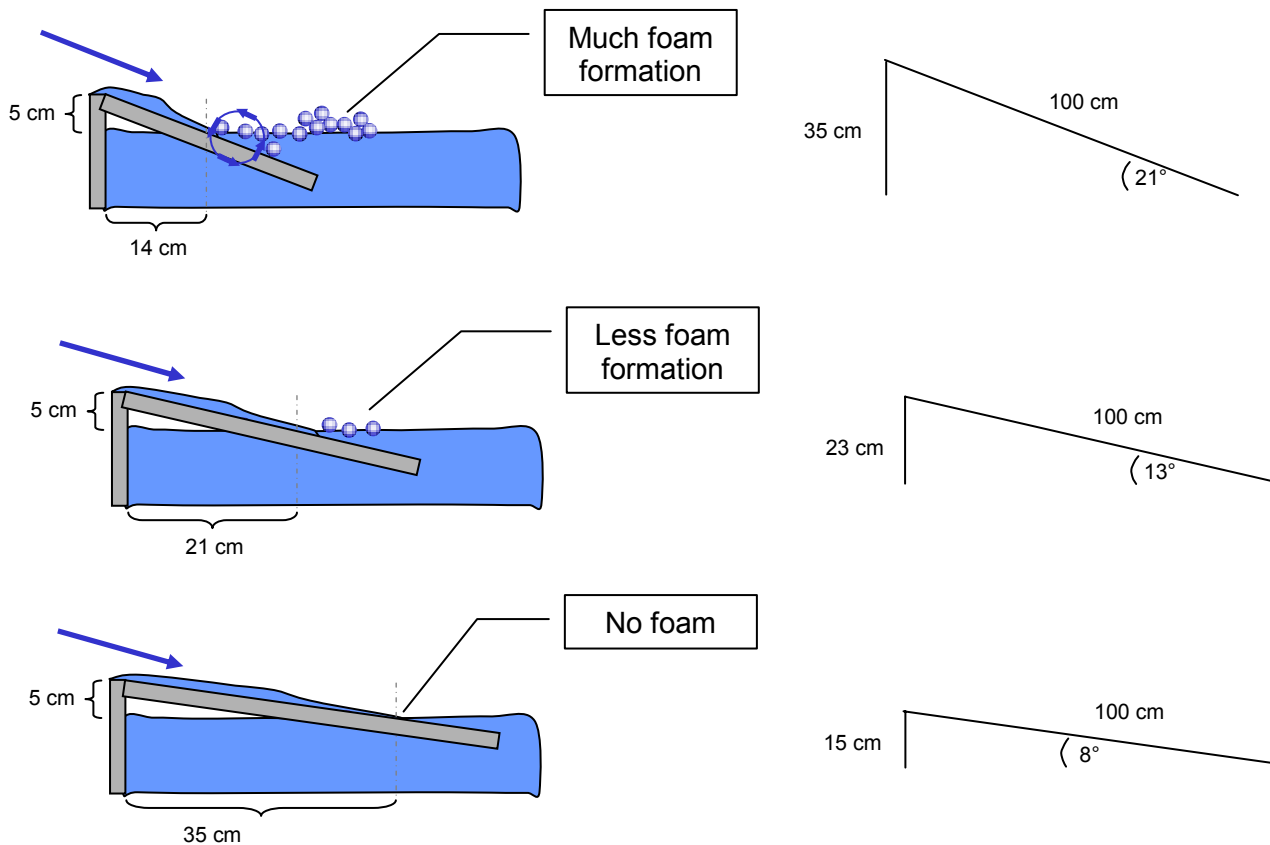
Inappropriate Design



Details:

Conclusion: Foam formation occurs as a result of turbulence when flowing/dropping solution hits another solution's surface.

This can be reduced by changing the slope angle for the solution to flow back from the upper to the lower tank:



It is essential for the process to run economically, to avoid foaming of either the Pre-Dips or the immersion tin bath. Foam is an indication of air in the solution leading to undesired oxidation processes. The consequence is a loss of valuable chemistry and formation of precipitates that require more frequent replenishments and equipment maintenance. This issue should therefore be seriously addressed when designing and manufacturing a horizontal line.

4. Rinse design

Spray rinses and high pressure rinses are not proven to be effective.

It is common thinking, that a high pressure rinse (e.g. with 20 bars) would increase the rinsing effect, especially for small aspect ratios. This is questionable, because

- a) water forms drops under pressure. These drops would be „thrown“ at the PCB with a specific speed. The drop diameters formed under pressure are actually higher than today's standard μ vias and blind vias. How can these drops better penetrate such vias? The water drop would rather be dispersed on the edges rather than moving into the vias.
- b) high pressure water rinsing could also lift the solder mask off the board, especially in case of a chemical attack on the edges of the mask (undercutting effect).

The results have been obtained in a practical study made by a major European PCB manufacturer in cooperation with one of the world's most experienced equipment manufacturer.

Independent from the immersion tin chemistry used, a flow rinse already provides a very good cleaning result.

1.5.3 Rinse water quality for ORMECON™ CSN FF and ORMECON™ CSN FF-W

The deposition quality significantly depends on the purity and cleanliness of the boards. Several rinses should ensure a good cleaning from chemical and other residues between process steps. The rinse water should therefore be kept extremely clean, especially in static rinses. Dirty or contaminated rinse waters, e.g. loaded with process chemicals or other substances, could affect the tin deposit quality. Constant and easy water exchange should be possible.

Except for the final rinse, all rinses could be operated with city water if the quality is in accordance with our specification (see specs below). Should the city water not be clean enough, DI water should be used. However the final rinse must definitely be operated with DI water to avoid ionic contamination from residues, e.g. salt or other substances, left on the board's surface prior to drying.

One parameter indicating the pollution of rinsing water is its conductivity. This parameter should be checked **regularly once a day**. The following table shows the limit values for each single rinsing step:

Module #	Place in process sequence	Water quality	Temperature	Conductivity allowed
Module 2	Rinse after ACL 7001	City water*	Room temp (10 – 35°C)	< 1,500 µS / cm ²
Module 4	Rinse after MET 7000	DI water	Room temp (10 – 35°C)	< 800 µS / cm ² **
Module 7	Rinse after CSN 7004	City water*	For CSN FF: 50 – 60°C For CSN FF-W: 25 – 35°C	< 800 µS / cm ² ***
Module 8	Final Rinse	DI water	For CSN FF: 50 – 60°C For CSN FF-W: 25 – 35°C	< 20 µS / cm ²

* only if quality is in accordance with specified purity (conductivity), otherwise DI water should be used.

** OMP 7001 is sensitive to chloride contamination, so chloride level of this rinse should not exceed 15 mg/L

*** If a pH control is not in use

Rinse waters used for the rinses should be in accordance with the following Water Quality Guideline:

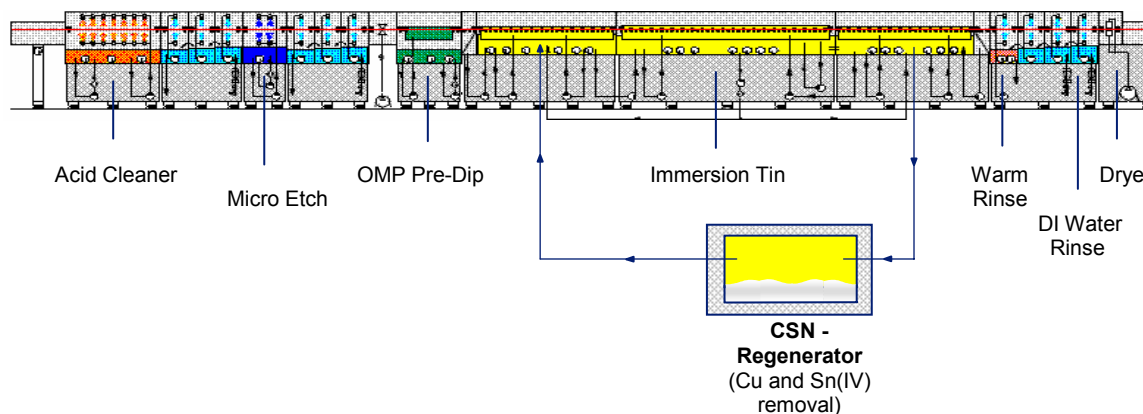
Parameter	Unit	City Water	DI Water
Temperature	°C	10 – 25	10 – 25
pH value		6.5 – 8.0	5.5 – 7.5
Hardness	°dH	< 10	0
COD	mg/L	< 5	< 2
TOC	mg/L	< 5	< 1
Conductivity	µS / cm ²	< 800	< 10
Free Chlorine	mg/L	< 0.3	0
Cations:			
Ca	mg/L	< 70	< 1
Mg	mg/L	< 15	< 0.100
Cu, Al, Zn	mg/L	< 0.5	< 0.100
Ni, Mn, Cr, Sn	mg/L	< 0.5	< 0.020
Fe, Pb	mg/L	< 0.2	< 0.020
NH ⁴⁺	mg/L	< 0.5	< 0.020
Anions			
Sulfate	mg/L	< 120	< 5
Chloride	mg/L	< 100	< 5
Nitrate	mg/L	< 10	< 0.5
Phosphate	mg/L	< 2	< 0.5
Silicate	mg/L	< 10	< 5
Fluoride	mg/L	< 1	< 0.5

Rinsing of complex layouts containing e.g. blind vias or solder mask plugged holes is difficult and rinsing with water only does not remove all process bath residues from the board. This could lead to a high ionic contamination and corrosion attack of the tin finish (especially around holes).

In order to improve final rinsing, Ormecon offers a rinse aid product, called RAD 7000 C. This is a biodegradable, alkaline, organic concentrate to be blended with water, which assists in the removal of residues from the board. An RAD 7000 bath not only helps to neutralize acidic residues left from the CSN 7004 immersion tin bath and therefore improves cleanliness significantly, it is also able to remove other residues and contaminants from the surface. It therefore increases the wettability of the tin, hence improving its multiple solderability (which could be disturbed by residues, especially after multiple heat cycles).

For further product details, please refer to the product's technical data sheet and the analysis procedure on pages 254 - 261 of this Process Guide.

1.5.4 Specification for CSN REGENERATOR for ORMECON™ CSN FF and ORMECON™ CSN FF-W lines



The CSN Regenerator is a piece of equipment that regenerates the immersion tin solution and significantly prolongs the solution's shelf life. It removes copper, Sn(IV) and organic contamination with a simple cooling effect and is operated batch-by-batch.

Copper accumulates in the CSN 7004 working solution over time and affects the tin deposit quality at concentrations of >8.5 g/L. It is normally diluted in the warm plating bath, but tends to crystallize when the temperature drops:

- Copper crystallizes at temperatures $< 21^{\circ}\text{C}$, depending on its concentration
- The amount of copper crystallizing is similar for the entire temperature range, only the time for complete precipitation of copper increases with higher temperatures.
- Tin also crystallizes at temperatures $< 7^{\circ}\text{C}$

The CSN Regenerator usually works with a temperature of $8 - 9^{\circ}\text{C}$, to make sure tin stays in solution, but copper is precipitated as fast as possible. For this temperature window, the regeneration process takes approx. 4 - 8 hours, depending on concentration and temperature prior to cooling. A precipitation at 20°C could take 2 – 5 days, depending on the heat transfer ability and design of the equipment used.

CSN Regenerator operation

The CSN Regenerator consists of a cooled container with a slow turning stirrer (see drawing). The copper rich CSN 7004 solution is collected in an intermediate tank, where it can cool down prior to being regenerated. It is then led into the cooling reactor of the CSN Regenerator. Depending on the operating conditions, the temperature of the copper rich plating solution varies between room temperature and 70°C prior to cooling. After the reactor is filled, the cooling thermostat and stirring device are started to cool down the solution. As soon as the desired temperature is reached the stirrer stops to let the precipitated copper complex settle in the cone of the reactor. Above 7°C no tin will precipitate from the solution!

After sedimentation, the upper clear phase is pumped out through a fine filter system into another intermediate tank for replenishment prior to re-use in the plating bath.

The sludge is separately collected in the sludge container below the reactor and should be dumped properly.

The tin bath should have a copper content of > 4 g/L prior to starting the regeneration process, to achieve a proper saturation level in the tin solution. With lower copper concentration the precipitation process will be inefficient and would require longer.

During the precipitation procedure, the solution will look very cloudy like orange juice with milk. But as the particles grow to form white crystals, they will finally precipitate. The weight/volume of the filtrate makes approx. 15% of the total solution volume used for regeneration. It should be dumped according to local regulations for waste containing metal.

The copper content of the clear phase has been dropped down to 1 - 3 g/L. The tin content should not change. If it is also decreased, the cooling temperature was too low. In this case tin needs to be added as a part of the necessary replenishment procedure.

Before re-using the clear, yellow solution an analysis and replenishment procedure is necessary. The copper has been precipitated as a copper complex. Therefore it is necessary to re-add the complexing agent that was removed with the copper. For this purpose CSN 7004 RG and CSN 7004 R are used. For replenishment details turn to pages 243 - 245.

After replenishment, the solution can be used as / added to the working solution. It has similar properties as fresh CSN 7004, except for a slightly higher copper content (1 - 3 g/L copper).

Sn(IV) removal

Besides copper, Sn(IV) and organic contamination are removed. Sn(IV) usually forms as a result of oxidation due to air incorporation into the working solution. The main issues with Sn(IV) are

- a) the transformation of Sn(II), which is necessary for the desired tin deposition, to Sn(IV) which does not form a metallic deposit. This makes more frequent additions of Sn(II) necessary, so it increases the running costs,
and
- b) the formation of a white crusty Sn(IV) precipitate, that deposits in tanks, pipes, pumps, etc. It significantly increases the maintenance level and sequence and could even destroy sensitive equipment parts (e.g. pumps).

Unlike other available immersion tin baths CSN 7004 does not have quality issues with increasing Sn(IV) content. Standard immersion tin solutions are affected in terms of deposition speed and deposit quality with a Sn(IV) content exceeding 3 g/L. CSN 7004 maintains its deposition velocity and deposit quality up to a Sn(IV) concentration of > 10 g/L. So from a quality control point of view, a Sn(IV) removal is not necessary.

However it should be removed to avoid issues with Sn(IV) precipitates. The online removal of copper and Sn(IV) in the CSN Regenerator process is a simple, cost effective procedure, that makes further special equipment for Sn(IV) treatment unnecessary.

Removal of organic contamination

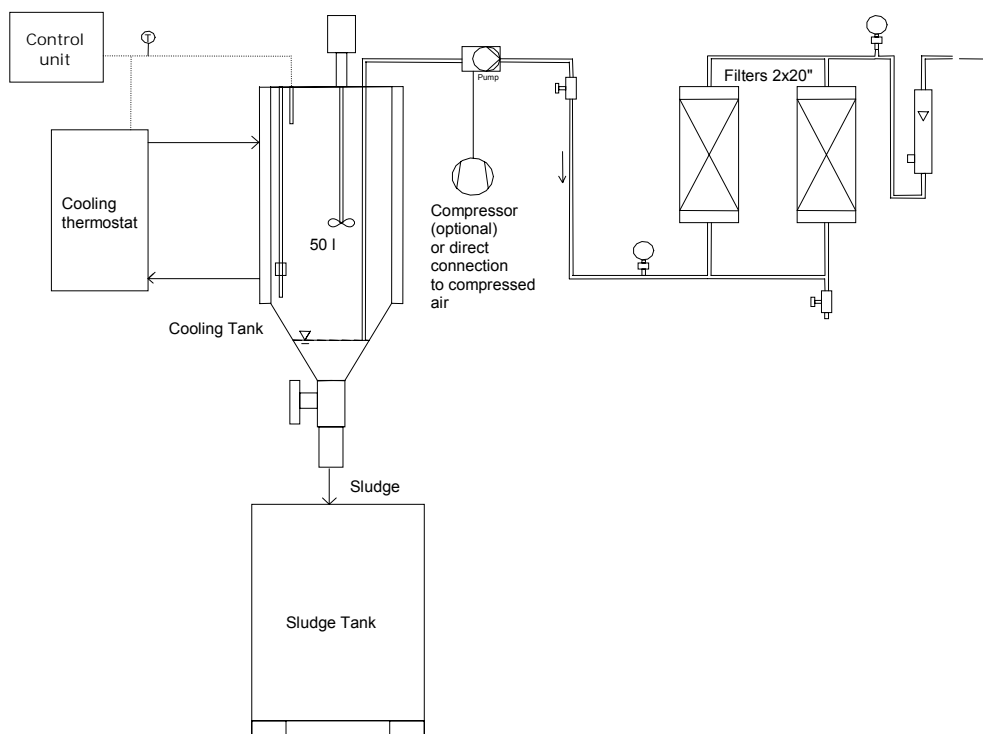
Organic contamination can lead to an irreversible destruction of the working solution (worst case), and usually affects the tin deposit quality even in low concentrations. The copper precipitation process in the CSN Regenerator helps to clean the working solution from organic contamination and helps to maintain the excellent quality of the ORMECON™ CSN FF and ORMECON™ CSN FF-W tin deposit.

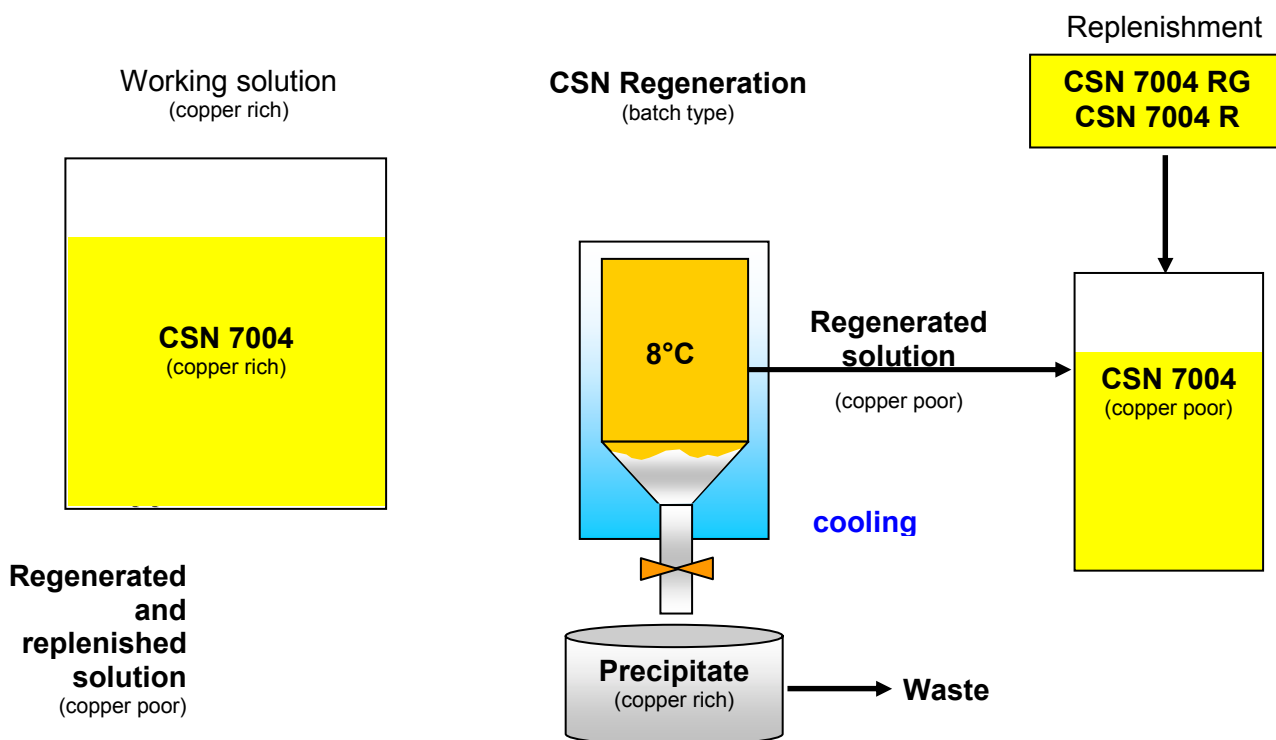
!	<p>The operation of the CSN Regenerator helps to significantly prolong the life of your CSN 7004 working solution. Dumping of chemistry due to high copper load becomes unnecessary. The CSN Regenerator reduces your chemical consumption by 20 – 30%, depending on individual parameters of the line (e.g. copper area, utilization, etc.).</p>	!
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Process sequence:

	Temp. [°C]	Time [h]	Stirring device	Cooling thermostat	Filter pump	
Filling cooling tank						Fill the glass tank (cooling tank) with copper rich immersion tin solution.
↓						
Cooling	8 °C	2-4 h (depending on temp. of the original solution)	On	On	Off	The solution is cooled down to appr. 8 - 9°C. To support cooling use the stirring device to improve heat transfer.
↓						
Sedimentation	8 °C	2-4 h (depending on copper conc. of the original solution)	Off	On	Off	Stop the stirrer and let the precipitates settle in the cone of the cooling tank.
↓						

Filtration clear phase	8 °C	5-10 min.	Off	Off	On	The clear phase is separated from the precipitated sludge in the cone by using the suction tube in the cooling tank. The clear phase is pumped through two 20 " filter units to complete cleaning of the solution.
↓						
Drain off sludge into the sludge container	-	1-2 min	Off	Off	Off	Drain off the sludge by opening the ball cock at the bottom of the cooling tank (appr. 15 Vol%).
↓						
Cleaning cooling tank	-	1-2 min.	Off	Off	Off	Residues are sprayed out with water from the top of the cooling tank.





Copper precipitation without CSN Regenerator

Copper removal can also be realized without the original CSN Regenerator. This is only recommended for the operation of vertical lines. For horizontal processing professional equipment is necessary.

For operating a vertical line it is recommended to use a separate tank / barrel for cooling down the working solution to crystallize excessive copper. It is not recommended to make the crystallization in the original tanks, because

- a) the precipitation process could take 2 – 5 days at room temperature (depending on heat transfer ability and design of equipment used), so the line can not be operated during this time.
- b) in regions with high outside temperature, a cooling of the working solution to 20 – 21°C can not be realized without artificial cooling.
- c) the high amount of precipitation sludge could block filters, pumps, pipes, etc.

A separate tank / barrel could be cooled, even to lower temperatures and the sludge would settle at the bottom, so the solution could be decanted easily to be filtered. No affect on the equipment could occur.

Example:

A 200 Liter PE-container was surrounded by a simple PVC-water pipe. Cold water at 12°C was used to cool the liquid down to 14°C within 2 days. This was necessary because the normal room temperature was close to 30°C.



Inside the simple “Regenerator”:

After 48 hours of cooling to 14°C, crystals had formed. Most of them precipitated at the bottom, some of them

float on top and some cause the liquid to appear cloudy. After carefully decanting, or filtering the liquid, it can be replenished and reused in the CSN 7004 working bath.



! When filtering the solution from the simple “Regenerator”, be aware that the amount of precipitation sludge is quite a lot, approx. 15% of the bath volume. Normal filter cartridges are not recommended. Use a big volume filter bag or decant the liquid carefully without filtering. The small amount of precipitate will be dissolved in the working solution and clears up when heated. But: analysis and replenishment of the decanted solution is necessary prior to re-using it as (part of the) working solution! !

For replenishment details turn to page 243.

1.6 Trouble Shooting Guide

Although ORMECON™ CSN FF and ORMECON™ CSN FF-W only have 4 active steps they must be closely watched to constantly produce high quality Immersion Tin circuit boards.

1.6.1 Sources of Immersion Tin problems

- ◆ Chemicals Solution Control
 Contaminants
- ◆ Substrate Manufacturing history of part
 Preplate treatment
 Soldermask
- ◆ Mechanical Temperature
 Filtration
 Agitation
 Sparging
 Part Orientation
 Placement of Replenishments
 Bath Loading

1.6.2 Problems with process chemistry

PROBLEM	POSSIBLE REASON	CORRECTIVE ACTION
Color change of MET 7000 bath from green to blue	a) Hydrogen peroxide concentration too high b) Concentration of MET 7000 S too low	a) Analyze and adjust hydrogen peroxide concentration together with other MET 7000 components according to replenishment procedure b) Add MET 7000 S
Black-green sediment in original OMP 7000 C / OMP 7001 container	Precipitation of green particles is a typical characteristic of this dispersion	Re-disperse sediment by stirring or shaking
OMP 7000 C contains non-dispersible agglomerates	Probably chemical reaction due to contamination.	Do not use nor filter product. Contact your local supplier
Color change of OMP 7000 / OMP 7001 process bath	Probably chemical reaction due to contamination.	Contact local technical support

PROBLEM	POSSIBLE REASON	CORRECTIVE ACTION
Sediment formation in OMP 7000 / OMP 7001 process bath	Normal sedimentation of a dispersion	Re-disperse sediment by stirring or circulation
CSN 7004 / CSN 7004 R containers contain white precipitates	Crystallization of tin due to low temperature and shortfall of solubility limit	a) Heat up container to 60°C and precipitates will re-dissolve b) If full container volumes are used, empty container into process bath and rinse out precipitates thoroughly with hot process solution.
Hot CSN 7004 process bath contains white precipitates	Sn(IV) crystal formation due to oxidation	a) Check / Exchange filters → Determine Sn concentration in the bath and replenish if necessary. b) Check for foaming indicating aeration of the bath (leading to excessive oxidation) and modify equipment to prevent foaming → Determine Sn concentration in the bath and replenish if necessary.
Cold CSN 7004 plating bath contains white / yellowish precipitates	Copper complex crystallization due to high copper content	Remove copper complex sludge from tank / module, analyze acidity, Sn, Cu, complexing agent of remaining solution and replenish with CSN 7004, CSN 7004 R and CSN 7004 RG
CSN 7004 plating bath contains brown precipitates	a) Contamination b) Result of local overheating and formation of decomposition products	a) make full analysis of plating bath and check solderability and tin thickness. If one feature is out of spec. Stop production and exchange the bath or contact your local technical support for assistance. b) Same procedure as a). Check equipment design (heater capacity, bath agitation, etc) prior to new make-up to prevent local overheating. Adjust heating procedure.

Copper content in CSN 7004 reaches stop value		<p>a) Exchange bath</p> <p>b) Use CSN Regenerator</p> <p>c) Pour (parts of) the plating solution into another tank and replace with fresh CSN 7004 solution to reduce copper content. Cool down separated copper rich solution to 12 - 14 °C and precipitate copper complex. Decant clear solution, replenish properly and use again for additions necessary for plating bath. (see details on pages 66 - 71)</p>
Iron content in CSN 7004 exceeds 5 ppm	Iron emitting source in contact with CSN 7004 plating solution (e.g. from rack, heater, metal screws or fasteners, metal bars, heating / cooling pipes, etc.)	Find and eliminate iron source immediately. (Partial) Exchange of the bath may be required.

1.6.3 Problems with tin deposit

PROBLEM	POSSIBLE REASON	CORRECTIVE ACTION
Skip plating	a) Improper cleaning / etching b) Improper rinsing	a) Check cleaner concentration, temperature and immersion time; check etch rate in micro etch. Inspect panels for grease, solder, soldermask residues, other residues. b) Each solution must be completely rinsed from the panel after each process step. Check rinse water quality (Specifications are given on page 64)

PROBLEM	POSSIBLE REASON	CORRECTIVE ACTION
Stray plating	a) Improper cleaning / etching b) Improper rinsing c) Interference with solder mask d) Isolated gray / brown stains in specific sections of the panel (e.g. along the edges) due to remains of CSN 7004 in „traps“, leading to local corrosion e) Improper OMP 7001 plating f) Agitation too low / too high	a) Check cleaner concentration, temperature and immersion time; check etch rate in micro etch. Inspect panels for grease, solder, soldermask residues, other residues. b) Each solution must be completely rinsed from the panel after each process step. Check rinse water quality (Specifications are given on pages 64) c) Check solder mask for compatibility or contact your tech. support d) Check / Optimize rinsing e) Check Ag deposit after OMP 7001 predip and adjust process bath conditions f) Adjust bath agitation
Gray tin deposit right after make-up (discoloration)	a) Bath has not been properly conditioned prior to use b) Cleaning solution residues left in the tanks / modules prior to make-up Other possible reasons are given in the next line (<i>Gray tin deposit right after plating</i>)	a) Condition CSN 7004 working solution following instructions given on page 29) b) Check beta of tin solution and other features. Bath may need to be (partially) exchanged.

PROBLEM	POSSIBLE REASON	CORRECTIVE ACTION
Gray tin deposit right after plating (discoloration)	c) Improper rinsing d) Improper etching e) Acidity of CSN 7004 too low f) Local overheating g) Improper (uneven) OMP 7001 plating	c) Each solution must be completely rinsed from the panel after each process step. Check rinse water quality (Specifications are given on pages 64) d) Check etch rate in micro etch. Check roughness after etching. Inspect panels for grease, solder, soldermask residues, other residues. e) Adjust acidity f) Use CSN 7004 CAT process. Optimize heater / tank configuration g) Check Ag deposit after OMP 7001 predip and adjust process bath conditions
Dark Gray / black deposit (discoloration)	a) Contamination of CSN 7004 plating bath with e.g. foreign metals like, iron, nickel, zinc, etc. b) Contamination of CSN 7004 plating bath with CEM-1 material c) Interference with solder mask / coverlay (adhesive)	a) Check bath on metal ion content. It may be necessary to exchange the bath. Eliminate source for contamination. Contact your local technical support for assistance. b) Exchange CSN 7004 plating bath c) See page 76

PROBLEM	POSSIBLE REASON	CORRECTIVE ACTION
Interference with solder mask / coverlay (adhesive)	a) Mask processing problem, undercuring	a) Use baking / UV post curing of panels to cure the mask. If that does not fix the problem, it may be a problem of pre-treatment prior to mask application. In this case adjust pre-treatment to improve mask adhesion. If this still does not solve the issue, the problem may be due to mask components and a general incompatibility (lack of chemical resistance) → see c)
	b) Excessive time in CSN 7004 bath	b) Check tin bath temperature and dwell time. Run panels only as long as needed for specified tin thickness
	c) Incompatibility of solder mask / coverlay (adhesive)	c) In case of incompatibility a change of the operating window of the CSN 7004 may help, e.g. lower temperature, longer dwell time. Make sure specified tin thickness is guaranteed. If this is still not a viable option, it may be necessary to change the mask or the coverlay / adhesive system to a more resistant type. Contact your local technical support for assistance.

PROBLEM	POSSIBLE REASON	CORRECTIVE ACTION
Ionic contamination	Improper rinsing	<ul style="list-style-type: none"> - Check all process rinses for correct temperature and cleanliness. Clean rinses if necessary and ensure sufficient fresh water supply to keep rinses clean - Check rinsing ability. Improve rinsing quality by e.g. exchanging nozzles, increasing pressure, use horizontal rinsing, ultrasonic, etc. - Use RAD 7000 for improved cleanliness
Yellowing after reflow	<p>a) Foreign metal contamination</p> <p>b) Organic contamination on the surface</p>	<p>a) Check tin bath for foreign metal contamination, e.g. iron, nickel, aluminum, antimony, etc. and exchange tin bath (partially) if metal contamination was confirmed. Eliminate source for metal contamination. Contact your local technical support for assistance.</p> <p>b)</p> <ul style="list-style-type: none"> - Check all process rinses for correct temperature and cleanliness. Clean rinses if necessary and ensure sufficient fresh water supply to keep rinses clean - Use RAD 7000 for improved cleanliness - Check process for overheating problem in the tin bath

PROBLEM	POSSIBLE REASON	CORRECTIVE ACTION
Yellowing after reflow (continued)	<p>c) Insufficient tin thickness</p> <p>d) excessive oxide formation on the surface</p>	<p>c) Check if pure tin thickness is sufficient for given reflow conditions (consider Sn loss due to diffusion at elevated temperatures. A minimum of 0.1µm of pure Sn needs to remain on the board after final reflow cycle. Check Sn thickness with GCM or SERA)</p> <p>d) High humidity usually leads to accumulated oxide formation. Check drying level of boards prior to stacking / storing / shipping and check storing conditions. Avoid humid conditions.</p>

1.7 FMEA Failure Mode and Effect Analysis

#	Process function / Requirements	Potential Failure Mode	Potential Effect(s) of Failure	Potential Cause(es) / Mechanism(s) of Failure	Current Process Controls	Recommended Actions	Responsibility & Target Completion Date	Actions taken (Date & Initials)
					Precautionary Measures	O S D RPN		
1	Feeding Module	➤ Scratches	➤ Optical Failure		Visual inspection	1 8 2 16		
2	Acid Cleaner Module Cleaner	➤ Insufficient cleaning	➤ Skip plating ➤ Dark stains / spots	➤ Insufficient dwell ➤ Insufficient concentration ➤ Insufficient temperature ➤ Insufficient agitation	➤ SPS ➤ m ² counter for dosing ➤ automatic niveau control in module ➤ automatic temp. controllers ➤ Manual temp. check ➤ Agitation check ➤ Lab analysis	1 1 1 5		
3	Cascade Rinse Water Rinse	➤ Improper rinsing	➤ Drag-out of / Contamination with Acid Cleaner ➤ Uneven micro etching	➤ High pollution level of rinse water ➤ Bad rinse water quality ➤ Insufficient fresh water supply	➤ SPS control of fresh water supply ➤ Automatic niveau control ➤ Temperature control ➤ pH measurement ➤ Conductivity measurement ➤ Lab analysis ➤ Protection against dry running	1 2 6 12		
4	Micro Etch Module Etching	➤ Etch rate too low ➤ Etch rate too high	➤ Poor deposit quality ➤ Poor solderability ➤ Skip plating ➤ Solder mask delamination ➤ Thin copper in the holes and on the pads ➤ Poor solderability ➤ Skip plating	➤ Insufficient dwell ➤ Insufficient concentration ➤ Insufficient temperature ➤ Excessive dwell ➤ Excessive concentration ➤ Excessive temperature ➤ Excessive concentration Cu	➤ SPS ➤ Automatic niveau control ➤ Photometer (Cu conc.) ➤ Automatic temperature controllers ➤ Manual temperature check ➤ Lab analysis ➤ Protection against dry running	3 7 1 21 2 10 1 20		
5	Cascade Rinse Water Rinse	➤ Improper rinsing	➤ Drag-out of / Contamination with micro etch ➤ Improper wetting by pre-dip ➤ Poor tin deposit quality	➤ High pollution level of rinse water ➤ Bad rinse water quality ➤ Insufficient fresh water supply	➤ SPS control of fresh water supply ➤ Automatic niveau control ➤ Temperature control ➤ pH measurement ➤ Conductivity measurement ➤ Lab analysis ➤ Protection against dry running	1 10 1 10		
6A	Pre-Dip OMP 7000	➤ Insufficient deposit	➤ Pre-Pip flocculation / precipitation ➤ poor wettability of tin bath ➤ Skip plating ➤ small grain size tin	➤ Concentration too low ➤ Foaming ➤ Excessive water evaporation ➤ Insufficient buffer dosing ➤ Contamination with Micro Etch ➤ Contamination with tin bath ➤ pH too high	➤ SPS ➤ Automatic niveau control ➤ Dosing control ➤ pH measurement ➤ Lab analysis	4 5 1 20		
O. Occurrence			S. Severeness			D. Detection		RPN: Risk priority number
Improbable			1			Improbable		10
Very seldom			2 – 3			Very seldom		8 – 9
Seldom			4 – 6			Seldom		5 – 7
Moderate			7 – 8			Moderate		3 – 4
Most likely			9 - 10			Most likely		1 - 2
								O x S x D

FMEA Failure Mode and Effect Analysis

#	Process function / Requirements	Potential Failure Mode	Potential Effect(s) of Failure	Potential Cause(es) / Mechanism(s) of Failure	Current Process Controls					Recommended Actions	Responsibility & Target Completion Date	Actions taken (Date & Initials)
					Precautionary Measures	O	S	D	RPN			
6B	Pre-Dip OMP 7001	➤ Ag deposit too thin	➤ Insufficient whisker-reducing effect	➤ Concentration too low ➤ Organic Metal flocculation / precipitation ➤ Foaming ➤ Temperature too low ➤ Excessive copper concentration	➤ SPS ➤ Automatic niveau control ➤ Dosing control ➤ Automatic temperature control ➤ Manual temperature check ➤ Lab analysis ➤ Ag thickness measurement	3	9	2	54			
		➤ Ag deposit too thick	➤ Insufficient tin deposit growth / tin deposit too thin ➤ Skip plating	➤ Concentration too high ➤ Temperature too high		3	9	2	54			
7.	Immersion Tin Modules Immersion Tin A, B,	➤ Acid concentration too high	➤ Solder mask attack	See „Potential Failure Mode“	➤ SPS controlled dosing ➤ Daily bath analysis ➤ Automatic niveau control ➤ Automatic temperature control ➤ Manual temperature check ➤ Protection against dry running ➤ Visual control ➤ Sn thickness measurement	2	8	2	32			
		➤ Acid concentration too low	➤ Hydrolysis ➤ Tin precipitation ➤ Insufficient tin deposit thickness		2	9	2	36				
		➤ Tin concentration too high	➤ No impact		2	8	2	32				
		➤ Tin concentration too low	➤ Decreasing tin deposit thickness		2	9	1	18				
		➤ Copper concentration too high	➤ Solderability drops		3	5	2	30				
		➤ Complexing agent too low	➤ Decreasing tin deposit thickness		2	5	4	40				
		➤ Filtration out of order	➤ Decreasing tin deposit thickness		2	5	2	20				
		➤ Frequency of pumps too high	➤ Agitated bath; foaming; deposit thickness drops		2	5	1	10				
		➤ Temperature too low	➤ Decreasing tin deposit thickness		2	9	2	36				
		➤ Temperature too high	➤ Excessive tin deposit thickness; solder mask attack		2	6	2	24				
8.	Cascade Rinse Water Rinse	➤ Improper rinsing ➤ Residue remains ➤ Ionic contamination	➤ Stains ➤ Spots ➤ Streaks ➤ Poor solderability	➤ High pollution level of rinse water ➤ Bad rinse water quality ➤ Insufficient fresh water supply ➤ Insufficient temperature	➤ SPS control of fresh water supply ➤ Automatic niveau control ➤ Temperature control ➤ pH measurement ➤ Conductivity measurement ➤ Lab analysis ➤ Protection against dry running ➤ Visual control	2	7	2	28			
O. Occurrence				S. Severeness		D. Detection				RPN: Risk priority number		
Improbable Very seldom Seldom Moderate Most likely				1 2 – 3 4 – 6 7 – 8 9 - 10		10 8 – 9 5 – 7 3 – 4 1 - 2				O x S x D		



FMEA Failure Mode and Effect Analysis

#	Process function / Requirements	Potential Failure Mode	Potential Effect(s) of Failure	Potential Cause(es) / Mechanism(s) of Failure	Current Process Controls					Recommended Actions	Responsibility & Target Completion Date	Actions taken (Date & Initials)
					Precautionary Measures	O	S	D	RPN			
9.	Drying Module	➤ Water spots ➤ Staining	➤ Cosmetic defect ➤ Optical Failure	➤ Improper air volume ➤ Improper air temperature ➤ Improper air pressure	➤ Temperature control ➤ Visual control	1	8	1	8			
10.	Solder mask attack	➤ Solder mask delaminates from copper at the mask / copper boundary	➤ Exposed copper		➤ Address to solder mask application unit: ➤ Keep to pretreatment and curing procedures	5	8	1	40			
O. Occurrence				S. Severeness		D. Detection				RPN: Risk priority number		
Improbable		1		Barely distinguishable		10				O x S x D		
Very seldom		2 – 3		Insignificant		8 – 9						
Seldom		4 – 6		Moderately significant		5 – 7						
Moderate		7 – 8		Significant		3 – 4						
Most likely		9 - 10		Crucial		1 - 2						

1.8 Inspection Check Lists for Horizontal Lines

Check List A Frequency: Each Shift

Check List B Frequency: Daily

Check List C Frequency: Weekly

Check List D Frequency: Monthly

These check lists can be followed and used to ensure a proper monitoring of the process and equipment.

1.8.1 ORMECON™ CSN FF / ORMECON™ CSN FF-W - Check List A

Frequency: **Each Shift**

Process Control

Actions	Done / Date / Time / Signature
1. Analyze liquids of all process modules (Acid Cleaner, Micro Etch, Pre-Dip, Immersion Tin)	
2. At beginning of each shift: run test coupons and determine tin plating properties.	
3. Check machine parameters set on PLC	
4. Check arising alarm reports	
5. Check automatic top-up system (if applicable)	
6. Check rinse water input	
7. Check function of auto dosing system	
8. Make manual replenishment according to lab analysis	

Maintenance

Actions	Done / Date / Time / Signature
9. Check function of all switches	
10. Visual inspection of all pumps	
11. Check function of all filtration units	

1.8.2 ORMECON™ CSN FF / ORMECON™ CSN FF-W - Check List B

Frequency: **Daily**

Process Control

Actions	Done / Date / Time / Signature
1. Sampling of process modules (at least daily)	
2. Measure the conductivity in the rinsing Modules	
3. Determination of etch rate	

Maintenance

Actions	Done / Date / Time / Signature
4. Check safety devices of the line	
5. Check all modules and tanks with respect to leakage	
6. Visual inspection of sieves in the immersion tin module	
7. Visual inspection of the transport system	
8. Check efficiency of squeeze rollers	
9. Visual control of spray pattern in rinsing cascades	
10. Check function of nozzles	
11. Check function of drying module	
12. Check function of extraction system	

1.8.3 ORMECON™ CSN FF / ORMECON™ CSN FF-W - Check List C

Frequency: **Weekly**

Process Control

Actions	Done / Date / Time / Signature
1. Measure the temperature in the modules	
2. Measure the temperature in the rinsing modules	

Maintenance

Actions	Done / Date / Time / Signature
3. Replace and clean the nozzles in the immersion tin modules	
4. Change filtration cartridge in the immersion tin modules (higher frequency may be necessary)	
5. Clean final rinse module	
A. Heating elements need to be switched off approx. 20 – 30 min. before start of the cleaning	
B. Close water inlet and leave bypass pipe between cascades open	
C. Drain old rinse water and clean sieves and dirt traps	
D. Rinse with water	
E. Fill up cascade with 5 – 10% NaOH in water	
F. Start circulation pumps and flooding of immersion trays and run for at least 60 minutes	
G. Clean nozzle heads	
H. Inspect transport rollers and clean if necessary?	
I. Remove impurities / residues in the module (manually if necessary)	
J. Drain NaOH solution	
K. Rinse with water until pH has turned neutral (pH should be similar to local water quality)	
L. Fill cascades with fresh water, according to recommended rinse water quality	
M. Adjust rinse water input into the cascades to nominal values?	

1.8.4 ORMECON™ CSN FF / ORMECON™ CSN FF-W - Check List D / 1

Frequency: **Monthly**

Maintenance

Actions	Done / Date / Time / Signature
1. Inspect heating capacity of heating elements	
2. Inspect dry run safety devices in all process modules and rinsing cascades	
3. Inspect driving chain between motor and driving axle	
4. Inspect driving axle with respect to wear and tear	
5. Check functioning of ball valves	

Cleaning

Actions	Done / Date / Time / Signature
6. Cleaning of drying module	
A. Cleaning carried out?	
B. Inspection of filter cloth?	
C. Check function of drying module	
7. Clean rinsing cascade after pre-treatment (Acid Cleaner and Micro Etch)	
A. Close water inlet and leave bypass pipe between cascades open	
B. Drain old rinse water and clean sieves and dirt traps	
C. Rinse with water	
D. Fill up cascade with 5 – 10% NaOH in water	
E. Start circulation pumps and flooding of immersion trays and run for at least 60 minutes	
F. Clean nozzle heads	
G. Inspect transport rollers and clean if necessary?	
H. Remove impurities / residues in the module (manually if necessary)	
I. Drain NaOH solution	
J. Rinse with water until pH has turned neutral (pH should be similar to local water quality)	
K. Fill cascades with fresh water, according to recommended rinse water quality	
L. Adjust rinse water input into the cascades to nominal values?	
Final approval for immersion tin process after chemical cleaning:	Date and Signature:

1.8.5 ORMECON™ CSN FF / ORMECON™ CSN FF-W - Check List D / 2

Frequency: **Monthly**

Module Cleaning (Pre-treatment)

Actions	Done / Date / Time / Signature
1. Turn the line into operation mode „OFF“ and start „Maintenance Program“ by turning maintenance key at the power cabinet	
2. Close valve of auto-top dosing	
3. Heating elements need to be switched off approx. 30 min. prior to emptying the modules?	
4. Remove chemicals	
5. Rinse modules with water	
6. Fill module with water, add NaOH to make a 5% solution and heat up to approx. 40°C. This cleaning step should be run for 4 – 12 h	
7. Drain NaOH solution and rinse with water	
8. Remove impurities / residues manually	
9. Inspect transport rollers and clean if necessary	
10. Fill module with water and run for 30 min. at room temperature	
11. Drain water and rinse with water again	
12. Fill module with water and add sulfuric acid to make-up a 2 – 3 % solution. Heat this up to 50 – 60°C and run for approx. 12 hours	
13. Drain H ₂ SO ₄ solution and rinse with water	
14. Final rinsing water should have neutral pH (or similar to local city water quality)	
15. Check filter system, pumps and piping system for residues and add new filter cartridges	
16. Concentrate valves have been opened again?	
17. Open valve of auto-top dosing	
18. Re-fill modules with chemicals	
Final approval for process module after chemical cleaning:	Signature:

1.8.6 ORMECON™ CSN FF / ORMECON™ CSN FF-W - Check List D / 3

Frequency: **Monthly**

Module Cleaning (OMP 7000 / OMP 7001 Pre-Dip)

Actions	Done / Date / Time / Signature
1. Turn the line into operation mode „OFF“ and start „Maintenance Program“ by turning maintenance key at the power cabinet	
2. Close valve of auto-top dosing	
3. Heating elements need to be switched off approx. 30 min. prior to emptying the modules?	
4. Remove OMP 7000 or OMP 7001 chemicals	
5. Rinse modules with water	
6. Fill module with water, add NaOH to make a 5% solution and heat up to approx. 40°C. This cleaning step should be run for 4 – 12 h	
7. Drain NaOH solution and rinse with water	
8. Remove impurities / residues manually. High pressure water-jet may be necessary to get rid of all green residues.	
9. Inspect transport rollers and clean if necessary	
10. Fill module with water and run for 30 min. at room temperature	
11. Drain water and rinse with water again	
12. Fill module with water and add sulfuric acid to make-up a 1 % solution (pH <3) and run at room temperature for approx. 12 hours	
13. Drain H ₂ SO ₄ solution and rinse with water	
14. Final rinsing water should have neutral pH (or similar to local city water quality)	
15. Check pumps and piping system for residues	
16. Concentrate valves have been opened again?	
17. Open valve of auto-top dosing	
18. Re-fill modules with chemicals	
Final approval for process module after chemical cleaning:	Signature:

1.8.7 ORMECON™ CSN FF / ORMECON™ CSN FF-W - Check List D / 4

Frequency: **Monthly**

Module Cleaning (Immersion Tin Modules)

Actions	Done / Date / Time / Signature
1. Turn the line into operation mode „OFF“ and start „Maintenance Program“ by turning maintenance key at the power cabinet	
2. Valve of auto-top dosing has been closed?	
3. Heating elements need to be switched off approx. 30 min. prior to emptying the modules?	
4. Pump immersion tin solution into the maintenance tanks? Are all pipes empty? Open all valves of the empty piping and close them again after complete emptying of piping system.	
5. Remove filter cartridges?	
6. First rinse water hose and opened concentrate outlets?	
7. Close outlets and production overflow and fill up modules with water	
8. Add sodium hydroxide to get a 5% solution and fill the modules to cleaning level	
9. All pumps in function with heating temperature set to 50 – 60°C	
10. Duration of cleaning sequence is min. 12 hours	
11. Heating elements have been switched off approx. 20 min. prior to draining the cleaning solution into the waste water system? Repeat empty piping procedure.	
12. Rinse the modules with water hose and clean the modules manually where necessary. Close the empty-piping valves.	
13. Close the concentrate outlet valves and re-fill the modules with water. Let the water circulate for at least 30 min. with all pumps running.	
14. Drain the rinse water and use a water hose for rinsing out residues. Lift the rollers and check for dirt traps and residues left here. Clean the sieves and other dirt traps.	
15. In case of sodium hydroxide left in the piping system, a second water rinsing step is necessary. Always empty the piping system as explained above. Don't forget to close the system afterwards.	
16. If no additional water cleaning is necessary, the second chemical cleaning step can be started. Close the concentrate outlet valves, fill the module	

with water and heat it up to 40°C.	
17. Add sodium persulfate (SPS) carefully. As a rule of thumb you may use 1 kg of SPS for 20 liters of water. Finally fill the modules to cleaning level.	
18. Start circulation pumps, the flooding of the immersion tray (set frequency controlled pumps to highest frequency) and the heating. Cleaning temperature should be 50 – 60°C	
19. Duration of the cleaning is min. 12 hours	
20. Switch off heating elements approx. 20 min. before draining cleaning solution to waste water treatment system	
21. Rinse the modules with water hose and clean the modules manually if necessary.	
22. Close the concentrate outlet valves and refill the module with water. Circulate water for at least 30 minutes with all pumps running.	
23. Repeat this rinsing procedure 3 – 4 times, until the final rinsing water has a pH of approx. 6 – 7 (depending on local city water quality)	
24. Change filter cartridges.	
Additional maintenance work during or after chemical cleaning	
25. Inspection of nozzles	
26. Inspection of heating pipes	
27. Inspection of sieves and dirt traps	
28. Inspection of immersion tin tray	
29. Completion of the chemical cleaning and re-start of the line	
A. Immersion Tin solution is pumped back into modules	
B. Level adjustment with CSN 7004	
C. Get back into „Production Mode“ and process solution will be mixed and heated to nominal temperature (68°C)	
D. De-aerate filter units and re-open auto-top dosing	
E. Check the line for leakage	
F. Take samples from process modules and analyze	
G. Process some copper clad laminate panels under production conditions, in order to clean the rollers from residues.	
H. Process test panels for Sn thickness analysis (plus Ag after Pre-Dip in case of CSN FF-W) and solderability check.	
Final approval for immersion tin process after chemical cleaning:	Date and Signature:

ORMECON™ CSN FF / ORMECON™ CSN FF-W

Immersion Tin Processes

2 Analysis Guide

2.1 Introduction

This analysis chapter of the Process Guide is designated to provide a survey about all necessary control parameters and correction actions in order to constantly run the processes within the given specifications and to ensure a perfect quality maintenance of the products. The given information is meant to make the operation and control of the processes as easy as possible. You will find in this section:

- Survey about all process parameters
- Analysis procedures for all process baths
- Replenishment procedures for all process baths
- Analysis protocols (for removal / copy and for using for the process operation)
- Analysis and replenishment records (for removal / copy and for using for the process operation)
- Replenishment tables with options to individually adjust to the customer's bath volumes, to make replenishments even easier (for removal / copy and for using for the process operation)
- Monitoring tables for each process bath (for removal / copy and for using for the process operation)

In case of further questions or remarks, please refer to your local tech support or Ormecon International's technical service @

+ 49 (0)40 60 41 06 – 0

or

pcb@ormecon.de

During the operation of **ORMECON™ CSN FF / ORMECON™ CSN FF-W** regular analysis and replenishment procedures are required to keep the process within specification (the relevant internals and procedures are given on the following pages).

Limit values and operating ranges of all **ORMECON™ CSN FF / ORMECON™ CSN FF-W** products are given on the next pages:

2.1.1 Operating window for process baths

Acid Cleaner ACL 7001 (Pre-Treatment)

Property for analysis	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method
Specific Gravity [g/cm ³]	1.02 – 1.06	1.04	1.03	1.05	1.02	1.06	# 1
Acidity [mol/L]	0.9 – 1.9	1.4	1.1	1.7	0.9	1.9	# 2 B
Temperature [°C]	40 - 50	45	43	47	40	50	--

Micro Etch MET 7000 (Pre-Treatment)

Property for analysis	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method
H ₂ SO ₄ concentr. [g/L]	130 – 210	175	150	195	130	210	# 16
H ₂ O ₂ concentr. [g/L]	15 – 40	25	20	30	15	40	# 3
Copper concentr. [g/L]	< 50	-	-	40	-	50	# 5 B / 6 / 7 B
Etch Rate [µm]	0.8 – 2.4	1.1	0.9	2.0	0.8	2.4	# 4
Color	green	green	-	-	-	blue	--
Temperature [°C]	28 - 42	35	30	40	28	42	--

Pre-Dip OMP 7000 (Organic Metal)

Property for analysis	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method
pH	1.3 – 2.7	2.0	1.5	2.5	1.3	2.7	# 10
Concentration OMP 7000 C [g/L]	20 – 65	50	25	60	20	65	# 8 / 9 A / 9 B
Temperature [°C]	15 – 30	25 (RT)*	17	28	15	30	--

* RT = room temperature

The replenishment instructions for each product given in this analysis guide need to be strictly adhered to.

Pre-Dip OMP 7001 (whisker-reduced)

Property for analysis	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method
Acidity [mol/L]	0.30 – 0.45	0.40	0.35	0.43	0.30	0.45	# 2 B
Specific Gravity[g/cm ³]	1.00 – 1.05	1.02	1.01	1.04	1.00	1.05	# 1
Conc. of whiskerred. component [mg/L]	90 – 250	150	100	220	90	250	# 18
Conc. Org. Metal [mg/L]	20 – 60	40	35	50	20	60	# 9 C
Complexing agent [g/L]	20 – 40	30	22	35	20	40	# 12 B
Copper [mg/L]	< 120	-	-	100		120*	# 5 C
Temperature [°C]	35 - 45	40	37	43	35	45	--

* if Org. Metal flocculates

The replenishment instructions for each product given in this analysis guide need to be strictly adhered to.

2.1.2 Vertical mode

Immersion Tin CSN 7004

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method #
Specific Gravity [g/cm ³] at 20°C at 60 – 70 °C	1.18 – 1.25 1.16 – 1.23	1.20 1.18	1.19 1.17	1.23 1.21	1.18 1.16	1.25 1.23	# 1
Acidity [mol/L]	4.0 – 6.0	4.5	4.2	5.5	4.0	6.0	# 2 A
Sn-Concentration [g/L]	6 – 24	9	7	23	6	24	# 11
Cu-Concentration [g/L]	< 8.5	-	-	8.0	-	8.5	# 5 A / 6 / 7 A
Fe-Concentration [ppm]	Max. 100	≤ 5	-	15	-	100	# 15
Complexing agent [g/L]	90 – 140	100	100	130	90	140	# 12 A / 13
Beta value [β]	0.70 - 0.90	0.80	0.75	-	0.70	-	# 17
Temperature [°C]	Max. 65	60	-*	64	-*	65	--

* value might have to be defined to achieve a minimum required tin thickness

2.1.3 Horizontal mode

Immersion Tin CSN 7004

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method #
Specific Gravity [g/cm ³] at 20°C at 60 – 70 °C	1.18 – 1.25 1.16 – 1.23	1.20 1.18	1.19 1.17	1.23 1.21	1.18 1.16	1.25 1.23	# 1
Acidity [mol/L]	4.0 – 6.0	4.5	4.2	5.5	4.0	6.0	# 2 A
Sn-Concentration [g/L]	6 – 24	9	7	23	6	24	# 11
Cu-Concentration [g/L]	< 8.5	-	-	8.0	-	8.5	# 5 A / 6 / 7 A
Fe-Concentration [ppm]	Max. 100	≤ 5	-	15	-	100	# 15
Complexing agent [g/L]	90 – 140	100	100	130	90	140	# 12 A / 13
Beta value [β]	0.70 - 0.90	0.80	0.75	-	0.70	-	# 17
Temperature [°C]	Max. 73 Min. 40	68	-*	70	-*	73	--

* value depends on minimum required tin thickness

2.2 Analysis of deposits

Besides regular analysis of process baths, it is also highly recommended to check the properties of the immersion tin surface finish on a regular basis.

Contrary to HASL, ORMECON™ CSN FF and ORMECON™ CSN FF-W provide a planar, matte and silvery white tin layer. The color should be even and homogeneously silver. In case of any issue with the optical appearance, e.g. brown, dark gray or even black color as well as stains and streaks, the process conditions need to be carefully checked (see Troubleshooting Guide on 73 - 76)

Besides optical appearance, it is important to check thickness and solderability. This is recommended after every 0.8 m² of panel per liter process bath or once a week (depending on what comes first). For the tests, a separate test board should be run through the line and analyzed for the features given below. One test board per batch is sufficient. Which particular tests are necessary to prove solderability, depends on the customer's requirements and specifications.

2.2.1 ORMECON™ CSN FF / ORMECON™ CSN FF-W tin deposit properties

Sn-Pb

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit
Layer thickness* [μm] Coulometric Method GCM / calibrated X-Ray (Spec Gravity Sn = 7.3g/cm ³)	0.20 – 1.50	The required thickness is dependant on the desired soldering conditions (See Classification Sheet on page 100)				
Solderability Test [%] 155°C / 4h (Solderwave) Interflux IF 2005 M (ERSA), Sn60Pb40	< 1.0	< 1.0	-	-	-	1.0
Wetting angle [°] Fresh 155°C / 4h Kolo 300-25 (Stannol), Sn60Pb40	0 - 95 0 - 105	75 75	- -	- -	- -	95 105
Wetting angle [°] Fresh 155°C / 4h Alpha NR330 (Alphametals), Sn60Pb40	0 - 55 0 - 55	30 30	- -	- -	- -	55 55

Pb-free

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit
Layer thickness* [μm] Coulometric Method GCM / calibrated X-Ray (Spec Gravity Sn = 7.3g/cm ³)	0.20 – 1.50	The required thickness is dependant on the desired soldering conditions (See Classification Sheet on page 100)				
Wetting angle [°] Fresh 155°C / 4h Alpha NR330 (Alphametals), Sn96.5Ag3.5	0 - 90 0 - 90	30 30	- -	- -	- -	90 90

*Layer thickness is a recommended value, it may be necessary to define a different range with specific customer

For ORMECON™ CSN FF-W it is necessary to regularly analyze the thickness of the applied silver layer from the Pre-Dip. If the silver layer is within the specified range, a whisker reduction effect can be guaranteed.

2.2.2 ORMECON™ CSN FF-W whisker-reducing Ag layer

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit
Layer thickness [nm] Coulometric Method GCM (S.G.Ag = 10.5 g/cm ³)	15 - 45	25	18	40	15	45

2.2.3 Classification of Tin Thickness and Solderability Specifications

Level	Tin Thickness Range [μm]	Soldering and Storage Conditions	
		Sn-Pb	Pb-Free
1	≥ 1.15	1 Year Storage + 7x Reflow	1 Year Storage + 4x Reflow
2	1.05 - 1.14	1 Year Storage + 6x Reflow	1 Year Storage + 3x Reflow
3	0.95 - 1.04	1 Year Storage + 5x Reflow	1 Year Storage + 2x Reflow
4	0.80 - 0.94	1 Year Storage + 4x Reflow	1 Year Storage + 1x Reflow Tin Thickness Range = 0.4 - 0.94 μm
5	0.72 - 0.79	1 Year Storage + 3x Reflow	
6	0.65 - 0.71	1 Year Storage + 2x Reflow	
7	0.40 - 0.64	1 Year Storage + 1x Reflow	
8	0.30 - 0.39	6 Months Storage + 1x Reflow	6 Months Storage + 1x Reflow
9	0.20 - 0.29	1x Reflow	1x Reflow

* Notes

- 1) Pure tin thickness was measured coulometric with a GCM; S.G. 7.3g/cm³
- 2) Storage at 22°C in a non corrosive atmosphere
- 3) These values give no guarantees; they are just recommendations.
Deviations due to different storage or reflow conditions are possible.

2.2.4 Analysis Methods for ORMECON™ CSN FF/FF-W

Analysis Method	Analysis procedure for...	Analysis applicable for...
# 1	Specific Gravity	ACL 7001 / MET 7000 / CSN 7004 / OMP 7000 / OMP 7001 / RCL 7000
# 2 A	Acidity (Titration)	CSN 7004
# 2 B	Acidity (Titration)	ACL 7001/ OMP 7001
# 2 C	Acidity (Titration)	OMP 7000
# 3	Hydrogen Peroxide (H ₂ O ₂) content (Titration)	MET 7000
# 4	Etch Rate	MET 7000
# 5 A	Copper (AAS)	CSN 7004
# 5 B	Copper (AAS)	MET 7000
# 5 C	Copper (AAS)	OMP 7001
# 6	Copper (Titration)	CSN 7004 / MET 7000
# 7 A	Copper (UV-Vis)	CSN 7004
# 7 B	Copper (UV-Vis)	MET 7000
# 8	Organic Metal (visual standards)	OMP 7000
# 9 A	Organic Metal - 20% solution (UV-Vis)	OMP 7000
# 9 B	Organic Metal - 100% solution (UV-Vis)	OMP 7000
# 9 C	Organic Metal - 100% solution (UV-Vis)	OMP 7001
# 10	pH	OMP 7000 / RAD 7000 / RCL 7000
# 11	Stannous Tin (Sn (II)) (Titration)	CSN 7004
# 12 A	Complexing agent (UV-Vis)	CSN 7004
# 12 B	Complexing agent (UV-Vis)	OMP 7001
# 13	Complexing agent (Titration)	CSN 7004
# 14	Tin oxide (Sn (IV)) (AAS)	CSN 7004
# 15	Iron (AAS)	CSN 7004
# 16	Sulfuric Acid content (Titration)	MET 7000
# 17	Beta of CSN 7004	CSN 7004
# 18	Silver (Ag) content (AAS)	OMP 7001
# 19	RAD 7000 C content (Titration)	RAD 7000
# 20	Conductivity	RCL 7000

2.2.5 Analysis chemicals and equipment for ORMECON™ CSN FF/FF-W

Analysis Chemicals p.a.	Analysis Equipment
DI water	<u>Lab equipment:</u>
Sulfuric Acid (10% dilution)	25mL volumetric flasks
Sulfuric Acid conc. (96%)	50 mL volumetric flasks
Ammonium Chloride (NH ₄ Cl)	100 mL volumetric flasks
25% Ammonia (NH ₄ OH)	250 mL volumetric flasks
35% Hydrogen Peroxide (H ₂ O ₂)	500 mL volumetric flasks
Sodium Acetate tri-hydrate	1000 mL volumetric flasks
50% Hypophosphorous acid	100 mL graduated cylinders
10% HCl	250 mL Erlenmeyer flasks
10% Nitric Acid (HNO ₃) solution	Peleusball
200 x 100 mm double sided copper clad laminate	0.1 mL pipettes
	0.5 mL pipettes
<u>Indicators p.a.:</u>	1.0 mL pipettes
Cresol Red (0.1g in 99.9 g Ethanol (20%))	2.0 mL pipettes
Ferrouin solution (e.g. from Merck)	5.0 mL pipettes
Methyl Orange solution (0.04 g in 100 mL Ethanol (20%))	10.0 mL pipettes
PAN indicator solution	20 mL pipettes
(1-(2-Pyridylazo)-2-naphtol 0.1% in Methanol (95-100%))	25 mL pipettes
Xenol Orange (1% ground with KNO ₃)	50.0 mL pipettes
Bromophenol blue (0.1g in 100 mL Ethanol (20%))	5.0 mL measuring pipettes
	Alternative for pipettes: transfer pipettes
<u>Titration solutions:</u>	25.0 mL burettes
0.1 mol/L Cer(IV) sulfate	UV-Vis cuvettes
1 mol/L NaOH	25 mL beaker
0.1 mol/L EDTA - 2 Na (Di-sodium salt of EDTA)	100 mL beaker
0.02 mol/L KMnO ₄	
1 mol/L HCl	<u>Analytical equipment:</u>
	Analytical Balance
<u>Standard solutions: for AAS</u>	AAS
Copper standard solution (1000 mg/L, e.g. CertiPur from Merck)	UV-Vis Spectrometer
Silver standard solution (1000mg/L)	pH meter
Iron standard solution (1000 mg/L)	Thermometer
Tin standard (1000 mg/L)	
	Tin thickness measurement:
<u>Standard solutions: for pH-Meter „Knick Portameas“</u>	GCM (preferably), SERA, Thix-tester;
Standard buffer solution for pH = 4.0	Alternatively X-RF
Standard buffer solution for pH = 7.0	
Standard buffer solution for pH = 9.0 (only if required by pH meter)	Solderability test unit (optional a) or b]):
	a] oven for aging (with sufficient peak temp.) + solder pot
	b] solder paste printing (stencil) + reflow oven (with sufficient profile)

2.3 General Analysis Guidelines

During the operation of the process, the baths suffer from drag-out and evaporation losses. These losses need to be observed very carefully and regular replenishments of lost ingredients are necessary to maintain a proper functionality of the process bath.

The process steps have different requirements for replenishment due to their specific composition, operation conditions and function. The necessary analysis requirements can be found in the related sections of this Process Guide.

All analysis procedures should only be made after compensation of evaporation losses. It is necessary to add water to the process bath first until specific gravity has reached its nominal value, prior to taking a sample. Otherwise the analysis would lead to false results.

The main component of most of the process baths used in the ORMECON™ CSN FF process is DI water. So it is the main component to be lost by evaporation and drag-out. Consequently for ACL 7001, MET 7000, OMP 7000, CSN 7004, RAD 7000 and RCL 7000, prior to taking out a sample for analysis, DI water should be added, until the original solution level has been reached. If you do not do this the analysis would provide wrong results in terms of concentration (higher than they originally would be) and the replenishment procedures derived from those results would be inaccurate. The bath level would constantly drop, and the baths would run out of specification.

Normally replenishment is made in two steps:

- a) General replenishment additions are made regularly without analysis in the course of operating the bath (e.g. after each small batch, every day prior to re-start, after a longer stand still period, etc.)
- b) An analysis and data driven full replenishment procedure is required after each **7 m² (75 ft²)** panel / liter bath volume. This applies only if regular replenishments, as described in A), have been made in between.

A full analysis of the CSN 7004 tin bath is highly recommended after each 1 m² (11 ft²) panel / liter bath volume, because the tin bath suffers more from evaporation losses, due to its high operating temperatures, and besides normal drag-out tin is actively removed from the solution due to plating on the boards. A more regular monitoring is therefore necessary!

Guidelines for both replenishment procedures can also be found in the product related sections of the Process Guide.

A survey for the general "rule-of-thumb additions" as described in A) is given on the following page. The replenishment quantities given here are regular addition quantities necessary to keep the bath within specification between analysis intervals. The quantities in the survey are also given in the respective product analysis chapter.

Replenishment procedures and quantities for special products, such as OMP 7075 STAB, CSN 7004 CAT, CSN 7004 RG, RAD 7000 C, etc. are also given in the respective section of the analysis guide.

2.3.1 Replenishment Survey for Process Baths

ACL 7001 Process Bath

DI water for level adjustment and evaporation loss substitution

ACL 7001 C	for vertical operations:	12 mL / m ² treated panel	1.1 mL / ft ² treated panel
	For horizontal operations:	10 mL / m ² treated panel	0.9 mL / ft ² treated panel

MET 7000 Process Bath

DI water for level adjustment and evaporation loss substitution

MET 7000 S	for vertical operations:	3 mL / m ² treated panel	0.3 mL / ft ² treated panel
	for horizontal operations:	1 mL / m ² treated panel	0.1 mL / ft ² treated panel

H ₂ SO ₄ (96%)	for vertical operations:	12 mL / m ² treated panel	1.1 mL / ft ² treated panel
	for horizontal operations:	4 mL / m ² treated panel	0.4 mL / ft ² treated panel

H ₂ O ₂ (35%)	for vertical operations:	8 mL / m ² treated panel	0.8 mL / ft ² treated panel
	for horizontal operations:	3 mL / m ² treated panel	0.3 mL / ft ² treated panel

OMP 7000 Process Bath

DI water for level adjustment and evaporation loss substitution

OMP 7000 C	for vertical operations:	5 mL / m ² treated panel	0.5 mL / ft ² treated panel
	for horizontal operations:	1 mL / m ² treated panel	0.1 mL / ft ² treated panel

OMP 7000 B	for vertical operations:	2.5 mL / m ² treated panel	0.23 mL/ft ² treated panel
	for horizontal operations:	0.5 mL / m ² treated panel	0.05 mL/ft ² treated panel

OMP 7001 Process Bath

Based on analysis only!

CSN 7004 Process Bath

DI water for level adjustment and evaporation loss substitution

CSN 7004 R	for horizontal operations:	45 mL / m ² treated panel	4.2 mL / ft ² treated panel
	for vertical operations:	35 mL / m ² treated panel	3.3 mL / ft ² treated panel

CSN 7004	for all operations:	80 - 120 mL / m ² treated panel	
		7.4 - 11.2 mL / ft ² treated panel	

Acid Cleaner for

ORMECON™ CSN FF / ORMECON™ CSN FF-W

2.4 ACL 7001 C

Product Description

ACL 7001 is an acidic copper cleaner with special degreasing properties, and is designed for pre-treatment use in the ORMECON™ CSN immersion tin processes. It cleans the circuit board surface from grease and oils, developer residues after solder mask application and other impurities.

ACL 7001 provides the following advantages:

- Efficient removal of residues from the surface
- Very good rinsability
- Long bath life
- Free of complexing agents
- **ACL 7001 C** is supplied as a concentrate for blending with DI water

Application and Make-up

ACL 7001 C is a concentrate that has to be mixed with DI-water. Standard concentration is 12.5 vol%. It can be increased to 15 vol% if necessary for better cleaning results. However, it should not exceed 15 vol%.

Make-up recommendation:

DI water 87.5 vol%

ACL 7001 C 12.5 vol%

For replenishment: The concentration of ACL 7001 solutions can be maintained with additions of **ACL 7001 C** concentrate.

Product Specification ACL 7001 C

Nominal values / standard parameters are given in brackets ().

State:	liquid
Odor:	slightly acidic, stingy
Color:	colorless
Specific Gravity:	1.28 – 1.32 g/cm ³ (1.30)
Acidity:	10.5 – 12 mol/L (11.3)
pH-Value:	highly acidic
Chem. Characterization:	aqueous acidic organic concentrate

Parameters of ACL 7001 Process Bath

Nominal values / standard parameters are given in brackets ().

Concentration of ACL 7001 C:	10 - 15 vol% (12.5)
Temperature Range:	40 to 50 °C (45)
Acidity:	0.9 – 1.9 mol/L (1.4)
Specific Gravity (density):	1.02 – 1.06 g/cm ³ (1.04)
Dwell Time:	1 - 3 min (2)
Agitation:	mild work agitation and solution agitation and filtration recommended
Application:	vertical: immersion / horizontal: immersion or spray

ACL 7001 C

Equipment Material

Tanks:	PP; do not use steel or other metals
Heaters:	Quartz or Teflon/PTFE
Racks / Baskets:	Can be metal structures, but need to be coated with a) pore-free black or green HALAR (do not use blue HALAR!) b) pore-free PP coated stainless steel Baskets can also be completely made from PP No metal is allowed in contact with the solution!
Ventilation:	Advised
Agitation:	Recommended
Filtration:	Advised

Storage Requirements

Do not store together with alkaline or cyanide products and avoid contact with them. Do not use metal containers. Keep original containers tightly closed. Ensure good air circulation.

Shelf Life

12 months from production date, if stored according to storage recommendations.

Waste Removal

ACL 7001 C is acidic and needs to be diluted and neutralized before discharge into the public sewage system, preferably in a common batch neutralization.

ACL 7001 cleaning solutions have to be decontaminated prior to being discharged into public sewage systems. Used **ACL 7001** solutions contain copper which needs to be precipitated in a common batch neutralization. The filter cake contains larger amounts of copper and must be treated as special waste. The local waste water regulations should be adhered to.

Packaging

5, 10 and 25 L PE containers. Other package sizes upon request.

Safety Recommendations

ACL 7000 C is highly acidic. For handling wear rubber gloves and eye protection, if possible also wear a rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on Safety Data Sheet.

ACL 7001

2.4.1 Operating window for process bath

Acid Cleaner **ACL 7001**

Property for analysis	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method #
Specific Gravity @ 20°C [g/cm³]	1.02 – 1.06	1.04	1.03	1.05	1.02	1.06	# 1
Acidity [mol/L]	0.90 – 1.90	1.40	1.10	1.70	0.90	1.90	# 2 B
Temperature [°C]	40 – 50	45	43	47	40	50	--

ACL 7001

2.4.2 Control and Analysis Procedures for Acid Cleaner

Control Procedures:

Nominal values / standard parameters are given in brackets ().

Acidity: 0.9 – 1.9 mol/L (1.4)
Specific Gravity (density): 1.02 – 1.06 g/cm³ (1.04)

Specific gravity (density) is directly correlated with acidity. Specific Gravity measurement is useful as a control procedure to check the state of the bath / replenishment result.

Replenishment is made with *ACL 7001 C*

Note: The main component of *ACL 7001* is water. For proper replenishment it is necessary to first compensate evaporation and drag-out losses by adding DI water. Compensating chemical losses should only be taken care of after this.

We recommend making regular additions to the *ACL 7001* process bath based on usage:

- Add DI water until the original level is almost reached. Do not fill up to the original level at this point, because space for *ACL 7001 C* addition is required.
- Add *ACL 7001 C* 12 (10)* mL / m² treated panel (1.1 (0.9)* mL / ft² of treated panel)
- Fill up to the original level with DI water, if still necessary.

* values given for horizontal processing

Perform complete bath analysis after 7 m² (75 ft²) of treated panel per liter bath volume. Then make bath corrections based on analysis following the given procedure:

1. Add DI water until the original level is almost reached
2. Take a sample and analyze acidity of the process bath solution
3. Add corresponding amount of *ACL 7001 C*
4. Fill up to original liquid level with DI water, if necessary, and re-check acidity.

The corresponding analysis methods are described below.

ACL 7001

Control and Analysis Procedures for Acid Cleaner

2.4.3 Analysis

Acidity Determination (Method # 2 B)

- Fill approx. 50 mL of DI water into a 250 mL Erlenmeyer flask
- Pipette 10 mL of ACL 7001 process solution into the flask
- Add DI water until the entire sample volume is 100 – 150 mL
- Add 8 – 10 drops of Cresol Red indicator solution
- Titrate with 1.0 mol/L sodium hydroxide solution (NaOH) from orange over yellow to the purple endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop ($=0.05$ mL), make two additional titrations.

Calculation:

$$\text{Acidity [mol/L]} = \text{Volume of 1.0 mol/L NaOH used} \times F / 10$$

Factor F = Factor of NaOH (if unknown, use F=1)

Replenishment:

Operating Range: 0.9 – 1.9 mol/L (1.4 mol/L nominal)

$$\text{Addition of ACL 7001 C [Liter]} = \frac{1.4 - \text{Acidity result [mol/L]}}{11.3} \times \text{bath volume [Liter]}$$

Additions of ACL 7001 C should be made to the bath to adjust acidity.

Specific Gravity Determination (Method # 1)

- The solution has to have a temperature of 20°C (if not, warm the solution up / cool it down to 20°C, before filling)
- Tare a dry 100 mL volumetric flask on an analytical balance
- Fill to mark with ACL 7001 working solution
- Record the mass of the ACL 7001 solution

→ Make 3 measurements and determine average value

Calculation:

$$\text{Specific Gravity (Density) [g/cm}^3\text{]} = \text{mass in g} / 100$$

We do not recommend making corrections to the ACL 7001 process bath based on specific gravity. This analysis is intended only as a measure of the state of the bath or to control the replenishment made.

ACL 7001

Analysis Protocol

- Evaluation of acidity by neutralization -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water
1.0 mol/L NaOH
Cresol Red
(0.1 g in 99.9 g of Ethanol 20%)

Equipment: 25 mL Burette
10 mL Pipette
3 x 250 mL Erlenmeyer flask

Procedure:

1. Fill approx. 50 mL of DI water into a 250 mL Erlenmeyer flask
2. Pipette 10 mL of ACL 7001 process solution into the flask
3. Add DI water until the entire sample volume is 100 – 150 mL
4. Add 8 – 10 drops of Cresol Red indicator solution
5. Titrate with 1.0 mol/L sodium hydroxide solution (NaOH) from orange over yellow to the purple endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation Acidity [mol/L] = Average volume of 1.0 mol/L used NaOH x F / 10

Factor F = Factor of KMnO_4 (if unknown, use F=1)

Analysis results: Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Average Consumption of NaOH: _____ mL

Acidity of ACL 7001 process bath : _____ mol/L

ACL 7001

Analysis Protocol

- Determination of Specific Gravity -

Date of analysis: _____ Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: ---**Equipment:** Analytic balance with 0.01 g precision
100 mL volumetric flask (calibrated)
Thermometer**Procedure:**

1. The solution has to have a temperature of 20°C (if not, warm the solution up / cool it down to 20°C) before filling
 2. Tare a dry 100 mL volumetric flask on an analytical balance
 3. Fill to mark with ACL 7001 working solution
 4. Record the mass of the ACL 7001 solution
- Make 3 measurements and determine average value

Calculation:
$$\text{Specific Gravity (Density) [g/mL]} = \text{mass [g] of ACL 7001 solution} / 100$$
Analysis results:

Weight of solution: _____ g Specific Gravity: _____ g/mL

Weight of solution: _____ g Specific Gravity: _____ g/mL

Weight of solution: _____ g Specific Gravity: _____ g/mL

Average Specific Gravity of ACL 7001 solution: _____ g/mL

ACL 7001

Analysis and Replenishment Record

Bath volume: _____ Liter Date of analysis: _____ . _____ . _____

Throughput: _____ m² / ft² Analyzer (initials): _____

Capacity / Liter: _____ m²/L or ft²/L (throughput : bath volume)

Date of installation: _____ . _____ . _____ Signature: _____

Physical Data:

	OK	NOK
Form:	watery	oily
Color:	colorless @ room temp	colored/hazy @ room temp
Appearance:	(white) hazy/cloudy @ 45°C	different
Foam formation:	slight	strong

Analysis results:

Acidity: _____ mol/L

Replenishment:

Operating Range: 0.9 – 1.9 mol/L (1.4 mol/L nominal)

Calculation of necessary replenishment quantity:

$$\text{Addition of ACL 7001 C [liter]} = \frac{1.4 - \text{Acidity result [mol/L]}}{11.3} \times \text{bath volume [liter]} = \underline{\hspace{2cm}} \text{ L}$$

Analysis and replenishment result for ACL 7001 process bath:

Bath is ok, no replenishment necessary

Bath needs to be replenished with _____ L of ACL 7001 C

Bath needs to be exchanged

Comments: _____

ACL 7001

Replenishment Table

Example for 100 liters of bath volume

Values related to your specific bath volume:

_____ liters

Acidity analysis result	Addition of ACL 7001 C [L]	Addition of ACL 7001 C [L]
1.4 mol/L	0.00	
1.3 mol/L	0.90	
1.2 mol/L	1.80	
1.1 mol/L	2.70	
1.0 mol/L	3.50	
0.9 mol/L	4.40	
0.8 mol/L	5.30	
0.7 mol/L	6.20	
0.6 mol/L	7.10	
0.5 mol/L	8.00	
0.4 mol/L	8.90	

Bath within spec

Calculation:

$$\text{Addition of ACL 7001 C [liter]} = \frac{1.4 - \text{Acidity result [mol/L]}}{11.3} \times \text{bath volume [liter]}$$

Micro Etch for

ORMECON™ CSN FF / ORMECON™ CSN FF-W

2.5 MET 7000

Product Description

MET 7000 is an acidic copper cleaner, based on hydrogen peroxide and sulfuric acid, stabilized with **MET 7000 S**. It is perfectly suitable for use prior to the tinning process, because it creates a smooth copper surface, which is important for the topography of the final tin finish.

MET 7000 offers the following advantages:

- Smooth and consistently even etch of the copper surface
- Prevents tarnishing prior to tinning process
- Very good rinsability
- Long life cycle
- Free of complexing agent
- **MET 7000 S** is only a stabilizer. 98 vol.% of the remaining necessary products can be submitted by the user (but are commodities used by PCB shops anyway)

Application and Make-up

MET 7000 S is a stabilizer for a hydrogen peroxide/ sulfuric acid based etching solution, that is added with only 2 vol.%.

Make-up recommendation:

DI water	81.5 Liters	or	81.5 kg
Sulfuric Acid (H ₂ SO ₄ , 96%)	10.0 Liters	or	18.5 kg
MET 7000 S	2.0 Liters	or	2.2 kg
Hydrogen Peroxide (H ₂ O ₂ , 35%)	6.5 Liters	or	7.4 kg

Submit $\frac{3}{4}$ of the DI water and slowly add sulfuric acid while stirring.

Caution: Exothermic reaction causes temperature increase (up to 40+°C possible!). Add **MET 7000 S** stabilizer while stirring and finally add hydrogen peroxide. Mix properly and fill up the volume with the remaining DI water quantity.

Attention: Please follow precaution measures for handling corrosive substances. Ensure proper mixing during and after blending steps.

Caution: Do not store or move readily blended etching solution in tightly sealed containers.

Product Specification MET 7000 S

Nominal values / standard parameters are given in brackets ().

State:	liquid
Odor:	slightly acidic, stingy
Color:	brownish
Specific Gravity:	1.10 – 1.15 g/cm ³ (1.12)
Acidity:	3.20 – 4.20 mol/L (3.50)
pH-Value:	highly acidic
Chem. Characterization:	aqueous acid solution

MET 7000

Process Parameters of MET 7000 process bath

Nominal values / standard parameters are given in brackets ().

Concentration:	2 vol.% MET 7000 S
Temperature Range:	28 - 42 °C (35°C)
Specific Gravity:	1.11 – 1.16 g/cm ³ (1.14)
Hydrogen Peroxide content (H ₂ O ₂):	15 – 40 g/L (25)
H ₂ SO ₄ content:	130 – 210 g/L (175)
Copper content:	< 50 g/L
Dwell Time:	1 - 2 min (95 sec.)
Etch rate:	0.8 - 2.4 µm (1.1 µm)
Application:	Immersion for vertical and horizontal processing

Equipment Material

Tanks:	PP; do not use steel or other metals
Heaters:	Quartz or Teflon/PTFE
Racks / Baskets:	Can be metal structures, but need to be coated with <ol style="list-style-type: none">pore-free black or green HALAR (do not use blue HALAR!)pore-free PP coated stainless steel Baskets can also be completely made from PP No metal is allowed in contact with the solution!
Ventilation:	Advised
Agitation:	Recommended
Filtration:	Advised

Storage Requirements (MET 7000 S)

Do not store together with alkaline or cyanide products and avoid contact with them. Do not store readily blended etching solution in tightly sealed containers (gas formation). Do not use metal containers. Keep original containers tightly closed. Ensure good air circulation.

Shelf Life (MET 7000 S)

12 months from production date, if stored according to storage recommendations.

Waste Removal (MET 7000 S)

MET 7000 S is acidic and needs to be neutralized before discharge into the public sewage system, preferably in a common batch neutralization.

MET 7000 etching solutions need to be decontaminated prior to being discharged into public sewage systems. Used **MET 7000** solutions contain copper which need to be precipitated in the common batch neutralization. The filter cake contains larger amounts of copper and must be treated as special waste. The local waste water regulations have to be adhered to.

Packaging (MET 7000 S)

5, 10 and 25 L PE containers. Other package sizes upon request.

MET 7000

Safety Recommendations

MET 7000 S, MET 7000 etching solution and its components are highly acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

2.5.1 Operating window for process bath

Micro Etch MET 7000

Property for analysis	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method #
Specific Gravity @ 20°C [g/cm ³]	1.11 – 1.16	1.14	1.12	1.15	1.11	1.16	# 1
H ₂ SO ₄ conc. [g/L]	130 – 210	175	150	195	130	210	# 16
H ₂ O ₂ conc. [g/L]	15 – 40	25	20	30	15	40	# 3
Copper conc. [g/L]	Max. 50	--	--	40	--	50	# 5 B / 6 / 7 B
Etch rate [µm]	0.8 – 2.4	1.1	0.9	2.0	0.8	2.4	# 4
Color	Green	Green	--	--	--	blue	--
Temperature [°C]	28 – 42	35	30	40	28	42	--

MET 7000

Control and Analysis Procedures for Micro Etch

The micro etching process is a very important process step, because it creates the active surface for the immersion tin application. Any irregularity on the copper surface left after micro etching will become obvious in the tin deposit. Also surface roughness plays a very important part. The rougher the surface, the less white the deposit. The smoother the surface, the whiter the tin finish.

Control Procedures:

Nominal values / standard parameters are given in brackets ().

Specific Gravity:	1.11 – 1.16 g/cm ³ (1.14)
Hydrogen Peroxide content (H ₂ O ₂):	15 – 40 g/L (25)
H ₂ SO ₄ content (Acidity):	130 – 210 g/L (175)
Copper content:	< 50 g/L
Dwell Time:	1 - 2 min (95)
Etch rate:	0.8 - 2.4 µm (1.1)
Color:	green

Note: Calculation of necessary *MET 7000 S* addition is based on determination of H₂O₂ content of the process bath. Every addition of hydrogen peroxide (H₂O₂) requires the addition of *MET 7000 S*.

Specific gravity (density) is directly correlated with acidity. Specific gravity measurement is useful as a control procedure to check the state of the bath / replenishment result.

Copper can be detected with 3 different analysis methods: with AAS, UV-Vis or Titration. Procedures are given for all of them. Please choose most the appropriate method.

Replenishment is made with *MET 7000 S*, H₂O₂, H₂SO₄

For proper replenishment it is necessary to first compensate evaporation losses by adding DI water. Compensating chemical losses should only be taken care of after this.

Process bath temperature adjustments may be necessary if etch rate is out of specification.

Excessive copper concentration can be reduced by cooling down the bath to ≤ 20°C. If copper is not / can not be removed regularly, the bath should be (partially) exchanged.

We recommend making regular additions to the *MET 7000* process bath based on usage:

- Add DI water until the original level is almost reached. Do not fill up to the original level at this point already, because space for hydrogen peroxide, sulfuric acid and *MET 7000 S* additions is required.
- Add *MET 7000 S* 3 (1)* mL / m² treated panel (0.3 (0.1)* mL / ft² of treated panel)
- Add H₂SO₄ (96%) 12 (4)* mL / m² treated panel (1.1 (0.4)* mL / ft² of treated panel)
- Add H₂O₂ (35%) 8 (3)* mL / m² treated panel (0.8 (0.3)* mL / ft² of treated panel)
- Fill up to the original level with DI water, if necessary.

* values given for horizontal processing

MET 7000

Control and Analysis Procedures for Micro Etch

Perform complete bath analysis after 7 m² (75 ft²) of treated panel per liter bath volume. Then make bath corrections based on analysis following the given procedure:

1. Add DI water until the original level is almost reached
2. Analyze hydrogen peroxide content of the process bath solution and replenish properly
3. Add corresponding amount of *MET 7000 S* and check color of process bath (has to be green)
4. Analyze sulfuric acid and replenish properly
5. Analyze copper content (0 – 50 g/L)
6. Fill up to original liquid level with DI water, if necessary.

2.5.2 Analysis

Hydrogen Peroxide (H₂O₂) Determination (Method # 3)

1. Add ~100 mL of DI water into an Erlenmeyer flask
2. Add 5 mL of diluted H₂SO₄ (10%)
3. Pipette 0.5 mL of the MET 7000 process solution into the Erlenmeyer flask
4. Add 1 – 2 drops of Ferroin indicator
5. Titrate with 0.1 mol/L Cer(IV)sulfate from red-orange to the light blue (nearly colorless) endpoint
6. note: it's important to titrate very slowly (drop by drop) because only 1-2 drops give the endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

$$\text{H}_2\text{O}_2 \text{ content [g/L]} = \text{Volume of 0.1 mol/L Cerium (IV) sulfate used [mL]} \times F \times 3.4$$

Factor F = Factor of Cerium (IV) sulfate (if unknown, use F=1)

Replenishment:

Operating range: 15 – 40 g/L (25 g/L nominal)

$$\text{Addition of H}_2\text{O}_2 \text{ [L]} = \frac{(25 - \text{H}_2\text{O}_2 \text{ content [g/L]}) \times 0.1 \times \text{bath volume [L]}}{\text{Concentr. of H}_2\text{O}_2 \text{ used for replenishment [\%]} \times \text{s.g. of H}_2\text{O}_2 \text{ used for replenishment}^*$$

*e.g. specific gravity (s.g.) of 35% H₂O₂ = 1.13; s.g. of 50% H₂O₂ = 1.19g/mL

$$\text{Addition of MET 7000 S [mL]} = (25 - \text{H}_2\text{O}_2 \text{ content [g/L]}) \times 0.8 \times \text{bath volume [L]}$$

Every addition of H₂O₂ required the addition of *MET 7000 S*. **For every g of missing (added) H₂O₂ (100%) 0.8 mL of MET 7000 S are necessary.** Dosing of H₂O₂ has an impact on the color of the process bath (should be green). Dark blue solutions either contain too much H₂O₂ or too little *MET 7000 S* stabilizer.

MET 7000

Control and Analysis Procedures for Micro Etch

Sulfuric Acid (H₂SO₄) Determination (Method # 16)

1. Fill 50 mL of DI water into a 250 mL Erlenmeyer flask
2. Pipette 2 mL of MET 7000 process solution into the Erlenmeyer flask
3. Add DI water until the sample volume is 100 – 150 mL
4. Add 1 – 2 drops of Methyl Orange indicator solution
5. Titrate with 1mol/L NaOH from rose (red) to the deep yellow endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

$$\text{H}_2\text{SO}_4 \text{ content [g/L]} = \text{Volume of 1.0 mol/L NaOH [mL]} \times F \times 24.5$$

$$\text{Factor F} = \text{Factor of NaOH (if unknown, use F=1)}$$

Replenishment:

Operating Range: 130 – 210 g/L (175 g/L nominal)

$$\text{Addition of H}_2\text{SO}_4 \text{ [liter]} = \frac{(175 - \text{H}_2\text{SO}_4 \text{ content [g/L]}) \times 0.1 \times \text{bath volume [L]}}{\text{Concentr. of H}_2\text{SO}_4 \text{ used for replenishment [\%]} \times \text{s.g. of H}_2\text{SO}_4 \text{ used for replenishment}^*$$

*e.g. specific gravity (s.g.) of 96% H₂SO₄ = 1.85

Etch Rate Determination (Method # 4)

1. Take 3 – 5 pieces of double-sided, undrilled copper clad laminate (size = 200 x 100 mm)
2. Immerse specimen in the MET 7000 bath in your line for 1 minute
3. Determine weight of the specimen on an analytical balance. Record this as W₁
4. Immerse specimen in the MET 7000 bath in your line for 1 minute
5. Rinse and dry specimen and record weight after processing as W₂

Calculation:

$$\begin{aligned} \text{weight loss [g]} &= W_1 \text{ [g]} - W_2 \text{ [g]} \\ \text{Etch Rate } [\mu\text{m}] &= \text{weight loss [g]} \times F \text{ } [\mu\text{m/g}] \\ \text{Factor F} &= 2.8 \mu\text{m} / \text{g} \end{aligned}$$

Action if Etch Rate out of spec:

Operating range: 0.8 – 2.4 μm (1.1 μm nominal)

- a) Etch Rate < 0.8 μm → increase bath temperature or
→ increase H₂O₂ content (together with MET 7000 S)
- b) Etch Rate > 2.4 μm → decrease bath temperature

Attention: Especially a fresh make up may show inconstant etch rates. In this case an addition of CuSO₄ up to a Cu concentration of 5 g/L may lead to a constant etch rate. Also Chlorides e.g. from the rinse before the MET may lower the etch rate.

MET 7000

Control and Analysis Procedures for Micro Etch

Copper Content Determination – Procedure 1 with AAS (GBC 908AA) (Method # 5 B)

Copper content is best determined with Atomic Absorption, but alternate methods are also given:

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

AAS Parameter:	Wavelength:	327.4 nm	Working range:	from 2.5 – 10 µg/mL
	Slit width:	0.2 nm	Sensitivity:	0.05 µg / mL
	Lamp current:	3.0 mA	Flame type:	Air Acetylene (oxidizing)

Procedure:

Preparation of standard solutions for calibration:

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 10 mL of the Cu standard solution (1000 mg/L) into a 100 mL volumetric flask.
2. Fill to 100 mL level with HNO₃ (10%)
3. Take 50 mL of this dilutes copper solution, fill into a 500 mL graduated flask and fill to level with HNO₃ (10%).
→ this is now a parent solution with 10 mg/L Cu, which is used to make further dilutions for standards:
4. Prepare the following calibration standards for the AAS measurement carefully, using the 10 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 2 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 4 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 6 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 8 mg/L (pipette 80 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 10 mg/L (100 mL parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the standard solution prior to use for the respective standard solution handling!

Preparation of the MET 7000 plating bath sample:

A minimum dilution of the MET 7000 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Fill 0.5 mL of MET 7000 plating solution into a 1000 mL volumetric flask
2. Fill up to 1000 mL level with HNO₃ solution (10%)
3. this is now a 1:2000 dilution

MET 7000

Control and Analysis Procedures for Micro Etch

AAS Measurement:

1. Set the AAS to a wavelength of 327.4 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R^2 . If $R^2 < 0.95$, all standard solutions need to be re-made freshly and measured again.
6. If R^2 is ok, measure the prepared MET 7000 plating bath sample
7. Record the Cu [$\mu\text{g/mL}$] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted MET 7000 plating solution exceed 10 $\mu\text{g/mL}$, an additional dilution of the sample is recommended, because this is out of the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Cu content calculation of the plating solution.

Calculation:

$$\text{Cu [g/L]} = (\mu\text{g/mL Cu from AAS}) \times 2$$

Copper Content Determination – Procedure 2 with titration (Method # 6)

* Preparation of ammonia/ammonium chloride buffer:

1. Fill a 1000 mL volumetric flask with approx. 300 mL of DI water
2. Dissolve 54 g of ammonium chloride (NH_4Cl) and 350 mL ammonia (NH_4OH 25%)
3. After complete solution, fill up to 1000 mL mark with DI water

Analysis procedure:

1. Fill a Erlenmeyer flask with approx. 50mL DI water
2. Pipette 5 mL of MET 7000 process solution into the Erlenmeyer flask
3. Add ammonium chloride buffer until pH is 8.5 (± 0.5) -> deep blue solution
4. Fill up to approx. 100 mL with DI water and mix well
5. Add 3 drops of PAN indicator solution
6. Titrate with 0.1 mol/L EDTA-2Na from deep blue to the green endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop ($=0.05$ mL), make two additional titrations.

Calculation:

$$\text{Copper content [g/L]} = \text{Volume of 0.1 mol/L EDTA-2Na solution used [mL]} \times F \times 1.27$$

MET 7000

Control and Analysis Procedures for Micro Etch

Copper Content Determination – Procedure 3 with UV Vis (this procedure is the least accurate, Method # 7B)

1. Pipette 20 mL of MET 7000 solution into a 100 mL volumetric flask
2. Fill up to 100 mL mark with DI water
→ this is now a 1:5 dilution
3. Calibrate UV Vis Spectrometer with DI water as base line
4. Fill cuvette with prepared MET 7000 plating bath sample
5. Check extinction at ~ 500 nm (minimum)
6. Check extinction at 805 nm
7. Read UV Vis display for results given for the two typical extinctions

→ Make 3 measurements and determine average value

Calculation:

$$\text{Cu concentration [g/L]} = (\text{Extinction @ 805 nm} - \text{Extinction @ 500 nm}) \times 26.88$$

Action if copper content is too high:

- a) (Partially) discard the bath
- b) Take out (e.g. by filtration) the copper crystals when the bath is cooled down to room temp (approx. 20°C). After removal of copper crystals, heat the bath back up, to continue with the process. Make a full analysis of ingredients and replenish if necessary prior to using the bath again.

Note: The copper content in the process bath should be below 50 g/L at all times. A concentration exceeding 50 g/L could lead to crystal precipitation on the printed circuit board surface. Copper concentrations above 30 g/L can already be easily detected, because copper crystals would precipitate with cooling down the process bath to $\leq 20^\circ\text{C}$. However these crystals would re-dissolve with warming up the bath again.

Specific Gravity Determination (Method # 1)

1. The solution has to have a temperature of 20°C (if not, warm the solution up / cool it down to 20°C, before filling)
2. Tare a dry 100 mL volumetric flask on an analytical balance
3. Fill to mark with MET 7000 working solution
4. Record the mass of the MET 7000 solution

→ Make 3 measurements and determine average value

Calculation:

$$\text{Specific Gravity (Density) [g/cm}^3\text{]} = \text{mass in g} / 100$$

We do not recommend making corrections to the MET 7000 process bath based on specific gravity. This analysis is intended only as a measure of the state of the bath or to control the replenishment made.

MET 7000

Analysis Protocol

- Evaluation of Hydrogen Peroxide (H₂O₂) content -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water
Sulfuric Acid (10% dilution)
Ferroin indicator
0.1 mol/L Cerium (IV) sulfate for Titration

Equipment: 100 mL Graduated cylinder
0.5 mL Pipette
3 x 250 mL Erlenmeyer flask
25mL Burette

Procedure:

- add ~ 100 mL of DI water into the Erlenmeyer flask
- add 5 mL of diluted H₂SO₄ (10%)
- Pipette 0.5 mL of the MET 7000 process solution into the Erlenmeyer flask
- add 1 – 2 drops of Ferroin indicator
- titrate with 0.1 mol/L Cerium (IV) sulfate from red-orange to the light blue (nearly colorless) endpoint

note: it is important to titrate very slowly (drop by drop) because only 1-2 drops give the endpoint (attention: if over titrated yellow)

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation: H₂O₂ content [g/L] = Av. volume of 0.1 mol/L of Cerium (IV) sulfate used [mL] x F x 3.4

Factor F = Factor of Cerium (IV) sulfate (if unknown, use F=1)

Analysis results: Consumption of Cerium (IV) sulfate: _____ mL

Consumption of Cerium (IV) sulfate: _____ mL

Consumption of Cerium (IV) sulfate: _____ mL

Consumption of Cerium (IV) sulfate: _____ mL

Consumption of Cerium (IV) sulfate: _____ mL

Average Consumption of Cerium (IV)sulfate: _____ mL

H₂O₂ content of MET 7000 solution : _____ g/L

MET 7000

Analysis Protocol

- Evaluation of H_2SO_4 content -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water
1 mol/L NaOH
Methyl Orange indicator solution
(0.04 g in 100 mL DI water)

Equipment: 25 mL Burette
2 mL Pipette
3 x 250 mL Erlenmeyer flask

Procedure:

- fill 50 mL of DI water into a 250 mL Erlenmeyer flask
- Pipette 2 mL of MET 7000 process solution into the Erlenmeyer flask
- add DI water until the sample volume is ~100 mL
- add 1 – 2 drops of Methyl Orange indicator solution
- titrate with 1.0 mol/L NaOH from rose (red) to the deep yellow endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation: H_2SO_4 content [g/L] = Average volume of 1.0 mol/L NaOH used [mL] x F x 24.5

Factor F = Factor of NaOH (if unknown, use F=1)

Analysis results: Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Average Consumption of NaOH: _____ mL

H_2SO_4 of MET 7000 solution : _____ g/L

MET 7000

Analysis Protocol

- Evaluation of Etch Rate -

Date of analysis: _____ Analyzer (initials): _____

Date of sample removal: _____ Signature: _____

Sample no.: _____

Equipment: 3 - 5 x double-sided copper clad laminate (size 200 x 100 mm)
Analytical balance

Procedure:

1. Take 3 – 5 pieces of undrilled double-sided copper clad laminate (size = 200 x 100 mm)
2. Immerse specimen in the MET 7000 bath in your line for 1 minute
3. Determine weight of the specimen on an analytical balance. Record this as W_1
4. Immerse specimen in the MET 7000 bath in your line for 1 minute
5. Rinse and dry specimen and record weight after processing as W_2

Calculation: weight loss [g] = W_1 [g] – W_2 [g]

Etch Rate [μm] = weight loss [g] x F [μm/g]

Factor F = 2.8 μm / g

Analysis results:

 W_1 : _____ g W_2 : _____ g W_1 : _____ g W_2 : _____ g W_1 : _____ g W_2 : _____ g W_1 : _____ g W_2 : _____ g W_1 : _____ g W_2 : _____ g

Average weight loss: _____ g

Etch Rate of MET 7000 solution : _____ μm

MET 7000

Analysis Protocol

- Determination of copper content with AAS -

For GCA 980AA (standards)

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Cu standard solution (1000 mg/L)
HNO₃ solution (10% p.a.)

Equipment: 500 mL volumetric flask
5 x 100 mL volumetric flask
PE bottles: 1x 50 mL, 5x 100 mL,
1x 250 mL
Pipettes: 1x 50 mL, 1x 20 mL,
1x 10 mL

AAS Parameter: Wavelength: 327.4 nm
Slit width: 0.2 nm
Lamp current: 3.0 mA

Working range: from 2.5 – 10 µg/mL
Sensitivity: 0.05 µg / mL
Flame type: Air Acetylene (oxidizing)

Procedure:**Preparation of standard solutions for calibration:**

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 10 mL of the Cu standard solution (1000 mg/L) into a 100 mL volumetric flask.
2. Fill to 100 mL level with HNO₃ (10%)
3. Take 50 mL of this dilutes copper solution, fill into a 500 mL graduated flask and fill to level with HNO₃ (10%).
→ this is now a parent solution with 10 mg/L Cu, which is used to make further dilutions for standards:
4. Prepare the following calibration standards for the AAS measurement carefully, using the 10 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 2 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 4 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 6 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 8 mg/L (pipette 80 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 10 mg/L (100 mL parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the standard solution prior to use for the respective standard solution handling!

MET 7000**Analysis Protocol****- Determination of copper content with AAS –****For GBC 908AA (measurement)**

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Cu calibration standards
(2, 4, 6, 8 and 10 mg/L)
HNO₃ solution (10% p.a.)

Equipment: 1000 mL volumetric flask
1x 0.5 mL Pipette

Preparation of the MET 7000 plating bath sample:

A minimum dilution of the MET 7000 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Fill 0.5 mL of MET 7000 plating solution into a 1000 mL volumetric flask
2. Fill up to 1000 mL level with HNO₃ solution (10%)
→ this is now a 1:2000 dilution

AAS Measurement:

1. Set the AAS to a wavelength of 327.4 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R^2 . If $R^2 < 0.95$, all standard solutions need to be re-made freshly and measured again.
6. If R^2 is ok, measure the prepared MET 7000 plating bath sample
7. Record the Cu [$\mu\text{g/mL}$] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted MET 7000 plating solution exceed 10 $\mu\text{g/mL}$, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Cu content calculation of the plating solution.

MET 7000**Analysis Protocol****- Determination of copper content with AAS –****For GBC 908AA (measurement continued)***Calculation:*

$$\text{Cu [g/L]} = (\mu\text{g/mL Cu from AAS}) \times 2$$

R² _____*Analysis results:*Cu from AAS: _____ $\mu\text{g/mL}$ Cu from AAS: _____ $\mu\text{g/mL}$ **Copper content of MET 7000 solution : _____ g/L**

MET 7000

Analysis Protocol

- Determination of copper content with Titration -

Date of analysis: _____ Analyzer (initials): _____

Date of sample removal: _____ Signature: _____

Sample no.: _____

Reagents: Ammonia/Ammonium chloride buffer*
 PAN indicator solution (1-(2-Pyridylazo)-2-naphtol)
 → 0.1% in Methanol (95 – 100%)
 0.1 mol/L EDTA-2Na (Di sodium salt of EDTA)

Equipment:
 3 x 250 mL Erlenmeyer flask
 5 mL Pipette
 25 mL Burette
 1000 mL Volumetric flask

Procedure: * Preparation of ammonia/ammonium chloride buffer:
 1. Fill a 1000 mL volumetric flask with approx. 300 mL of DI water
 2. Dissolve 54 g of ammonium chloride (NH₄Cl) and 350 mL ammonia (NH₄OH 25%)
 3. After complete solution, fill up to 1000 mL mark with DI water

Analysis procedure:
 1. Pipette 5 mL of MET 7000 process solution into the Erlenmeyer flask
 2. Add ammonium chloride buffer until pH is 8.5 (± 0.5) → deep blue solution
 3. Fill up to approx. 100 mL with DI water and mix well
 4. Add 3 drops of PAN indicator solution
 5. Titrate with 0.1 mol/L EDTA-2Na from deep blue to the green endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation: Copper content [g/L] = Consumption of 0.1 mol/L EDTA-2Na solution [mL] x F x 1.27
 Factor F = Factor of EDTA-2Na solution (if unknown, use F=1)

Analysis results: Consumption of EDTA: _____ mL
 Consumption of EDTA: _____ mL
 Consumption of EDTA: _____ mL
 Consumption of EDTA: _____ mL
 Consumption of EDTA: _____ mL

Average Consumption of EDTA: _____ mL

Copper content of MET 7000 solution : _____ g/L

MET 7000

Analysis Protocol

- Determination of copper content with UV-Vis - (not as accurate as AAS and titration)

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water

Equipment: UV-Vis Spectrometer
UV-Vis cuvette
20 mL Pipette
100 mL Volumetric flask

Procedure:

1. Pipette 20 mL of MET 7000 solution into a 100 mL volumetric flask
2. Fill up to 100 mL mark with DI water
-> this is now a 1:5 dilution
3. Calibrate UV Vis Spectrometer with DI water as base line
4. Fill cuvette with prepared MET 7000 plating bath sample
5. Check extinction at ~ 500 nm (minimum)
6. Check extinction at 805 nm
7. Read UV Vis display for results given for the two typical extinctions

→ Make 3 measurements and determine average value

Calculation: Cu concentration [g/L] = (Extinction @ 805 nm - Extinction @ 500 nm) x 26.88

Analysis results: Extinction: 805nm 500nm

UV-Vis result: _____

UV-Vis result: _____

UV-Vis result: _____

Average UV-Vis result: _____

Copper content of MET 7000 solution : _____ g/L

MET 7000**Analysis Protocol****- Determination of Specific Gravity -**

This measurement is not a frequently required analysis. It is only used as a helpful control tool, to check, of the bath is in specification or to control your replenishment.

Do not make additions to the MET 7000 process bath based on specific gravity!

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: ---**Equipment:** Analytic balance with 0.01 g precision
100 mL Volumetric flask
Thermometer**Procedure:**

1. The solution has to have a temperature of 20°C (if not, warm the solution up / cool it down to 20°C, before filling)
2. Tare a dry 100 mL volumetric flask on a balance
3. Fill to mark with MET 7000 working solution
4. Record the mass of the MET 7000 solution

→ Make 3 measurements and determine average value

Calculation:

Specific Gravity (Density) [g/mL] = mass of MET 7000 solution [g] / 100

Analysis results:

Weight of solution: _____ g

Specific Gravity: _____ g/mL

Weight of solution: _____ g

Specific Gravity: _____ g/mL

Weight of solution: _____ g

Specific Gravity: _____ g/mL

Average Specific Gravity of MET 7000 solution: _____ g/mL

MET 7000

Analysis and Replenishment Record #1 of 4

Bath volume: _____ Liter Date of analysis: ____ . ____ . ____

Throughput: _____ m² / ft² Analyzer (initials): _____

Capacity / Liter: _____ m²/L or ft²/l (throughput : bath volume)

Date of installation: ____ . ____ . ____ Signature: _____

Physical Data:

	OK	Caution	NOK
Form:	watery		oily
Color:	green	blue green green blue	blue others
Appearance:	clear / slightly opaque	opaque / hazy	different
Foam formation:	no	yes	

Analysis procedure:

Etch Rate

Etch rate result = _____ μm

Operating range: 0.8 – 2.4 μm

Nominal value: 1.1 μm

Action if Etch Rate out of spec:

c) *Etch Rate < 0.8 μm → increase bath temperature
or
→ increase H₂O₂ content (together with MET 7000 S)*

d) *Etch Rate > 2.4 μm → decrease bath temperature*

MET 7000

Analysis and Replenishment Record #2 of 4

Date of analysis: ____ . ____ . ____

Signature: _____

H₂O₂ Hydrogen Peroxide

→ H₂O₂ content = _____ g/L

Operating range: 15 – 40 g/L

Nominal value: 25 g/L

Calculation:

$$\text{Addition of H}_2\text{O}_2 \text{ [liter]} = \frac{(25 - \text{H}_2\text{O}_2 \text{ content [g/L]}) \times 0.1 \times \text{bath volume [L]}}{\text{Concentr. of H}_2\text{O}_2 \text{ used for replenishment [\%]} \times \text{s.g. of H}_2\text{O}_2 \text{ used for replenishment}^*$$

*e.g. specific gravity (s.g.) of 35% H₂O₂ = 1.13; s.g. of 50% H₂O₂ = 1.19g/mL**Result: _____ liter of H₂O₂ have to be added**

$$\text{Addition of MET 7000 S [mL]} = (25 - \text{H}_2\text{O}_2 \text{ content [g/L]}) \times 0.8 \times \text{bath volume [L]}$$

Result: _____ mL of MET 7000 S have to be added

Every addition of H₂O₂ required the addition of *MET 7000 S*. **For every g of missing (added) H₂O₂ (100%) 0.8 mL of *MET 7000 S* are necessary.**

Dosing of H₂O₂ has an impact on the color of the process bath (should be green). Dark blue solutions either contain too much H₂O₂ or too little *MET 7000 S* stabilizer.

MET 7000

Analysis and Replenishment Record #3 of 4

Date of analysis: ____ . ____ . ____

Signature: _____

H₂SO₄ Sulfuric Acid

→ H₂SO₄ content = _____ g/L

Operating Range: 130 – 210 g/L

Nominal value: 175 g/L

Calculation:

$$\text{Addition of H}_2\text{SO}_4 \text{ [liter]} = \frac{(\text{175} - \text{H}_2\text{SO}_4 \text{ content [g/L]}) \times 0.1 \times \text{bath volume [L]}}{\text{Concentr. of H}_2\text{SO}_4 \text{ used for replenishment [\%]} \times \text{s.g. of H}_2\text{SO}_4 \text{ used for replenishment}^*$$
*e.g. specific gravity (s.g.) of 96% H₂SO₄ = 1.85**Result: _____ liter of H₂SO₄ have to be added**

Copper content determination with AAS, UV-VIS or Titration

→ Copper content = _____ g/L

Nominal value: < 50 g/L*Action if copper content is too high:*

Take out (e.g. by filtration) the copper crystals when the bath is cooled down to room temp. after removal of copper crystals, heat the bath back up to continue with the process. Make a full analysis of ingredients and replenish if necessary prior to using the bath again.

Note: The copper content in the process bath should be below 50 g/L at all times. A concentration exceeding 50 g/L could lead to crystal precipitation on the printed circuit board surface. Copper concentrations above 30 g/L can already be easily detected, because copper crystals would precipitate with cooling down the process bath to ≤ 20°C. However these crystals would re-dissolve with warming up the bath again.

MET 7000

Analysis and Replenishment Record #4 of 4

Date of analysis: ____ . ____ . ____

Signature: _____

Density / Specific Gravity (Analysis only for control - not frequently necessary)➔ Specific Gravity of MET 7000 = _____ g/cm³Operating range: 1.11 – 1.16 g/cm³Nominal value: 1.14 g/cm³**Overall analysis and replenishment result for MET 7000 process bath :**

Bath is ok, no replenishment necessary

Bath needs to be replenished with _____ mL of H₂O₂
_____ mL of MET 7000S
_____ mL of H₂SO₄

Bath needs to be exchanged

Copper needs to be removed

Temperature needs to be increased to _____ °C decreased to _____ °C

Comments: _____

MET 7000

Replenishment Table

H₂O₂ (Hydrogen Peroxide) / MET 7000 S

Example for 100 liter bath volume

Values for your bath volume:

_____ liter

Analysis result H ₂ O ₂ content [g/L]	Addition of e.g. H ₂ O ₂ (35%) [L]	Addition of <i>MET 7000 S</i> [mL]	Addition of H ₂ O ₂ (____%) [L]	Addition of <i>MET 7000 S</i> [mL]
5	5.1	1600		
6	4.8	1520		
7	4.6	1440		
8	4.3	1360		
9	4.0	1280		
10	3.8	1200		
11	3.5	1120		
12	3.3	1040		
13	3.0	960		
14	2.8	880		
15	2.5	800		
16	2.3	720		
17	2.0	640		
18	1.8	560		
19	1.5	480		
20	1.3	400		
21	1.0	320		
22	0.8	240		
23	0.5	160		
24	0.3	80		

Bath within spec

MET 7000

Replenishment Table

H_2SO_4 (Sulfuric Acid)

Example for 100 liter bath volume

Values for your bath volume:

_____ liter

Analysis result H_2SO_4 content [g/L]	Addition of e.g. H_2SO_4 (96%) [L]	Addition of H_2SO_4 (____%) [L]
50	7.0	
60	6.5	
70	5.9	
80	5.3	
90	4.8	
100	4.2	
110	3.7	
120	3.1	
130	2.5	
140	2.0	
150	1.4	
160	0.8	
170	0.3	

Bath within spec



MET 7000

Monitoring Record

<u>Bath:</u> Micro Etch MET 7000		<u>Bath volume:</u> _____ liter			<u>New make-up:</u> 81.5 liters of DI water 2.0 liters of MET 7000 S		10.0 liters of H ₂ SO ₄ (96%) 6.5 liters of H ₂ O ₂ (35%)	
Date	Analysis Result				Analysist's Initials	Corrections		Additions made (Initials)
	H ₂ O ₂ [g/L]	H ₂ SO ₄ [g/L]	Copper [g/L]	Etch rate [μm]		1. Addition per bath volume 2. New make-up 3. Comments	Initials	
	15 - 40 (25)	130 - 210 (175)	< 50	0.8 - 2.4 (1.1)				

Organic Metal Pre-Dip for ORMECON™ CSN FF

2.6 OMP 7000

Product Description

OMP 7000 is the unique Organic Metal containing Pre-dip of ORMECON™ CSN FF. It is an aqueous dispersion for further pre-treatment of the copper surface following acid cleaner and micro etch.

OMP 7000 provides the following advantages:

- Efficient anti-tarnishing effect as the main prerequisite for a homogeneous white tin surface finish (no stains, no spots)
- Provides a catalyst for the immersion tin deposition process, to ensure very a dense, big grain size tin deposit.
- Long bath life
- Free of complexing agents
- Easy to replenish
- Supplied as concentrate to be mixed with DI water

Application and Make-up

OMP 7000 is made from two concentrated components **OMP 7000 C** (main concentrate) and **OMP 7000 B** (buffer solution). The concentrates have to be mixed with DI water for make-up. 5 vol.% **OMP 7000 C** and 2.5 vol.% **OMP 7000 B** are used.

Make-up recommendation:

DI water	92.5 Liter	or	92.5 kg
OMP 7000 B	2.5 Liter	or	2.50 kg
OMP 7000 C	5.0 Liter	or	5.0 kg

Submit approx. 70 Liters of DI water and add **OMP 7000 B** buffer solution while stirring. Now add **OMP 7000 C** concentrate, mix properly again and add the remaining quantity of water.

Attention: It is essential to add water first and mix it with the buffer solution prior to concentrate addition. Otherwise the Organic Metal will irreversibly flocculate and precipitate.

OMP 7000 C and **OMP 7000 B** are not compatible with each other in their concentrated form and should therefore never be mixed prior to being dissolved in water.

OMP 7000 C contains small particles that tend to settle during storage. Before taking any **OMP 7000 C** out of the original container, please check for sediments and make sure all precipitation is re-dispersed by powerful shaking or stirring. If not, errors will occur in bath make-up and/or replenishment due to varying Organic Metal concentrations in the quantity taken out.

For replenishment: The concentration of the OMP 7000 bath can be maintained with additions of **OMP 7000 C** and **OMP 7000 B**.

OMP 7000

Product Specification OMP 7000 C

Nominal values / standard parameters are given in brackets ()

State:	liquid
Odor:	slightly acidic
Color:	dark green, opaque
Specific Gravity:	0.98 - 1.05 g/cm ³ (1.00)
pH-Value:	1.50 – 2.50 (2.00)
Chem. Characterization:	aqueous Organic Metal dispersion

OMP 7000 C contains small particles. Do not filter the concentrate nor the readily blended process bath. Filtering would remove the Organic Metal in the OMP 7000 and destroy the pre-dip bath. However a circulation pump is recommended to avoid sedimentation.

Product Specification OMP 7000 B

Nominal values / standard parameters are given in brackets ()

State:	liquid
Odor:	slightly acidic, stingy
Color:	colorless to brownish
Specific Gravity:	1.05 – 1.07 g/cm ³ (1.06)
Acidity:	1.70 – 2.00 mol/L (1.80)
pH-Value:	highly acidic
Chem. Characterization:	aqueous acid solution

Process Parameters of the OMP 7000 process bath:

Nominal values / standard parameters are given in brackets ()

Concentration OMP 7000 C:	5 vol. % 20 – 65 g/L (50)
Concentration OMP 7000 B:	2.5 vol. %
pH	1.3 – 2.7 (2.0)
Color	green
Temperature Range:	15 – 30°C
Dwell Time:	approx. 1 min.
Application:	Immersion for vertical and horizontal mode

OMP 7000 C

Equipment Material

Tanks:	PP; do not use steel or other metals
Heaters:	Quartz or Teflon/PTFE
Racks / Baskets:	Can be metal structures, but need to be coated with <ol style="list-style-type: none">pore-free black or green HALAR (do not use blue HALAR!)pore-free PP coated stainless steel Baskets can also be completely made from PP No metal is allowed in contact with the solution!
Ventilation:	Advised
Agitation:	Recommended (avoid foaming)
Circulation:	Continuous circulation is recommended to avoid sedimentation
Filtration:	Do <u>not filter</u> the concentrate nor the readily blended process bath. Filtering would remove the Organic Metal in the OMP 7000 and destroy the pre-dip bath.

Storage Requirements

Do not store together with alkaline or cyanide products and avoid contact with them. Direct contact to alkaline media would immediately cause an irreversible change of the Organic Metal. This becomes obvious by a color change from green to blue and could occur in **OMP 7000 C** or the readily blended OMP 7000 process bath. Do not use metal containers. Keep original containers tightly closed. Ensure good air circulation.

Shelf Life

12 months from production date, if stored according to storage recommendations.

Waste Removal

OMP 7000 C, **OMP 7000 B** and the readily blended OMP 7000 bath can go to a common batch neutralization. The filter cake contains larger amounts of the Organic Metal and should be treated as special waste. The local waste water regulations should be adhered to.

Packaging

1, 5 and 25 L PE containers. Other package sizes upon request.

Safety Recommendations

OMP 7000 C and **OMP 7000 B** are acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

OMP 7000

2.6.1 Operating window for process bath

Pre-Dip **OMP 7000** (Organic Metal)

Property for analysis	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method #
pH	1.3 – 2.7	2.0	1.5	2.5	1.3	2.7	# 10
Organic Metal conc. [g/L]	20 - 65	50	25	60	20	65	9A / 9B
Temperature [°C]	15 – 30	25	17	28	15	30	--

OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

2.6.2 Control Procedures

The Organic Metal Pre-dip is a unique process bath that ensures a homogeneous pre-treatment of the copper surface, to minimize defects in the tin deposit (streaks, stains, etc). It also ensures a big grain size immersion tin deposit topography, which is the reason for ORMECON™ CSN FF's superior properties. Proper maintenance of the Pre-dip process bath is essential for a high reliability of the surface finish.

The working bath make up is 2.5 vol% OMP 7000 B buffer mixed into DI water, then add 5 vol% OMP 7000 C concentrate. **ALWAYS SHAKE THE OMP 7000 C CONCENTRATE BEFORE USE.** The addition of buffer will give a pH working range of 1.3-2.7. Running a pH higher than 2.7 will make the solution unstable and ineffective.

The preferred method of control is a simple color analysis.

If a stagnant rinse is used after the OMP 7000 bath, then the rinse should be changed daily. An overflow rinse after OMP 7000 is preferred.

Concentration OMP 7000 C:	20 – 65 g/L (50 g/L nominal)
pH	1.3 – 2.7 (2.0 nominal)
Color	green
Optical Appearance	slightly hazy/opaque, no flocculates

Replenishment is made with *OMP 7000 C*, *OMP 7000 B*

Note: The main component of OMP 7000 is water. For proper replenishment it is necessary to first compensate evaporation and drag-out losses by adding DI water. Compensating chemical losses should only be taken care of after this.

We recommend making regular additions to the OMP 7000 process bath based on usage:

1. Add DI water until the original level is almost reached. Do not fill up to level at this point, because room for OMP 7000 C and OMP 7000 B addition is required. However, due to low evaporation losses, the addition of DI water may not be necessary every time.
2. Add OMP 7000 C 5.0 (1.0)* mL / m² treated panel (0.50 (0.10)* mL / ft² of treated panel)
3. Add OMP 7000 B 2.5 (0.5)* mL / m² treated panel (0.25 (0.05)* mL / ft² of treated panel)
4. Fill up to the original level with DI water, if necessary.

* values given for horizontal processing

OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

Perform complete bath analysis after 7 m² (75 ft²) of treated panel per liter bath volume. Then make bath corrections based on analysis following the given procedure:

- 1) Add DI water until original level is almost reached. Since the OMP 7000 process bath is operated at low temperatures, evaporation losses should be low.
- 2) Analyze OMP 7000 C concentration and replenish properly with OMP 7000 C (operating range for OMP 7000 C concentration = 20 – 65 g/L)
- 3) Maintain content of buffer OMP 7000 B. For every mL addition of OMP 7000 C, 0.5 mL of OMP 7000 B must be added. Check pH. (Operating range for pH = 1.3 – 2.7)
- 4) Fill up to original liquid level with DI water, if necessary.

2.6.3 Analysis

OMP 7000 strength determination (visual comparison by color standard) (Method # 8)

Due to the color of OMP 7000 and the direct dependence between color intensity and OMP 7000 C concentration, it is possible to determine the concentration of the process bath by visual comparison. This method is not as precise as a UV-Vis measurement and requires a trained eye (with some experience), but is a viable concentration measurement option, because of the broad process window. An exact measurement of the OMP 7000 C concentration is desired, but not necessary to operate the process bath within spec.

Operating range: 20 – 65 g/L (50 g/L nominal)

Analysis preparation:

1. First add DI water and fill up the tank to almost the original level (prior to taking samples for analysis). This is important, because otherwise the working bath sample would be too concentrated and could lead to false results and wrong replenishment recommendation.
2. Mix the process bath thoroughly (e.g. by stirring, circulation pump, etc.) and make sure there is no sediment left in the tank
3. Prepare two fresh standard solutions
with an OMP 7000 C content of 20 g/L = the minimum operating concentration
with an OMP 7000 C content of 65 g/L = the maximum operating concentration
4. Take a process bath sample and compare against standard solutions

A) Preparation of a 20 g/L standard solution

1. submit 50 – 70 mL of DI water into a 100 mL volumetric flask
2. add 1.0 mL of OMP 7000 B (Buffer solution) and mix thoroughly
3. now add 2.0 mL of OMP 7000 C (the green concentrate) and mix thoroughly again.

OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

ALWAYS SHAKE THE OMP 7000 C CONCENTRATE BEFORE USE. If material is taken out with sediments still at the bottom, the standard solution does not contain the proper concentration and the entire analysis and replenishment procedure will become wrong.

4. Carefully fill up the flask exactly to the 100 mL level mark with DI water and mix again.

It is necessary to submit water first and adjust pH with OMP 7000 B buffer solution, because OMP 7000 C and OMP 7000 B are not compatible with each other in their concentrated form (the buffer would irreversibly destroy the concentrate)

B) Preparation of a 65 g/L standard solution

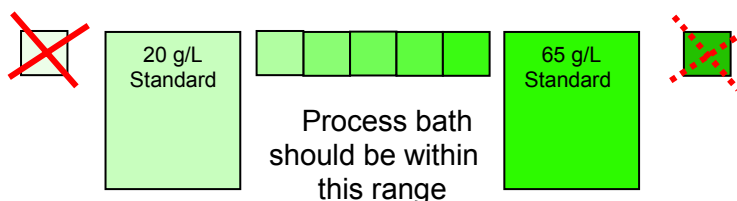
1. submit 50 – 70 mL of DI water into a 100 mL volumetric flask
2. add 3.25 mL of OMP 7000 B (Buffer solution) and mix thoroughly
3. now add 6.5 mL of OMP 7000 C (the green concentrate) and mix thoroughly again.

ALWAYS SHAKE THE OMP 7000 C CONCENTRATE BEFORE USE. If material is taken out with sediments still at the bottom, the standard solution does not contain the proper concentration and the entire analysis and replenishment procedure will become wrong.

4. Carefully fill up the flask exactly to the 100 mL level mark with DI water and mix again.

It is necessary to submit water first and adjust pH with OMP 7000 B buffer solution, because OMP 7000 C and OMP 7000 B are not compatible with each other in their concentrated form (the buffer would irreversibly destroy the concentrate)

- A) Fill a 10 mm cuvette or proper alternative glass receptacle with each of the two standard Solutions.
- B) Fill another 10 mm cuvette with the OMP 7000 process bath.
- C) Visually compare color of the working bath to the standard solutions. The color of the working bath should be within the color range of the standards.
- D) Replenishment is necessary if the process bath is thinner than the minimum concentration (lighter in color = lower in strength).
- E) If the process bath is thicker than the maximum standard (stronger in color), no action is necessary, because the process bath will be diluted during operation anyway.



OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

It is highly recommended to make new standards for every analysis, because the Organic Metal tends to settle over time and the accuracy of the color strength can not be guaranteed for "old" standard solutions.

It is essential for the visual comparison, that all samples (standard solutions and process bath) are examined in similar, and more important, small receptacles. It is recommended to use small cuvettes or small test tubes. Receptacle with big volumes are not suitable, because the color of the examined sample may be too strong to make a color differentiation possible. Similar sized receptacles are necessary to make comparisons possible.

A visual inspection is less accurate than a UV-Vis measurement and it is impossible to determine the exact concentration of the OMP 7000 C in the process bath. However it is possible to check if the bath is working within the operating range (which is quite broad).

As a matter of fact, it is not possible to achieve an exact figure for the necessary replenishment quantity of OMP 7000 C and OMP 7000 B. So the replenishment of the two components is empiric. It is recommended to add OMP 7000 B and OMP 7000 C to a smaller quantity of the process bath to determine a proper replenishment quantity, if the process bath is at the lower end of the operating range or too thin.

Example:

1. Fill 1000 mL of the OMP 7000 working bath into a suitable receptacle.
2. Add a few mL OMP 7000 B, stir well
3. Add the double quantity of OMP 7000 C and stir again.
4. Note the added amounts of OMP 7000 B and OMP 7000 C
5. Fill a small portion into a 10 mm cuvette (or alternative receptacle) and compare against the two standard solutions.
6. Repeat this procedure until the color of the process bath sample achieved desired strength.
7. Transfer the entire addition amount of OMP 7000 B and OMP 700 C to your process bath quantity
8. Make the addition to your process bath with corresponding quantities of OMP 7000 B and OMP 7000 C.

Important Remark:

For replenishing the OMP 7000 process bath add OMP 7000 B (buffer) first while stirring thoroughly. Only then add the corresponding amount of OMP 7000 C and mix well again.

After addition of OMP 7000 B and OMP 7000 C, add DI water to process bath until original level mark is reached (if still necessary).

OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

OMP 7000 strength determination (UV Vis analysis) (Method # 9 A & 9 B)

Due to the color of OMP 7000 and the direct dependence between color intensity and OMP 7000 C concentration, it is possible to determine the concentration of the process bath by visual measurement. UV-Vis spectrometers provide data about light absorption on the Organic Metals in the liquid. Optical density of the probe is directly correlated with the OMP 7000 C concentration. Due to its characteristic color, the Organic Metal provides a typical extinction peak at around 430 nm. The strength of this peak can be directly transferred into a concentration value of the OMP 7000 C with the equation given in this section.

Due to a wide peak plateau of the peak, the measurement range is between 400 and 450 nm.

Operating range: 20 – 65 g/L (50 g/L nominal)

Analysis preparation:

1. First add DI water and fill up the tank to almost the original level (prior to taking samples for analysis). This is important, because otherwise the working bath sample would be too concentrated and could lead to false results and wrong replenishment recommendation.
2. Mix process bath thoroughly (e.g. by stirring, circulation pump, etc.) and make sure there is no sediment left in the tank
3. use a DI water baseline as zero calibration for the measurements (for spectrometers that can only measure one wave length at a time, it is necessary to calibrate with a DI water baseline prior to each measurement)
4. Prepare a fresh standard solution with 50 g/L of OMP 7000 C concentration (=nominal value) , dilute it to 20% (see *Procedure details* for make-up) and measure with UV-Vis spectrometer
5. Take a process bath sample and compare against standard solutions
6. Use only 10 mm cuvettes for measuring process bath samples

There are two different types of spectrometers:

- a) Spectrometers which can only measure one wave length at a time
- b) Spectrometers which are able to measure a full UV-Vis spectrum (200 – 1000 nm) in one go

Remark:

The extinction peaks may vary by a few nm, due to bath conditions.

For UV-Vis spectrometer that can cover a full spectrum, please take the highest peak value, even though it may not be exactly at 430 nm.

For UV-Vis spectrometer that can only detect one wavelength at a time, it is recommended to use a filter for 430 nm. If such filters are not available, chose filters as close to the recommended wavelength as possible (400 - 450 nm).

OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

For one wavelength spectrometer it may occur, that the filters do not cover the exact high of the peak. In this case the measurement will not provide accurate results, leading to an imprecise replenishment of the process bath. But even though the target concentration of the process bath is 50 g/L, the operating window allows 20 – 65 g/L. This provides enough security for replenishments made based on imprecise UV-Vis measurements.

A) Preparation of a 50 g/L standard solution

1. submit 50 – 70 mL of DI water into a 100 mL volumetric flask
2. add 2.5 mL of OMP 7000 B (Buffer solution) and mix thoroughly
3. now add 5.0 mL of OMP 7000 C (the green concentrate) and mix thoroughly again.

ALWAYS SHAKE THE OMP 7000 C CONCENTRATE BEFORE USE. If material is taken out with sediments still at the bottom, the standard solution does not contain the proper concentration and the entire analysis and replenishment procedure will become wrong.

4. Carefully fill up the flask exactly to the 100 mL level mark with DI water and mix again.

It is necessary to submit water first and adjust pH with OMP 7000 B buffer solution, because OMP 7000 C and OMP 7000 B are not compatible with each other in their concentrated form (the buffer would irreversibly destroy the concentrate)

Some UV-Vis units can handle extinction levels of 2.0 given by a 100% standard (no dilution necessary). In this case stop with standard solution preparation here (after A.4) and continue with B). The analysis procedure and calculation method for a 100% standard is given on page 153.

5. Dilute the standard by pipetting 20 mL of the fresh standard solution to a new 100 mL volumetric flask and fill up with DI water to the 100 mL level mark

The standard solution has now been diluted to 20 %. A dilution is necessary, because some UV-Vis instruments are imprecise at higher extinction levels.

It is essential for the accuracy of your measurement to prepare this standard with the utmost care and precision. Remember: The replenishment of your process bath is based on the measurement results.

B) Measure a DI base line, using a 10 mm cuvette

C) Measure the extinction of the diluted 50 g/L standard solution at 430 nm, using a 10 mm cuvette

This is necessary to check your UV-Vis spectrometer and measurement procedure for accuracy.

If the peak maxima are met exactly (most likely with full spectrum spectrometers), the following value should be detected:

at 430 nm 0.4 ± 0.05

These values usually represent a 50 g/L standard (which has been diluted to 20%)

OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

If you do not reach these values, there are two reasons:

- Sediments of OMP 7000 C may still be at the bottom of the original container, that has not been fully re-dispersed prior to taking out the sample. If this is the case, shake the container again to make sure all sediment and precipitation is gone and prepare a new diluted standard following the procedure given in step 1).
- Your UV-Vis measurement did not exactly meet the peak's maxima. If this is the case, note the extinction values you obtained and use them as a reference for your process bath measurement (see step D).

D) Measure the extinction of the actual OMP 7000 process bath

It is essential to use the same dilution factor as for the standard solution, to compare the results, so:

1. Pipette 20 mL of the original OMP 7000 process bath of the tank into a 100 mL volumetric flask. Make sure no sediments are in the tank prior to removing the sample, because this would also lead to wrong results.
2. Fill up to 100 mL level mark with DI water and mix again thoroughly.
3. Measure the extinction of your diluted process bath sample at 430 nm (using DI water as a blank)

The measured result represents the concentration of a diluted process bath (20%). To get the real process bath concentration it is necessary to calculate it using the following equation:

$$\text{Concentration} = \text{Extinction at } \sim 430 \text{ nm} / 0.4 \times 50$$

This equation applies to UV-Vis measurements that met the exact peak's maxima (achieving the recommended extinction results given in step 3).

If other extinction values were achieved in step 3 due to imprecise detection of the peaks, they should be used in the equation instead of 0.4:

$$\text{Concentration} = \frac{\text{Extinction at } \sim 430 \text{ nm}}{\text{Extinction from standard measurement (step C)}} \times 50$$

These equations can not be used, if differences in extinction results were caused by sediment remains in the OMP 7000 C container prior to sample removal.

OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

Calculation:

The Organic Metal concentration value of the process bath can be calculated as follows

$$\text{OMP 7000 C conc.} = \text{Extinction at 400 - 450 nm} / 0.4 \times 50$$

Replenishment:

$$\text{Addition of OMP 7000 C [L]} = \frac{(50 - \text{OMP 7000 C conc.}) \times \text{bath volume [L]}}{1000}$$

$$\text{Addition of OMP 7000 B [L]} = \frac{\text{Addition of OMP 7000 C [L]}}{2}$$

Important Remark:

For replenishing the OMP 7000 process bath add OMP 7000 B (buffer) first while stirring thoroughly. Only then add the corresponding amount of OMP 7000 C and mix well again.

After addition of OMP 7000 B and OMP 7000 C, add DI water to process bath until original level mark is reached (if necessary).

pH Determination (Method # 10)

1. Adjust OMP 7000 process bath sample to 25°C temperature
2. Switch on pH-meter
3. Start calibration mode
4. calibrate the pH-meter according to the manufacturers instructions
5. Rinse electrode with DI water thoroughly
6. Immerse electrode into OMP 7000 process bath sample and measure pH

→ Make 3 pH measurements and use average value for replenishment procedure determination.

Calculation:

Read pH value from display

OMP 7000

Control and Analysis Procedures for Organic Metal Pre-Dip

Normally the pH is automatically adjusted when adding OMP 7000 B and OMP 7000 C for OMP 7000 C concentration adjustment. So it is usually not required (and not recommended) to add an additional amount of OMP 7000 buffer solution for pH adjustment. If the pH is out of specification after proper replenishment of OMP 7000 C and OMP 7000 B, please contact your local Ormecon International representative for technical assistance.

Visual Appearance Determination

OMP 7000 is a dispersion of very fine particles of the Organic Metal. It is not a solution. After a down time of e.g. 3 days approx. 30 % of the particles are precipitated as sediment. This is normal and the particles can be easily re-dispersed by stirring, circulation pumping or shaking. The sediment needs to be re-dispersed prior to using the process bath to ensure a proper concentration of the OMP 7000 C in to the OMP 7000 when processing PCBs.

Never filter the process bath! The Organic Metal particles would be irreversibly removed from the bath, which would cause a break-down and a significant decrease of color strength.

The OMP 7000 process bath has to be dark green at all times. It is necessary to regularly check the color vs. a standard (see page 144). The bath needs to be observed carefully for color changes, haziness and flocculation. The Organic Metal is a chemically active substance, sensitive to chemical influences:

Color changes usually occur with pH changes.

Haziness and flocculation/agglomeration could be caused by contamination with chemicals or foreign particles (e.g. metal ion contamination, shavings/filings, etc.)

All appearance changes indicate an affection of the bath's functionality. Not only do the superior properties of the ORMECON™ CSN FF process become lost, but e.g. flocculation/agglomeration of the Pre-dip should be avoided, because such agglomerates could get stuck in holes, causing severe damage to the PCB.

Some changes of the physical state of the bath are irreversible, so please contact your local Ormecon International representative in case any of the above described changes are observed.

OMP 7000**2.6.4 Analysis Protocol****- Determination of Organic Metal with UV-Vis - (using a 20% solution)**

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water**Equipment:** UV-Vis Spectrometer
UV-Vis cuvette
20 mL Pipette
100 mL Volumetric flask

Procedure:

1. Pipette 20 mL of the OMP 7000 process bath in a 100 mL volumetric flask
2. Fill up to 100 mL level mark with DI water and mix thoroughly
3. Fill a UV-Vis cuvette (10 mm) with DI water and measure as a blank
4. Fill a UV-Vis cuvette (10 mm) with the prepared OMP 7000 solution
5. Measure absorbance (Abs.) at **-430 nm** (any wavelength between 400 and 450 is ok)
6. Read UV Vis display for results

→ Make 3 measurements and determine average value

Calculation: $\text{OMP 7000 C concentration [g/L]} = \text{Extinction (Abs.) @ 400 - 450 nm} / 0.4 \times 50$ **Analysis results:** UV-Vis result: _____

UV-Vis result: _____

UV-Vis result: _____

Average UV-Vis result: _____**OMP 7000 C content of OMP 7000:** _____ g/L

OMP 7000**Analysis Protocol****- Determination of Organic Metal with UV-Vis - (using a 100% solution)**

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents:**Equipment:** UV-Vis Spectrometer
UV-Vis cuvette

Procedure:

1. Remove 10 – 100 mL of the OMP 7000 process bath and fill them into a beaker
2. Fill a UV-Vis cuvette (10 mm) with DI water and measure as a blank
3. Fill a UV-Vis cuvette (10 mm) with the prepared OMP 7000 solution
4. Measure absorbance (Abs.) at **~430 nm** (any wavelength between 400 and 450 is ok)
5. Read UV Vis display for results

→ Make 3 measurements and determine average value

Calculation: OMP 7000 C concentration [g/L] = Extinction (Abs.) @ 400 - 450 nm x 25**Analysis results:** UV-Vis result: _____

UV-Vis result: _____

UV-Vis result: _____

Average UV-Vis result: _____**OMP 7000 C content of OMP 7000:** _____ g/L

OMP 7000**Analysis Protocol****- Determination of pH value -**

Date of analysis: _____ Analyzer (initials): _____

Date of sample removal: _____

Sample no.: _____ Signature: _____

Reagents: Standard buffer solutions
(according to manufacturer)**Equipment:** pH-meter, e.g. "Knick Portameas"
2 (3) x 100 mL beaker**Procedure:**

1. Adjust OMP 7000 process bath sample to 25°C temperature
2. Switch on pH-meter
3. Start calibration mode
4. calibrate the pH-meter according to the manufacturers instructions
5. Rinse electrode with DI water thoroughly
6. Immerse electrode into OMP 7000 process bath sample and measure pH

→ Make 3 pH measurements and use average value for replenishment procedure determination.

Calculation: Read pH value from display**Analysis results:** pH result: _____ pH result: _____

pH result: _____

Average pH result: _____

Normally the pH is automatically adjusted when adding OMP 7000 B and OMP 7000 C for OMP 7000 C concentration adjustment. So it is usually not required (and not recommended) to add an additional amount of OMP 7000 buffer solution for pH adjustment. If the pH is out of specification after proper replenishment of OMP 7000 C and OMP 7000 B, please contact your local Ormecon International representative for technical assistance.

OMP 7000

Analysis and Replenishment Record #1 of 3

Bath volume: _____ Liter Date of analysis: _____ . _____ . _____

Throughput: _____ m² / ft² Analyzer (initials): _____

Capacity / Liter: _____ m²/L or ft²/L (Throughput / bath volume)

Date of installation: _____ . _____ . _____ Signature: _____

Physical Data:

OK

Caution

NOK

Form:	water		
Color:	dark green	blue green yellow green	blue others
Appearance:	clear / slightly opaque	opaque / hazy	different
Flocculation:	no	little	significant
Sediment:	no	little	significant
Foam formation after shaking:	no	yes	
Odor:	acetic	different	

Analysis procedure:

UV-Vis Measurement for 20% dilution (see description pages 152 ff))

1. Pipette 20 mL of the original OMP 7000 process bath of the tank into a 100 mL volumetric flask (no sediments in tank allowed prior to removing the sample)
2. Fill up to 100 mL level mark with DI water and mix again thoroughly.
3. Measure the extinction of your diluted process bath sample at 400 - 450 nm

		Operating range	Nominal value		
Extinction at 430 nm	<input type="text"/>	0.16 – 0.52	0.4 ± 0.05	ok	nok

Calculation:

OMP 7000 C conc. = Extinction (Abs.) @ ~ 430 nm / 0.4 x 50 = _____ g/L

		Operating range	Nominal value		
Concentration [g/L]	<input type="text"/>	20 – 65 g/L	50 g/L	ok	nok

OMP 7000

Analysis and Replenishment Record #2 of 3

Date of analysis: ____ . ____ . ____

Signature: _____

Analysis procedure (continued):

UV-Vis Measurement for 100% process bath

1. Take out 10 - 100 mL of the original OMP 7000 process bath of the tank and fill into a beaker (no sediments in tank allowed prior to removing the sample)
2. Fill a UV-Vis cuvette (10 mL) with this solution
3. Measure the extinction of this process bath sample at 400 - 450 nm

		Operating range	Nominal value		
Extinction at ~430 nm	<input type="text"/>	0.80 – 2.60	2.0 ± 0.05	ok	nok

Calculation:

$$\text{OMP 7000 C conc.} = \text{Extinction (Abs.) @ } \sim 430 \text{ nm} \times 25 = \text{_____ g/L}$$

		Operating range	Nominal value	
Concentration [g/L] nok	<input type="text"/>	20 – 65 g/L	50 g/L	ok

Calculation:

$$\text{Addition of OMP 7000 C [L]} = \frac{(50 - \text{OMP 7000 C conc.} \times \text{bath volume [L]})}{1000}$$

Result: _____ liter of OMP 7000 C have to be added

$$\text{Addition of OMP 7000 B [L]} = \frac{\text{Addition of OMP 7000 C [L]}}{2}$$

Result: _____ liter of OMP 7000 B have to be added

OMP 7000

Analysis and Replenishment Record #3 of 3

Date of analysis: ____ . ____ . ____

Signature: _____

Analysis procedure (continued):

pH value (pH-meter measurement)				
		Operating range	Nominal value	
pH	<input type="text"/>	1.3 – 2.7	2.0	ok nok

Overall analysis and replenishment result for OMP 7000 process bath :

Bath is ok, no replenishment necessary

Bath needs to be replenished with _____ L of OMP 7000 C

_____ L of OMP 7000 B

Bath needs to be exchanged

Comments: _____

OMP 7000

Replenishment Table

OMP 7000 C and OMP 7000 B

Example for 100 liter bath volume

Values for bath volume

_____ liter

Analysis result OMP 7000 C concentr. [g/L]	Addition of OMP 7000 C [L]	Addition of OMP 7000 B [L]	Addition of OMP 7000 C [L]	Addition of OMP 7000 B [L]
45	0.50	0.25		
40	1.00	0.50		
35	1.50	0.75		
30	2.00	1.00		
25	2.50	1.25		
20	3.00	1.50		
15	3.50	1.75		
10	4.00	2.00		
5	4.50	2.25		

Bath within spec



OMP 7000

2.6.5 Monitoring Record

Bath: Pre-Dip OMP 7000		Bath volume: _____ liter		New make-up: 92.5 liters of DI water 2.65 liters of OMP 7000 B		5.0 liters of OMP 7000 C	
Date	Analysis Result			Analysist's Initials	Corrections		Additions made (Initials)
	OMP 7000 C conc. [g/L]	pH value	Color		1. Addition per bath volume 2. New make-up 3. Comments	Initials	
	20 – 65 (50)	1.3 – 2.7 (2.0)	dark green				

Stabilizer for OMP 7000 Pre-Dip in ORMECON™ CSN-FF

2.7 OMP 7075 STAB

Product Description

OMP 7075 STAB is a stabilizer for operating the Organic Metal containing Pre-Dip OMP 7000. The OMP 7000 Pre-Dip is a dispersion and contains fine particles of the green Organic Metal. With air being inserted into the OMP 7000 process bath, the Organic Metal could oxidize, flocculate and precipitate at the bottom of the tank/module and on equipment parts. Foaming is an indication of air insertion and should be avoided under all circumstances. Oxidation of the Organic Metal results in agglomeration and precipitation, which drives the running costs up and could lead to contamination on the board.

The addition of **OMP 7075 STAB** helps to re-disperse flocculated Organic Metal in the OMP 7000 Pre-Dip bath. However foaming should be reduced / avoided prior to using **OMP 7075 STAB**, otherwise regular STAB additions will be necessary.

Application

OMP 7075 STAB is a ready to use additive for OMP 7000, that is used with 0.1 - 0.5 vol or wgt %.

Indicators for the necessity of **OMP 7075 STAB** are:

- constant foaming in the OMP 7000 process bath
- extraordinary high consumption of OMP 7000 C
- visual precipitation of the Organic Metal

OMP 7075 STAB can also be used for cleaning the OMP 7000 Pre-Dip tank/module. For details, please see Cleaning Instructions in this Process Guide.

Note: Use of **OMP 7075 STAB** first leads to heavy foaming after addition to the process bath. This foaming does not have an effect on the quality and the foam will settle shortly

Product Specification

Nominal values / standard parameters are given in brackets ().

State:	liquid
Color:	brownish
Specific Gravity:	0.99 - 1.05 g/cm ³ (1.00)
Acidity:	0.25 - 0.50 mol/L (0.35)
pH value:	pH = ~1
Chem. Characterization:	aqueous solution



OMP 7000 with artificial air agitation for 14 hours:

- heavy foaming
- flocculation of the Organic Metal and significant concentration reduction



OMP 7000 before air agitation:

- perfect dispersion
- Concentration: 110%



OMP 7000 after air agitation:

- obvious flocculation
- Concentration: 41%

OMP 7075 STAB

Storage Requirements

Do not store together with basic or cyanide products and avoid contact with them. Do not use metal containers. Keep original containers tightly closed. Ensure good air circulation.

Shelf Life

12 months from production date, if stored according to storage recommendations.

Waste Removal

OMP 7075 STAB is acidic and has to be diluted and neutralized prior to discharge in the public sewage system. The local waste water regulations have to be adhered to.

Packaging

1 and 5 L PE containers. Other package sizes upon request.

Safety Recommendations

OMP 7075 STAB is acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

Whisker-reducing Organic Metal Pre-Dip for ORMECON™ CSN FF-W

2.8 OMP 7001

Product Description

OMP 7001 is the unique whisker-reducing Pre-Dip of ORMECON™ CSN FF-W. It is an aqueous dispersion which deposits a metallic nano layer on the copper surface before the tin plating. The nano layer greatly reduces tin whisker formation from the plated tin deposit. It also contains Ormecon's Organic Metal which provides additional benefits to the tin finish.

OMP 7001 provides the following advantages:

- Deposition of a metallic nano layer on the copper surface
- Efficient anti-tarnishing effect as the main prerequisite for a homogeneous white tin surface finish (no stains, not spots)
- Provides a catalyst for the immersion tin deposition process.
- Easy to replenish

Application

OMP 7001 is a ready-to-use product. It is used for make-up and replenishment of the operating bath.

Attention: **OMP7001** contains small particles. **Do not filter the product.** However a circulation pump is recommended to avoid sedimentation.

The **OMP 7001** particles tend to settle during storage. Before taking any **OMP 7001** out of the original container, please check for sediments and make sure all precipitation is re-dispersed by powerful shaking or stirring. If not, errors will occur in bath make-up and/or replenishment due to varying Organic Metal concentrations in the quantity taken out.

Product Specification

Standard values are given in brackets ().

State:	liquid
Odor:	slightly acidic
Color:	light green
Specific Gravity:	1.00 – 1.03 g/cm ³ (1.02)
Acidity:	0.30 – 0.45 mol/L (0.40)
pH:	< 2.0
Conc. "whisker-reducing" agent:	190 – 250 mg/L (200)
Conc. Organic Metal:	30 - 60 mg/L (40)
Complexing agent:	25 – 40 g/L (30)
Chem. Characterization:	aqueous acidic Organic Metal plating bath

OMP 7001

Process Parameters

Standard parameters are given in brackets ().

Specific Gravity:	1.00 - 1.05 g/cm ³ (1.02)
Acidity:	0.30 - 0.45 mol / l (0.40)
Concentration "whisker-reducing" agent:	90 – 250 mg / l (150)
Concentration Organic Metal:	20 – 60 mg/L (40)
Complexing agent:	20 – 40 g/L (30)
Temperature Range:	35 – 45°C (40°C)

Treatment Time:	approx. 1 min. (45 sec.)
Application:	Immersion for vertical and horizontal mode

Equipment Material

Tanks:	PP; do not use steel or other metals.
Racks / Baskets:	Can be metal structures, but need to be coated with a) pore-free black or green HALAR (do not use blue HALAR!) b) pore-free PP coated stainless steel Baskets can also be completely made from PP No metal is allowed in contact with the solution!
Heaters:	Quartz or Teflon/PTFE (specific power < 2 W/cm ²).

Storage Requirements

Do not store together with alkaline or cyanide products and avoid contact with them. Direct contact to alkaline media would immediately cause an irreversible change of the Organic Metal. This becomes obvious by a color change from green to blue and could occur in **OMP 7001**. Do not use metal containers. Keep original containers tightly closed. Ensure good air circulation.

Shelf Life

12 months from production date, if stored according to storage recommendations.

Waste Removal

OMP 7001 can go to common batch neutralization. The filter cake contains larger amounts of the Organic Metal and whisker-reducing agent and should be treated as special waste. The local waste water regulations should be adhered to.

Packaging

5, 25 and 200 L PE containers. Other package sizes upon request.

Safety Recommendations

OMP 7001 is acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

OMP 7001

2.8.1 Operating window for process bath

Pre-Dip **OMP 7001** (Organic Metal and Whisker-reducing agent)

Property for analysis	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method
Acidity [mol/L]	0.30 – 0.45	0.4	0.37	0.43	0.30	0.45	# 2 B
Concentration of whisker-reducing component [mg/L]	90 – 250	150	100	200	90	250	# 18
Concentration Organic Metal [mg/L]	20 – 60	40	35	50	20	60	# 9 C
Cu-Concentration [mg/L]	< 120	-	-	100	-	120*	# 5 C
Specific Gravity [g/cm ³]	1.00 – 1.05	1.02	1.01	1.04	1.00	1.05	# 1
Temperature [°C]	30 – 45	40**	37	43	35	45	--
Complexing agent [g/L]	20 – 40	30	22	33	20	40	# 12 B

* or if Organic Metal flocculation occurs

** The nominal value depends on the equipment configuration. During make-up the temperature window will be adjusted to the recommended silver thickness requirement. The set temperature should not vary by $\pm 3^{\circ}\text{C}$ (control limits), resp. $\pm 5^{\circ}\text{C}$ (stop limits) during the subsequent operation of the process. A later correction however is allowed to adjust deposition thickness if out of spec.

Analysis of whisker-reducing deposit (Ag layer after Pre-Dip)

ORMECON™ CSN FF-W whisker-reducing layer

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit
Coulometric Measurement * [nm]	15 – 45**	25	18	40	15	45

*(Spec. Gravity Ag = 10.5 g/cm³)

**Silver layers > 45nm do not necessarily lead to technical disadvantages. If the tin layer is within specifications and there no other negative effects production can be continued.

OMP 7001

2.8.2 Control and Analysis Procedures for whisker-reducing Pre-Dip

Note: The Organic Metal Pre-dip is a unique process bath that ensures a homogeneous pre-treatment of the copper surface, to minimize defects in the tin deposit (streaks, stains, etc) and reduce whisker growth. It also ensures a big grain size immersion tin deposit topography, which is the reason for ORMECON™ CSN FF-W's superior properties. Proper maintenance of the Pre-dip process bath is essential for a high reliability of the surface finish.

As the OMP 7001 bath is used, copper will be dissolved into solution. A copper content above 120 mg/L will cause the Organic Metal to flocculate (agglomerate), precipitate and most probably settle at the bottom of the tank. This will become easily visible by a loss of the Pre-Dip's green color. If the process bath becomes clear, the OMP 7001 is no longer functional and must be replaced.

Standard parameters are given in brackets ()

Specific Gravity	1.00 – 1.05 g/cm ² (1.02)
Acidity	0.30 – 0.45 mol/L (0.40)
Ag-Concentration	90 – 250 mg/L (150)
Organic Metal Concentration	20 – 60 mg/L (40)
Cu-Concentration	< 120 mg/L
Complexing Agent	20 – 40 g/L (30)

Replenishment of process bath is made with: OMP 7001 or OMP 7001 R

The replenishment is determined by the copper content of the working bath which is building up during operation. Addition of fresh solution is necessary to

- replenish dragged out silver and Organic Metal
- to keep the copper content of the working bath below 120 mg/L

There are two ways for replenishment:

- with OMP 7001 recommended for operations with an average exposed copper area of 30 – 40 %
- with OMP 7001 R recommend for operations with an average exposed copper area of > 40%

Replenishment rules for both possibilities are described in the following pages.

OMP 7001

Control and Analysis procedures for whisker-reducing Pre-Dip

Replenishment with OMP 7001

This is a two-phase procedure.

Phase 1: Replenishment of freshly made-up solution until Cu content reaches 120 mg/L

After make-up, the fresh OMP 7001 working bath does not contain any copper, so the replenishment is dedicated to keep specific gravity, silver, complexing agent and Organic Metal content within spec.

Evaporation losses are replaced by addition of DI water. Use acidity or specific gravity analysis results to determine the necessary addition quantity. After adjusting the acidity with DI water, drag out losses are replaced with fresh OMP 7001 by adjusting the level of the solution (approx. 100 mL per m² of treated panel is expected).

Since copper content is critical for the bath, it must be thoroughly checked every 0.2 m² of treated panel per liter bath volume.

For an average exposed copper area of 20% this replenishment procedure is good enough to keep the copper content below the stop limit of 120 mg/L. As soon as the copper content reaches ≥ 120 mg/L the second phase of replenishment applies, because further dilution with fresh solution is required to lower the copper content.

Phase 2: Replenishment by “feed and bleed”

If the copper content reaches ≥ 120 mg/L, fresh OMP 7001 is necessary to bring and keep the copper content below this limit. For this purpose a defined quantity of the working bath has to be removed (“bleed”) and substituted by a similar quantity of fresh OMP 7001 (“feed”). The exact quantity of necessary fresh OMP 7001 is dependant on equipment and circuit parameter and could vary. It should be carefully determined with the help of regular analysis (every 0.2 m² panel / liter bath volume). As a rule of thumb 150 mL per m² of produced panel should be added. An addition of fresh OMP 7001 is recommended every 10 m² panel.

Please note that the replenishment quantity for copper dilution includes the quantity necessary for drag-out replenishment.

Example: After producing 10 m² of panel, the level of the working bath is approx. 1 liter lower than before, due to drag-out losses. For copper dilution an addition of 1.5 liters are necessary. 1 liter is used to fill up drag-out losses, which only leaves 0.5 l to be replaced with “feed and bleed”.

If the copper content still increases despite a “feed and bleed” replenishment with 150 mL/m² panel, the replenishment quantity has to be increased as well. If the copper content continuously drops while using 150 mL/m², the quantity can be decreased.

As soon as a general specific “feed and bleed” amount has been determined, the frequency for copper analysis can be reduced to once a day.

Due to subjective equipment and board design it is not possible to recommend an overall “feed and bleed” replenishment quantity. This has to be determined specifically. The replenishment quantity may change if the exposed copper area changes and should be adapted.

OMP 7001

Control and Analysis procedures for whisker-reducing Pre-Dip

Replenishment with OMP 7001 R

This procedure is based on the throughput of exposed copper:

For each 0.2 m² of exposed copper per liter bath volume, 7.5% of the operating bath have to be removed and substituted by 25% **OMP 7001 R** and 75% DI water.

Example:

Average exposed Cu surface of plated panels:	20 % / m ²
OMP 7001 process bath volume :	100 liter

0.2 m² Cu / 100 liter bath = 20 m² Cu (100%)

→ an average of 20% Cu is equivalent to 100 m² panel surface

According to this example the operating bath has to be replenished with OMP 7001 after every 100 m² of panel surface. The necessary replenishment quantities are given below.

Quantity determination for OMP 7001 R replenishment:

7.5% of the process bath / liter volume have to be exchanged:

→ For a bath with 100 liter volume, this is equivalent to the substitution of 7.5 Liter OMP 7001 process solution with

- 1.9 liters of **OMP 7001 R**
- 5.6 liters of DI water.

Replenishment procedure/sequence: (Data for the given example are shown in brackets)

- remove 7.5% OMP 7001 / liter bath volume (7.5 liters)
- substitute 25% of this volume with **OMP 7001 R** (1.9 liters)
- Fill up to original bath level with DI water (should be equivalent to 75% of the removed process bath volume plus compensation of evaporation losses) (5.6 liters + „X“)

It is important to keep the circulation pump running during the **OMP 7001 R** and DI water addition, to ensure a good and fast mixture.

For calculation of your specific replenishment intervals, please refer to the sheet „Calculation of Replenishment Intervals“.

OMP 7001

Control and Analysis procedures for whisker-reducing Pre-Dip

Two analysis procedures are necessary to determine the replenishment:

- a) Acidity / specific gravity to determine DI water addition
- b) Copper content to determine the amount of necessary OMP 7001 / OMP 7001 R

While the following three analysis procedures should be carried out to monitor and control the accuracy of the replenishment:

- c) Silver content
- d) Organic Metal content
- e) Complexing agent content

With regular additions of fresh OMP 7001 or OMP 7001 R solution according to the replenishment recommendations given previously, all parameters / components should stay within the operating window. An analysis of silver, complexing agent and Organic Metal content is only necessary for monitoring purpose, to make sure the OMP 7001 process bath is within the operating window, not for adjustment of the working solution. Control of the appearance (green and clear color) is a quick visible indicator for proper condition of the OMP 7001 working bath.

The analysis methods are described on the following pages.

OMP 7001

2.8.3 Analysis

Specific Gravity (Density) Determination (Method # 1)

1. The solution has to have a temperature of 20°C (if not, warm the solution up / cool it down to 20°C, before filling)
2. Tare a dry 100 mL volumetric flask on an analytical balance
3. Fill to mark with OMP 7001 working solution
4. Record the mass of the OMP 7001 solution

→ Make 3 measurements and determine average value

Calculation:

$$\text{Specific Gravity (Density) g/cm}^3 = \text{mass of OMP 7001 solution [g]} / 100$$

Operating range of specific gravity @ 20°C : 1.00 – 1.05 g/cm³ (1.02 g/cm² nominal)

Action if specific gravity is too high: add DI water to process solution and check specific gravity during DI water addition.

Action if specific gravity is too low: evaporate water and check specific gravity during evaporation process

OMP 7001

Control and Analysis procedures for whisker-reducing Pre-Dip

Acidity Determination (Method # 2 B)

1. Fill approx. 50 mL of DI water into a 250 mL Erlenmeyer flask
2. Pipette 10 mL of ACL 7001 process solution into the flask
3. Add DI water until the entire probe volume is 100 – 150 mL
4. Add 8 – 10 drops of Cresol Red indicator solution
5. Titrate with 1.0 mol/L sodium hydroxide solution (NaOH) from orange over yellow to the purple endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop ($=0.05$ mL), make two additional titrations.

Calculation:

$$\text{Acidity [mol/L]} = \text{Volume of 1.0 mol/L NaOH used} \times F / 10$$

Factor F = Factor of NaOH (if unknown, use F=1)

Operating range of acidity : 0.35 – 0.45 mol/L (0.40 mol/L nominal)

Copper Content Determination – with AAS (GBC 908AA) (Method # 5 C)

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper). Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

AAS Parameter:	Wavelength: 327.4 nm	Working range: from 2.5 – 10 µg/mL
	Slit width: 0.2 nm	Sensitivity: 0.05 µg / mL
	Lamp current: 3.0 mA	Flame type: Air Acetylene (oxidizing)

Procedure:

Preparation of standard solutions for calibration:

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 10 mL of the Cu standard solution (1000 mg/L) into a 100 mL volumetric flask.
2. Fill to 100 mL level with HNO₃ (10%)
3. Take 50 mL of this dilutes copper standard solution, fill into a 500 mL volumetric flask and fill to level with HNO₃ (10%).
→ this is now a parent solution with 10 mg/L Cu, which is used to make further dilutions for standards:
4. Prepare the following calibration standards for the AAS measurement carefully, using the 10 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 2 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 4 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 6 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))

OMP 7001

Control and Analysis procedures for whisker-reducing Pre-Dip

8 mg/L (pipette 80 mL parent solution into a 100 mL volumetric flask and fill up with HNO₃ solution (10%))

10 mg/L (100% parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the solutions prior to use for the respective standard solution handling!

Preparation of the OMP 7001 bath sample:

A minimum dilution of the OMP 7001 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Pipette 10 mL of OMP 7001 plating solution into a 100 mL volumetric flask
2. Fill up to 100 mL level with HN O₃ solution (10%)
→ this is now a 1:10 dilution

AAS Measurement:

1. Set the AAS to a wavelength of 327.4 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R². If R² < 0.95, all standard solutions need to be re-made freshly and measured again.
6. If R² is ok, measure the prepared OMP 7001 plating bath sample
7. Record the Cu [µg/mL] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted OMP 7001 plating solution exceed 10 µg/mL, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Cu content calculation of the plating solution.

Calculation:

$$\text{Cu [g/L]} = (\mu\text{g/mL Cu from AAS}) \times 10$$

OMP 7001

Control and Analysis procedures for whisker-reducing Pre-Dip

Whisker Reducing Agent (Ag) Determination – with AAS (GBC 908AA) (Method # 18)

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper). Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

AAS Parameter:	Wavelength:	328.1 nm	Working range:	from 1.4 – 5.5 µg/mL
	Slit width:	0.5 nm	Sensitivity:	0.03 µg / mL
	Lamp current:	4.0 mA	Flame type:	Air Acetylene (oxidizing)

Procedure:

Preparation of standard solutions for calibration:

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 25 mL of the Ag standard solution (1000 mg/L) into a 50 mL volumetric flask.
2. Fill to 50 mL level with HNO₃ (10%)
3. Take 25 mL of this dilute silver solution, fill into a 250 mL volumetric flask and fill to level with HNO₃ (10%).
→ this is now a solution with 50 mg/L Ag, which is used to make further dilutions for standards:
4. Take 50 mL of this dilute silver solution, fill into a 500 mL volumetric flask and fill to level with HNO₃ (10%).
→ this is now a parent solution with 5 mg/L Ag, which is used to make further dilutions for standards:
5. Prepare the following calibration standards for the AAS measurement carefully, using the 5 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 1.5 mg/L (pipette 30 mL parent solution into a 100 mL volumetric flask and fill up to level with HNO₃ solution (10%))
 - 2.5 mg/L (pipette 50 mL parent solution into a 100 mL volumetric flask and fill up to level with HNO₃ solution (10%))
 - 3.0 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill up to level with HNO₃ solution (10%))
 - 5 mg/L (100% parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the solutions prior to use for the respective standard solution handling!

Preparation of the OMP 7001 bath sample:

A minimum dilution of the OMP 7001 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Pipette 1 mL of OMP 7001 plating solution into a 50 mL volumetric flask
2. Fill up to 50 mL level with HNO₃ solution (10%)
→ this is now a 1:50 dilution

OMP 7001

Control and Analysis procedures for whisker-reducing Pre-Dip

AAS Measurement:

9. Set the AAS to a wavelength of 328.1 nm
10. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
11. Optimize the flame according to equipment manufacturer's recommendation
12. Start measuring standard solutions with AAS
13. Check R^2 . If $R^2 < 0.95$, all standard solutions need to be re-made freshly and measured again.
14. If R^2 is ok, measure the prepared OMP 7001 plating bath sample
15. Record the Ag [$\mu\text{g/mL}$] readings from the AAS
16. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Ag content.

Should the measured value of the diluted OMP 7001 plating solution exceed 5 $\mu\text{g/mL}$, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Ag content calculation of the plating solution.

Calculation:

$$\text{Ag [g/L]} = (\mu\text{g/mL Ag from AAS}) \times 50$$

Complexing agent determination - with UV Vis (Method # 12 B)

1. Pipette 5 mL of OMP 7001 process bath into a 1000 mL volumetric flask
2. Fill up to 1000 mL level with DI water
3. Pipette 10 mL of this diluted solution into another 1000 mL volumetric flask
4. Fill up to 1000 mL level with DI water
5. Fill a UV-Vis cuvette (10 mm) with this solution
6. Measure absorbance (Abs.) at **232 nm**, using DI water as a blanc
7. Read UV Vis display for results

→ Make 3 measurements and determine average value

Calculation:

$$\text{Complexing agent concentration [g/L]} = \text{Extinction (Abs.) @ 232 nm} \times 136$$

OMP 7001

Control and Analysis procedures for whisker-reducing Pre-Dip

Organic Metal determination - with UV Vis (Method # 9 C)

1. Remove 10 – 100 mL of the OMP 7001 process bath and fill them into a beaker
2. In case the bath is hazy / milky let the white clouds settle until the solution is clear
3. Fill a UV-Vis cuvette (10 mm) with this clear solution (Note: haziness will lead to false results)
4. Measure absorbance (Abs.) at **400 – 450 nm**, using DI water as a blank (any wavelength between 400 and 450 is ok)
5. Read UV Vis display for results

→ Make 3 measurements and determine average value

Calculation:

Organic Metal concentration [mg/L] = Extinction (Abs.) @ 400 - 450 nm x 240

OMP 7001

Analysis Protocol

- Determination of Specific Gravity -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: ---**Equipment:** Analytic balance with 0.01 g precision
100 mL Volumetric flask
Thermometer**Procedure:**

1. The solution has to have a temperature of 20°C (if not, warm the solution up / cool it down to 20°C) before filling
2. Tare a dry 100 mL volumetric flask on an analytical balance
3. Fill to mark with OMP 7001 working solution
4. Record the mass of the OMP 7001 solution

→ Make 3 measurements and determine average value

Calculation:
$$\text{Specific Gravity (Density) [g/mL]} = \text{mass of OMP 7001 solution [g]} / 100$$
Analysis results:

Weight of solution: _____ g

Specific Gravity: _____ g/mL

Weight of solution: _____ g

Specific Gravity: _____ g/mL

Weight of solution: _____ g

Specific Gravity: _____ g/mL

Average Specific Gravity of OMP 7001 solution: _____ g/mL

OMP 7001

Analysis Protocol

- Evaluation of acidity by neutralization -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water
1 mol/L NaOH
Cresol Red
(0.1 g in 100 g of Ethanol 20%)

Equipment: 25 mL burette
10 mL Pipette
3 x 250 mL Erlenmeyer flask

- Procedure:**
1. Fill approx. 50 mL of DI water into a 250 mL Erlenmeyer flask
 2. Pipette 10 mL of OMP 7001 process bath into the flask
 3. Add DI water until the entire sample volume is ca. 100 mL
 4. Add 8 – 10 drops of Cresol Red indicator solution
 5. Titrate with 1 mol/L NaOH from yellow to purple

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation: Acidity [mol/L] = Average volume of 1.0mol/L NaOH used / 10

Factor F = Factor of NaOH (if unknown, use F=1)

Analysis results: Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Average Consumption of NaOH: _____ mL

Acidity of OMP 7001 bath : _____ mol/L

OMP 7001

Analysis Protocol

Determination of copper content with AAS – for GBC 908AA (standards)

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)
Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____ Analyzer (initials): _____

Date of sample removal: _____ Signature: _____

Sample no.: _____

Reagents: Cu standard solution (1000 mg/L)
HNO₃ solution (10% p.a.)

Equipment: 500 mL volumetric flask
5x 100 mL volumetric flask
PE bottles: 1x 50 mL
5x 100 mL
1x 250 mL
Pipettes: 1x 50 mL
1x 20 mL
2x 10 mL

AAS Parameter: Wavelength: 327.4 nm
Slit width: 0.2 nm
Lamp current: 3.0 mA

Working range: from 2.5 – 10 µg/mL
Sensitivity: 0.05 µg / mL
Flame type: Air Acetylene (oxidizing)

Procedure: **Preparation of standard solutions for calibration:**

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 10 mL of the Cu standard solution (1000 mg/L) into a 100 mL volumetric flask.
2. Fill to 100 mL level with HNO₃ (10%)
3. Take 50 mL of this dilutes copper solution, fill into a 500 mL graduated flask and fill to level with HNO₃ (10%).
→ this is now a parent solution with 10 mg/L Cu, which is used to make further dilutions for standards:
4. Prepare the following calibration standards for the AAS measurement carefully, using the 10 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 2 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 4 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 6 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 8 mg/L (pipette 80 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 10 mg/L (100% parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the solutions prior to use for the respective standard solution handling!

OMP 7001

Analysis Protocol

Determination of copper content with AAS for GBC 908AA (measurement)

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Cu calibration standards
(2, 4, 6, 8 and 10 mg/L)
HNO₃ solution (10% p.a.)

Equipment: 100 mL volumetric flask
10 mL pipettes

AAS Parameter: Wavelength: 327.4 nm
Slit width: 0.2 nm
Lamp current: 3.0 mA

Working range: from 2.5 – 10 µg/mL
Sensitivity: 0.05 µg / mL
Flame type: Air Acetylene (oxidizing)

Preparation of the OMP 7001 plating bath sample:

A minimum dilution of the OMP 7001 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Fill 10 mL of OMP 7001 plating solution into a 100 mL volumetric flask
2. Fill up to 100 mL level with HNO₃ solution (10%)
→ this is now a 1:10 dilution

AAS Measurement:

1. Set the AAS to a wavelength of 327.4 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R². If R² < 0.95, all standard solutions need to be re-made freshly and measured again.
6. If R² is ok, measure the prepared OMP 7001 plating bath sample
7. Record the Cu [µg/mL] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted OMP 7001 plating solution exceed 10 µg/mL, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Cu content calculation of the plating solution.

OPM 7001

Analysis Protocol

Determination of copper content with AAS for GBC 908AA (measurement continued)

Calculation:

$$\text{Cu [mg/L]} = (\mu\text{g/mL Cu from AAS}) \times 10$$

R^2 _____

Analysis results:

Cu from AAS: _____ $\mu\text{g/mL}$

Cu from AAS: _____ $\mu\text{g/mL}$

Copper content of OPM 7001 solution : _____ mg/L

OMP 7001

Analysis Protocol

Determination of whisker-reducing agent (Ag) with AAS for GBC 908AA (standards)

This measurement is only necessary as a control measurement.

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Ag standard solution (1000 mg/L)
HNO₃ solution (10% p.a.)

Equipment: 500 mL volumetric flask
1 x 250 mL volumetric flask
3 x 100 mL volumetric flask
1 x 50 mL volumetric flask
PE bottles: 1x 500 mL, 3x 100 mL,
1x 250 mL
Pipettes: 1x 50 mL, 1x 25 mL,
1 x 20 mL , 1x 10 mL,

AAS Parameter: Wavelength: 328.1 nm
Slit width: 0.5 nm
Lamp current: 4.0 mA

Working range: from 1.4 – 5.5 µg/mL
Sensitivity: 0.03 µg / mL
Flame type: Air Acetylene (oxidizing)

OMP 7001

Analysis Protocol

Determination of whisker-reducing agent (Ag) with AAS for GBC 908AA (standards continued)***Procedure: Preparation of standard solutions for calibration:***

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 25 mL of the Ag standard solution (1000 mg/L) into a 50 mL volumetric flask.
2. Fill to 50 mL level with HNO₃ solution (10%)
3. Take 25 mL of this dilutes silver solution, fill into a 250 mL volumetric flask and fill to level with HNO₃ solution (10%).
→ this is now a solution with 50 mg/L Ag, which is used to make further dilutions for standards:
4. Take 50 mL of this dilutes silver standard solution, fill into a 500 mL volumetric flask and fill to level with HNO₃ solution (10%).
→ this is now a parent solution with 5 mg/L Ag, which is used to make further dilutions for standards:
5. Prepare the following calibration standards for the AAS measurement carefully, using the 5 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 1.5 mg/L (pipette 30 mL parent solution into a 100 mL volumetric flask and fill up to level with HNO₃ solution (10%))
 - 2.5 mg/L (pipette 50 mL parent solution into a 100 mL volumetric flask and fill up to level with HNO₃ solution (10%))
 - 3 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill up to level with HNO₃ solution (10%))
 - 5 mg/L (100% parent solution)

Note: rinse all pipettes and bottles with a small quantity of the standard solution prior to use for the respective standard solution handling!

OMP 7001

Analysis Protocol

- Determination of whisker-reducing agent (Ag) with AAS - (measurement)

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Ag calibration standards
(1,5; 2,5; 3 and 5 mg/L)
HNO₃ solution (10% p.a.)

Equipment: 50 mL volumetric flask
Pipettes: 1x 1mL

AAS Parameter: Wavelength: 328.1 nm
Slit width: 0.5 nm
Lamp current: 4.0 mA

Working range: from 1.4 – 5.5 µg/mL
Sensitivity: 0.03 µg / mL
Flame type: Air Acetylene (oxidizing)

Preparation of the OMP 7001 plating bath sample:

A minimum dilution of the OMP 7001 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Fill 1 mL of OMP 7001 plating solution into a 50 mL volumetric flask
2. Fill up to 50 mL level with HNO₃ solution (10%)
→ this is now a 1:50 dilution

AAS Measurement:

1. Set the AAS to a wavelength of 328.1 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R². If R² < 0.95, all standard solutions need to be re-made freshly and measured again.
6. If R² is ok, measure the prepared OMP 7001 plating bath sample
7. Record the Ag [µg/mL] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted OMP 7001 plating solution exceed 5 µg/mL, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Ag content calculation of the plating solution.

OMP 7001

Analysis Protocol

- Determination of whisker-reducing agent (Ag) with AAS - (measurement continued)

Calculation:

$$\text{Ag [mg/L]} = (\mu\text{g/mL Ag from AAS}) \times 50$$

R² _____

Analysis results:

Ag from AAS: _____ $\mu\text{g/mL}$

Ag from AAS: _____ $\mu\text{g/mL}$

Whisker-reducing agent (Ag) content of OMP 7001 solution : _____ **mg/L**

OMP 7001**Analysis Protocol****- Determination of complexing agent with UV-Vis -**

This measurement is only necessary as a control measurement.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water**Equipment:** UV-Vis Spectrometer
UV-Vis cuvette
2 x 1000 mL Volumetric flask
2x 5 mL Pipette
10 mL Pipette

Procedure:

1. Pipette 5 mL of the process bath into a 1000 mL volumetric flask
2. Fill to 1000 mL level mark with DI water
3. Pipette 10 mL of this diluted solution into another 1000 mL volumetric flask
4. Fill up to 1000 mL level with DI water
5. Fill a UV-Vis cuvette (10 mm) with this solution
6. Measure absorbance (Abs.) at **232 nm** (200 – 300 nm), using DI water as a blanc
7. Read UV Vis display for results

→ Make 3 measurements and determine average value

Calculation: Complexing agent concentration [g/L] = Extinction (Abs.) @ 232 nm x 136

Analysis results: UV-Vis result: _____

UV-Vis result: _____

UV-Vis result: _____

Average UV-Vis result: _____

Complexing agent content of OMP 7001: _____ g/L

OMP 7001

Analysis Protocol

- Determination of Organic Metal with UV-Vis -

This measurement is only necessary as a control measurement.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents:

Equipment: UV-Vis Spectrometer
UV-Vis cuvette

- Procedure:**
1. Remove 10 – 100 mL of the OMP 7001 process bath and fill them into a beaker
 2. In case the bath is hazy/milky let the white clouds settle until the solution is clear
 3. Fill a UV-Vis cuvette (10 mm) with this clear solution (Note: haziness will lead to false results)
 4. Measure absorbance (Abs.) at **400 – 450 nm**, using DI water as a blank (any wavelength between 400 and 450 is ok)
 5. Read UV Vis display for results

→ Make 3 measurements and determine average value

Calculation: Organic Metal concentration [mg/L] = Extinction (Abs.) @ 400 - 450 nm x 240

Analysis results: UV-Vis result: _____

UV-Vis result: _____

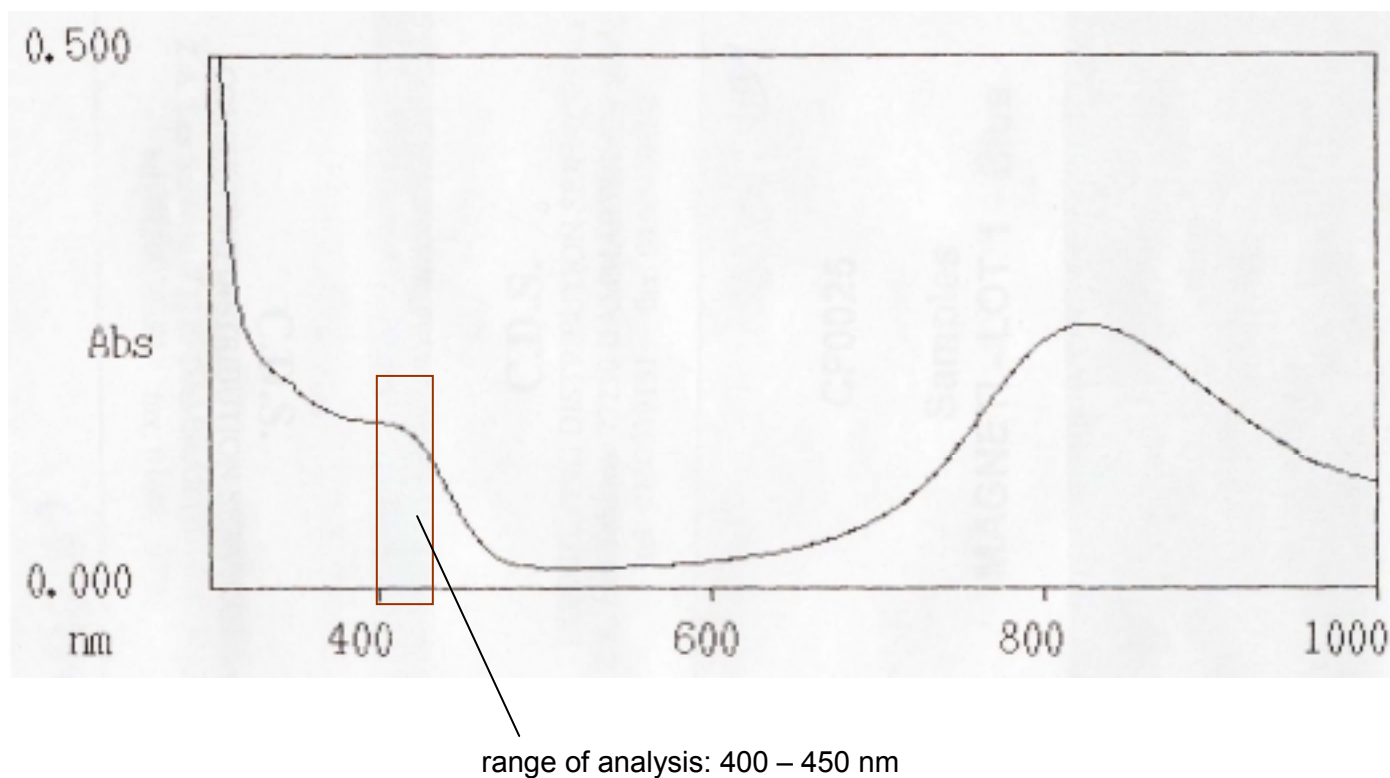
UV-Vis result: _____

Average UV-Vis result: _____

Organic Metal content of OMP 7001: _____ mg/L

OMP 7001

Typical UV-Vis spectrum of an OMP 7001 process bath



OMP 7001

Analysis and Replenishment Record #1 of 3

Bath volume: _____ Liter Date of analysis: ____ . ____ . ____

Throughput: _____ m² / ft² Analyzer (initials): _____

Capacity / Liter: _____ m²/L or ft²/L (Throughput / bath volume)

Date of installation: ____ . ____ . ____ Signature: _____

Physical Data:

	OK	Caution	NOK
Form:	watery		oily
Color:	green	blue green yellow green	blue others
Appearance:	clear / slightly opaque	opaque / hazy	different
Flocculation:	no	little	significant
Sediment:	no	little	significant
Foam formation after shaking:	no	yes	
Odor:	acetic	different	

Analysis procedure:

Acidity	
→ Acidity = _____ mol/L	
Operating Range: 0.30 – 0.45 mol/L Nominal value: 0.40 mol/L	
Action if acidity is too high:	add DI water to process solution and check acidity during DI water addition.
Action if acidity is too low:	evaporate water and check acidity during evaporation process

OMP 7001

Analysis and Replenishment Record #2 of 3

Date of analysis: ____ . ____ . ____

Signature: _____

Copper content determination with AAS

→ Copper content = _____ mg/L

Operating range for copper content: < 120 mg/L

Action if copper content is too high:

If the copper content exceeds the given value substitute a suitable amount of the process bath by fresh OMP 7001 or OMP 7001 R solution (depending on preference)

Rule of thumb for replenishment with OMP 7001:

- add 150 mL / m² to the process bath after every 10 m² of panel.

Rule for replenishment with OMP 7001 R:

- For each 0.2 m² of exposed copper per liter bath volume, remove 7.5% of the operating bath and substituted 25% of this volume with **OMP 7001 R** and 75% with DI water.

Further details are given in the “OMP 7001 Control and Analysis Procedure” section and in the “Calculation of OMP 7001 R Replenishment Intervals” table.

Whisker-reducing agent (Ag) (only necessary to check if process bath is within spec)

→ Whisker-reducing agent (Ag) = _____ mg/L

Operating Range: 90 - 250 mg/L

Nominal value: 150 mg/L

OMP 7001

Analysis and Replenishment Record #3 of 3

Date of analysis: ____ . ____ . ____

Signature: _____

Complexing agent	(only necessary to check if process bath is within spec)
→ Complexing agent concentration in OMP 7001 = _____ g/L	
Operating range :	20 – 40 g/L
Nominal value:	30 g/L

Organic Metal content	(only necessary to check if process bath is within spec)
→ Organic Metal content in OMP 7001 = _____ mg/L	
Operating range :	20 – 60 mg/L
Nominal value:	40 mg/L

Overall analysis and replenishment result for OMP 7001 process bath :

Bath is ok, no replenishment necessary

Water needs to be added / evaporated

Bath needs to be replenished with liters of OMP 7001

Bath needs to be exchanged

Comments: _____

OMP 7001

Calculation of Replenishment Intervals for OMP 7001 R

For each 0.2 m² of exposed copper per liter bath volume, remove 7.5% of the operating bath and substituted 25% of this volume with **OMP 7001 R** and 75% with DI water.

Average exposed copper surface of processed boards: (A1) _____ % / m²

OMP 7001 process bath volume : (A2) _____ liters

0.2 m² x (A2) _____ liters → (A3) _____ m² Cu (100%)

(A3) _____ m² Cu (100%) x (A1) _____ % = (A4) _____ m²

An addition of OMP 7001 R is necessary after (A4) _____ m² of panel.

→ OMP 7000 process bath quantity to be removed

7.5% of process bath volume = (A2) _____ liters x 7.5% = (A5) _____ liters

→ Replenishment quantities

OMP 7001 R: 25% (¹/₄) of the removed process bath quantity

(A5) _____ liters / 4 = (A6) _____ liters

DI water 75% (³/₄) of the removed process bath volume

(A5) _____ liters / 4 x 3 = (A7) _____ liters

Fill out this sheet once, using your specific operating conditions (tank volumes and average exposed Cu area), and you'll get a general rule for your specific OMP 7001 replenishment intervals and quantities!



OMP 7001

Monitoring Record

<u>Bath:</u> Pre-Dip OMP 7001		<u>Bath volume:</u> _____ liter		<u>New make-up:</u> 100 % of OMP 7001			
Date	Analysis Result			Analysist's Initials	Corrections		Additions made (Initials)
	Organic Metal [mg/L]	Silver [mg/L]	Copper [mg/L]		1. Addition per bath volume 2. New make-up 3. Comments	Initials	

OMP 7001 R

2.8.4 Replenishment for whisker-reducing Pre-Dip ORMECON™ CSN FF-W

Product Description

The OMP 7001 Pre-Dip loses some of its ingredients during operation, due to drag-out and plating. In order to maintain good operating conditions and to ensure proper whisker-reduction, it is necessary to regularly monitor and replenish the OMP 7001 process bath.

OMP 7001 R is a concentrated Organic Metal dispersion for replenishment, that adds back missing components to the process bath.

Attention: **OMP 7001 R** contains small particles. Do not filter the product.

Replenishment Procedure

Replenishment of the OMP 7001 process bath is not made based on analysis, but with a general procedure.

Besides drag-out and plating losses, the OMP 7001 process bath suffers from copper accumulation. Since there is no copper removal procedure available, a regular addition of fresh solution is necessary to stabilize the copper content..

OMP 7001 R is used to replenish lost components and to stabilize the copper concentration in the OMP 7001 process bath.

For each 0,2 m² of exposed copper / liter process bath volume, 7,5% of the OMP 7001 plating solution have to be removed and are substituted by 25% **OMP 7001 R** and 75% DI water.

Example:

Average exposed copper area on PCBs: 20% / m²

OMP 7001 process bath volume: 100 Liters

$$0.2 \text{ m}^2 \text{ Cu} / 100 \text{ Liter bath} = 20 \text{ m}^2 \text{ Cu (100\%)}$$

➔ with 20% Cu = 100 m² PCB surface area

In this case the replenishment procedure has to be made after every 100 m² of processed board surface area.

7.5% of the plating bath per liter volume have to be exchanged:

➔ For a 100 Liter bath, 7.5 Liters of OMP 7001 process solution have to be replaced by

- 1.9 Liters of **OMP 7001 R**
- 5.6 Liters of DI water.

Replenishment Sequence

(Numbers of the above example are given in brackets)

- Take out 7.5% OMP 7001 / liter bath volume (7.5 Liters)
- Add 25% of this volume of **OMP 7001 R** to the process bath (1.9 Liters)
- Fill up with DI water to nominal level

Note: Operate circulation pump when making the **OMP 7001 R** and DI water addition, to ensure a fast and good distribution in the process bath.

OMP 7001 R

Product Specification

Standard values are given in brackets ().

State:	liquid
Odor:	slightly acidic
Color:	green
Specific Gravity:	1.03 – 1.15 g/cm ³ (1.08)
Acidity:	1.50 – 1.80 mol / l (1.60)
Organic Metal content:	120 - 240 mg/L (160)
Conc. “whisker-reducing” agent:	700 – 900 mg/L (800)
Chem. Characterization:	aqueous acidic Organic Metal replenisher

Storage Requirements

Do not store together with alkaline or cyanide products and avoid contact with them. Direct contact to alkaline media would immediately cause an irreversible change of the Organic Metal. This becomes obvious by a color change from green to blue and could occur in **OMP 7001 R**. Do not use metal containers. Keep original containers tightly closed. Ensure good air circulation.

Shelf Life

12 months from production date, if stored according to storage recommendations.

Waste Removal

OMP 7001 R can go to common batch neutralization. The filter cake contains larger amounts of the Organic Metal and whisker reducing agent and should be treated as special waste (heavy metal containing). The local waste water regulations have to be adhered to.

Packaging

5, 10, and 25 L PE containers. Other package sizes upon request.

Safety Recommendations

OMP 7001 R is acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

Immersion Tin for ORMECON™ CSN FF / ORMECON™ CSN FF-W

2.9 CSN 7004

Product Description

CSN 7004 is a ready-to use Immersion Tin bath that deposits a highly planar, metallic tin layer selectively on the copper surface.

CSN 7004 offers the following advantages:

- High surface planarity with minimal thickness variation among pads
- Lead-free surface finish
- Suitable for lead-free technology
- Ideal for fine line technology and complex layouts
- Suitable for press-fit technology
- Suitable for flex circuits (even in reel-to-reel application)
- Can be used as Etch Resist
- Easy to re-work
- Suitable for vertical and horizontal processing
- Very good rinsability
- Low density / viscosity, therefore good wettability of plugged holes and blind vias
- Excellent solder mask compatibility
- Long life cycle, especially with CSN Regenerator

Application

CSN 7004 is a ready-to-use solution that is supplied in two components: **CSN 7004-1** and **CSN 7004-2** need to be mixed in a 9 : 1 volume ratio prior to use.

Product Specification CSN 7004-1 and CSN 7004-2

Nominal values / standard parameters are given in brackets ().

	7004-1	7004-2
State:	liquid	liquid
Odor:	slightly acidic	slightly acidic
Color:	yellowish	colorless
Specific Gravity:	1.18 - 1.22 g/cm ³ (1.20)	1.10 - 1.15 g/cm ³ (1.13)
Tin concentration:	20 - 27 g/L (22)	--
Acidity:	4.0 - 4.9 mol/L (4.5)	4.0 - 4.5 mol/L (4.25)
complexing agent:	90 - 140 g/L (115)	--
pH-Value:	acidic	acidic
chem. Characterization:	aqueous tin salt solution	aqueous tin salt solution

CSN 7004

Process Parameters CSN 7004 process bath

Nominal values / standard parameters are given in brackets ().

Make-up:	90% by vol CSN 7004-1 + 10% by vol CSN 7004-2
Temperature range (horizontal):	40°C - 70 °C (68°C)
Temperature range (vertical):	40°C - 65°C (60°C)
Dwell time (horizontal):	7 - 11 min. (8 min.) for SnPb soldering 12 - 18 min. (14 min.) for Pb-free soldering
Dwell time (vertical):	9 - 15 min. (12 min.) for SnPb soldering 16 - 27 min. (21 min.) for Pb-free soldering
Application:	Immersion for vertical and horizontal processing

For vertical module operation:

Specific Gravity [g/cm ³]	
@ 20°C	1.18 – 1.25 g/cm ² (1.20 g/cm ³)
@ 60 – 70 °C	1.16 – 1.23 g/cm ² (1.18 g/cm ³)
Acidity [mol/L]	4.0 – 6.0 mol/L (4.5 mol/L)
Sn-Concentration [g/L]	6 – 24 g/L (9 g/L)
Cu-Concentration [g/L]	< 8.5 g/L
Complexing agent [g/L]	90 – 140 g/L (100 g/L)

For horizontal module operation:

Specific Gravity [g/cm ³]	
@ 20°C	1.18 – 1.25 g/cm ² (1.20 g/cm ³)
@ 60 – 70 °C	1.16 – 1.23 g/cm ² (1.18 g/cm ³)
Acidity [mol/L]	4.0 – 6.0 mol/L (4.5 mol/L)
Sn-Concentration [g/L]	8 – 24 g/L (13 g/L) Single Modul or Modul A
Sn-Concentration [g/L]	6 – 24 g/L (9 g/L) Modul B and Modul C
Cu-Concentration [g/L]	< 8.5 g/L
Complexing agent [g/L]	80 – 140 g/L (100 g/L) Modul A
Complexing agent [g/L]	90 – 140 g/L (100 g/L) Single Modul, Modul B and Modul C

For replenishment: Please see Process Guide for details. For replenishment purposes CSN 7004-1 + CSN 7004-2 are used in addition to CSN 7004 R and CSN 7004 RG.

CSN 7004

Equipment Material

Tanks:	PP; do not use steel or other metals
Heaters:	Quartz or Teflon/PTFE maximum surface temperature 75°C Recommended: Ormecon heating system
Racks / Baskets:	Can be metal structures, but need to be coated with a) pore-free black or green HALAR (do not use blue HALAR!) b) pore-free PP coated stainless steel Baskets can also be completely made from PP No metal is allowed in contact with the solution!
Ventilation:	Advised
Agitation:	Recommended
Circulation:	Continuous circulation is recommended to avoid local overheating
Filtration:	Continuously through 50 and 20 µm PP filters.

Storage Requirements

Do not store together with alkaline or cyanide products and avoid contact with them. Do not use metal containers. CSN 7004 products could precipitate at temperatures < 10°C and totally re-dissolve at higher temperatures (Note: for heating up the solution to dissolve the precipitates do not exceed +60°C, because this could irreversibly damage the solution). Make sure all precipitates are resolved prior to use or are fully transferred to the bath tank, when using full container volumes. Keep original containers tightly closed. CSN 7004 products are sensitive to light. They should be stored in tinted containers in a cool dark place, at temperatures between 10 and 30 °C. Avoid direct exposure to sun light. Ensure good air circulation.

Shelf Life

CSN 7004-1 and CSN 7004-2 have a shelf-life of 12 months from production date, if stored according to storage recommendations.

Waste Removal

CSN 7004 is an aqueous solution containing organics and tin metal salts. In the process of activating copper clad material, some copper may be removed and dissolved in solution. The spent working solution of CSN 7004 may be treated by adjusting the pH of the solution to a pH above 10 with dilute caustic soda. Allow the precipitate to settle. Filter the solution and make a final pH adjustment between 6 and 8 with diluted sulfuric acid. Complex cracking is also required, before sending the spent solution to the sewer. Consult with local officials for further waste disposal regulations.

Packaging CSN 7004-1

25 and 180 L PE containers. Other package sizes upon request.

Packaging CSN 7004-2

5, 10 and 20 L PE containers. Other package sizes upon request.

Safety Recommendations

CSN 7004-1 and **CSN 7004-2** are acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

CSN 7004

2.9.1 Operating window for process bath

Vertical mode

Immersion Tin CSN 7004

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method #
Specific Gravity [g/cm ³] at 20°C at 60 – 70 °C	1.18 – 1.25 1.16 – 1.23	1.20 1.18	1.19 1.17	1.23 1.21	1.18 1.16	1.25 1.23	# 1
Acidity [mol/L]	4.0 – 6.0	4.5	4.2	5.5	4.0	6.0	# 2A
Sn-Concentration [g/L]	6 – 24	9	7	23	6	24	# 11
Cu-Concentration [g/L]	< 8.5	-	-	8.0	-	8.5	# 5 A / 6 / 7 A
Fe-Concentration [ppm]	Max. 100	≤ 5	-	15	-	100	# 15
Complexing agent [g/L]	90 – 140	100	100	130	90	140	# 12 A / 13
Beta value [β]	> 0.70	0.8	0.75	-	0.70	-	# 17
Temperature [°C]	Max. 65	60	-*	64	-*	65	-

* Value might have to be defined to achieve a minimum required tin thickness. At temperatures < 40°C Cu starts to precipitate and could damage the line especially the pumps.

CSN 7004

Horizontal mode

Immersion Tin CSN 7004

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit	Analysis Method #
Specific Gravity [g/cm ³] at 20°C at 60 – 70 °C	1.18 – 1.25 1.16 – 1.23	1.20 1.18	1.19 1.17	1.23 1.21	1.18 1.16	1.25 1.23	# 1
Acidity [mol/L]	4.0 – 6.0	4.5	4.2	5.5	4.0	6.0	# 2A
Sn-Concentration [g/L] Single Modul / Modul A Modul B / Modul C	8 – 24 6 – 24	13 9	9 7	23 23	8 6	24 24	# 11
Cu-Concentration [g/L]	< 8.5	-	-	8.0	-	8.5	# 5 A / 6 / 7 A
Fe-Concentration [ppm]	Max. 100	≤ 5	-	15	-	100	# 15
Complexing agent [g/L] / Modul A Single Modul / Modul B / Modul C	80 - 140 90 – 140	100 100	90 100	130 130	80 90	140 140	# 12 A / 13
Beta value [β]	> 0.70	0.8	0.75	-	0.70	-	# 17
Temperature [°C]	Max. 73	68	-*	70	-*	73	-

* Value might have to be defined to achieve a minimum required tin thickness. At temperatures < 40°C Cu starts to precipitate and could damage the line especially the pumps.

CSN 7004

2.9.2 Operating window for tin deposit

ORMECON™ CSN FF / ORMECON™ CSN FF-W tin deposit properties

Sn-Pb

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit
Layer thickness* [μm] Coulometric Method GCM (Spec Gravity Sn = 7.3g/cm ³)	0.20 – 1.50	The required thickness is dependant on the desired soldering conditions (See Classification Sheet on page 202)				
Solderability Test [%] 155°C / 4h (Solderwave) Interflux IF 2005 M (ERSA), Sn60Pb40	< 1.0	< 1.0	-	-	-	1.0
Wetting angle [°] Fresh 155°C / 4h Kolo 300-25 (Stannol), Sn60Pb40	0 - 95 0 - 105	75 75	- -	- -	- -	95 105
Wetting angle [°] Fresh 155°C / 4h Alpha NR330 (Alphametals), Sn60Pb40	0 - 55 0 - 55	30 30	- -	- -	- -	55 55

Pb-free

	Range	Nominal Value	Lower Control Limit	Upper Control Limit	Lower Stop Limit	Upper Stop Limit
Layer thickness* [μm] Coulometric Method GCM (Spec. Gravity Sn = 7.3g/cm ³)	0.20 – 1.50	The required thickness is dependant on the desired soldering conditions (See Classification Sheet on page 202)				
Wetting angle [°] Fresh 155°C / 4h Alpha NR330 (Alphametals), Sn96.5Ag3.5	0 - 90 0 - 90	30 30	- -	- -	- -	90 90

*Layer thickness is a recommended value, it may be necessary to define a different range with specific customer

CSN 7004

Classification of Tin Thickness and Solderability Specifications

Level	Tin Thickness Range [μm]	Soldering and Storage Conditions	
		Sn-Pb	Pb-Free
1	≥ 1.15	1 Year Storage + 7x Reflow	1 Year Storage + 4x Reflow
2	1.05 - 1.14	1 Year Storage + 6x Reflow	1 Year Storage + 3x Reflow
3	0.95 - 1.04	1 Year Storage + 5x Reflow	1 Year Storage + 2x Reflow
4	0.80 - 0.94	1 Year Storage + 4x Reflow	1 Year Storage + 1x Reflow Tin Thickness Range = 0.4 - 0.94μm
5	0.72 - 0.79	1 Year Storage + 3x Reflow	
6	0.65 - 0.71	1 Year Storage + 2x Reflow	
7	0.40 - 0.64	1 Year Storage + 1x Reflow	
8	0.30 - 0.39	6 Months Storage + 1x Reflow	6 Months Storage + 1x Reflow
9	0.20 - 0.29	1x Reflow	1x Reflow

* Notes

- 1) Pure tin thickness was measured coulometric with a GCM; S.G. 7.3g/cm³
- 2) Storage at 22°C in a non corrosive atmosphere
- 3) These values give no guarantees; they are just recommendations. Deviations due to different storage or reflow conditions are possible.

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

During normal operation, the CSN 7004 immersion tin bath suffers from high evaporation losses in addition to normal drag-out. At the same time, the tin concentration is regularly reduced due to tin plating on the processed boards. The copper concentration constantly increases as boards are processed. Regular analysis and replenishment is essential to ensure a long-term reliability and high quality of the Immersion Tin deposit.

Nominal values / standard parameters are given in brackets ().

Specific Gravity

at 20°C	1.18 – 1.25 g/cm ³ (1.20)
at 60 – 70 °C	1.16 – 1.23 g/cm ³ (1.20)

Acidity 4.0 – 6.0 mol/L (4.5)

Sn-Concentration 6 – 24 g/L (9)
vertical line and horizontal line (Modul B and Modul C)
8 – 24 g/L (13)
horizontal line (Single Modul and Modul A)

Cu-Concentration max. 8.5 g/L

Complexing agent 90 – 140 g/L (100)
vertical line and horizontal line (Single Modul, Modul B and Modul C)
80 – 140 g/L (100)
horizontal line (Modul A)

Replenishment is made with *CSN 7004 (readily mixed)*, *CSN 7004 R (readily mixed)* and *CSN 7004 RG* (if CSN Regenerator is used)

Regular analysis of CSN 7004 is recommended after each 1 m² (11 ft²) of treated panel surface per liter bath volume.

We recommend making regular additions to the CSN 7004 process bath based on usage:

1. Compensate drag-out losses with addition of CSN 7004 (readily mixed from CSN 7004-1 and CSN 7004-2 in a 9 : 1 volume ratio). Add 80 – 120 mL / m² panel (7.4 – 11.2 mL / ft²)
2. Compensate evaporation losses with DI water by adding DI water until the original level is almost reached. Do not fill up to level at this point, because room for CSN 7004 R and CSN 7004 RG addition is required.
3. Add CSN 7004 R 45 (35)* mL / m² treated panel (4.2 (3.3)* mL / ft² of treated panel)
4. Fill up to the original level with readily mixed, fresh CSN 7004 solution, if necessary.

* values given for horizontal processing

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Perform complete bath analysis after **1 m²** (11 ft²) of treated panel per liter bath volume. Then make bath corrections based on analysis following the given procedure:

- 1) Measure specific gravity (density) of tin solution and adjust with addition of DI water

Operating range of specific gravity @ 20°C = 1.18 – 1.25 g/cm³

Operating range of specific gravity @ 60 – 70°C = 1.16 – 1.23 g/cm³

- 2) Measure copper content. If copper content is within spec, continue with 3)

Operating range for copper content: < 8.5 g/L for vertical and horizontal operation

If the copper content exceeds the given values, take out approx. 5 - 15% of the process solution and compensate with a similar quantity of fresh CSN 7004 (readily mixed).

Note: If the CSN Regenerator is available, all quantities removed from the CSN 7004 process baths can be regenerated and used again. If no regeneration is possible, such quantities have to be disposed of.

The use of the CSN Regenerator substitutes the describes “feed-and-bleed” procedure. If copper is removed from the solution regularly, replenishment of complexing agent with CSN 7004 RG becomes necessary, instead of exchanging parts of the process bath with fresh CSN 7004 solution. Details can be found in the Analysis and Replenishment Record.

- 3) Measure tin content and replenish with CSN 7004 R

Operating range for tin concentration: 6 – 24 g/L for vertical and horizontal operation (Modul B and Modul C)

8 – 24 g/L for horizontal operation (Single Modul and Modul A)

- 4) Fill up to original level with readily mixed fresh CSN 7004 solution.

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

2.9.3 Analysis

Specific Gravity (Density) Determination (Method # 1)

5. The solution has to have a temperature of 20°C (if not, warm the solution up / cool it down to 20°C, before filling)
6. Tare a dry 100 mL volumetric flask on an analytical balance
7. Fill to mark with CSN 7004 working solution
8. Record the mass of the CSN 7004 solution

→ Make 3 measurements and determine average value

Calculation:

$$\text{Specific Gravity (Density) g/cm}^3 = \text{mass of CSN 7004 solution [g]} / 100$$

Operating range of specific gravity @ 20°C : 1.18 – 1.25 g/cm³ (1.20 g/cm² nominal)

Operating range of specific gravity @ 60 – 70°C : 1.16 – 1.23 g/cm³ (1.18 g/cm³ nominal)

Action if specific gravity is too high: add DI water to process solution and check specific gravity during DI water addition.

Action if specific gravity is too low: evaporate water and check specific gravity during evaporation process

Please note that specific gravity analysis in the lab should be made with tempered process solution at 20°C. The operating range for 60 – 70°C is only given as a guideline for online density control in the line (e.g. as a control of the auto top-up)!

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Copper Content Determination – Procedure 1 with AAS (GBC 908AA) (Method # 5 A)

Copper Content is best determined with Atomic Absorption, but alternate methods are also given:

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

AAS Parameter:	Wavelength: 327.4 nm	Working range: from 2.5 – 10 µg/mL
	Slit width: 0.2 nm	Sensitivity: 0.05 µg / mL
	Lamp current: 3.0 mA	Flame type: Air Acetylene (oxidizing)

Procedure:

Preparation of standard solutions for calibration:

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 10 mL of the Cu standard solution (1000 mg/L) into a 100 mL volumetric flask.
2. Fill to 100 mL level with HNO₃ (10%)
3. Take 50 mL of this dilutes copper standard solution, fill into a 500 mL volumetric flask and fill to level with HNO₃ (10%).
 - this is now a parent solution with 10 mg/L Cu, which is used to make further dilutions for standards:
4. Prepare the following calibration standards for the AAS measurement carefully, using the 10 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 2 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 4 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 6 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 8 mg/L (pipette 80 mL parent solution into a 100 mL volumetric flask and fill up to mark with HNO₃ solution (10%))
 - 10 mg/L (100% parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the solutions prior to use for the respective standard solution handling!

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Preparation of the CSN 7004 plating bath sample:

A minimum dilution of the CSN 7004 plating bath is necessary. Further dilution may be required because of AAS conditions.

3. Fill 5 mL hypophosphorous acid (50% p.a.) into a 500 mL volumetric flask
4. Pipette 1 mL of CSN 7004 plating solution into the 500 mL volumetric flask
5. Fill up to 500 mL level with HN O₃ solution (10%)
→ this is now a 1:500 dilution

In case the copper level in the plating bath sample is very high, it may be appropriate to use a higher dilution (1:1000). In this case the sample should be prepared as follows:

6. Fill 5 mL hypophosphorous acid (50% p.a.) into a 1000 mL volumetric flask
7. Pipette 1 mL of CSN 7004 plating solution into the 1000 mL volumetric flask
8. Fill up to 1000 mL level with HNO₃ solution (10%)

It may be useful to prepare both dilutions of a CSN 7004 process bath to get the best and most reliable result.

AAS Measurement:

17. Set the AAS to a wavelength of 327.4 nm
18. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
19. Optimize the flame according to equipment manufacturer's recommendation
20. Start measuring standard solutions with AAS
21. Check R². If R² < 0.95, all standard solutions need to be re-made freshly and measured again.
22. If R² is ok, measure the prepared CSN 7004 plating bath sample
23. Record the Cu [µg/mL] readings from the AAS
24. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted CSN 7004 plating solution exceed 10 µg/mL, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Cu content calculation of the plating solution.

Calculation:

$$\text{Cu [g/L]} = (\mu\text{g/mL Cu from AAS}) \times 0.5 \quad (\text{for the 1:500 dilution of the CSN 7004 sample})$$

$$\text{Cu [g/L]} = (\mu\text{g/mL Cu from AAS}) \times 1 \quad (\text{for the 1:1000 dilution of the CSN 7004 sample})$$

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Copper Content Determination – Procedure 2 with Titration (Method # 6)

Preparation of ammonium/ammonium chloride buffer solution

1. Fill a 1000 mL volumetric flask with approx. 300 mL of DI water
2. Dissolve 54g of ammonium chloride (NH₄Cl) and 350 mL ammonium (NH₄OH 25%)
3. After complete solution add DI water to 1000 mL level mark

Analysis procedure:

1. Mix 5 mL of CSN 7004 process bath sample with ~50 mL of DI water in a 250 mL Erlenmeyer flask
2. Add 5.0 mL ammonium/ammonium chloride buffer and mix well
3. Check pH and add more buffer if necessary to adjust pH = 8 - 9
4. Then add hydrogen peroxide (35%) (max. 25 mL) until the solution starts to foam and wait 2 - 3 minutes
5. Add 3 - 5 drops of PAN indicator and mix thoroughly
6. Titrate with 0.1 mol/L EDTA-2Na (Di sodium salt of EDTA) from purple to a mint green endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

Cu content [g/L] = Average volume of 0.1 mol/L EDTA-2Na solution used [mL] x 1,27 x F

Factor F = Factor of EDTA (if unknown, use F = 1)

Copper Content Determination – Procedure 3 with UV Vis (this procedure is the least accurate)

(Method # 7 A)

1. Calibrate UV Vis Spectrometer with ammonia 25% as base line
2. Fill 1.0 mL CSN 7004 process solution into a beaker
3. Add 10 mL ammonia 25% solution
4. Add 0.2 mL hydrogen peroxide solution (35%) and mix with a dry glass stick
5. Filter this solution through a dry blackband filter (12 - 25 μ m fineness)
6. Fill this prepared CSN 7004 solution in an UV-Vis cuvette
7. Wait 1 minute before measuring the probe
8. Check extinction at ~ 635 nm
9. Check extinction at ~ 450 nm
10. read UV Vis display for results given for the two typical extinctions

→ Make 3 measurements and determine average value

Calculation:

$$\text{Cu concentration [g/L]} = \frac{(\text{Extinction @ 635 nm} - \text{Extinction @ 450 nm} - 0.025)}{0.0744}$$

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Action if copper content is too high:

If the copper content exceeds the given values, take out approx. 5 - 15% of the process solution and compensate with a similar quantity of fresh CSN 7004 (readily mixed).

Note: If the CSN Regenerator is available, all quantities removed from the CSN 7004 process baths can be regenerated and used again. If no regeneration is possible, such quantities have to be disposed.

CSN Regenerator:

The copper content in the process bath can be reduced by “freezing” copper out of the solution. This would substitute the above described “feed-and-bleed” procedure and will limit chemical consumption in addition to prolonging the bath life. The CSN Regenerator process will force the precipitation of a copper complex from the tin solution. Besides copper also a part of the tin solution’s complexing agent would be precipitated. This complexing agent is essential for a high tin deposit quality and has to be replenished. It is therefore necessary to analyze the complexing agent concentration in the tin solution after each copper removal and replenish thoroughly with CSN 7004 RG. For procedure see page 212:

Stannous Tin Content (Sn²⁺) Determination (Method # 11)

Preparation of sodium acetate buffer solution

1. Fill a 1000 mL volumetric flask with approx. 500 mL of DI water
2. Dissolve 550g of sodium acetate tri-hydrate
3. After complete solution add DI water to 1000 mL level mark

Analysis procedure:

1. Add 50 mL of DI water to a 250 mL Erlenmeyer flask
2. Add 5.0 mL hypophosphorous acid (50%) with measuring pipette and mix
3. Pipette 5.0 mL of the CSN 7004 working solution into the flask
4. Slowly add acetate buffer solution until pH reaches 4.0 (+/- 0.5) (with approx. 10 – 30 mL)
5. Add DI water until sample volume is 100 – 150 mL
6. Add 1 - 2 micro-spatula tips of grinded Xenol Orange indicator
7. Titrate with 0.1 mol/L EDTA-2Na (Di sodium salt of EDTA) from purple to a light yellow endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

Tin (Sn²⁺) content [g/L] = Average volume of 0.1 mol/L EDTA-2Na solution used [mL] x 2.374

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Replenishment of Stannous Tin (Sn^{2+})

Vertical and horizontal line (Modul B and C):

Maintain the level of stannous tin between 6 and 24 g/L, nominal 9 g/L. Use CSN 7004-R Replenisher to increase and maintain the tin concentration. Mix 90% CSN 7004-R1 with 10% of CSN 7004-R2 by volume prior to addition to the bath.

The calculation for replenishment of tin is shown below and is based on adding Replenisher to achieve a tin concentration of 9 g/L (nominal value)

Horizontal line (Single Modul and Modul A):

Maintain the level of stannous tin between 8 and 24 g/L, nominal 13 g/L. Use CSN 7004-R Replenisher to increase and maintain the tin concentration. Mix 90% CSN 7004-R1 with 10% of CSN 7004-R2 by volume prior to addition to the bath.

The calculation for replenishment of tin is shown below and is based on adding Replenisher to achieve a tin concentration of 13 g/L (nominal value)

It may be possible that the calculated necessary quantity of CSN 7004-R to be added would exceed the total bath volume. In this case it is necessary to remove a certain quantity from the process bath before adding the calculated quantity of CSN 7004-R.

Example procedure (bath volume = 100 Liters)

1. Check and adjust specific gravity or acidity of the process bath by adding or removing DI water
2. Analyse copper content. If the Cu concentration exceeds the specified limit, remove 5 – 15% of the process bath volume.
3. Analyse stannous tin concentration and calculate necessary CSN 7004-R replenishment quantity. Should the necessary quantity exceed the available tank space, remove the quantity difference from the process bath first before adding CSN 7004-R.

Example:

If the bath volume after step 2 is only 75 Liters and the necessary quantity of CSN 7004-R is 30 Liters, 5 Liters of the CSN 7004 process bath need to be removed prior to adding 30 Liters of CSN 7004-R

4. After addition of CSN 7004-R, fill up any missing volume with fresh CSN 7004 solution until the bath is back at 100 Liter volume.

Operating range for tin concentration:	6 – 24 g/L	for vertical and horizontal operation (Modul B and C)
	8 – 24 g/L	for horizontal operation (Single Modul and Modul A)
Nominal value for tin concentration:	9 g/L	for vertical and horizontal operation (Modul B and C)
	13 g/L	for horizontal operation (Single Modul and Modul A)

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Calculation of necessary replenishment quantity:

$$\text{Addition of CSN 7004 R [L]} = \frac{[9 \text{ g/L} - \text{Sn concentr.}] \times \text{Bath volume [L]}}{36}$$

(for 9 g/L nominal)

$$\text{Addition of CSN 7004 R [L]} = \frac{[13 \text{ g/L} - \text{Sn concentr.}] \times \text{Bath volume [L]}}{32}$$

(for 13 g/L nominal)

Acidity Determination (Method # 2 A)

1. Fill approx. 50 mL of DI water into a 250 mL Erlenmeyer flask
2. Pipette 1 mL of CSN 7004 process bath into the flask
3. Add DI water until the entire sample volume is 100 – 150 mL
4. Add 8 – 10 drops of Cresol Red indicator solution (0.1% Cresol Red in 100 g Ethanol(20%))
5. Titrate with 1.0 mol/L NaOH from yellow to the purple endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (≈ 0.05 mL), make two additional titrations.

Calculation:

$$\text{Acidity [mol/L]} = \text{Average volume of 1.0 mol/L NaOH used [mL]}$$

If the CSN 7004 bath is in good working condition, the acidity should be between 4.0 and 6.0 mol/L (4.5 mol/L nominal). We do not recommend making corrections to the bath based on acidity analysis.

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Complexing Agent Determination

This measurement is only necessary, if copper is removed from the CSN 7004 solution by crystallization (CSN Regenerator) or as a control measurement, if tin deposit quality is out of specification, even though acidity, density, tin and copper content are within specified range.

A low content of complexing agent slows the tin deposition speed down.

Complexing agent determination - Procedure 1 with UV Vis (Method # 12 A)

8. Add approx. 500 mL of DI water into a 1000 mL volumetric flask
9. Add 5 mL hypophosphoric acid (50%) with 5mL measuring pipette
10. Pipette 5 mL of CSN 7004 process bath
11. Fill up to 1000 mL level with DI water
12. Pipette 10 mL of this diluted solution into another 1000 mL volumetric flask
13. Fill up to 1000 mL level with DI water
14. Fill a UV-Vis cuvette (10 mm) with this solution
15. Measure absorbance (Abs.) at **232 nm**, using DI water as a blanc
16. Read UV Vis display for results

→ Make 3 measurements and determine average value

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Calculation:

Complexing agent concentration [g/L] = Extinction (Abs.) @ 232 nm x 136

Operating range of extinction measurement @ 232 nm : 0.59 – 1.03

Nominal value for extinction @ 232 nm: 0.74

Operating range of complexing agent in the bath: 90 – 140 g/L (100 g/L nominal)

Calculation of necessary replenishment quantity:

Addition of CSN 7004 RG [liter] = (100 – complexing agent concentr.) x 0.01 x bath volume [L]

Complexing agent determination - Procedure 2 with Titration (Method # 13)

(not as accurate as UV-Vis)

Besides the complexing agent, this titration also detects stannous tin (Sn^{2+}) and copper. It is therefore necessary to determine stannous tin and copper prior to this Titration in order to subtract them from the titration's result.

For subtraction of Sn^{2+} and Cu it is necessary to transfer the concentration values given in [g/L] into mol/L. This is achieved by the following equations:

$$\text{Sn}^{2+} [\text{mol/L}] = \text{Sn}^{2+} [\text{g/L}] / 118.7 [\text{g/mol}]$$

$$\frac{1}{2} \text{ Cu } [\text{mol/L}] = \text{ Cu } [\text{g/L}] / 63.5 [\text{g/mol}] / 2$$

Titration procedure:

1. Add approx. 100 mL of DI water to a 250 mL Erlenmeyer Flask
2. Add 5 mL concentrated sulfuric acid (H_2SO_4) (96%) and mix
3. Pipette 0.1 mL of the CSN 7004 working solution into the flask and mix
4. Add 1 - 2 drops of Ferroin Indicator and mix thoroughly
5. Titrate slowly with 0.02 mol/L KMnO_4 (=0.1 N KMnO_4) to the colorless endpoint (has to stay colorless for nearly 15 sec.)

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

Complexing agent [g/L] =

$$(\text{Quantity of used } \text{KMnO}_4 [\text{mL}] \times F) \times 0.395 - \text{Sn}^{2+} [\text{mol/L}] - \frac{1}{2} \text{ Cu } [\text{mol/L}] \times 76.13$$

Factor F = Factor (Titer) of KMnO_4 (if unknown, use F=1)

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

It is essential for the accuracy of the analysis, that the KMnO_4 solution is fresh (max. one week old). Otherwise the result varies significantly due to a chemical change of the KMnO_4 solution.

Operating range of complexing agent: 80 – 140 g/L (100 g/L nominal)

Calculation of necessary replenishment quantity:

Addition of CSN 7004 RG [liter] = (100 – complexing agent concentr.) x 0.01 x bath volume [L]

Example calculation:

KMnO_4 consumption:	4.0 mL	Factor (Titer) of KMnO_4 = 0.98
Sn^{2+} concentration:	18.0 g/L	
Cu concentration:	8.0 g/L	
Bath volume:	100 Liters	

$\text{Sn}^{2+} [\text{mol/L}] = 18.0 \text{ g/L} / 118.7 [\text{g/mol}] = 0.152 \text{ mol/L}$

$\frac{1}{2} \text{ Cu } [\text{mol/L}] = 8.0 \text{ g/L} / 63.5 [\text{g/mol}] / 2 = 0.063 \text{ mol/L}$

Complexing agent [g/L] = [(4.0 mL x 0.98 x 0.395) - 0.152 mol/L - 0.063 mol/L] x 76.13 = 101.5 g/L

Sn (IV) determination (GBC 908AA) (Method # 14)

Tin oxidizes during the operation of CSN 7004 and Sn(II) is transformed into Sn(IV) . These tin oxides can cause quality problems, especially because they make the bath hazy and make the plating rate of the bath drop significantly. If this is the case, the Sn(IV) content can be measured with a subtractive method. First the total Sn content of the bath sample is measured. Then the stannous tin (Sn_{2+}) content is detected by titration (method # 11). Subtracting the Sn(II) from the total Sn concentration gives the Sn(IV) content in the plating bath sample.

This measurement is not a regular analysis method. It is only necessary, if e.g. the CSN 7004 bath is cloudy/hazy or the plating rate drops significantly.

It's very difficult to determine Sn(IV) with Atomic Absorption Spectroscopy because during the measurement carbon, which forms as a by product, can block the burner and lead to false results. This is the reason why this method isn't completely checked yet.

AAS Parameter:

Wavelength:	235.5 nm
Slit width:	0.5 nm
Lamp current:	5.0 mA

Working range:	from 35 – 135 $\mu\text{g/mL}$
Sensitivity:	0.72 $\mu\text{g} / \text{mL}$
Flame type:	Nitrous oxide-acetylene (reducing slightly luminous)

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for tin)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Procedure:

Preparation of standard solutions for calibration:

All dilutions are made in HCl solution (w = 10%) with p.a. quality

1. Pipette 50 mL of the Sn standard solution (1000 mg/L) into a 250 mL volumetric flask.
2. Fill to 200 mL level with HCl (10%).
→ this is now a parent solution with 200 mg/L Sn, which is used to make further dilutions for standards:
3. Prepare the following calibration standards for the AAS measurement carefully, using the 200 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HCl solution)
 - 40 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))
 - 60 mg/L (pipette 30 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))
 - 80 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))
 - 100 mg/L (pipette 50 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))
 - 120 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))

Note: rinse all pipettes and bottles with a small quantity of each of the standard solution prior to use for the respective standard solution handling!

Preparation of the CSN 7004 plating bath sample:

A minimum dilution of the CSN 7004 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Add 5 mL hypophosphorous acid (50% p.a.) into a 100 mL volumetric flask
2. Pipette 1 mL of CSN 7004 plating solution into the 100 mL volumetric flask
3. Fill up to 100 mL level with HCl solution (10%)
→ this is now a 1:100 dilution
In case the tin level in the plating bath sample is very high, it may be appropriate to use a higher dilution (1:500). In this case the sample should be prepared as follows:
4. Fill 5 mL hypophosphorous acid (50% p.a.) into a 500 mL volumetric flask
5. Pipette 1 mL of CSN 7004 plating solution into the 500 mL volumetric flask
6. Fill up to 500 mL level with HCl solution (10%)

It may be useful to prepare both dilutions of a CSN 7004 process bath to get the best and most reliable result.

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

AAS Measurement:

1. Set the AAS to a wavelength of 235.5 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R^2 . If $R^2 < 0.95$, all standard solutions need to be re-made freshly and measured again.
6. If R^2 is ok, measure the prepared CSN 7004 plating bath sample
7. Record the Sn [$\mu\text{g/mL}$] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted CSN 7004 plating solution exceed 120 $\mu\text{g/mL}$, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Cu content calculation of the plating solution.

Calculation: $\text{Sn(IV) [g/L]} = (\text{ppm Sn} \times 0.5) - (\text{Sn(II) from titration})$ (for the 1:500 dilution of the CSN 7004 sample)

$\text{Sn(IV) [g/L]} = (\text{ppm Sn} \times 0.1) - (\text{Sn(II) from titration})$ (for the 1:100 dilution of the CSN 7004 sample)

Iron (Fe) determination with AAS (GBC 908AA) (Method # 15)

Iron is a contaminant, that can affect the tin deposit quality and solderability performance of the ORMECON™ CSN FF and FF-W surface finish. Especially fresh iron is a risk.

If iron is suspected to be the reason for a quality issue, this analysis should be used to detect iron in the plating bath.

Besides, it is recommended to use this analysis with a two day frequency for one week after initial make-up of a fresh CSN 7004 bath to determine, if an iron source is present.

This measurement is not a regular analysis method. It is only necessary, if e.g. an iron contamination is suspected due to quality problems such as discoloration or solderability decrease.

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for iron)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

AAS Parameter: Wavelength: 248.3 nm
 Slit width: 0.2 nm
 Lamp current: 7.0 mA

Working range: from 2 – 9 $\mu\text{g/mL}$
 Sensitivity: 0.05 $\mu\text{g} / \text{mL}$
 Flame type: Air Acetylene (oxidizing)

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Procedure:

Preparation of standard solutions for calibration:

All dilutions are made in HNO_3 solution (w = 10%) with p.a. quality

1. Pipette 10 mL of the Fe standard solution (1000 mg/L) into a 100 mL volumetric flask.
2. Fill to 100 mL level with HNO_3 (10%)
3. Take 50 mL of this diluted iron solution, fill into a 500 mL volumetric flask and fill to 500 mL level with HNO_3 (10%)
→ this is now a parent solution with 10 mg/L Fe, which is used to make further dilutions for standards:
4. Prepare the following calibration standards for the AAS measurement carefully, using the 10 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO_3 solution)
 - 2 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill to level with HNO_3 solution (10%))
 - 4 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill to level with HNO_3 solution (10%))
 - 6 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill to level with HNO_3 solution (10%))
 - 8 mg/L (pipette 80 mL parent solution into a 100 mL volumetric flask and fill to level with HNO_3 solution (10%))
 - 10 mg/L (100% parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the standard solution prior to use for the respective standard solution handling!

Preparation of the CSN 7004 plating bath sample:

A minimum dilution of the CSN 7004 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Fill 2 mL hypophosphorous acid (50% p.a.) into a 25 mL volumetric flask
2. Fill 12.5 mL of CSN 7004 plating solution into the 25 mL volumetric flask
3. Fill up to 25 mL level with HNO_3 solution (10%)

AAS Measurement:

1. Set the AAS to a wavelength of 248.3 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R^2 . If $R^2 < 0.95$, all standard solutions need to be re-made freshly and measured again.
6. If R^2 is ok, measure the prepared CSN 7004 plating bath sample
7. Record the Fe [$\mu\text{g/mL}$] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Fe content.

CSN 7004

Control and Analysis Procedures for Immersion Tin bath

Should the measured value of the diluted CSN 7004 plating solution exceed 9 µg/mL, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Fe content calculation of the plating solution.

Calculation:

$$\text{Fe [mg/L]} = (\mu\text{g/mL Fe from AAS}) \times 2 \quad (\text{or any other dilution factor used for the CSN 7004 sample})$$

Beta value [β] (Method # 17)

The beta value is a quality value invented by Ormecon GmbH to specifically determine the plating properties of a CSN 7004 process bath. β is a function of the deposition of a specific pure tin thickness at a defined tin bath temperature and a defined dwell time.

Operating window: $\beta = > 0.7$ (Nominal value = ≥ 0.8)

A drop of the beta value below 0.7 usually indicates that the bath has suffered damage. Possible reasons for a beta drop could be overheating, contamination by organics or foreign metals, excessive Sn(IV) formation, etc. This is normally seen in a decreasing quality of the deposit. So in case of a problem with the plated tin layer thickness, it is recommended to check the beta value of the working solution. During and after the corrective action to eliminate the source of the damage, the beta should be checked again, in order to prove the efficiency of the correction. The beta should increase to the specified range of > 0.7 .

The beta should also be used as the benchmark for CSN 7004 conditioning after process installation. Storage and transportation can temporarily affect the plating conditions of the fresh CSN 7004 solution, resulting in a low beta value. However conditioning is required after initial make-up and the beta should be back in specification within 24 hours.

In order to determine the beta correctly, tin thickness, tin bath temperature and dwell time need to be measured with utmost care. Using wrong data for the beta calculation could lead to false results and wrong interpretations. Provisions for an accurate beta determination are:

- Check exact temperature of the plating bath manually with a thermometer. Do not rely on temperature readings from the equipment.
- Control dwell times precisely with manual stop watch
- Measure tin deposit with calibrated coulometric instrument (preferably GCM).

Refer to the Beta Table in the Analysis Protocol section. This is set for a bath temperature of 65°C and 20 min. plating time and can be easily used to determine the beta value of your CSN 7004 plating solution (keep to these conditions thoroughly to get reliable results).

A beta value below 0.7 indicates a damage to the bath, e.g. due to overheating, organic contamination, etc. The root cause for this damage needs to be found and eliminated. A plating solution with a beta below 0.7 can possibly be saved with CSN 70004 CAT if the damage is caused by overheating. In case of other reasons for the damage, the plating bath needs to be (partially) exchanged until the source is finally eliminated

CSN 7004

Analysis Protocol

- Determination of Specific Gravity -

Date of analysis: _____ Analyzer (initials): _____

Date of sample removal: _____ Signature: _____

Sample no.: _____

Reagents: ---**Equipment:** Analytic balance with 0.01 g precision
100 mL Volumetric flask
Thermometer**Procedure:**

1. The solution has to have a temperature of 20°C (if not, warm the solution up / cool it down to 20°C) before filling
2. Tare a dry 100 mL volumetric flask on an analytical balance
3. Fill to mark with CSN 7004 working solution
4. Record the mass of the CSN 7004 solution

→ Make 3 measurements and determine average value

Calculation:
$$\text{Specific Gravity (Density) [g/mL]} = \text{mass of CSN 7004 solution [g]} / 100$$
Analysis results:

Weight of solution: _____ g Specific Gravity: _____ g/mL

Weight of solution: _____ g Specific Gravity: _____ g/mL

Weight of solution: _____ g Specific Gravity: _____ g/mL

Average Specific Gravity of CSN 7004 solution: _____ g/mL

CSN 7004

Analysis Protocol

- Determination of copper content with AAS - for GBC 908AA (standards)

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)
Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Cu standard solution (1000 mg/L)
 HNO₃ solution (10% p.a.)

Equipment: 500 mL Volumetric flask
 5 x 100 mL Volumetric flask
 PE bottles: 1x 50 mL, 5x 100 mL,
 1x 250 mL
 Pipettes: 1x 50 mL, 1x 20 mL,
 1x 10 mL

AAS Parameter: Wavelength: 327.4 nm
 Slit width: 0.2 nm
 Lamp current: 3.0 mA

Working range: from 2.5 – 10 µg/mL
 Sensitivity: 0.05 µg / mL
 Flame type: Air Acetylene (oxidizing)

Procedure:**Preparation of standard solutions for calibration:**

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 10 mL of the Cu standard solution (1000 mg/L) into a 100 mL volumetric flask.
2. Fill to 100 mL level with HNO₃ (10%)
3. Take 50 mL of this dilutes copper standard solution, fill into a 500 mL graduated flask and fill to level with HNO₃ (10%).
 → this is now a parent solution with 10 mg/L Cu, which is used to make further dilutions for standards:
4. Prepare the following calibration standards for the AAS measurement carefully, using the 10 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 2 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 4 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 6 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 8 mg/L (pipette 80 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 10 mg/L (100% parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the standard solution prior to use for the respective standard solution handling!

CSN 7004

Analysis Protocol

- Determination of copper content with AAS - For GBC 908AA (measurement)

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for copper)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Cu calibration standards
(2, 4, 6, 8 and 10 mg/L)
Hypophosphorous acid (50% p.a.)
HNO₃ solution (10% p.a.)

Equipment: 2x 1000 mL Volumetric flask
1x 1 mL Pipette

AAS Parameter: Wavelength: 327.4 nm
Slit width: 0.2 nm
Lamp current: 3.0 mA

Working range: from 2.5 – 10 µg/mL
Sensitivity: 0.05 µg / mL
Flame type: Air Acetylene (oxidizing)

Preparation of the CSN 7004 plating bath sample:

A minimum dilution of the CSN 7004 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Fill 5 mL hypophosphorous acid (50% p.a.) into a 500 mL volumetric flask
 2. Pipette 1 mL of the CSN 7004 plating solution into the 500 mL volumetric flask
 3. Fill up to 500 mL level with HNO₃ solution (10%)
- this is now a 1:500 dilution

In case the copper level in the plating bath sample is very high, it may be appropriate to use a higher dilution (1:1000). In this case the sample should be prepared as follows:

4. Fill 5 mL hypophosphorous acid (50% p.a.) into a 1000 mL volumetric flask
5. Fill 1 mL of the CSN 7004 plating solution into the 1000 mL volumetric flask
6. Fill up to 1000 mL level with HNO₃ solution (10%)

It may be useful to prepare both dilutions of a CSN 7004 process bath to get the best and most reliable result.

CSN 7004

Analysis Protocol

- Determination of copper content with AAS - For GBC 908AA (measurement continued)

AAS Measurement:

1. Set the AAS to a wavelength of 327.4 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R^2 . If $R^2 < 0.95$, all standard solutions need to be re-made freshly and measured again.
6. If R^2 is ok, measure the prepared CSN 7004 plating bath sample
7. Record the Cu [$\mu\text{g/mL}$] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted CSN 7004 plating solution exceed 10 $\mu\text{g/mL}$, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Cu content calculation of the plating solution.

Calculation:

$$\text{Cu [g/L]} = (\mu\text{g/mL Cu from AAS}) \times 500 / 1000 \quad (\text{for the 1:500 dilution of the CSN 7004 sample})$$

$$\text{Cu [g/L]} = (\mu\text{g/mL Cu from AAS}) \times 1000 / 1000 \quad (\text{for the 1:1000 dilution of the CSN 7004 sample})$$

R^2 _____

Analysis results:

Cu from AAS: _____ $\mu\text{g/mL}$

Cu from AAS: _____ $\mu\text{g/mL}$

Copper content of CSN 7004 solution : _____ g/L

CSN 7004

Analysis Protocol

- Determination of copper content with Titration -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Ammonium/ammonium chloride buffer solution*
Hydrogen Peroxide (35%) H_2O_2
0.1 mol/L EDTA-2Na (Di sodium salt of EDTA)
PAN indicator $C_{15}H_{11}N_3O$ (0.1% in methanol)
DI water

Equipment: 25 mL Burette
1000 mL Volumetric flask
5 mL Pipette
3 x 250 mL Erlenmeyer flask

Procedure:*** Preparation of ammonium/ammonium chloride buffer solution**

1. Fill a 1000 mL volumetric flask with approx. 300 mL of DI water
2. Dissolve 54 g of ammonium chloride (NH_4Cl) and 350 mL ammonium (NH_4OH 25%)
3. After complete solution add DI water to 1000 mL level mark

Analysis procedure:

1. Fill ~50 mL DI water into a 250 mL Erlenmeyer flask
2. Pipette 5 mL of CSN 7004 process bath sample in the Erlenmeyer flask
3. Add 5.0 mL ammonium/ammonium chloride buffer and mix well
4. Check pH and add more buffer if necessary to adjust pH = 8 - 9
5. Then add Hydrogen Peroxide (35%) (max. 25 mL) until the solution starts to foam and wait 2 - 3 minutes
6. Add 3 - 5 drops of PAN indicator and mix thoroughly
7. Titrate with 0.1 mol/L EDTA-2Na (Di sodium salt of EDTA) from purple to a mint green endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

Copper content [g/L] = Average volume of 0.1 mol/L EDTA-2Na used [mL] x 1,27 x F

Factor F = Factor of EDTA (if unknown, use F=1)

Analysis results:

Consumption of EDTA: _____ mL

Consumption of EDTA: _____ mL

Consumption of EDTA: _____ mL

Consumption of EDTA: _____ mL

Consumption of EDTA: _____ mL

Average Consumption of EDTA: _____ mL

Copper content of CSN 7004 solution : _____ g/L

CSN 7004

Analysis Protocol

- Determination of copper content with UV-Vis -

(not as accurate as AAS and titration)

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Ammonium 25%
Hydrogen Peroxide 35%

Equipment: UV-Vis Spectrometer
UV-Vis Cuvette
0.5 mL Pipette
5.0 mL Pipette
0.1 mL Pipette
25 mL beaker

Procedure:

1. Calibrate UV Vis Spectrometer with ammonia 25% as base line
2. Fill 1.0 mL CSN 7004 process solution into a 25 mL beaker
3. Add 10 mL ammonia 25% solution
4. Add 0.2 mL hydrogen peroxide solution (35%) and mix with a dry glass stick
5. Filtrate through Blackband filter (12 – 25 µm fineness)
6. Fill a UV-Vis Cuvette with this prepared CSN 7004 solution
7. Wait 1 minute before measuring the sample
8. Check extinction at ~ 635 nm
9. Check extinction at ~ 450 nm
10. read UV Vis display for results given for the two typical extinctions

→ Make 3 measurements and determine average value

Calculation: Cu concentration [g/L] =
$$\frac{(\text{Extinction @ 635 nm} - \text{Extinction @ 450 nm} - 0.025)}{0.0744}$$

Analysis results: UV-Vis result: _____ (Extinction)

UV-Vis result: _____ (Extinction)

UV-Vis result: _____ (Extinction)

Average UV-Vis result: _____

Copper content of CSN 7004 solution : _____ g/L

CSN 7004

Analysis Protocol

- Determination of Stannous tin (Sn^{2+}) concentration -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Sodium acetate buffer solution*
 Hypo phosphoric acid, 50% p.a.
 0.1 mol/L EDTA-2Na (Di sodium salt of EDTA)
 Xenol Orange (1% grinded with KNO_3)
 DI water

Equipment: 25 mL Burette
 5 mL Pipette
 5 mL Measuring pipette
 3 x 250 mL Erlenmeyer flask
 1000 mL Volumetric flask

Procedure:* Preparation of sodium acetate buffer solution

1. Fill a 1000 mL volumetric flask with approx. 500 mL of DI water
2. Dissolve 550g of sodium acetate tri-hydrate
3. After complete solution add DI water to 1000 mL level mark

Analysis procedure:

1. Fill 50 mL of DI water to a 250 mL Erlenmeyer flask
2. Add 5.0 mL hypophosphorous acid (50%) with measuring Pipette and mix
3. Pipette 5.0 mL of the CSN 7004 working solution into the flask
4. Slowly add acetate buffer solution until pH reaches 4.0 (+/- 0.5) (with approx. 10 – 30 mL)
5. Add DI water until sample volume is 100 – 150 mL
6. Add 1 - 2 micro-spatula tips of grinded Xenol Orange indicator
7. Titrate with 0.1 mol/L EDTA-2Na (Di sodium salt of EDTA) from purple to a light yellow endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation: Tin (Sn^{2+}) content [g/L] = Average volume of 0.1 mol/L EDTA-2Na used [mL] x 2.374 x F
 Factor F = Factor of EDTA (if unknown, use F=1)

Analysis results:

Consumption of EDTA: _____ mL

Consumption of EDTA: _____ mL

Consumption of EDTA: _____ mL

Consumption of EDTA: _____ mL

Consumption of EDTA: _____ mL

Average Consumption of EDTA: _____ mL

Stannous Tin (Sn^{2+}) content of CSN 7004 solution : _____ g/L

CSN 7004

Analysis Protocol

- Evaluation of acidity by neutralization -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water
1 mol/L NaOH
Cresol Red
(0.1 g in 100 g of Ethanol 20%)

Equipment: 25 mL burette
1 mL pipette
3 x 250 mL Erlenmeyer flask

Procedure:

1. Fill approx. 50 mL of DI water into a 250 mL Erlenmeyer flask
2. Pipette 1 mL of CSN 7004 process bath into the flask
3. Add DI water until the entire probe volume is 100 – 150 mL
4. Add 8 – 10 drops of Cresol Red indicator solution
5. Titrate with 1.0 mol/L NaOH from yellow to the purple endpoint

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation: Acidity [mol/L] = Average volume of 1.0 mol/L NaOH solution used [mL] x F

Factor F = Factor of NaOH (if unknown, use F=1)

Analysis results: Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Consumption of NaOH: _____ mL

Average Consumption of NaOH: _____ mL

Acidity of CSN 7004 bath : _____ mol/L

CSN 7004**Analysis Protocol****- Determination of complexing agent with UV-Vis -**

This measurement is only necessary, if copper is removed from CSN 7004 solution by crystallization (CSN Regenerator) or as a control measurement, if tin deposit quality is out of specification, even though acidity, density, tin and copper content are within specified range.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water
Hypo phosphoric acid (50%)

Equipment: UV-Vis Spectrometer
UV-Vis cuvette
2 x 1000 mL Volumetric flask
5 mL Pipette
10 mL Pipette
5 mL Measuring pipette

Procedure:

1. Fill approx. 500 mL into a 1000 mL volumetric flask
2. Add 5 mL hypophosphorous acid (50%) with 5mL measuring pipette
3. Pipette 5 mL of CSN 7004 process bath into the flask
4. Fill up to 1000 mL level with DI water
5. Pipette 10 mL of this diluted solution into another 1000 mL volumetric flask
6. Fill up to 1000 mL level with DI water
7. Fill a UV-Vis cuvette (10 mm) with this solution
8. Measure absorbance (Abs.) at **232 nm**, using DI water as a blank
9. Read UV Vis display for results

→ Make 3 measurements and determine average value

Calculation: Complexing agent concentration [g/L] = Extinction (Abs.) @ 232 nm x 136

Analysis results: UV-Vis result: _____ nm

UV-Vis result: _____ nm

UV-Vis result: _____ nm

Average UV-Vis result: _____ nm**Complexing agent content of CSN 7004 solution :** _____ g/L

CSN 7004

Analysis Protocol

- Determination of complexing agent with Titration - (not as accurate as UV-Vis)

This measurement is only necessary, if copper is removed from CSN 7004 solution by crystallization (CSN Regenerator) or as a control measurement, if tin deposit quality is out of spec., even though acidity, density, tin and copper content are within specified range.

Besides the complexing agent, this titration also detects stannous tin (Sn^{2+}) and copper. It is therefore necessary to determine stannous tin and copper prior to this titration in order to subtract them from the titration's result.

For subtraction of Sn^{2+} and Cu it is necessary to transfer the concentration values given in [g/L] into mol/L. This is achieved by the following equations:

$$\text{Sn}^{2+} [\text{mol/L}] = \text{Sn}^{2+} [\text{g/L}] / 118.7 [\text{g/mol}] \quad \text{and}$$

$$\frac{1}{2} \text{Cu} [\text{mol/L}] = \text{Cu} [\text{g/L}] / 63.5 [\text{g/mol}] / 2$$

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water
 H_2SO_4 conc. (96%)
 Ferroin Indicator
 KMnO_4 c=0.02 mol / L

Equipment: 25 mL Burette
 0.1 mL Pipette
 5 mL Pipette
 250 mL Erlenmeyer flask

Procedure: **Only use fresh KMnO_4 solution to ensure accuracy of measurement!!!**

1. Fill approx. 100 mL of DI water to a 250 mL Erlenmeyer Flask
2. Add 5 mL concentrated sulfuric acid (H_2SO_4) (96%) and mix
3. Pipette 0.1 mL of the CSN 7004 working solution into the flask and mix
4. Add 1 - 2 drops of Ferroin Indicator and mix thoroughly
5. Titrate slowly with 0.02 mol/L KMnO_4 (=0.1 N KMnO_4) from rose to the colorless endpoint (has to stay colorless for nearly 15 sec.)

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

$$\text{Complexing agent [g/L]} = ((\text{Quantity of used } \text{KMnO}_4 [\text{mL}] \times F) \times 0.395 - \text{Sn}^{2+} [\text{mol/L}] - \frac{1}{2} \text{Cu} [\text{mol/L}]) \times 76.13$$

Factor F = Factor of KMnO_4 (if unknown, use F=1)

Analysis results:Consumption of KMnO_4 : _____ mLConsumption of KMnO_4 : _____ mLConsumption of KMnO_4 : _____ mLConsumption of KMnO_4 : _____ mLConsumption of KMnO_4 : _____ mL**Average KMnO_4 consumption** _____ mL**Complexing agent content of CSN 7004 solution :** _____ g/L

CSN 7004

Analysis Protocol

- Determination of Sn(IV) - with AAS GBC 908AA (standards)

Tin oxidizes during the operation of CSN 7004 and Sn(II) is transformed into Sn(IV). These tin oxides can cause quality problems, especially because they make the bath hazy and make the plating rate of the bath drop significantly. If this is the case, the Sn(IV) content can be measured with a subtractive method. First the total Sn content of the bath sample is measured. Then the stannous tin (Sn^{2+}) content is detected by titration (method # 11). Subtracting the Sn(II) from the total Sn concentration gives the Sn(IV) content in the plating bath sample.

This measurement is not a regular analysis method. It is only necessary, if e.g. the CSN 7004 bath is cloudy/hazy or the plating rate drops significantly.

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for tin)

Should the working range of your AAS vary from the conditions given above, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: HCl solution (10 %)
Sn standard solution (1000 mg/L)

Equipment: Volumetric flask: 1 x 250 mL,
5 x 100 mL
Pipettes: 1 x 50 mL, 1 x 20 mL
1 x 10 mL
PE-bottles: 1x 100 mL,
5x 100 mL

It's very difficult to determine Sn(IV) with Atomic Absorption Spectroscopy because during the measurement carbon, which forms as a by product, can block the burner and lead to false results. This is the reason why this method isn't completely checked yet.

AAS Parameter:	Wavelength: 235.5 nm	Working range: from 35 – 135 µg/mL
	Slit width: 0.5 nm	Sensitivity: 0.72 µg / mL
	Lamp current: 5.0 mA	Flame type: Nitrous oxide-acetylene (reducing slightly luminous)

CSN 7004**Analysis Protocol****- Determination of Sn(IV) - (with AAS GBC 908AA, Standards continued)****Procedure:****Preparation of standard solutions for calibration:**

All dilutions are made in HCl solution (w = 10%) with p.a. quality

1. Pipette 50 mL of the Sn standard solution (1000 mg/L) into a 250 mL volumetric flask.
2. Fill to 200 mL level with HCl (10%).
→ this is now a parent solution with 200 mg/L Sn, which is used to make further dilutions for standards:
3. Prepare the following calibration standards for the AAS measurement carefully, using the 200 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HCl solution)
 - 40 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))
 - 60 mg/L (pipette 30 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))
 - 80 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))
 - 100 mg/L (pipette 50 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))
 - 120 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill to level with HCl solution (10%))

Note: rinse all pipettes and bottles with a small quantity of each of the standard solution prior to use for the respective standard solution handling!

CSN 7004

Analysis Protocol

- *Determination of Sn(IV) - (with AAS GBC 908AA, measurement)*

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for tin)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice.

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: HCl solution (10 %)
Sn calibration standards
(40, 60 80 100 and 120 mg/L)

Equipment: 1 x 500 mL Volumetric flask
1 x 100 mL Volumetric flask
1 x 1 mL Pipette

It's very difficult to determine Sn(IV) with Atomic Absorption Spectroscopy because during the measurement carbon, which forms as a by product, can block the burner and lead to false results. This is the reason why this method isn't completely checked yet.

AAS Parameter: Wavelength: 235.5 nm
Slit width: 0.5 nm
Lamp current: 5.0 mA

Working range: from 35 – 135 µg/mL
Sensitivity: 0.72 µg / mL
Flame type: Nitrous oxide-acetylene
(reducing slightly luminous)

Preparation of the CSN 7004 plating bath sample:

A minimum dilution of the CSN 7004 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Fill 5 mL hypophosphorous acid (50% p.a.) into a 100 mL volumetric flask
 2. Pipette 1 mL of CSN 7004 plating solution into the 100 mL volumetric flask
 3. Fill up to 100 mL level with HCl solution (10%)
- this is now a 1:100 dilution

In case the tin level in the plating bath sample is very high, it may be appropriate to use a higher dilution (1:500). In this case the sample should be prepared as follows:

4. Add 5 mL hypophosphorous acid (50% p.a.) into a 500 mL volumetric flask
5. Fill 1 mL of CSN 7004 plating solution into the 500 mL volumetric flask
6. Fill up to 500 mL level with HCl solution (10%)

It may be useful to prepare both dilutions of a CSN 7004 process bath to get the best and most reliable result.

CSN 7004

Analysis Protocol

- *Determination of Sn(IV) - (with AAS GBC 908AA, measurement continued)*

AAS Measurement:

1. Set the AAS to a wavelength of 235.5 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R^2 . If $R^2 < 0.95$, all standard solutions need to be re-made freshly and measured again.
6. If R^2 is ok, measure the prepared CSN 7004 plating bath sample
7. Record the Sn [$\mu\text{g/mL}$] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Cu content.

Should the measured value of the diluted CSN 7004 plating solution exceed 120 $\mu\text{g/mL}$, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Cu content calculation of the plating solution.

Calculation: $\text{Sn(IV) [g/L]} = (\text{ppm Sn} \times 0.5) - (\text{Sn(II) from titration})$ (for the 1:500 dilution of the CSN 7004 sample)

$\text{Sn(IV) [g/L]} = (\text{ppm Sn} \times 0.1) - (\text{Sn(II) from titration})$ (for the 1:100 dilution of the CSN 7004 sample)

R^2 _____

Analysis results:

Sn from AAS: _____ $\mu\text{g/mL}$

Sn from AAS: _____ $\mu\text{g/mL}$

Sn(IV) content of CSN 7004 solution : _____ g/L

CSN 7004

Analysis Protocol

Determination of Fe contamination with AAS - GBC 908AA (standards)

This measurement is not a regular analysis method. It is only necessary, if e.g. the immersion tin deposit becomes gray / discolored and / or solderability drops significantly.

Iron is a contaminant, that can affect the tin deposit quality and solderability performance of the ORMECON™ CSN FF and FF-W surface finish. Especially fresh iron is a risk.

If iron is suspected to be the reason for a quality issue, this analysis should be repeated in a two day interval, in order to determine any change in the iron concentration of the tin bath. If the iron content in the CSN 7004 solution increases over time, a source for fresh iron emission is available, continuously "bleeding" fresh iron into the plating solution.

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for iron)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Fe standard solution (1000 mg/L)
HNO₃ solution (10% p.a.)

Equipment: 500 mL volumetric flask
5 x 100 mL volumetric flask
PE bottles: 1x 50 mL, 4x 100 mL,
1x 250 mL
Pipettes: 1x 50 mL, 1x 20 mL,
2x 10 mL

AAS Parameter: Wavelength: 248.3 nm
Slit width: 0.2 nm
Lamp current: 7.0 mA

Working range: from 2 – 9 µg/mL
Sensitivity: 0.05 µg / mL
Flame type: Air Acetylene (oxidizing)

CSN 7004

Analysis Protocol

*Determination of Fe contamination with AAS – GBC 908AA (standards continued)**Procedure:***Preparation of standard solutions for calibration:**

All dilutions are made in HNO₃ solution (w = 10%) with p.a. quality

1. Pipette 10 mL of the Fe standard solution (1000 mg/L) into a 100 mL volumetric flask.
2. Fill to 100 mL level with HNO₃ (10%)
3. Take 50 mL of this diluted iron standard solution, fill into a 500 mL volumetric flask and fill to 500 mL level with HNO₃ (10%)
→ this is now a parent solution with 10 mg/L Fe, which is used to make further dilutions for standards:
4. Prepare the following calibration standards for the AAS measurement carefully, using the 10 mg/L parent solution and store in clean PE bottles:
 - 0 mg/L (10% HNO₃ solution)
 - 2 mg/L (pipette 20 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 4 mg/L (pipette 40 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 6 mg/L (pipette 60 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 8 mg/L (pipette 80 mL parent solution into a 100 mL volumetric flask and fill to level with HNO₃ solution (10%))
 - 10 mg/L (100% parent solution)

Note: rinse all pipettes and bottles with a small quantity of each of the standard solution prior to use for the respective standard solution handling!

CSN 7004

Analysis Protocol

Determination of Fe contamination with AAS - GBC 908AA (measurement continued)

This measurement is not a regular analysis method. It is only necessary, if e.g. the immersion tin deposit becomes gray / discolored and / or solderability drops significantly.

Iron is a contaminant, that can affect the tin deposit quality and solderability performance of the ORMECON™ CSN FF and FF-W surface finish. Especially fresh iron is a risk.

If iron is suspected to be the reason for a quality issue, this analysis should be repeated in a two day interval, in order to determine any change in the iron concentration of the tin bath. If the iron content in the CSN 7004 solution increases over time, a source for fresh iron emission is available, continuously "bleeding" fresh iron into the plating solution.

Attention: Adhere to your specific AAS instruction guide (especially with regards to the concentrations of the ideal detection window for iron)

Should the working range of your AAS vary from the conditions given, dilutions of the standard solutions and the plating bath need to be adjusted according to the equipment manufacturer's advice

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Fe standard solution
(2, 4, 6, 8 and 10 mg/L)
HNO₃ solution (10% p.a.)

Equipment: 25 mL Volumetric flask
2 mL Measuring pipette
10 mL Pipette
2.5 mL Pipette

AAS Parameter: Wavelength: 248.3 nm
Slit width: 0.2 nm
Lamp current: 7.0 mA

Working range: from 2 – 9 µg/mL
Sensitivity: 0.05 µg / mL
Flame type: Air Acetylene (oxidizing)

Preparation of the CSN 7004 plating bath sample:

A minimum dilution of the CSN 7004 plating bath is necessary. Further dilution may be required because of AAS conditions.

1. Fill 2 mL hypophosphorous acid (50% p.a.) into a 25 mL volumetric flask
2. Pipette 12.5 mL of CSN 7004 plating solution into the 25 mL volumetric flask
3. Fill up to 25 mL level with HN O₃ solution (10%)

AAS Measurement:

1. Set the AAS to a wavelength of 248.3 nm
2. Switch it on at least 60 minutes prior to measuring (detector needs approx. 60 min. to reach operating temperature)
3. Optimize the flame according to equipment manufacturer's recommendation
4. Start measuring standard solutions with AAS
5. Check R². If R² < 0.95, all standard solutions need to be re-made freshly and measured again.
6. If R² is ok, measure the prepared CSN 7004 plating bath sample
7. Record the Fe [µg/mL] readings from the AAS
8. After sample measurement, a control measurement should be run with the standard solution coming closest to the plating bath's Fe content.

Should the measured value of the diluted CSN 7004 plating solution exceed 9 µg/mL, an additional dilution of the sample is recommended, because this is off the optimal measuring range and accuracy is critical. It is important to consider any additional sample dilution for the final Fe content calculation of the plating solution.

Calculation:

$$\text{Fe [mg/L]} = (\mu\text{g/mL Fe from AAS}) \times 2 \quad (\text{or any other dilution factor used for the CSN 7004 sample})$$

R² _____

Analysis results:

Fe from AAS: _____ µg/mL

Fe from AAS: _____ µg/mL

Fe content in CSN 7004 solution : _____ mg/L

CSN 7004

Analysis Protocol

- Determination of CSN 7004 beta value [β] -

Date of analysis: _____ Analyzer (initials): _____

Date of sample removal: _____ Signature: _____

Sample no.: _____

Please note: Temperature and times have to be absolutely accurate to ensure a correct result. The temperature of the tin bath must be 64.5 – 65.5 °C during the full duration of immersion time. Manual temperature measurement is advised. The immersion time must be within a tolerance of +/- 5 sec. Tin thickness must be measured coulometrically, preferably with GCM. If another instrument is used, calibration with GCM is required. Calibration values can be included on the table below. X-Ray instruments are normally not suitable for beta determination because their error is too high (~ 10-25%).

Temperature	Dipping time	Sn-Thickness GCM	Sn-Thickness customer instrument		beta of CSN 7004
			SERA / GCM / others		
°C	min	[μ m]	[μ m]	[pinch]	[β]
65	20	0,97			0,74
65	20	0,98			0,75
65	20	0,99			0,76
65	20	1,01			0,77
65	20	1,02			0,78
65	20	1,03			0,79
65	20	1,04			0,80
65	20	1,06			0,81
65	20	1,07			0,82
65	20	1,08			0,83
65	20	1,10			0,84
65	20	1,11			0,85
65	20	1,12			0,86
65	20	1,14			0,87
65	20	1,15			0,88
65	20	1,16			0,89
65	20	1,18			0,90
65	20	1,19			0,91
65	20	1,20			0,92
65	20	1,21			0,93
65	20	1,23			0,94
65	20	1,24			0,95
65	20	1,25			0,96
65	20	1,27			0,97
65	20	1,28			0,98
65	20	1,29			0,99
65	20	1,31			1,00
65	20	1,32			1,01
65	20	1,33			1,02
65	20	1,35			1,03

Beta value of CSN 7004 plating solution : _____

CSN 7004

Analysis and Replenishment Record #1 of 4

Bath volume: _____ Liter Date of analysis: _____ . _____ . _____

Throughput: _____ m² / ft² Analyzer (initials): _____

Capacity / Liter: _____ m²/L or ft²/L (throughput : bath volume)

Date of installation: _____ . _____ . _____ Signature: _____

Physical Data:

	OK	Caution	NOK
Form:	liquid	white precipitation	
Color:	slightly yellow	others	
Appearance:	clear / slightly opaque	opaque / hazy	different
Foam formation:	yes	no	
Odor:	slightly sulfuric	sulfuric	H ₂ S odor

Analysis procedure:

Analysis and adjustment of Specific Gravity is the first step in the CSN 7004 replenishment procedure:

Density / Specific Gravity

➔ Average density of CSN 7004 = _____ g/cm³

Operating range of specific gravity @ 20°C : 1.18 – 1.23 g/cm³

Operating range of specific gravity @ 60 – 70°C : 1.16 – 1.25 g/cm³

Nominal value @ 20°C: **1.20 g/cm³**

Nominal value @ 60 – 70°C: **1.18 g/cm³**

Action if specific gravity is too high: add DI water to process solution and check specific gravity during DI water addition.

Action if specific gravity is too low: evaporate water and check specific gravity during evaporation process

CSN 7004

Analysis and Replenishment Record #2 of 4

Date of analysis: _____ . _____ . _____

Signature: _____

After Specific Gravity adjustment, copper content has to be determined and measured for reduction have to be executed as described if applicable.

Copper content determination with AAS, UV-VIS or Titration

→ Copper content = _____ g/L

Operating range for copper content: < 8.5 g/L for vertical and horizontal operation

Action if copper content is too high:

If the copper content exceeds the given values, take out approx. 5 - 15% of the process solution and compensate with a similar quantity of fresh CSN 7004 (readily mixed).

Note: If the CSN Regenerator is available, all quantities removed from the CSN 7004 process baths can be regenerated and used again. If no regeneration is possible, such quantities have to be disposed.

CSN Regenerator:

The copper content in the process bath can be reduced by "freezing" copper out of the solution. This would substitute the above described "feed-and-bleed" procedure and will limit chemical consumption in addition to prolonging the bath life. The CSN Regenerator process will force the precipitation of a copper complex from the tin solution. Besides copper also a part of the tin solution's complexing agent would be precipitated. This complexing agent is essential for a high tin deposit quality and has to be replenished. It is therefore necessary to analyze the complexing agent concentration in the tin solution after each copper removal and replenish thoroughly with CSN 7004 RG. For procedure see page 212:

CSN 7004

Analysis and Replenishment Record #3 of 4

Date of analysis: _____ . _____ . _____

Signature: _____

Complexing agent

→ Complexing agent concentration in CSN 7004 = _____ g/L

Operating range of complexing agent concentration :

for vertical and horizontal line (Single Modul, Modul B and C): 90 – 140 g/L

for horizontal line (Modul A): 80 – 140 g/L

Nominal value of complexing agent concentration: **100 g/L**

Calculation of necessary replenishment quantity:

Addition of CSN 7004 RG [liter] = (100 – complexing agent concentr.) x 0.01 x bath volume [L]

Result: _____ liter of CSN 7004 RG have to be added

Tin replenishment with CSN 7004 R is the third step in the tin bath replenishment procedure.

Tin content (Sn)

→ Tin concentration in CSN 7004 = _____ g/L

Operating range for tin concentration:

for vertical and horizontal line (Modul B and C): 6 – 24 g/L

for horizontal line (Single Modul, Modul A): 8 – 24 g/L

Nominal value for tin concentration: **9 g/L** for vertical and horizontal operation (Modul B and C)
13 g/L for horizontal operation (Single Modul and Modul A)

Calculation of necessary replenishment quantity:

Addition of CSN 7004 R [L] =
$$\frac{[9 \text{ g/L} - \text{Sn concentr.}] \times \text{Bath volume [L]}}{36}$$

 (for 9 g/L nominal)

Addition of CSN 7004 R [L] =
$$\frac{[13 \text{ g/L} - \text{Sn concentr.}] \times \text{Bath volume [L]}}{32}$$

 (for 13 g/L nominal)

Result: _____ liter of CSN 7004 R have to be added

Note: Mix CSN 7004-R1 and CSN 7004-R2 in a 9:1 volume ratio before adding to the process bath.

CSN 7004

Analysis and Replenishment Record #4 of 4

Date of analysis: ____ . ____ . ____

Signature: _____

Acidity

→ Acidity = _____ mol/L

Operating Range: 4 – 6.0 mol/L

Nominal value: 4.5 mol/L

Acidity should be ok, if bath is replenished properly on a regular basis. Measurement is just necessary to check.

Last replenishment step is the addition of fresh CSN 7004 solution to fill up the bath to its original level.

Beta value

→ β = _____

Operating Range: > 0.7

Nominal value: ≥ 0.8

Overall analysis and replenishment result for CSN 7004 process bath :

Bath is ok, no replenishment necessary

Addition / Evaporation of water is necessary to adjust specific gravity

_____ Liters of process bath have to be removed to reduce Cu content.

_____ Liters process bath have to be removed to make space for CSN 7004-R

Bath needs to be replenished with _____ L CSN 7004 RG

_____ L CSN 7004 R (readily mixed)

Bath needs to be exchanged

Comments: _____

CSN 7004

2.9.4 Replenishment Table

CSN 7004 R (for 9 g/L nominal)

Example for 100 liter bath volume

Values for your bath volume:

_____ liter

Analysis result Tin (Sn) content [g/L]	Addition of CSN 7004 R (readily mixed)[L]	Addition of CSN 7004 R (readily mixed) [L]
13	0.0	
12	0.0	
11	0.0	
10	0.0	
9	0.0	
8	2.8	
7	5.6	
6	8.3	
5	11.1	
4	13.9	
3	16.7	

Bath within spec

The functionality of the tin bath is critical below a tin content of 6 g/L of bath volume, even with replenishment. So it is recommended to exchange the bath.

This replenishment table aims to adjust the nominal level of stannous tin in the CSN 7004 process bath (= 9 g/L). Setting a higher tin concentration up to a max. of 24 g/L is of course possible.

CSN 7004 R (for 13 g/L nominal)

Example for 100 liter bath volume

Values for your bath volume:

_____ liter

Analysis result Tin (Sn) content [g/L]	Addition of CSN 7004 R (readily mixed)[L]	Addition of CSN 7004 R (readily mixed) [L]
13	0.0	
12	3.1	
11	6.3	
10	9.4	
9	12.5	
8	15.6	
7	18.8	
6	21.9	
5	25.0	
4	28.1	
3	31.3	

Bath within spec

The functionality of the tin bath is critical below a tin content of 8 g/L of bath volume, even with replenishment. So it is recommended to exchange the bath.

This replenishment table aims to adjust the nominal level of stannous tin in the CSN 7004 process bath (= 13 g/L). Setting a higher tin concentration up to a max. of 24 g/L is of course possible.

CSN 7004

Replenishment Table

CSN 7004 RG

Example for 100 liter bath volume

Values for your bath volume:

_____ liter

Analysis result Complexing agent content [g/L]	Addition of CSN 7004 RG [L]	Addition of CSN 7004 RG [L]
140	0	
130	0	
120	0	
110	0	
100	0	
90	10	
80	20	
70	30	
60	40	
50	50	

Bath within spec

CSN 7004

2.9.5 Monitoring Record

		<u>Bath:</u> CSN 7004		<u>Bath volume:</u> _____ liter		<u>New make-up:</u> 9 vol.% CSN 7004-1 1 vol.% CSN 7004-2			
Date	Analysis Result					Analysist's Initials	Corrections		Additions / Corrections made (Initials)
	Tin content [g/L]	Copper content [g/L]	Spec. Gravity [g/cm ³]	Acidity [mol/L]	Beta value [β]		1. Addition per bath volume 2. New make-up / (partial) bath exchange 3. CAT process made 4. Comments	Initials	
	6 - 24	< 8.5	1.18 – 1.25 (20°C) 1.16 - 1.23 (65°C)	4.0 – 6.0	0.7 - 1.1				

Immersion Tin Replenishment for ORMECON™ CSN FF / ORMECON™ CSN FF-W

2.10 CSN 7004 R

Product Description

An Immersion Tin bath loses tin during operation due to the deposition. It also loses other components due to drag-out. In order to maintain good operating conditions and to ensure proper tin deposition, it is necessary to regularly monitor and replenish the tin bath CSN 7004 to keep it within the specified range. For this purpose replenishment products are necessary, which are added in a certain quantity, depending on the components missing.

For regular replenishment procedures CSN 7004 R is used. It is a tin concentrate that replaces used tin, but also adds other necessary bath components lost due to drag-out.

If the CSN Regenerator is used for continuous copper and Sn(IV) removal from the process solution, an additional replenishment product is necessary:

CSN 7004 RG Regeneration Replenisher

Further information about this product is given in the specific technical product data sheets.

Application

CSN 7004 R consists from two components: **CSN 7004-R1** and **CSN 7004-R2**, which have to be mixed in a 9:1 volume ratio prior to use.

As a rule of thumb an average CSN 7004 R replenishment quantity is 3.5 – 4.5 Liters / 10 m² can be expected for horizontal processing. The actually required quantity depends on the character and quantity of boards processed. CSN 7004 R quantity necessary for vertical processing is hard to predict, because it is also very much dependant on the line design and operation conditions, that usually vary more than horizontal processing conditions.

Further information about replenishment procedures and necessary CSN 7004 R see page 210

Product Specification

Standard values are given in brackets ()

	CSN 7004-R1	CSN 7004-R2
State:	liquid	liquid
Odor:	slightly acidic	slightly acidic
Color:	yellowish	colorless
Specific Gravity:	1.23 – 1.27 g/cm ³ (1.25)	1.09 – 1.12 g/cm ³ (1.10)
Acid Normality:	4.5 - 5.6 mol/L (5.0)	2.9 - 3.6 mol/L (3.4)
Tin content:	45 – 55 g/L (50)	--
Complexing agent:	100 – 150 g/L (127)	--
Iron:	≤ 100 ppm	≤ 100 ppm
Chem. Characterization:	aqueous tin salt solution concentrate	aqueous tin salt solution concentrate
Tin content (mixed 9:1):	40 – 50 g/L (45)	
Complexing agent (mixed 9:1):	90 – 135 g/L (114)	

CSN 7004 R

Storage Requirements

Do not store together with alkaline or cyanide products and avoid contact with them. Do not use metal containers. CSN 7004 products could precipitate at temperatures $< +10^{\circ}\text{C}$. They totally re-dissolve at higher temperatures (Note: for heating up the solution to dissolve the precipitates do not exceed $+60^{\circ}\text{C}$, because this could irreversibly damage the solution). Make sure all precipitates are resolved prior to use or are fully transferred to the bath tank, when using full container volumes. Keep original containers tightly closed. CSN 7004 products are sensitive to light. They should be stored in tinted containers in a cool dark place, at temperatures between 10 and 30°C . Avoid direct exposure to sun light. Ensure good air circulation.

Shelf Life

CSN 7004-R1 and **CSN 7004-2** have a shelf-life of 12 months from production date, if stored according to storage recommendations.

Waste Removal

CSN 7004-R1 is an aqueous solution containing tin salts, a complexing agent and other components. Unused process chemicals should go to common batch neutralization and waste treatment. Complex cracking is required. **CSN 7004-R2** is an acid aqueous solution and should also go to a common batch neutralization.

Packaging CSN 7004-R1

25 and 180 l PE containers. Other package sizes upon request.

Packaging CSN 7004-R2

5, 10 and 20 l PE containers. Other package sizes upon request.

Safety Recommendations

CSN 7004-R1 and **CSN 7004-R2** are acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

Immersion Tin Regeneration Replenishment for ORMECON™ CSN FF / ORMECON™ CSN FF-W

2.11 CSN 7004 RG

Product Description

During operation of an Immersion Tin bath copper naturally accumulates in the process solution, while tin is constantly taken out. CSN 7004 R helps to replenish lost tin in order to keep the immersion tin bath within specification. However copper concentration is building-up and could become critical at a certain concentration, because it involves the risk of copper enclosures in the tin deposit. This would affect solderability at a certain point and should therefore be avoided.

There are two ways to react on an increasing copper concentration in the tin solution:

- exchange the bath when the copper concentration has reached the specified limit.
- Remove copper from the tin solution regularly with the help of the *CSN REGENERATOR*.

The constant exchange of the tin bath due to limited copper load, is an important driver for high process cost. Using the *CSN REGENERATOR* helps to save process chemicals, significantly prolongs the life circle of an immersion tin bath and therefore cuts costs.

During the regeneration process the process solution is cooled down and a copper complex precipitates from the solution. This loss of complexing agent and other ingredients makes an additional replenishment component necessary:

CSN 7004 RG Regeneration Replenisher

CSN 7004 R mainly replenishes lost tin, which is always necessary when operating a tin bath, while **CSN 7004 RG** especially replenishes ingredients lost after using the *CSN REGENERATOR*.

Application

CSN 7004 RG is a ready to use product that has to be used in combination with *CSN 7004 R* after copper removal with *CSN REGENERATOR*. Its necessary addition is based on a complexing agent analysis (average concentration = 100 g/L).

As a rule of thumb an average **CSN 7004 RG** replenishment quantity of 10 mL/L can be expected for each missing g/L of complexing agent.

Further information about replenishment procedures and necessary **CSN 7004 RG** quantities see page 212.

Product Specification

Standard values are given in brackets ()

State:	liquid
Odor:	slightly acidic
Color:	colorless
Specific Gravity:	1.00 – 1.20 g/cm ³ (1.09)
Compl. agent concentration:	80 – 140 g/L (100)
Acidity:	1.00 – 5.00 mol/L (2.20)
Chem. Characterization:	aqueous complexing agent concentrate

CSN 7004 RG

Storage Requirements

Do not store together with alkaline or cyanide products and avoid contact with them. Do not use metal containers. **CSN 7004 RG** is sensitive to light. They should be stored in tinted containers in a cool dark place, at temperatures between 10 and 30 °C. Avoid direct exposure to sun light. Ensure good air circulation.

Shelf-life

12 months after production date, if stored according to storage recommendations.

Waste Removal

CSN 7004 RG is an aqueous solution containing complexing agents and other components. Unused process chemicals should go to common batch neutralization and waste treatment. Complex cracking is required, even for rinse waters.

Packaging

10, 25 and 200 L PE containers. Other package sizes upon request.

Safety Recommendations

CSN 7004 RG is acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

Decontamination Catalyst for

ORMECON™ CSN FF / ORMECON™ CSN FF-W

2.12 CSN 7004 CAT

Product Description

Any Immersion Tin bath is subject to an aging process over time during which degradation products are formed in the solution. This is a normal time and temperature depending process. The degradation products could be a poison for some of the Immersion tin bath ingredients and the tin deposit. Accumulation of degradation products in the tin bath solution could result in a streaky, dark, even black tin deposit. Temperature plays an important part in this aging process. High temperature accelerates the aging of the bath.

The formation of degradation products is not obvious. They tend to accumulate in the tin solution, especially in surface spheres, but usually remain unseen (except for the occasional occurrence of an oily looking film on the solution's surface). An indirect indication of degradation product formation is a significant loss of Immersion Tin deposit quality.

Usually the bath is perfectly stable at room temperature. Degradation starts at temperatures higher than 40°C. The following conditions could accelerate the degradation of a tin bath solution:

- Storage temperatures of > 40°C over 24 hours and more. This is especially critical, because gaseous degradation products can not evaporate from tightly sealed containers. This could lead to a low quality immersion tin deposit already after initial make-up.
- A strong local heater (Surface temperature > 75°C) in combination with insufficient bath circulation. This includes the risk of local overheating and local formation of degradation products that may accumulate in the solution over time.
- Insufficient air circulation (e.g. breakdown of exhaust system) limits the air exchange and help to keep gaseous products in solution.

All these issues should generally not occur when the goods are transported and stored properly and the equipment is perfectly adjusted to the process' requirements.

CSN 7004 CAT is not a product to compensate insufficient equipment. It can only be a temporary repair measure.

CSN 7004 CAT is only designed to decontaminate *CSN 7004* when suffering from aging. Any other reason that may also cause streaky, dark or black deposit, e.g. contamination with metal ions, contamination from solder masks or CEM-1 base material, rough etching, bad or polluted rinses or any other unspecified treatment, are beyond the influence of *CSN 7004 CAT* and can not be cured.

If a tin bath leads to an unspecified deposit quality regularly, it is necessary to check on equipment and handling procedures. Ormecon International and its global network will assist you to run the ORMECON™ CSN FF and ORMECON™ CSN FF-W process with the best possible result.

Application

CSN 7004 CAT consists from two different components: **CSN 7004-CAT1** and **CSN 7004-CAT2**, which have to be mixed in a 1 : 1 volume ratio prior to use. A blend of these two products has a limited stability and loses its effect when stored for more than two hours.

Add 3 vol.% of the readily mixed *CSN 7004 CAT* to the warm degraded tin bath (usual process temperature) and stir well while adding. Stop filtering the bath for the decontamination period, but keep the circulation pumps running constantly to support the decontamination process. Depending on the bath circulation *CSN 7004 CAT* reacts with the entire tin bath volume within a 24 hour period. The result could be a white precipitation, which makes the bath look slightly hazy.

CSN 7004 CAT

After the 24 hour decontamination period, start filtering the bath again and remove the white precipitated particles to get the bath back to a clear liquid. Change the filters and clean the pump properly after the bath is clear again.

During aging and the decontamination process with *CSN 7004 CAT* important ingredients of the tin bath get lost. They need to be replenished immediately after using *CSN 7004 CAT*. For this purpose it is necessary to add 3.6% of *CSN 7004 R* (R1 and R2 mixed in the usual 9:1 volume ratio) to the decontaminated tin solution.

After this procedure it is recommended to make a full analysis of the process bath and make another addition of *CSN 7004 R* if necessary. It is also recommended to check the tin deposit on test boards regarding appearance, thickness and solderability prior to going back to mass production.

It is recommended to use *CSN 7004 CAT* only up to 3 times during a life cycle of a tin bath. After that, it needs to be exchanged.

Make-up

Example:

Volume of degraded tin bath	100 Liters	
Necessary quantity of <i>CSN 7004 CAT</i> (1 : 1 blend)	3.0 Liters	(1.5 Liters each of CSN 7004-CAT1 and CSN 7004-CAT2)
Necessary quantity of <i>CSN 7004 R</i> (9 : 1 blend)	3.6 Liters	(3.24 Liters of CSN 7004-R1 and 0.36 Liters of CSN 7004-R2)

The catalyst is only effective after mixing **CSN 7004-CAT1** and **CAT2** in a 1 : 1 volume ratio.

The replenisher is only effective after mixing **CSN 7004-R1** and **R2** in a 9:1 volume ratio.

Attention: **CAT1** and **CAT2** should only be mixed immediately prior to addition and only in the quantity needed, because the storage time of the blend is only very limited.

For decontamination of an aged tin bath 3 vol.% of the *CSN 7004 CAT* (1:1 blend) are necessary.

Decontamination is completed after 24 hours. It is then necessary to replenish degraded ingredients to re-balance the tin bath. For this purpose 3.6% of *CSN 7004 R* (9:1 blend) is necessary.

CSN 7004 process bath should now be back to normal high quality. Please check solution with full analysis and tin deposit on appearance, thickness and solderability.

Product Specification

Standard values are given in brackets ()

	CSN 7004 CAT1	CSN 7004 CAT2
State:	liquid	liquid
Color:	green	slightly blue
Odor:	odorless	slightly acidic
Specific gravity:	1.00 - 1.10 g/cm ³ (1.04)	1.00 - 1.10 g/cm ³ (1.04)
Acidity:	1.0 - 2.5 mol/L (1.4)	1.0 - 2.5 mol/L (1.5)
Organic Metal content:	90 - 130 % (100)	--
Active Component content:	--	70 - 120 % (100)
Chem. Characterization:	aqueous dispersion concentrate	aqueous acidic concentrate

CSN 7004 CAT

Storage Requirements

Do not store together with alkaline or cyanide products and avoid contact with them. Do not use metal containers. **CSN 7004-CAT1** and **CSN 7004-CAT2** should not be stored as a blend, because stability is very limited. Both products are sensitive to light and should be stored in tainted containers in a cool dark place, at temperatures between 10 and 30 °C. Avoid direct exposure to sun light. Ensure good air circulation.

Shelf-life

12 months after production date, if stored according to storage recommendations.

Waste Removal

CSN 7004-CAT1 and **CSN 7004-CAT2** are acidic aqueous solutions that should go to a common batch neutralization.

Packaging

5 and 25 L PE containers. Other package sizes upon request.

Safety Recommendations

CSN 7004-R1 and **CSN 7004-R2** are acidic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

Rinse Aid for ORMECON™ CSN FF / ORMECON™ CSN FF-W

2.13 RAD 7000 C

Product Description

RAD 7000 C is a biodegradable, alkaline, organic concentrate used to assist in the removal of residues following the CSN 7004 immersion tin bath and prior to soldering and fusing processes using fluxes. **RAD 7000 C** works without the use of silicone de-foamers.

Application

RAD 7000 C can be used in aqueous in-line spray cleaning or dip systems. 1 – 25% solutions of **RAD 7000 C** are usually added to a heated, re-circulating rinse section to facilitate cleaning in penetrating crevices. **RAD 7000 C** also significantly reduces the ionic contamination on a printed circuit board, because the rinse aid helps to neutralize the acidic residues from the immersion tin bath on the board. Rinsing with **RAD 7000 C** is always followed by thorough water rinsing to ensure removal of the rinse aid solution from the board surface. De-ionized water should be used in the final rinse to achieve a high order of ionic cleanliness.

RAD 7000 treatment is especially recommended for complex board designs, blind vias and solder mask plugged holes.

Make-up recommendation:

DI water	99.0 – 75.0 vol%
RAD 7000 C	1.0 – 25.0 vol%

For replenishment: The concentration of RAD 7000 solutions can be maintained with additions of the **RAD 7000 C** concentrate.

Product Specification

Nominal values / standard parameters are given in brackets ().

State:	liquid
Odor:	slightly ammonia
Color:	clear light amber
Specific Gravity:	0.96 - 1.05 g/cm ³ (0.99)
pH value:	pH = ~12
Chem. Characterization:	base organic concentrate

Process Parameters

Nominal values / standard parameters are given in brackets ().

Concentration of RAD 7000 C:	1.0 – 25.0 vol.% (depending on desired cleaning effect)
Temperature Range:	40 - 60 °C (50°C)
pH (1% in water):	pH = ~ 9.5
pH (25% in water):	pH = ~12

RAD 7000

Process Parameters (continued.)

Dwell Time:	1 - 3 min (2 min.)
Agitation:	mild work agitation, solution agitation and filtration recommended
Application:	vertical: immersion
	horizontal: immersion or spray

Control Procedures

We recommend replacing the RAD 7000 bath when ionic contamination levels become unacceptable. The RAD 7000 bath should be replaced at least once per month. It is possible to analyze RAD 7000 through an acid-base Titration. Refer to the Process Guide' analysis procedures for details.

Equipment Material

Tanks:	PVC or PP; do not use steel or other metals.
Racks / Baskets:	Can be metal structures, but need to be coated with a) pore-free black or green HALAR (do not use blue HALAR!) b) pore-free PP coated stainless steel Baskets can also be completely made from PP No metal is allowed in contact with the solution!
Heaters:	Stainless steel, Quartz or Teflon/PTFE.

Storage Requirements (RAD 7000 C)

Do not store together with acidic or cyanide products and avoid contact with them. Do not use metal containers. Keep original containers tightly closed. Ensure good air circulation.

Shelf Life (RAD 7000 C)

12 months from production date, if stored according to storage recommendations.

Waste Removal (RAD 7000 C)

RAD 7000 C is basic and has to be diluted and neutralized before discharge in public sewage system, preferably in a common batch neutralization. The local waste water regulations have to be adhered to.

Packaging (RAD 7000 C)

1, 5, and 25 L PE containers. Other package sizes upon request.

Safety Recommendations (RAD 700 C)

RAD 7000 C is strongly basic. For handling wear rubber gloves and eye protection, if possible also wear rubber apron. In case of skin contact rinse thoroughly with plenty of cold water. In case of eye contact immediately rinse with plenty of water and consult a physician. Adhere to the information on the Safety Data Sheet.

RAD 7000

Control and Analysis Procedures for RAD 7000

Rinsing of complex layouts containing e.g. blind vias or solder mask plugged holes is difficult and rinsing with water only may not remove all process bath residues or other contaminants from the board. This could lead to a high ionic contamination, corrosion attack of the tin finish (especially around holes), yellowing after reflow, etc.

In order to improve final rinsing, Ormecon offers a rinse aid product, called RAD 7000 C. This is a biodegradable, basic (pH >7), organic concentrate to be blended with water, which assists in the removal of residues from the board. An RAD 7000 process bath not only helps to neutralize acidic residues left from the CSN 7004 immersion tin bath and therefore improves cleanliness significantly. It is also able to remove other residues and contaminants from the surface, so it increases the wettability of the tin hence improving its multiple solderability (which could be disturbed by residues, especially after multiple heat cycles).

RAD 7000 C can be easily applied by blending with DI water. The concentration can vary between 1 and 25%, depending on the necessary or desired level of cleanliness. In case of suspicion of residues affecting multiple solderability, the most efficient concentration to remove such residues and improve multiple solderability has to be evaluated.

In case of cleaning issues, the necessary RAD 7000 C concentration can be determined with ionic contamination measurements.



A highly concentrated RAD 7000 bath has a very high pH and may lead to attack on solder mask or coverlay. If an attack occurs after the RAD 7000 rinsing, the concentration needs to be reduced.

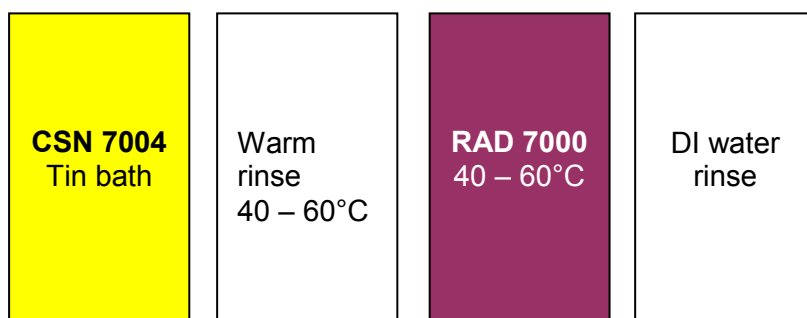


Replenishment of a RAD 7000 rinse is made with *RAD 7000 C*, based on acid-base Titration.

Regular analysis of an RAD 7000 rinse should be made once a day, as neutralization occurs during operation due to dilution. We recommend replacing the RAD 7000 bath when ionic contamination levels become unacceptable. The RAD 7000 bath should be replaced at least once per month.

An RAD 7000 rinse is usually used after the warm rinse following the CSN 7004 bath (see drawing below). It is also operated at elevated temperatures between 40 – 60°C and should be followed by further rinses, to ensure the removal of RAD 7000 from the board. The final rinse has to be operated with DI water to achieve a high order of ionic cleanliness, which is a standard for the ORMECON™ CSN FF and CSN FF-W processes anyway.

Minimum configuration:



RAD 7000

Control and Analysis Procedures for RAD 7000

2.13.1 Analysis

Concentration of RAD 7000 C Determination (Method # 19)

1. Pipette a 5 mL sample of the RAD 7000 working bath into an Erlenmeyer flask
2. Add ~ 100 mL of DI water
3. Add 5 – 10 drops of Bromophenol blue indicator
4. Titrate with 1 mol/L HCl from purple to yellow solution

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

Concentration of RAD 7000 C [%] = Average consumption of HCl x 1.745 x F

Factor F = Factor (Titer) of HCl (if unknown, use F=1)

pH Determination (Method # 10)

7. Adjust RAD 7000 process bath sample to 25°C temperature
8. Switch on pH-meter
9. Start calibration mode
10. Calibrate the pH-meter according to the manufacturers instructions
11. Rinse electrode with DI water again
12. Immerse electrode into RAD 7000 process bath sample and measure pH

→ Make 3 pH measurements and use average value for replenishment procedure determination.

Calculation:

Read pH value from display

RAD 7000

Analysis Protocol

- Evaluation of RAD 7000 C concentration by Titration -

Date of analysis: _____

Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: DI water
1 mol/L HCl
Bromophenol blue indicator

Equipment: 25 mL Burette
5 mL Pipette
3 x 250 mL Erlenmeyer flask

Procedure:

1. Pipette a 5 mL sample of the RAD 7000 working bath into an Erlenmeyer flask
2. Add ~ 100 mL of DI water
3. Add 5 – 10 drops of Bromophenol blue indicator
4. Titrate with 1 mol/L HCl from purple to yellow solution

→ Make 3 titrations. If the results differ by more than ± 1 drop (=0.05 mL), make two additional titrations.

Calculation:

Concentration of RAD 7000 C [%] = Average volume of 1 mol/L HCl solution used x 1.745 x F

Factor F = Factor (Titer) of HCl (if unknown, use F=1)

Analysis results:

Consumption of HCl: _____ mL

Consumption of HCl: _____ mL

Consumption of HCl: _____ mL

Consumption of HCl: _____ mL

Consumption of HCl: _____ mL

Average Consumption of HCl: _____ mL**RAD 7000 C concentration : _____ %**

RAD 7000**Analysis Protocol****- Determination of pH value -**

Date of analysis: _____ Analyzer (initials): _____

Date of sample removal: _____

Signature: _____

Sample no.: _____

Reagents: Standard buffer solutions
(according to manufacturer)**Equipment:** pH-meter, e.g. "Knick Portameas"
2 (3) x 100 mL beaker**Procedure:**

1. Adjust RAD 7000 process bath sample to 25°C temperature
2. Switch on pH-meter
3. Start calibration mode
4. Calibrate pH-Meter according to the manufacturers instructions
5. Rinse electrode with DI water again
6. Immerse electrode into RAD 7000 process bath sample and measure pH

→ Make 3 pH measurements and use average value for replenishment procedure determination.

Calculation: Read pH value from display**Analysis results:** pH result: _____

pH result: _____

pH result: _____

Average pH result: _____

RAD 7000

Analysis and Replenishment Record #1 of 1

Bath volume: _____ Liter Date of analysis: _____ . _____ . _____

Throughput: _____ m² / ft² Analyzer (initials): _____

Capacity / Liter: _____ m²/L or ft²/L (throughput : bath volume)

Date of installation: _____ . _____ . _____ Signature: _____

Physical Data:

	OK	Caution	NOK
Form:	liquid	white precipitation	
Color:	colorless/slightly amber	others	
Appearance:	clear	opaque / hazy	different
Foam formation:	no	yes	

Analysis procedure:

RAD 7000 C content

→ RAD 7000 C concentration = _____ %

Operating range: 1 – 25% (depending on desired cleaning effect)

Result: _____ liter of RAD 7000 C have to be added

Overall analysis and replenishment result for RAD 7000 bath :

Bath is ok, no replenishment necessary

Bath needs to be replenished with _____ liters RAD 7000 C

Bath needs to be exchanged

Comments: _____



RAD 7000

Monitoring Record

<u>Bath:</u> RAD 7000 Bath		<u>Bath volume:</u> Liter		<u>New make-up:</u> 75 – 99 vol% DI water 1 – 25 vol% RAD 7000 C		
Date	Analysis Result		Analysist's Initials	Corrections		Additions made (Initials)
	RAD 7000 C content [%]	pH		1. Addition per bath volume 2. New make-up 3. Comments	Initials	
	1 - 25	9.5 - 12				

2.14 Safety Recommendations

When handling dangerous substances the suitable safety measures for self protection as well as the protection of others and the environment are to be met. The applicable S and R regulations according to the content of the chemical law and the safety regulations are to be adhered to.

All ORMECON™ CSN products are (highly) acidic solutions. Prevent contact with eyes, skin and mucous membrane. When handling the products wear safety clothing such as acid resistant coats, gloves and safety goggles. In case of contact, affected parts must be thoroughly rinsed with plenty of water and contaminated clothing needs to be removed. In case of eye contact, swallowing and inhaling of acid vapors, consult a physician immediately. When handling ORMECON™ CSN products adhere to the detailed product safety data sheets.

Until being properly disposed, ORMECON™ CSN solutions should not be mixed with other chemicals. Not even smaller volumes. In combination with oxidizing substances gas can be formed, which is injurious to health.

2.15 Specification and Warranty

Ormecon GmbH guarantees constant composition of the products and functionality in accordance with the technical information up to the time of delivery and opening of the packages. In its designated state ORMECON™ CSN FF and ORMECON™ CSN FF-W provide surface finish properties for printed circuit boards as defined in the technical data sheets and this Process Guide.

This does not relieve the user from the need to conduct his own experiments and tests. No warranty is given with respect to properties of products manufactured using ORMECON™ CSN FF and/or ORMECON™ CSN FF-W.

All process chemicals are delivered on the basis of the “Delivery conditions of the electroplating industry” and the “General delivery conditions for products and services of the electroplating industry”, and especially on the basis of IPC – A 600 C-D.

The user may assume that if the instructions for use are followed precisely, the properties described in this Process Guide will result.

Necessary quality assurance

The user guarantees that the products provided for ORMECON™ CSN FF and ORMECON™ CSN FF-W will be checked carefully immediately after receipt. Any defects found at this point have to be reported to Ormecon GmbH without any delay.

Contamination of the baths with impurities must be avoided under any circumstances. Fresh water must be used for rinsing. Circulation water must be treated and must not contain any excessive or unusual impurities.

During production, the process checks set out in this Process Guide have to be executed at regular intervals. The treated surface area needs to be documented.

All quality control and quality assurance steps and results need to be documented in accordance with DIN ISO 9001.

2.16 Product liability

Ormecon GmbH does not accept any liability for any use beyond our control.

Product liability shall be accepted for a faulty delivery, that could not have been detected with receipt check or quality controls during production and which falls within the responsibility of Ormecon GmbH as the supplier. Furthermore it is necessary for acceptance of product liability, that it can be proven to the supplier, that no deviation from the process rules and quality assurance rules took place during use of the products.