Operating Instructions for

the Acid Pickling Solution

Regeneration Unit MECER

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User Manual
Revision 1, five-stage sump mixer

Table of contents

1 INTRODUCTION

This section includes information on safety and proper handling of the MECER installation. The following topics will be considered:

- Safety factors
- When will the MECER installation arrive
- Handling the MECER installation

1.1 SAFETY FACTORS

Work on the MECER installation involves the use of hazardous materials. Their brief overview, together with an overview of the causes of electric shock related to electroextraction, are included in this section.

1.1.1 INFORMATION SYMBOLS USED IN THIS INSTRUCTION

The following symbols are used in this manual to warn the user about potentially dangerous conditions:

warning

THE WARNING SIGN DRAWS ATTENTION TO THE PRODUCTION TECHNOLOGY, VIOLATION OF WHICH CAN LEAD TO INJURY OR DEATH.

carefully

THE DANGER SIGN DRAWS ATTENTION TO PRODUCTION TECHNOLOGY, VIOLATION OF WHICH MAY LEAD TO DAMAGE TO THE EQUIPMENT.

note

This symbol highlights the important additional information provided by the various procedures.

1.1.2 WORKING WITH KEROSENE

The organic solution for the MACER installation called MX 90 consists of an organic extractant dissolved in kerosene. Kerosene with an ignition temperature of 71 ° C is considered a flammable liquid. Whenever possible, avoid inhaling its vapors and prolonged contact with the skin. The MX 90 reagent must be stored at a temperature between +5°C and +40°C.

The PBM for the MX 90 reagent is included in the Application.

1.1.3 WORKING WITH SULFURIC ACID

Sulfuric acid is an aggressive liquid that burns the skin and eyes, and irritates the mucous membranes. Avoid contact by using appropriate protective equipment, such as a protective mask and gloves.

When sulfuric acid is poured into the water, heat will be produced, which can lead to boiling and splashing. Acid should be added to the water slowly and carefully. NEVER add water to concentrated acid.

We recommend that the PBM for sulfuric acid be obtained from the supplier and added to the Application for reference in the event of an emergency.

1.1.4 WORKING WITH ELECTROLYZERS

Copper is deposited on the cathode of the electrolyzer at a low voltage with a high direct current. Since this is a low voltage, the electric current cannot overcome the large resistance of human skin. Therefore, there is a small chance of electric shock when touching copper busbars or electrodes. However, a large amount of energy passing through the tires and electrolytes generates and increases their temperature.

Caution should be exercised when using metal objects (e.g. tools) around electrolytic cells. A short circuit between positive and negative tires can cause sparking and the release of a large amount of heat, which can lead to burns. PLEASE NOTE that there may also be a temporary disconnection of the mains voltage supply from the rectifier due to the countercurrent effect of the battery from the electrolyzer.

During the operation of the electrolyzers, oxygen and acid vapors will be produced. Therefore, electrolytic cells must be provided with mechanical ventilation. To reduce the impact on the body, do not remove the covers from the electrolytic cells during operation.

See Section 1.1.3 "Working with sulfuric acid" for more information on precautions with acidic electrolyte.

1.2 WHEN WILL THE INSTALLATION ARRIVE MECER

Inspect the outside of all shipping containers for damage. If the equipment was damaged during transportation, you should immediately contact the shipping company and your Sigma representative.

Compare the contents of each container with the packing list(s) and the original order. Inspect all parts for visible damage. If you have any questions regarding damage or missing parts, contact a Sigma representative immediately.

1.3 HANDLING THE MECER INSTALLATION

Many components of the Mecer installation are made of PVC. Be careful when working on the installation so as not to damage these components.

carefully

DO NOT ALLOW HEATING OF PCBS TO A TEMPERATURE ABOVE 60 ° C. DO NOT ALLOW DIRECT SUNLIGHT TO FALL ON PCBS COMPONENTS FOR A LONG PERIOD OF TIME.

When placing the blocks on permanent installation sites, it is necessary to make sure that all the bearing supports of each of them are firmly installed on the floor and fitted to each other.
For example, the liquid extraction unit has spillway edges that must be installed strictly horizontally.

carefully

DO NOT USE LOW MOLECULAR WEIGHT ORGANIC SOLVENTS TO CLEAN PLASTIC INSTALLATION DETAILS. THIS CAN LEAD TO THE FRAGILITY OF PVC PARTS AND "TURBIDITY" PLASTIC SURFACES. TO REMOVE SUCH FOR SPILLS LIKE GREASE OR OIL, WE RECOMMEND USING ISOPROPANOL OR METHANOL AND THEN RINSING WITH WATER.

SOAPS, DETERGENTS AND OTHER CLEANERS THAT CONTAIN SURFACTANTS AFFECT THE SURFACE TENSION OF THE ORGANIC EXTRACTANT AND PREVENT PHASE SEPARATION IN THE LIQUID EXTRACTION UNIT. IF THESE SUBSTANCES ARE USED FOR CLEANING, DO NOT ALLOW THEM TO COME INTO CONTACT WITH PROCESS FLUIDS OR CONTAMINATE THEIR TANKS. ALWAYS RINSE THOROUGHLY WITH WATER.

Do not step on plastic pipes and do not use them as "handles" when lifting.

Do not apply excessive force when opening and closing the ball valves.

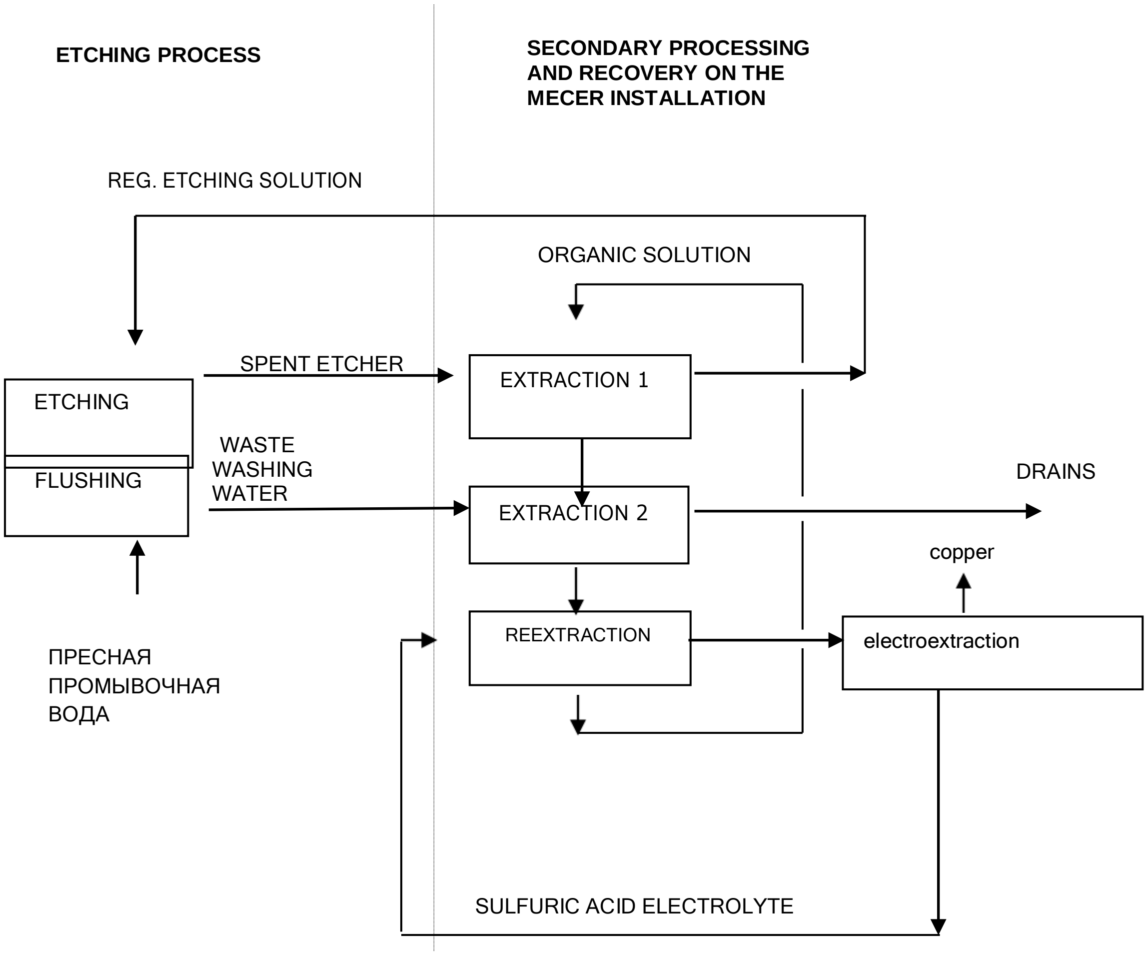
2 How does the MECER installation work ™

This section contains the commissioning of the MECER installation. It includes an explanation of the process known as liquid extraction. The following topics are considered:

- Overview
- What is liquid extraction?
- Operation of the blocks on the MECER installation
- Chemical reactions

2.1 OVERVIEW

A simplified diagram of the technological process of secondary processing and restoration at the Maker installation together with the etching installation is shown in Figure 2-1. (For a more detailed overview of the process at the Mecer installation, see the scheme of pipe strapping and instrumentation).



Img 2-1 Flow diagram of the technological process at the Mecer installation

As shown, two primary chemical operations are used for the process at the Mecer plant to recover copper from the contours of the etching chemical solution:

Liquid extraction (LGE), in which copper is removed from the spent pickling solution and washing water and transferred to an acidic electrolyte.

Electroextraction (EE), in which copper is removed from the acidic electrolyte to obtain high-quality metallic copper.

The method of electroextraction is well known in electroplating production. Liquid extraction, however, is not so well known. Therefore, before we discuss the work on the MECER installation, let's look at the liquid extraction process.

2.2 WHAT IS LIQUID EXTRACTION?

Liquid extraction (LGE) is a process that, if necessary, can be used for selective transfer (or "extraction") of a component from one solution to another. In order for the liquid extraction process to take place, the solution containing the component and the solution into which the component will be extracted should not be amenable to mixing, that is, the two solutions do not mix and remain separated into phases.

During the metal extraction process at the MECER plant, the target component is metal ions or a metal-ion compound dissolved in an aqueous solution. (At the Mecer installation, the component is copper dissolved in an etching solution). The aqueous solution is mixed with an immiscible organic reagent containing an active extractant. The active extractant is a chemical compound that, like a magnet, attracts metal ions more strongly than an aqueous solution does. When two solutions are mixed, metal ions pass from an aqueous solution to an organic solution. The amount of passing metal ions is directly related to the amount of active extractant in the organic solution.

After the metal ions have entered the organic solution, how do they recover in a form suitable for operation? Typical liquid extraction operations involve a second stage of the process called "reextraction". At this stage, an organic reagent containing metal ions is mixed with an acidic solution that is unable to mix. The acid has an even stronger "attraction" of metal ions than the organically active extractant, and therefore metal ions are "re-extracted" from the organic reagent into acid, in which they can be extracted as pure metal.

Then, the combination of extraction and re-extraction effectively transfers metal ions from (1) the initial aqueous solution to (2) the organic solution (3) the acid solution.

In the extraction and re-extraction processes, two solutions are mixed at high speed, breaking them into tiny droplets and ensuring good contact between them.
Mixing creates a dispersion similar to shaking bottles of salad dressing.

When you stop shaking the bottle and let it stand for a few minutes, the dispersion divides the contents into two layers, or phases, a less dense or more "light" phase on top and a denser or more "heavy" phase on the bottom. In the case described above, its own weight is used for separation. Small and medium-sized Mecer installations use gravity, and large installations use centrifugal forces to increase the separation rate. In installations with a centrifuge, the organic and aqueous phases are mixed in a mixer and then separated when the centrifuge is reached. The liquid with the highest density, i.e. the aqueous solution, moves away to the wall, while the organic solution ends up in the middle of the extractor.

2.3 OPERATION OF THE BLOCKS ON THE MECER INSTALLATION

During the etching process, unprotected metallic copper deposited on the printed circuit board is dissolved in the etching bath. From a chemical point of view, metallic copper is oxidized to divalent copper or copper (II) ions. This happens in two stages:

Stage 1: Dissolution of electrodeposited copper

Cu2+ + Cu => 2 Cu+
(divalent copper ions) (metallic copper)
(monovalent copper ions)

At this stage of the process, the unprotected copper is dissolved on the printed circuit board, the accumulation of monovalent copper ions in the etching solution. Eventually, this formation slows down and the etching process would finally stop if there were no second stage.

Stage 2: Re-oxidation of monovalent copper

2 Cu+ + ½ O + 2H+ => 2 Cu+ HO
(monovalent copper ions) (divalent copper ions)

This stage includes processes in the oxidation reactor and the etching chamber. The step depends on the presence of oxygen in the etching solution of the re-oxidation of monovalent copper ions to divalent copper back, or ions (II) copper. Then, there will be more divalent copper ions to oxidize metallic copper in step 1, resulting in more monovalent copper ions. Further, the two-stage process forms a circular contour, therefore, when creating appropriate conditions, the process of etching ions it can continue.

Optimal etching conditions are achieved with a content of 180-200 g/l of copper in the etching solution. But as the etching continues, the etching solution becomes more and more saturated with copper. To maintain the copper concentration in the optimal range, it is necessary to regularly remove the copper-saturated (spent) pickling solution and replace it with hydrochloric acid and water.

When the MECER plant is integrated with the etching line, the spent etching solution is "regenerated" (excess copper is removed), and then returned to the etching plant. In addition, the unit regulates the HCl concentration and redox potential in the etching bath to maintain the bath in optimal etching conditions.
The integrated MECER unit also recovers copper from waste washing water. The pH value of the waste washing water is regulated during the extraction process by adding sodium hydroxide. Therefore, it is impossible to return the treated washing water to the washing cycle.

The integrated MAKER unit regenerates the spent pickling solution, recovers copper from the spent washing water and regulates the conditions for baths with pickling solution. How the installation copes with these tasks is described in the following sections. For ease of understanding, the installation operation is divided into three main functions:

1. **Integration with the etching process line** to obtain stable and highly efficient etching by controlling the addition of a regenerated pickling solution of hydrochloric acid, and washing water to the pickling chamber, as well as oxygen gas to the oxidation reactor.

2. **Liquid extraction (LJ)** to transfer excess copper from the spent pickling solution and washing water to the acid electrolyte.

3. **Electroextraction (EE)** to remove copper from an acidic electrolyte to obtain high-quality metallic copper.

The operation of the MECER installation is automatically controlled by a programmable logic controller (PLC). During normal operation, when the MECER unit is integrated with the etching process line, the liquid extraction unit it starts working whenever the spent pickling solution for regeneration is received, and the electroextraction unit starts working when the concentration of copper in the electrolyte exceeds the set value.

2.3.1 INTEGRATION WITH THE ETCHING PROCESS LINE

The integration of the MECER plant with the etching process line leads to some changes in the control and operation of the etching plant. These changes are important not only to maintain optimal etching conditions, but also to maintain optimal operating conditions of the MECER plant. Three important automatic etching control systems are described below:

2.3.1.1 Copper concentration in pickling solution and acidity

A device is installed in the MECER installation that continuously monitors the acidity in the pickling bath. The signals from this device are used to control the input of the regenerated pickling solution and synchronously a small amount of hydrochloric acid into the pickling bath. At the same time, it regulates the copper content and acidity to maintain an optimal level.

The control system also contains a density sensor to measure the long-term effect of the water balance on the composition of the pickling solution. If the density tends to increase the washing water, then it is automatically fed into the stock of the pickling solution to maintain the density at a constant level.

In addition, the control system contains a redox potential (ORP) device. This potential is associated with the content of monovalent copper ions in the etching solution. With an increase in the content, the ORP drops. In order to maintain the redox potential for a certain optimal interval, check valves are activated to control the entry of gaseous oxygen into the oxidation reactor.

All additions to the turnover of the etching solution of the combined etching and oxidation process line of the reactor leads to overflow of the spent etching solution to the connected pumping station for further transfer to the corresponding main intermediate tank.

2.3.1.2 *Concentration of sodium chlorate in the pickling solution*

The pickling solution has a certain sodium chloride content to maintain the pickling performance. This content is analyzed in accordance with the program of work with chemical analyses. Due to losses due to entrainment, this content will decrease very slowly. Based on the analytical values, some volumes of pure sodium chloride salt are added at regular intervals to the salt-dissolving station to restore this content.

2.3.1.3 *Contamination of the washing*

When transferring printed circuit boards from the etching plant to the washing stage, they transfer (or "take out") with them a certain amount of etching solution, polluting the washing water with copper. Deionized water and hydrochloric acid are added to the washing water module, which dilutes the circulating water in the water intake, reducing the total concentration of copper.

The drain of the flushing module enters the appropriate intermediate tank, and then goes to the liquid extraction unit of the Mecer installation for copper extraction.

2.3.2 LIQUID EXTRACTION AT THE MECER PLANT

Liquid extraction is carried out in five mixers/settling tanks, while the first and second blocks are used to extract copper from the spent pickling solution, the third block is used to re-extract copper from the spent washing water, and the fourth and fifth blocks are used to distill copper from the organic solution to the sulfuric acid electrolyte. Each mixer/sump consists of one part called a mixer and one part called a sump. Extraction and re-extraction takes place in the mixer part, from where the dispersed mixture enters the sump part, where it is divided into two phases.

2.3.2.1 *The first mixer-sump and the second mixer- sump*

The spent pickling solution is transferred from the etching plant to an intermediate tank for the spent pickling solution, from where it is pumped to the second mixing/settling unit. After the pickling solution passes through the second mixing/settling unit, it is pumped into the first mixing/settling unit. The MX 90 reagent, which is an organic extractant, is fed from the storage tank into the first mixing/settling unit, from where it is poured into the second mixing/settling unit. This configuration maximizes the extraction of copper from the etching solution. The etching solution that has passed the first and second mixing/settling blocks is called a regenerated etching solution. It contains approximately 20 g/l less copper than the spent pickling solution.

The MX 90 reagent saturated with copper flows into the mixing chamber of the third mixing/settling unit.

2.3.2.2 *Third mixer/sump*

The spent flushing water is transferred from the flushing module of the etching plant to an intermediate tank for spent flushing water, from where it is pumped into a pre-mixing tank, in which its pH is regulated by adding sodium hydroxide.
The waste washing water coming from the intermediate tank is also diluted with washing water that has already passed the third mixing/settling unit. This configuration is necessary to achieve a good ratio between the amount of organic phase and the amount of aqueous phase in the third mixer/sump. The washing water is pumped from the pre-mixing tank into the mixer of the third mixer/settling tank, where it is mixed with the MX 90 reagent from the second mixing/settling unit. Copper is extracted or transferred to the organic phase.

The dispersed mixture enters the part of the sump, where it is divided into two phases. The washing water flowing from the third mixing/settling unit, and which depends on the level in the pre-mixing tank, or flows back into the pre-mixing tank or into another container, from where it is sent for wastewater treatment.
The MX 90 copper-saturated reagent flows into the fourth extraction unit for reextraction.

2.3.2.3 *Fourth mixer/sump*

In the fourth mixing chamber, the MX 90 copper-saturated reagent is mixed with a sulfuric acid electrolyte pumped from the electroextraction unit (EE). As described in Section 2.2, "What is liquid extraction?", copper is attracted by acid and flows from an organic solution to the electrolyte.

The dispersed mixture enters the part of the sump, where it is divided into two phases. The MX 90 reagent, already significantly freed from copper, flows into the fifth mixer. The acid electrolyte enriched with copper flows by gravity into the transfer tank, and then is transferred to the circulation tank of the electroextraction unit to extract copper in the form of metallic copper.

2.3.2.4 *Fifth mixer/sump*

In the fifth mixing chamber, the MX 90 reagent is again mixed with the sulfuric acid electrolyte pumped from the electroextraction unit (EE). Copper is attracted by acid and transferred from the organic solution to the electrolyte.
The dispersed mixture enters the part of the sump, where it is divided into two phases. The MX 90 reagent with an already significantly reduced copper concentration content flows by gravity into the storage tank, from where it is pumped into the first mixing/settling unit. The acid electrolyte enriched with copper flows by gravity into the transfer tank, and then is transferred to the circulation tank of the electroextraction unit to extract copper in the form of metallic copper.

2.3.3 ELECTRICAL EXTRACTION AT THE MECER INSTALLATION

The sulfuric acid electrolyte circulates to one or more electrolyzers. The circulation pump minimizes the risk of density gradients in the sulfuric acid electrolyte. The DC voltage supplied from the rectifier through accelerating and cathode electrodes placed in an acidic solution ensures the deposition of copper in solution in the form of pure metal on the cathodes.
The flow of current through the electrolytic cells is controlled to maintain the concentration of copper in the electrolyte within a certain range.

As in the etching plant, the concentration of copper affects the density of the electrolyte: the more copper in the electrolyte, the heavier or denser it becomes. Therefore, a density reading sensor is installed on the electroextraction unit. As the concentration of copper in the electrolyte increases, its density increases accordingly.

When the density in the electrolyte exceeds the set value, the rectifier adjusts the rate to a higher current strength, which leads to a higher rate of copper deposition. When the density decreases below the set value, the rectifier returns to a weak current sufficient to prevent reverse coating.

2.4 CHEMICAL REACTIONS

Etching and chemical processes at the Maker installation are reflected in the following equations:

A. **Etching of metallic copper from printed circuit boards:**

$\mathrm{Cu}^{0} + \mathrm{Cu}^{2+} \rightarrow 2\,\mathrm{Cu}^{+}$ (dissolution)

$2\,\mathrm{Cu+} + \frac{1}{2}\,\mathrm{O2} + 2\,\mathrm{H+} \rightarrow 2\,\mathrm{Cu2+} + \mathrm{H2O}$ (oxidation)

These two equations can be reduced to one equation:

$\mathrm{Cu}^{0} + \mathrm{Cu}^{2+} + \frac{1}{2}\,\mathrm{O}_2 + 2\,\mathrm{H}^{+} \rightarrow 2\,\mathrm{Cu}^{2+} + \mathrm{H}_2\mathrm{O}$

B. **Extraction** copper from spent pickling solution or spent washing water:

$\mathrm{Cu}^{2+} + 2\,\mathrm{RH}_{(opr)} \rightarrow \mathrm{CuR}_{2(opr)} + 2\,\mathrm{H}^{+}$

C. **Reextraction** copper from organic solution to electrolyte:

$\mathrm{CuR}_{2(opr)} + \mathrm{H2SO4} \Rightarrow \mathrm{CuSO}_4 + 2\,\mathrm{RH}_{(opr)}$

D. **Electroextraction** metallic copper from electrolyte:

$\mathrm{Cu}^{2+} + 2\,e^{-} \Rightarrow \mathrm{Cu}^{o}$ (на катодах)

$\mathrm{H}_2\mathrm{O} \Rightarrow 2\,\mathrm{H}^{+} + \frac{1}{2}\,\mathrm{O}_2 + 2\,e^{-}$ (на анодах)

3 Installation Description MECER

This section presents the components of the MECER installation. In most cases, a graphic describing the components is included, as well as an explanation of how the block works. The following topics are considered:

- Overview
- Liquid Extraction Unit
- Electrical extraction unit
- Intermediate tanks
- Integration of the MECER installation and the etching installation
- Process control system

3.1 OVERVIEW

The MECER installation in working condition contains the following blocks:

- Liquid extraction unit with five mixing/settling units. Each mixing/settling unit contains one or more combined tanks, which are used as a storage tank for the chemical medium of organic extraction (reagent MX 90), a pre-mixing tank, transfer tanks for solutions that have passed the mixing/settling units, etc.
- Electrical extraction unit (EE) with one or more electrolyzer, one circulation tank and one rectifier. (The number of electrolytic cells depends on the capacity of the installation)
- Three intermediate tanks for collecting spent pickling solution, regenerated pickling solution and spent washing water.
- Equipment for the integration of the MECER installation with the etching process line.
- A process control system consisting of a programmable logic controller (PLC), an operator interface panel, and various sensors and instrumentation (for example, control valves, level sensors, conductivity sensors, etc.).

A more detailed description of the equipment is given in the following sections.

3.2 LIQUID EXTRACTION UNIT

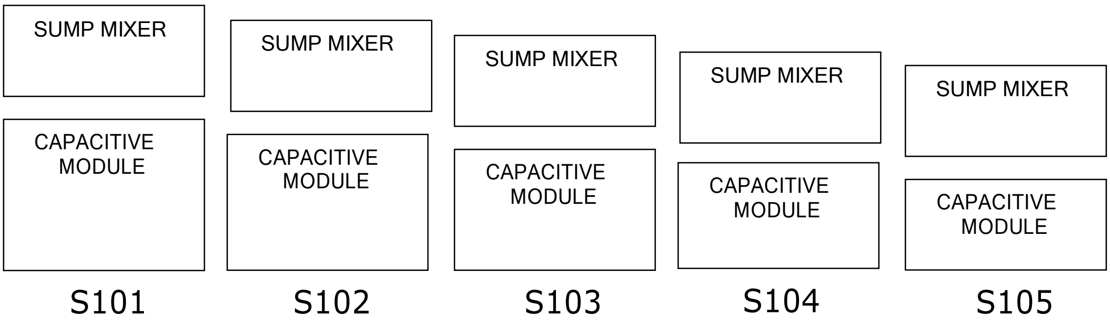


Figure 3-1. Liquid extraction unit

The liquid extraction unit contains the following equipment (see Figure 3-1):

- Five liquid extraction units (S101, S102, S103, S104 and S105) for extraction of copper from spent pickling solution, spent washing water and copper re-extraction from organic extractant.
    - Each liquid extraction unit includes a capacitive module and a mixing-settling module. One capacitive module is used as a storage tank for organic extractant. Other capacitive modules are used as intermediate tanks and transfer tanks.
      Transfer tanks are used to transfer regenerated pickling solution, waste washing water and saturated acid from the liquid extraction unit to their destination. These tanks can also be used as settling tanks described in section 3.2.2.

3.2.1 SUMP MIXER (S101, S102, S103, S104, S105)

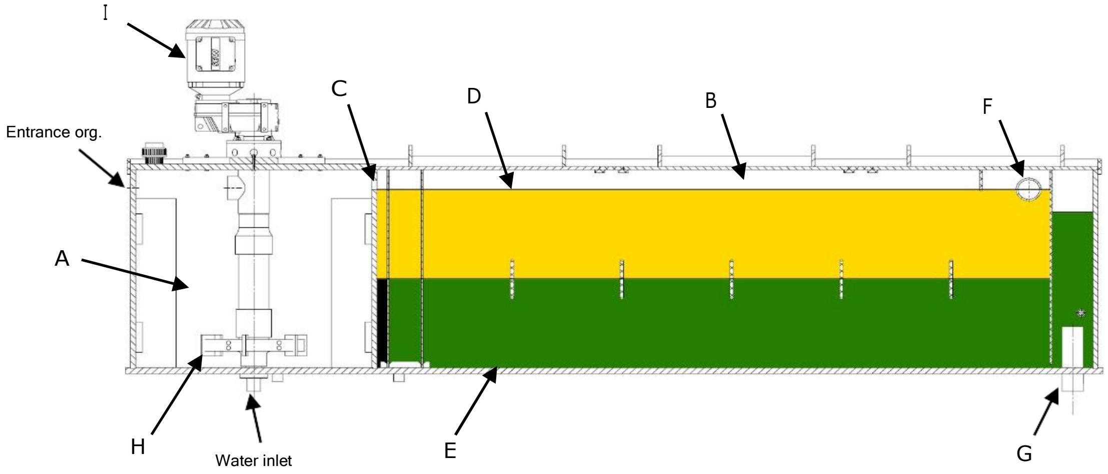


Figure 3-2. Sump mixer

Five settling mixers (see Figure 3-2) are part of five extraction modules (S101, S102, S103, S104 and S105).
Aqueous and organic solutions enter the mixing chamber (A), where they are mixed at a constant rate. The mixture will be fed through the gap (C) in the sedimentation chamber (B).

In the sedimentary chamber, the mixture separates, forming two phases. The upper phase (D) is a solution with a lower density (organic), and the lower phase (E) is a solution with a higher density (aqueous). The organic phase flows out through the bypass pipe (F). The water phase flows out through the hole in the bottom and through the pipe (G).

In the center of the mixing chamber there is an agitator PVV (H).
The mixer is driven by a motor with an adjustable speed (I).

3.2.2 SETTLING TANKS

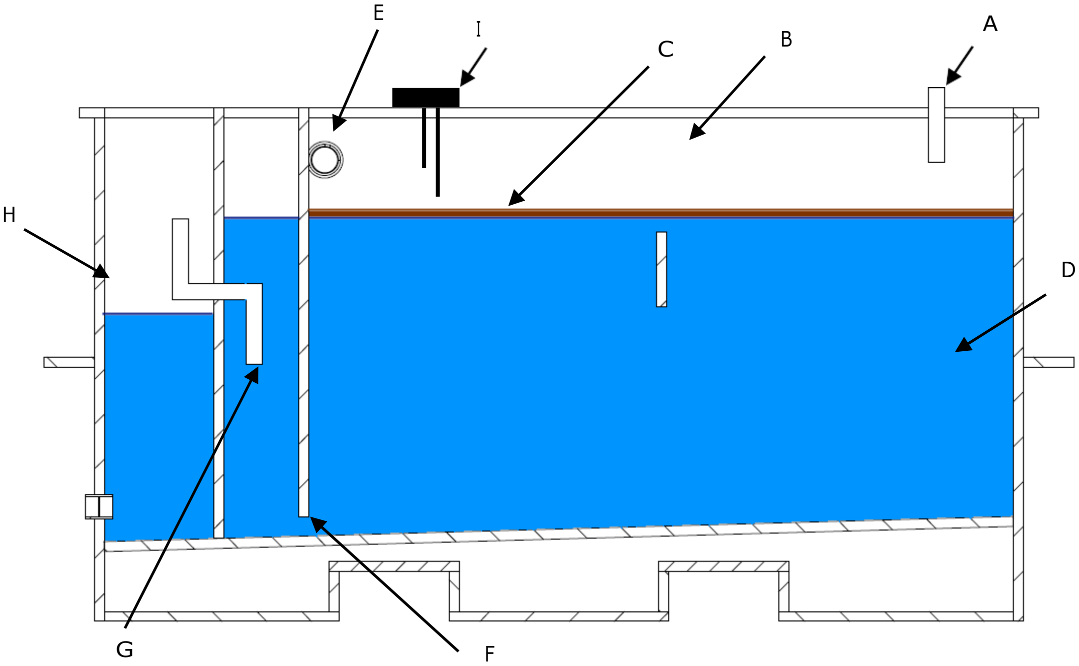


Figure 3-3. Sump tank

A typical layout of the transfer tank included in the capacitive modules is shown in Figure 3.3. Aqueous solutions from the sump mixer are fed through the pipe (A) into the sedimentation chamber (B). During the start-up mode of the liquid extraction units, the aqueous solution contains a small amount of organic solution.

In the sedimentary chamber, the mixture separates (precipitates), forming two phases. The upper phase (C) is a solution with a lower density (organic), and the lower phase (D) is a solution with a higher density (aqueous). The organic phase flows out of the tank through a horizontal pipe (E). The water phase flows under the partition (F) and flows through the pipe (G) into the water transfer tank (H).

Conductivity sensors (I) are installed in the sedimentary chamber. To maintain the consistency of the organic phase at a minimum level, a predetermined technological sequence called drainage is automatically started at the installation with a certain interval during which most of the organic phase flows through the pipe (E) back into the storage tank of the organic phase.

3.2.3 ORGANIC PHASE STORAGE TANK (T105)

The MX 90 organic reagent is contained in a tank built into one of the capacitive modules. The MX90 reagent is pumped through a filter to the first liquid extraction unit (the SAME unit), from where it flows into the second, third, fourth and fifth RE unit before it returns back to the storage tank. A small pump (P 105B) connected to the lowest point of the inclined bottom of the tank removes any kind of liquid that can be fed into the organic solution and returns it back to the S104.

A level sensor and a conductivity sensor are installed inside the storage tank.

3.2.4 TRANSFER TANKS

The regenerated pickling solution flows from the extraction unit S101 into the transfer tank T106. The waste washing water flows from the extraction unit S103 into the transfer tank T107. The saturated acid from the extraction unit S104 flows into the transfer tank T111, and the saturated acid from the extraction unit S105 flows into the transfer tank T112. Pumps connected to each transfer tank pump liquids to the appropriate destination. Pipe fittings are installed at the inlet and outlet of each pump, which allows easy disassembly of the pump for maintenance purposes.

Each transfer tank is equipped with level sensors and conductivity sensors, which are used to control the operation of pumps installed on transfer tanks.

3.3 ELECTRICAL EXTRACTION UNIT

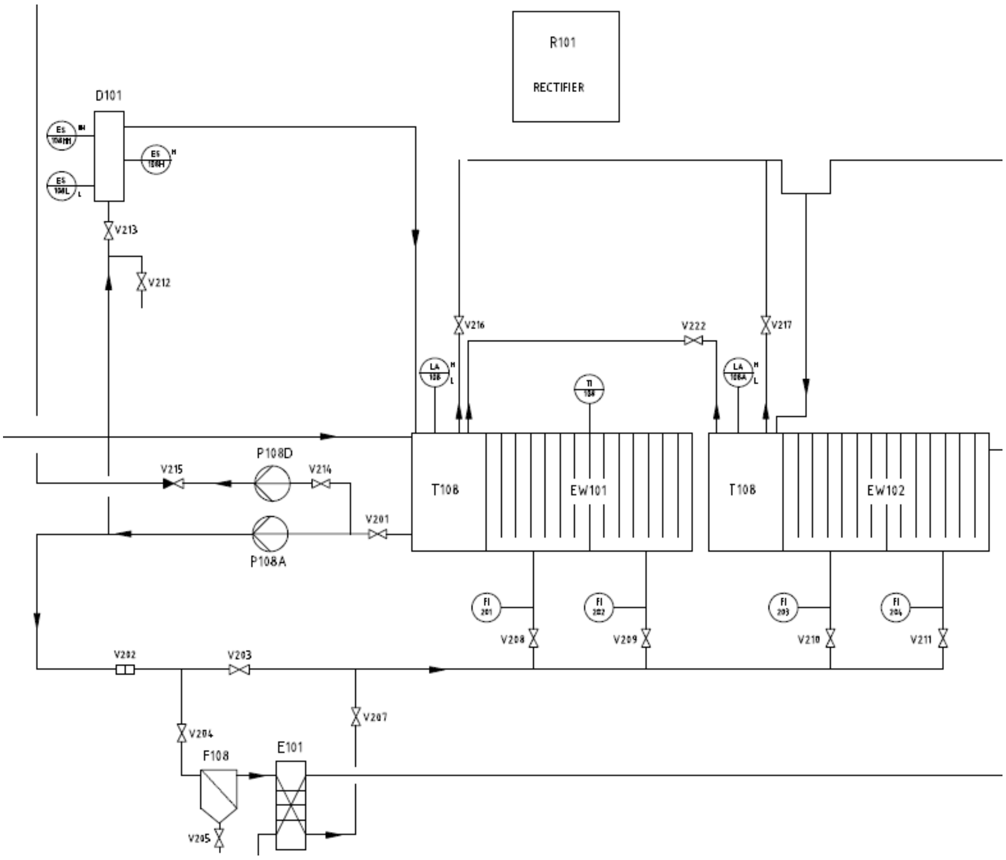


Figure 3-4. Electroextraction unit (sample of the type)

The electroextraction unit consists of the following equipment:

- One or more electrolyzers (EW101). Each electrolyzer consists of:
    - PCB tank divided into two or more:
    - Circulation tank for electrolyte (T108)
- Rectifier (R101)
- Density control unit
- Heat exchanger (depends on capacity)

3.3.1 CIRCULATION CAPACITY/ELECTROLYZERS

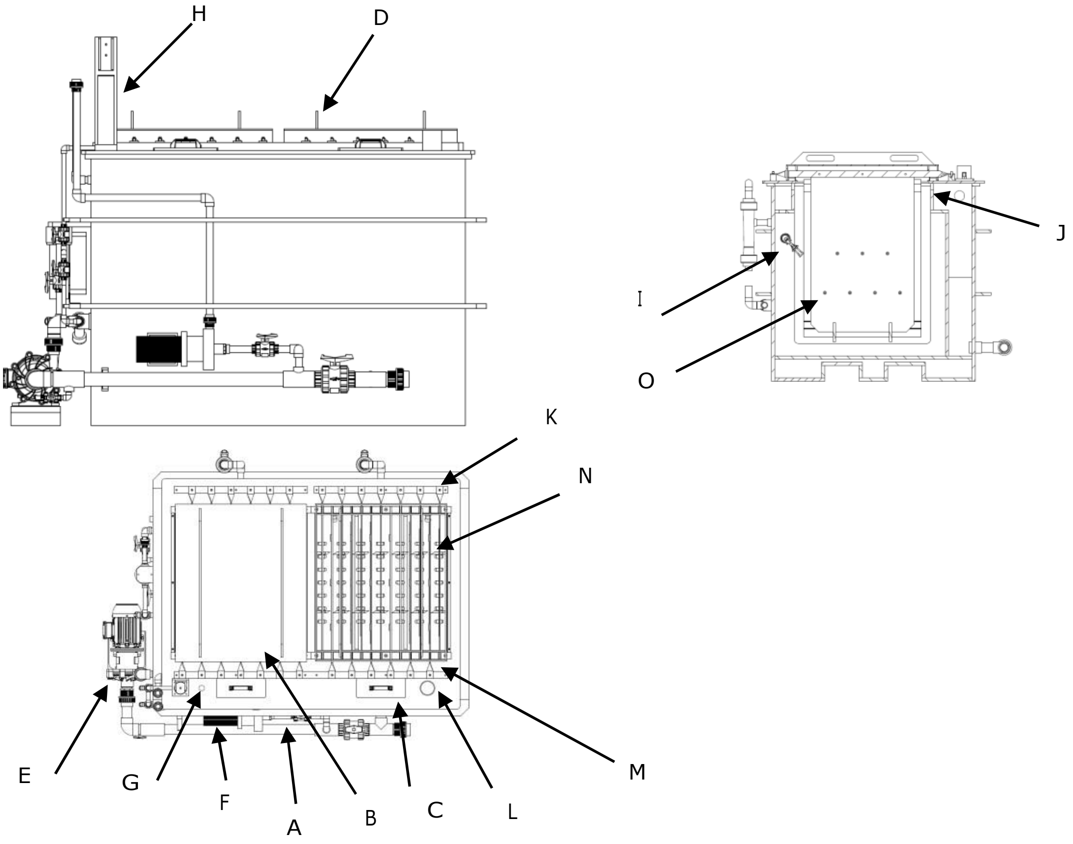


Figure 3-5. Electrolysis tank (sample type)

The electrolysis tank is divided into two or more compartments: the circulating tank (A) of the electrolyte and one or more electrolyzers (B). The circulating tank has an inspection hatch (C) and covers each electrolyzer (D).

The circulation tank has one centrifugal pump (E), which pumps the electrolyte solution in a closed system through electrolyzers and one or more centrifugal pumps (F), which provide the housing unit with electrolyte. The density control unit (H), level alarms and ventilation connection (L) are also installed on the top of the tank.

The circulation tank has one centrifugal pump (E), which pumps the electrolyte solution through a closed system through the electrolyte enters the electrolyzer through ejectors located along the electrolytic cell (I). The overflow threshold in each electrolytic cell (J) allows continuous return of the electrolyte to the circulation tank.

Each electrolyzer is equipped with 7 stable-sized anodes (K) and 6 copper cathodes (M). The anodes are bolted to copper strips that run across the entire width of the electrolytic cell. The strips are bolted at right angles to the copper busbar (N). Sheets of copper cathodes are bolted to copper strips running across the entire width of the electrolytic cell and located between the anode plates. The cathode strips are bolted to the second bus.

In order to avoid contact between the cathodes and the anodes, which leads to a short circuit, a plastic coupling (O) is put on each anode. The couplings ensure a minimum distance between the electrodes.

3.3.2 RECTIFIER

When the MECER unit is operating in automatic mode, the rectifier operates on two current loads: a rated load for copper deposition, and a "grounded" load to prevent the cathode from dissolving. As soon as the signals from the density control unit (high or low concentration of copper in the electrolyte) are received, the PLC will switch between high and low load on the rectifier.

The description of the rectifier supplied with the MECER installation is contained in the Appendix.

3.3.3 DENSITY CONTROL UNIT

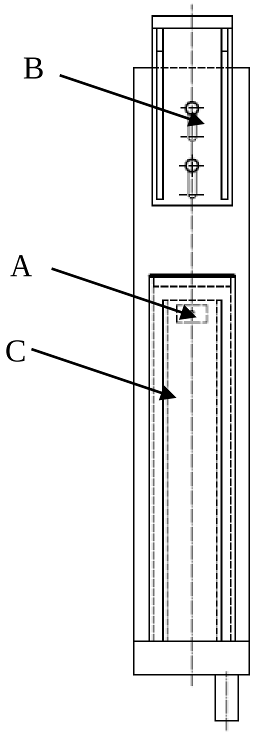


Figure 3-6. Density control unit

The concentration of copper in the electrolyte circulating in the electroextraction unit is monitored and controlled by the density control unit.

A small flow of electrolyte deviates from the outlet of the electrolyte circulation pump to the unit. The electrolyte passes through the valve into the chamber (A) equipped with a hydrometer. As the concentration of copper in the electrolyte increases, the density increases accordingly, resulting in an increase in the value of the hydrometer. Three inductive level sensors (B) detect the location of the hydrometer and send signals to the PLC. The signals are used to control the current strength from the rectifier and, if necessary, to turn off the liquid extraction unit. The settings of the three sensors correspond to approximate copper concentrations of 20, 25 and 32 g/l.

The electrolyte flows through the upper part of the chamber into the outer chamber (C) and returns to the circulation tank.

3.4 INTERMEDIATE TANKS

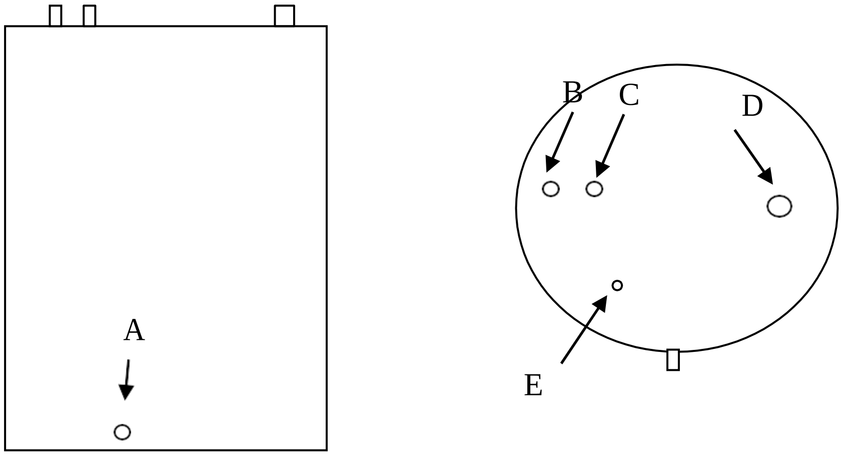


Figure 3-7. Standard intermediate tank

The etching process line will not always work at the same time as the MECER installation. Therefore, there is a need for intermediate tanks for storing spent pickling solution (T 101), regenerated pickling solution (T102) and spent washing water (T103). Intermediate tanks can be supplied by Sigma or the customer. This section and Figure 3-7 describe an example of common intermediate tank components.

The transfer of liquid from the intermediate tank is carried out using one or more pumps connected to one or more outlets at the bottom of the tank (A). The tanks are equipped with centrifugal pumps and flow control devices. If the liquid level in the intermediate tank has the potential to exceed the liquid level in the tank into which the liquid is being pumped, an exhaust pipe is installed on the side of the pump outlet to the upper part of the intermediate tank (B).

The upper part of each tank supports one or more liquid inlets (C), a ventilation duct (D) and a level sensor connection (E).

3.5 INTEGRATION OF THE MECER INSTALLATION WITH THE ETCHING INSTALLATION

The integration of the MAKER installation with the etching installation requires the presence of instrumentation and equipment for monitoring etching conditions in the etching process line. The necessary equipment is classified as follows:

- The etching solution control unit, which uses its own tools or signals from external devices to monitor and control the status of the etching plant, pH, redox potential, copper concentration in the etching solution at the etching plant, as well as control over the addition of gaseous oxygen, hydrochloric acid, washing water and regenerated etching solution at the etching plant.

3.5.1 ETCHING SOLUTION CONTROL UNIT

The etching solution control unit monitors and controls the following:

- The pH value of the pickling solution, which sends a signal to the PLC to start and stop the feeding system, which provides the supply of regenerated pickling solution and hydrochloric acid to the etching plant.

- The density of the pickling solution, which sends a signal to the PLC to start and stop the supply system, which provides the supply of flushing water to the etching plant.

- Status of the etching plant to determine whether reagents need to be added.

Depending on the layout of the etching plant and the MAKER installation, a pumping station may be required to transfer the spent etching solution and spent flushing water to intermediate tanks.

3.5.1.1 Control of copper concentration

The concentration of copper in the etching solution is maintained at the desired level by adding a regenerated pickling solution of hydrochloric acid to the etching unit at a signal from the pH indicator. The supply of regenerated pickling solution and hydrochloric acid to the etching plant will be carried out until the indicator pH will not drop below the setpoint.

3.5.1.2 Density control

The installation uses signals from the density control unit or from an external device to control the input of flushing water to the etching module. When the high-density sensor is activated, the supply of flushing water to the etching module is activated.
The feed stops when the high-density sensor is deactivated.

3.5.1.3 Control of the redox value

During the etching process, the redox value of the etching solution will gradually decrease due to an increase in Cu^+. Too high a concentration of Cu^+ ions will have a negative effect on the etching rate.
The signal from the redox value is used to control the addition of gaseous oxygen to the oxidation reactor, in which Cu^+ is oxidized to Cu^{2+}. The supply of gaseous oxygen to the oxidation reactor remains active until the measured redox value exceeds the required setpoint.

3.5.1.4 Status of the etching unit

In order to reduce the risk of accidents during service and maintenance at the etching plant, the addition of reagents to the etching plant is allowed only when the etching plant is operating normally.

4 Process control system

Most of the parameters for installing MECER can be set from the control panel. The control panel is also used to handle the alarm. The manual for the process control system can be found in the appendix.

5 Commissioning

This section includes information on the safety and commissioning of the Mecer ® installation. The following topics will be considered:

- Source materials
- Commissioning

5.1 SOURCE MATERIALS

To prepare for the launch of the MECER installation, the following materials are required:

MX90 reagent (launcher) - provided by Sigma. (supplied with the MECER installation).

MX90 reagent (additional agent) - provided by Sigma. (supplied with the MECER installation).

Sulfuric acid - provided by the customer. It is used to compose the electrolyte in the electroextraction unit.

Deionized water - provided by the customer. It is used to compose the electrolyte in the electroextraction unit.

The spent pickling solution is provided by the customer. The intermediate tank of the spent pickling solution must be filled before the start-up.

Waste washing water - provided by the customer. The intermediate tank of the spent washing water must be filled before the start-up.

The customer must also provide the equipment necessary to transfer these materials to the appropriate containers (for example, rotary pumps, hoses, etc.).

5.2 COMMISSIONING

Commissioning will be carried out under the supervision of a Mercer representative and will include the following:

- Assembly or assembly control (depending on the scope of delivery)
- Pressure testing and leakproofness test
- Mechanical drive testing
- Checking the operation of electrical equipment
- Final cleaning of the equipment
- Filling the installation with the source material
- Operation and configuration of the unit LE
- Эксплуатация и настройка блока ЕЕ
- Operation and adjustment of the oxidation unit
- Operation and setup of the etching process line

In order to facilitate the launch process, the customer must ensure that:

1. The power supply to the installation is provided,
2. The ventilation system is working,
3. Raw materials are available and
4. The person or persons responsible for the operation of the installation are present during the start-up process.

6 OPERATION AND MAINTENANCE

This section contains information on the automatic operation of the MECER installation using instrumentation.

This section also contains recommendations for routine maintenance to ensure the effective operation of the MECER installation.

Operation and maintenance includes:

Operation of the installation

Selective method of installation control and chemical analysis

Installation maintenance

6.1 OPERATION OF THE INSTALLATION

The process control system is designed for automatic operation of the MECER installation units. The control system also partially controls the etching operations. The information on the operation of the installation given in this section contains most of the functions involved.

The process control system works as follows:

6.1.1 MAIN CONTROL PANEL

note

Disabling the MACER installation will stop the supply of regenerated pickling solution, hydrochloric acid and oxygen gas to the etching process line.

The START button "START" - on the touch screen of the display starts the housing unit. Accordingly, the stop button "STOP" - disables the housing unit. Automatic restart is performed after the "normal" shutdown of the RE unit (for example, reloading the tank with spent pickling solution, high Cu content in the electrolyzer).

If problems occur (for example, the tank level alarm or motor current protection is triggered), some critical equipment may be switched off automatically and finally, the relevant parts of the installation will be turned off. Such "abnormal" events initiate the triggering of an emergency alarm and an emergency sound signal, as well as the issuance of a fault identification message on the operator interface panel:

> The alarm will be sounded during the entire duration of the malfunction. After the fault is removed, the alarm is turned off.
>
> The alarm sound will continue to sound until the RESET button on the touch screen is pressed. If it is impossible to remove the fault, the sound signal will not be restarted.
>
> The error message will remain on the operator interface panel until the alarm sounds, and until it is disabled by pressing the RESET button on the touch screen.

After identifying the fault, refer to Section 7, "Troubleshooting" to determine how to fix the problem.

6.1.2 LIQUID EXTRACTION UNIT

When the "START" button is pressed, the electric motors installed on the sump mixer (S101, S102, S103, S104 and S105) and the feed pumps for spent pickling solution, spent washing water, acid and organic solution will be started automatically as soon as certain conditions are met (see Section 6.1.2.1 "Operating conditions for the housing unit"). Electric motors and pumps are switched on immediately. Thus, the liquid extraction unit becomes fully operational and begins to produce a regenerated pickling solution and an acid electrolyte saturated with copper.

A frequency-controlled converter regulates the speed of the mixers. The speed of the mixer will be set during start-up and should not be changed unless specifically notified by Sigma.

The regenerated etching solution of the extraction unit S101 comes from the extraction unit through tank T106, the treated washing water of the extraction unit S103 comes from the extraction unit through tank T107, the copper-saturated electrolyte of the extraction unit S104 comes from the extraction unit through tank T111 and the copper-saturated electrolyte of the extraction unit S105 comes from the extraction unit through tank T112.

6.1.2.1 Operating conditions of the unit LE.

The following conditions must be met before the block starts working LE:

1. The concentration of copper electrolyte in the electroextraction unit should be at the level of the upper limit of the alarm «HIGH ALARM»,

2. The tank with the spent pickling solution (T101) should not be empty «EMPTY»,

3. The intermediate tank with regenerated pickling solution (T102) should not be full «FULL»,

4. The transfer tank with waste washing water (T107) should not be full "FULL"

5. The acid tank in the electroextraction unit (T108) must be neither full "FULL" nor empty «EMPTY»,
6. The transfer tank with regenerated pickling solution (T106) should not be full «FULL»,
7. The transfer tank with the spent washing water (T107) should not be full «FULL»,
8. The saturated acid transfer tank (T111) must not be full «FULL»,
9. The saturated acid transfer tank (T112) must not be full «FULL»,
10. The storage tank (T105) with the MX 90 reagent must be neither full "FULL" nor empty «EMPTY»,
11. The storage tank with NaOH (T113) should not be empty «EMPTY»,
12. The electrolyte circulation pump (P108A) should work,
13. All current protection devices must not be switched off,
14. The conductivity sensor in the tank T105 should not react to high water levels.

6.1.2.2 Reasons for disabling the block LE

The shutdown conditions can be "normal" or "abnormal". "Abnormal" events will trigger an alarm on the control panel and an audible alarm, as well as an error message on the operator interface panel. The shutdown conditions, each of which will cause the housing unit to stop, are as follows:

Stop caused by normal events:

1. The tank with the spent pickling solution (T101) is empty «EMPTY»,
2. The button is pressed «STOP».

Stop caused by abnormal events:

The concentration of copper in the electrolyte of the EE unit is at the upper limit of the alarm «HIGH ALARM».

1. The intermediate tank with regenerated pickling solution (T102) is filled «FULL»,
2. The tank with waste washing water (T103) is empty «EMPTY».

3. The transfer tank with regenerated pickling solution (T106) is filled «FULL»,

4. The transfer tank with regenerated washing water (T107) is full «FULL»,

5. The transfer tank with saturated acid (T111) is full «FULL»,

6. The transfer tank with saturated acid (T112) is full «FULL»,

   The tank (T105) with the MX 90 reagent must be neither full "FULL" nor empty «EMPTY»,

7. Storage tank with NaOH (T113) empty «EMPTY»,

8. The electrolyte circulation pump (P108A) must be stopped,

9. All protective means of electric motors of the pump or mixer must be activated.

10. All conductivity sensors in mixing/settling units must respond to high conductivity.

6.1.2.3 *Re-extraction from the organic phase*

The purpose of reextraction from the organic phase is to purify the organic solution from copper and pollutants in order to maintain it in an active state and ensure optimal performance.
Sigma provides that the re-extraction program from the organic phase should be carried out at least twice a month or if:

- The copper content in Part A of the organic phase industrial test test is higher than 3 g/l.
- Before stopping the installation for a period of more than 3 days.

note

The more often the reextraction is performed, the better.

During the re-extraction cycle from the organic phase, the unit operates in a special re-extraction mode without dosing the spent pickling solution or spent washing water. Please note that the unit does not regenerate any pickling solution and does not process any washing water during this period.

6.1.3 ELECTRICAL EXTRACTION UNIT

The main switch representing the rectifier on the control panel turns the rectifier on and off. Please note that the P108A pump will work as long as it is not switched off manually from the control panel.

The acid pump can be stopped for maintenance with the circulation switch "CIRCULATION ON/OFF" (the normal position of the switch is "ON"). This is a virtual switch and it is located on the screen for the electrical extraction unit.

6.1.3.1 *Reasons for disabling EE*

The operation of the electric extraction unit is automatically stopped (i.e. the circulation pump and rectifier are switched off) as a result of one of the following malfunctions that trigger the alarm:

1. Disconnecting any motor protection device on the control panel.initiating the activation of the alarm:

2. Activation of the emergency stop button «STOP».

3. Turning off the circulation switch «CIRCULATION».

4. The acid tank in the electroextraction unit (T108) must be neither full "FULL" nor empty «EMPTY»,

note

In each case, there is an automatic shutdown of the housing unit LE.

6.1.3.2 *Rectifier operation*

The current strength from the rectifier is regulated automatically by signals from the EE density control unit. The principles of control are as follows:

1. The copper concentration is at a low "LOW" level. The grounding load is supplied with precautions to avoid dissolution of the cathode. This is a "normal" situation.

   The concentration of copper is at a high level of "HIGH". The rated load corresponding to the performance of the electrolyzers is supplied. This is a "normal" situation.

   The copper concentration is at the level of the upper limit of the "HIGH ALARM" alarm. The rated load is applied and the operation of the housing unit stops. In this case, the SAME block is automatically reset when the copper concentration returns to a high "HIGH" level. This is an "abnormal" situation.

note

The current from the rectifier can be supplied only when the circulation pump is running. Therefore, the function of the P108A pump is vital for the entire process.

6.1.4 INTEGRATION WITH THE ETCHING PROCESS LINE

Power is supplied to the control system equipment on the etching process line (for example, pH meter, pumps) using the main switch on the main panel of the Mecer installation.

The PLC controls the transfer of process fluids between the etching line and the Mecer installation. These automatic feeding systems are described below:

6.1.4.1 Regenerated pickling solution for the pickling plant

The signals from the pH meter installed on the etching unit are used to control the pump P102, which transmits the regenerated etching solution from the intermediate tank T102 to the etching unit. See additional information in Section 3.5.1, "Etching Solution Control Unit».

note

At start-up, the regenerated pickling solution pump will operate for the minimum number of seconds set when the device is put into operation. This is done to reduce the physical wear and tear caused by repeated starts and stops.

The pump P102 stops unconditionally when one of the following conditions occurs:

1. The pH value on the etching unit is below the required setpoint. This is a "normal" situation.

   The etching process line has been disconnected. This is a "normal" situation.

2. The intermediate tank with regenerated pickling solution (T102) is empty "EMPTY", This is an "abnormal" situation.

   The tank with the spent pickling solution (T101) or the transfer tank (T109) is filled "FULL". This is an "abnormal" situation.

3. Disconnecting any motor protection device on the control panel. This is an "abnormal" situation.

4. Activation of the emergency stop button "STOP". This is an "abnormal" situation.
   .

6.1.4.2 Supply of hydrochloric acid to the etching plant

Hydrochloric acid is pumped to the etching plant using a dosing pump. The dosing pump works simultaneously with the P102 pump. The required dosage is determined during installation and commissioning at the Mecer installation.
The dosing pump stops unconditionally when one of the following conditions occurs:

1. The etching unit has been disabled. This is a "normal" situation.

2. The intermediate tank with hydrochloric acid (T602) is empty "EMPTY", This is an "abnormal" situation.

3. Activation of the emergency stop button "STOP". This is an "abnormal" situation.

6.2 SELECTIVE METHOD OF PLANT CONTROL AND CHEMICAL ANALYSIS

To control the process, certain technological flows should be regularly analyzed in accordance with the program set out in Table 6-1. Simple procedures for each analysis are contained in the Sections 6.2.1 - 6.2.12.

Record the results in the log (see Figure 6-1 for an example). Keep the logbook together with a link in case of a problem situation.

Compare the results with the normal value given in Table 6-1. If the results of the analysis exceed the specified limits, refer to Section 7.6, "Chemical problems" for troubleshooting.

Table 6-1. Sample Analysis program

Sample	Sample from:	Analysis for:	Chapter	Normal value/limits	When? [1]
Used pickling solution	Etching plant	Copper	6.2.1	180 - 200 g/l	Daily
		Total chlorine	6.2.2	7,5 - 8,3 mol/l	Weekly
		Free acidity	6.2.3	0,02 - 0,05 mol/l	Daily
		Sodium Chloride	6.2.4	3,3 - 3,5 mol/l	Every month.
		Sulphate	6.2.9	< 0,2 mol/l	Every month.
Reg. etching solution	LE block	Copper	6.2.1	160 - 180 g/l	Daily
		Free acidity	6.2.3	0.5 - 0.65	Daily
Waste washing water	LE block	Copper	6.2.5	< 5 g/l	Daily
Waste washing water	LE block	pH		2 - 3	Daily
Acidic electrolyte	Electrolyzer	Copper [2]	6.2.6	20 - 28 g/l [2]	Weekly
		All sulfuric acid [2]	6.2.7	180 - 200 g/l [2]	Weekly
		Chlorine	6.2.8	< 200 mg/l	Weekly
MX 90	Organic phase storage tank T105	Organic phase performance	6.2.10	Часть A: 0 - 3 g/l Часть B: 10 - 13 g/l	In a month

[1] When the results of the analysis go beyond the established limits and operational problems arise, it will be necessary to conduct more frequent analyses to investigate the problem.

[2] Compare the results of the analysis of copper and sulfuric acid with formula 6-3. The result must be within the "allowed operating range»

Concentration of copper and sulfuric acid in the electrolyte

Calculated ratio:

The total amount of sulfuric acid should be in the range of 180 - 200 g/l. The components are "free acid" (to be determined) and "bound acid" (associated with $CuSO_4$).
The bound acid corresponds to 1.54 of the concentration of copper.

Example:
Free acid 140 g/l; copper 30 g/l.

140 + (1,54 x 30) = **186,2** g/l (OK. внутренний диапазон 180 – 200)

Formula 6-2. Acceptable range for electrolyte analysis

6.2.1 COPPER IN SPENT AND REGENERATED PICKLING SOLUTION

Reagents

0,100 g*mol EDTK
25%- ammonia solution (25% NH_4OH)
Murexide indicator

Equipment

Burette (10 ml. with 0.05 ml scale division)
Pipettes (1, 5 and 20 ml)
Erlenmeyer flask (300 ml)
Measuring cylinder (100 ml)
Measuring flask (100 and 250 ml)

1. Analytical procedure

Add 0.500 ml of the etching solution sample to a 250 ml Erlenmeyer flask and dilute with 100 ml of distilled water. Add 1 ml of 25% ammonia solution. After this addition, the color of the solution will change from bluish-green to dark blue. Before proceeding, make sure that the solution is light and transparent. Add the murexide indicator on the tip of the spoon. Titrate the EDTC solution until the color changes to dark purple. Measure the volume in ml of the EDTC solution used (X).

Result: Copper (g/l) = X x **12,709**

6.2.2 Total chloride in the spent and regenerated pickling solution

Reagents

0,1N silver nitrate
Concentrated nitric acid, 65 %
25% ammonia solution (25% NH_4OH)

Equipment

pH selective silver chloride electrode for the determination of chloride ions (Ag/AgCl)
Magnetic stirrer and stirrer anchor
Burette (orange glass, 10 ml. with 0.05 ml scale division)
Beaker (100 ml)
Measuring flask (250 ml)
Measuring cylinder (25 ml)
Pipettes (5 ml)

1. Sample preparation

Add 5 ml of pickling solution and 5 ml of 25% ammonia solution to a 250 ml measuring flask and fill to the mark with distilled water. Shake well.

2. Preparation of a solution of 10% nitric acid

Carefully add 15 ml of concentrated nitric acid, HNO_3, to 85 ml of distilled water.

3. Analytical procedure

Place the beaker with the magnetic stirrer anchor inside on the magnetic stirrer. Add 5 ml of the prepared sample solution and 20 ml of 10% nitric acid solution in 100 ml of deionized water. Start mixing and insert the chloride ion electrode into the solution. Count the potential (mV) on the pH meter.

Titrate with a solution of silver nitrate in 1 ml doses. Wait approximately 30 seconds between each dose until the solution is balanced. Write down and plot the mV readings opposite the accumulated added volume of silver nitrate in ml. Continue adding silver nitrate at a 1 ml dose until the potential change exceeds 10 mV per 1 ml. Reduce the dose amount to 0.1 ml and continue recording and plotting the results of the mV until you reach the point at which the change in potential levels ends after passing the interval of a steep change. (The potential distribution curve will continue to grow slowly from the beginning. This will be followed by a sharp upward climb followed by a gradual leveling).
Result: Use a ruler to determine the point on the titration curve whose tangent has the greatest slope.
Use this point, called the inflection point, and the corresponding amount of silver nitrate consumption in ml (X) in the following equations: Chlorine (g/l) = X x 35.45

Alternative to the method 6.2.2

DETERMINATION OF TOTAL CHLORINE IN SPENT AND REGENERATED PICKLING SOLUTION

Reagents

Silver Nitrate (0.1 N)
pH 4 acetate buffer
Dichlorofluorescein indicator
Sodium Acetate
Acetic acid

Equipment

Burette (10 ml, scale division min. 0.05 ml with orange glass)
Erlenmeyer flask (250, 500 ml)
Measuring flask (wedge flask 250 ml)
Measuring cylinder (10 ml)
Pipette (5 ml)

1. Sample preparation

Add 5 ml of pickling solution and approximately 5 ml of 25% ammonia solution (if necessary) to a wedge flask (250 ml) and fill with distilled water to the pointer.

SHAKE WELL

2. Preparation of pH 4 acetate buffer (500 ml)

- Add 450 ml of distilled water and stir with a magnetic stirrer anchor in a 500 ml Erlenmeyer flask.
- Add 11 g of sodium acetate.
- Stir until completely dissolved and read the pH reading.
- While stirring, bring the pH by adding acetic acid (approximately 50 ml) to pH 4.

3. Analytical procedure

- Place 5 ml of the prepared sample with a pipette into a 250 ml Erlenmeyer flask.
- Add approximately 100 ml of distilled water.
- Add approximately 10 ml pH 4 acetate buffer.
- Add 4-6 drops of the dichlorofluorescein indicator.
- Titrate 0.1 N silver nitrate until the color changes from green to pink.

4. Calculations

Each milliliter of 0.1 N $AgNO_3$ corresponds to 35.46 g/l **Cl**

6.2.3 FREE ACIDITY IN PICKLING SOLUTION

Reagents

0,100 g*mol NaOH

Equipment

Optional - automatic titration instruments with pH electrode and recording device Magnetic Stirrer and Stirrer Anchor Measuring Flask (250 ml)
Pipettes

1. Operating procedure

Pour 100 ml or the appropriate amount of distilled water into the Erlenmeyer flask. If it is necessary to analyze the etching solution, add 20.0 ml (V ml according to the formula below) of the etching solution sample. If it is necessary to analyze the etching solution, add 20.0 ml (V ml according to the formula below) of the etching solution sample. Start the mixing device and start the titration procedure by dosing a standard caustic soda solution. At the same time, enter the data of the electrode potential from the recording device in the log opposite the accumulated added volume of the titrating solution.

Initially, the electrode potential will react slowly while the reaction increases when the end point of titration is reached. After passing the mentioned point, the reaction will decrease and when constructing a potential response curve opposite the added titrating volume, the corresponding inflection point will be determined. This will give the desired volume of equivalence X ml.

The passage of the titration endpoint during the titration process can be easily observed, since copper hydroxide deposition will soon follow and the titrimetric procedure will be stopped.

The free acidity of the pickling solution is calculated as follows

$[H^+]$ = X x 0,1/V mol/l

6.2.4 SODIUM CHLORATE IN PICKLING SOLUTION

Reagents

Appropriate standard sodium chloride solution for calibration purposes
Appropriate acid dilution solution, e.g. 0.01 g*mol HNO_3 or 0.01g*mol HCl

Equipment

Atomic absorption or emission instrument

Sample preparation

The sample of the pickling solution is diluted with a given dilution coefficient depending on the parameters of the devices and the operating mode of the installation. When a given level of sodium concentration of the order of 2 mol/ l is reached, the actual dilution coefficient will be of the order of 103 to 104. Any of the above proposed solutions can be used as a diluting solution.

The order of work

The atomic absorption device can be started either in absorption or emission mode. In the first case, a sodium lamp generates a light beam with a wavelength characteristic of sodium. The beam passes through the combustion flame, into which a certain small sample stream is sprayed. In proportion to the content of sodium atoms, part of the brightness of the light beam will be absorbed. The intensity level of the light passing to the optical detector of the device gives a certain signal value associated with the absorption of light.

If the emission mode of operation is preferred, the solution sample is sprayed, as described above, into the combustion flame, resulting in the emission of characteristic sodium light with an intensity proportional to the sodium content. The light detector will give the appropriate signal.

The device is calibrated by spraying a standard solution with a known sodium chloride content. Optionally, these standards also contain approximately the same amount of copper chloride as the etching solution sample being analyzed. This is done in order to minimize any deviation of the quantitative determination during the analytical procedure, which may be caused by the content of copper chloride.

The concentration of sodium chloride of the sprayed sample is estimated from the calibration curve. By simply multiplying by the above dilution coefficient, the sodium chloride content in the etching solution sample is obtained.

6.2.5 OPPER IN WASTE WASHING WATER

Reagents

0,1 g*mol ЭДТК
25% ammonia solution (25% NH_4OH)
Murexide indicator (1 part of murexide in 100 parts NaCl)

Equipment

Burette (10 ml. with a division scale of 0.05 ml)
Pipettes (1 and 20 ml)
Erlenmeyer flask (300 ml)

1. Operating procedure

Add 1 ml of 25% ammonia solution to 100 ml of distilled water in an Erlenmeyer flask. Add 20 ml of waste washing water. Check that the solution is light blue and transparent. Add the murexide indicator on the tip of the spoon.
Titrate the EDTC solution until the color changes to dark purple. Measure the volume in ml of the EDTC solution used (X).

Result: Copper (g/l) = X x **0,3177**

6.2.6 COPPER IN ACIDIC ELECTROLYTE

Reagents

0,1 g*mol EDTC
25% ammonia solution (25% NH_4OH)
The murexid indicator

Equipment

Burette (10 ml. with a division scale of 0.05 ml)
Pipettes (1 and 2 ml)
Measuring cylinder (100 ml)
Erlenmeyer flask (300 ml)

1. Operating procedure

Add 2 ml of electrolyte to the Erlenmeyer flask and dilute with 100 ml of distilled water. Add 2 ml of 25% ammonia solution. Stir the solution and visually check that it is light and transparent without accidental precipitation. Add the murexide indicator on the tip of the spoon. Titrate the EDTC solution until the color changes to dark purple. Measure the volume in ml of the EDTC solution used (X).

Result: Copper (g/l) = X x **3,177**

6.2.7 SULFURIC ACID IN AN ACIDIC ELECTROLYTE

In the long term, the concentration of free sulfuric acid in the electrolyte will slowly decrease. Under normal operating conditions, it may be necessary to restore the concentration of free acid several times a year. The need for this is monitored once a week after the next analysis of copper and acid in the electrolyte.

Analysis of copper: [Cu] g/l
Analyses of free sulfuric acid: [H_2SO_4] g/l

The total parameter of sulfuric acid is calculated as follows:[H_2SO_4] + 1,54 x [Cu] g/l

As long as the total sulfuric acid index remains within the range of 180-200 g/l, no action is required. If the upper value of 200 g/l is exceeded, then deionized water must be added to the circulating capacity of the electrolyte. This situation should never occur unless too much sulfuric acid is added to the installation by mistake.

When the lower value is reached, a volume of concentrated sulfuric acid equal to 1% of the total volume of the electrolyte will be added to the installation. This will increase the total sulfuric acid parameter by about 20 g/l.

Reagents

1,00 N sodium hydroxide
Methyl orange indicator (0.1% in water)

Equipment

Burette (25 ml. with a division scale of 0.1 ml)
Pipettes (5 ml)
Erlenmeyer flask (300 ml)
Measuring cylinder (100 ml)

1. Analytical procedure

Add 5 ml of electrolyte to the Erlenmeyer flask and dilute with 100 ml of distilled water. Add 5 drops of methyl orange indicator and titrate with sodium hydroxide solution until the color changes from red to yellow-orange. Measure the volume in ml of sodium hydroxide used (X).

Result: Sulfuric acid (g/l) = X x **9,80**

6.2.8 CHLORINE IN ACIDIC ELECTROLYTE

Reagents

0,01N silver nitrate
Concentrated nitric acid, 65 %

Equipment

pH with selective silver chloride electrode for the determination of chloride ions (Ag/AgCl)
Magnetic stirrer and stirrer anchor
Burette (25 ml. with a division scale of 0.1 ml)
Beaker (200 ml)
Measuring cylinder (25 ml)
Pipettes (5 and 50 ml)

1. Preparation of a solution of 10% nitric acid

Carefully add 15 ml of concentrated nitric acid to 85 ml of distilled water.

2. Analytical procedure

Place the beaker with the magnetic stirrer anchor inside on the magnetic stirrer. Add 50 ml of acidic electrolyte and 20 ml of 10% nitric acid solution. Dilute 100 ml of distilled water. Start mixing and insert the chloride ion electrode into the solution. Count the potential (mV) on the pH meter.

Titrate with a solution of silver nitrate in 1 ml doses. Wait approximately 30 seconds between each dose until the solution is in balance. Write down and plot the mV readings opposite the accumulated added volume of silver nitrate in ml.
Continue adding silver nitrate at a 1 ml dose until the potential change exceeds 10 mV per 1 ml. Reduce the dose amount to 0.1 ml and continue recording and plotting the results of the mV until you reach the point at which the change in potential levels ends after passing the interval of a steep change. (The potential distribution curve will continue to grow slowly from the beginning. Then a sharp upward climb will follow, followed by a gradual leveling).

Result: Use the ruler to determine the point on the titration curve whose tangent has the greatest slope. Use this point, called the inflection point, and the corresponding amount of silver nitrate consumption in ml (X) in the following equations:

Chloride ions (mg/l) = X x **7,09**

6.2.9 SULFATES IN THE SPENT PICKLING SOLUTION

Reagents

Concentrated hydrochloric acid
Barium chloride solution (50 g $BaCl_2$ x $2H_2O$ per liter of deionized wate)
Silver Nitrate solution
Sodium chloride solution (200 g $NaClO_3$ per liter of deionized water)

Equipment

pH meter or indicator paper
Magnetic Stirrer/electric cooker and magnetic stirrer anchors
Thermometer
Chemical glasses (500 ml)
Measuring cylinder (100 ml)
Pipettes (50 ml)
filter funnel
Filter paper (watman No. 542)
Drying Oven
Balance

1. Sample preparation

Pour 50 ml of the spent pickling solution with a pipette into a 500 ml beaker. Add 100 ml of deionized water.
Stir. Add 5 ml of concentrated HCl. Check the redox potential (ORP) of the solution. If the ORP is below + 600 Mv, slowly add a solution of sodium chloride with constant stirring to the protection and ventilation conditions.
If the ORP is above + 600 mV, follow the procedure below.

2. Analytical procedure

Нагреть раствор до температуры кипения. Добавить 50 мл кипящего раствора хлористого бария и хорошо перемешать. Охладить до 90 - 80°C и поддерживать при этой температуре до тех пор, пока не образуется белый осадок. Проверить, чтобы произошло полное осаждение путем добавления горячего раствора хлористого бария в прозрачную надосадочную жидкость. Прекращение выпадение осадка означает завершение реакции. Дайте осесть осадку.

Weigh dry filter paper (dried at 150°C). Write down the weight in grams as W1. Filter the solution through filter paper.

The filtrate should be transparent. Rinse the sediment with deionized water until it is free of chlorine. To check for chlorine, collect about 20 ml of washing water and add a few drops of silver nitrate solution. If a white precipitate forms, then chlorine is still present. Dry the sludge and filter paper to a constant weight in the oven at 150°C for about one hour. Weigh and record the weight in grams as W2.

Result: Sulfate (mol/l) = (W2 – W1) x 20/233.4

6.2.10 INDUSTRIAL TESTS OF ORGANIC EXTRACTANT (MX90)

Reagents

3 g/mol of sulfuric acid

Equipment

Dividing funnel (300 ml)
Chemical glasses (200 and 300 ml)
Measuring cylinder (100 ml)

1. Analytical procedure, part A

1. Collect a sample (> 100 ml) of organic extractant from the organic outlet at the stage of the fifth mixer-sump. The unit must be operated for at least one hour.

2. Pour 100 ml of the sample into the dividing funnel and add 20 ml of 3 g* molar sulfuric acid. Shake well for 10 minutes. Wait for the separation of the two phases (approximately 5 minutes). Make sure that the organic phase has acquired a yellow-brown tint, indicating the complete re-extraction of copper from the organic phase. If this does not happen, then contact a Sigma representative.

3. Drain the water and leave the acid (lower phase), but keep the organic phase in the funnel. Analyze the copper in the drained acid. Note that the value for the concentration of copper in acid must be divided by a factor of 5 to obtain the concentration of copper in the organic sample taken, which is Part A!

Result: the concentration of copper in the organic phase should be <3 g/l.

2. Analytical procedure, Part B

1. Add 100 ml of the spent pickling solution to the organic phase in the funnel and shake for 2 minutes. Wait for the separation of the two phases (approximately 5 minutes). Collect the pickling solution into a measuring cylinder and make sure that you have received almost all 100 ml of the pickling solution. Remove the pickling solution, but keep the organic phase in the funnel.

2. Add another 100 ml of the spent pickling solution to the funnel and shake for 2 minutes. Wait for the separation of the two phases (approximately 5 minutes). Drain the water and collect 100 ml of the pickling solution and remove it as indicated above, but keep the organic phase in the funnel.

3. Add 100 ml of waste washing water and shake lightly for a minute (the purpose of this action is simply to wash off and dilute the remaining drops of pickling solution that could remain on the inner wall of the funnel). Wait until the phases are completely separated. Drain the water, collect 100 ml of washing water and remove it (the aqueous solution may have a bluish color due to a small concentration of non-extracted copper).

4. Measure the amount of remaining organic solution and add the same amount of 3 g* molar sulfuric acid to the funnel and shake for 10 minutes. Make sure that the organic phase has acquired a deep yellow-brown hue, indicating the complete re-extraction of copper from the organic phase. If this does not happen, then contact a Sigma representative. Wait until the phases are completely separated.

5. Analyze the acid solution for the presence of copper.

   Result: The copper concentration value obtained in accordance with paragraph 5 is a measure of the maximum concentration of copper that can be obtained in the organic phase. It can be taken as the maximum loading capacity of copper in the organic phase.

   The resulting maximum loading capacity should be in the range from 10 to 13 g/l.

note

Complete phase separation should take less than 8-10 minutes. If the separation took longer, then contact your Sigma representative.

6.2.11 VERIFICATION OF ORGANIC SEPARATION/INVOLVEMENT

Equipment

Imhoff funnel (dimensional funnel)

The order of work

Take a sample of 2000 ml of organic phase, put it in the Imhoff funnel and count the amount of aqueous solution in the funnel after 24 hours.

Selection points

- Tank with organic phase T105
- Organic phase bypass pipes from the extraction unit.

Result:

The amount of aqueous solution in 2000 ml of the organic phase should be <1 ml.

6.3 MAINTENANCE OF THE INSTALLATION

The continuous operation of the MECER installation requires regular maintenance, as described in the following sections.

6.3.1 PROCESS MATERIALS: MANUAL ADDITION

Over time, evaporation and entrainment will lead to a decrease in the volume of some process fluids. An auxiliary quantity should be added for each liquid to maintain proper concentration and volumes.

6.3.1.1 Adding the MX 90 reagent to the unit LE

Evaporation ultimately leads to a situation with a low level of organic extractant (MX 90) in the storage tank (T105). In this case, the block stops automatically. Before adding fresh organic solution to the tank, make sure that the following conditions are met:

1. The depth of the organic phase in the sump compartment of each of the tanks T106, 107, T111 and T112 is approximately less than 20 mm.

2. There is practically no accumulation of organic solution in the LE circulation tank.

   If the above statements are correct, enough fresh MX 90 reagent should be added to half fill the tank.

   To restart the LE unit, press the "SX START" button on the control panel.

6.3.1.2 ADDING SULFURIC ACID TO THE EE UNIT

Entrainment and loss of acid vapors in the electroextraction unit can eventually lead to a loss of sulfuric acid concentration in the electrolyte, especially since the total volume of the electrolyte is regularly restored by deionized water. The need to add sulfuric acid is determined by analyzing the electrolyte sample for sulfuric acid and the concentration of copper. Two analyses should be carried out on the same sample of the solution. Analyses should be performed once a week (see Sections 6.2.6 and 6.2.7 for analytical procedures)

Use formula 6-2 to determine whether the test results are within their own boundaries. As long as the result is within the "acceptable" operating range, do not take any action. If the operating point falls below the "permissible" operating range, add concentrated sulfuric acid to the EE unit. If the operating point is above the "permissible" operating range, add deionized water.

Adding concentrated sulfuric acid from one percent to the total volume of the electrolyte in the unit will increase the concentration of sulfuric acid by approximately 20 g/l.

Before adding concentrated acid, turn off the housing unit. Add the acid directly to the EE circulation tank through the viewing window. Continuous operation of the circulation pump will ensure proper mixing of the acid.

warning

SULFURIC ACID CAN CAUSE BURNS TO THE SKIN AND EYES, AS WELL AS CAUSE IRRITATION OF THE MUCOUS MEMBRANE. CARE SHOULD BE TAKEN AND APPROPRIATE PROTECTIVE EQUIPMENT SHOULD BE USED WHEN ADDING ACID TO THE EE TANK. FOR MORE INFORMATION, PLEASE REFER TO THE PBM.

ADD NO MORE THAN 10 TO 15 LITERS OF ACID AT A TIME TO PREVENT EXCESSIVE HEATING AND SPLASHING. WAIT 15 MINUTES BEFORE THE NEXT ADDITION, UNTIL THE INSTALLATION DOES NOT MIX.

After adding all the acid, wait one hour until mixing is performed on the unit before starting the analysis of a new sample of sulfuric acid and copper concentration. If the new analysis values are within the acceptable operating range according to formula 6-2, restart the block LE and continue operation.

After a whole day of operation, repeat the analytical control procedure.

6.3.2 CLEANING TOOL/MAINTENANCE/REPLACEMENT

The effective operation of the MECER installation depends on regular cleaning and maintenance of process equipment and sensors. Record all maintenance activities in the maintenance log (a sample log is shown in Figure 6-7).

Maintenance schedule:

Daily

	Chapter
Checking the interfacial level of settling tanks	6.3.2.16
Checking the operation of the density controller	6.3.2.1
Checking the flow meter adjustment	6.3.2.5
Checking the temperature in the EE unit	
Checking the functioning of the water drainage pump P105B	
Checking the operation of mixers (visually)	
Leak check on all equipment (visually)	
Cleaning from drops and spills	6.3.7

If necessary:

	Chapter
Replacement of copper cathodes	6.3.3
Filter	6.3.2.8
Anodes	6.3.2.12
Service/Equipment replacement	6.3.2.15

Weekly:

	Chapter
Cleaning of the coarse filter EE	6.3.2.7
Checking the formation of dendrites	6.3.2.11
EE current measurement	6.3.2.13
Checking the pH meter/ORP tool on the etching unit and the LE unit	6.3.2.2
Cleaning of equipment (crystallization)	6.3.7

Monthly:

	Chapter
Cleaning and checking of level sensors	6.3.2.3
Cleaning of flow meters	6.3.2.5
Cleaning of conductivity sensors	6.3.2.6

Every 2 months

	Chapter
Calibration of dosing pumps	6.3.2.4

Twice a year:

	Chapter
Cleaning the rectifier	6.3.2.14

Annually:

	Chapter
Emptying and cleaning of equipment	6.3.7
Emptying and cleaning of intermediate tanks	

Figure 6-6. Maintenance schedule

Weekly maintenance record.....

Daily

	Completed - Signature						
	Mon.	Tue.	Wedn.	Thur.	Frid.	Satur.	Sun.
Checking the interfacial level of settling tanks							
Checking the operation of the density controller							
Checking the flow meter adjustment							
Checking the temperature in the EE unit							
Checking the functioning of the water drainage pump P105B							
Checking the operation of mixers (visually)							
Leak check on all equipment (visually)							
Checking the dosage of hydrochloric acid							
Cleaning from drops and spills							

If necessary:

	Date	Signature
Replacement of copper cathodes		
Filter		
Anodes		
Service/Equipment replacement		

Weekly:

	Date	Signature
Checking the density controller on the etching unit		
Cleaning of the coarse filter EE		
Checking the formation of dendrites		
EE current measurement		
Checking the pH meter/ORP tool on the etching unit and the block LE		
Cleaning of equipment (crystallization)		

Notes:

Figure 6-7. Example of a maintenance log - weekly

Maintenance record by year....

Monthly:

										Chapter	
Cleaning and checking of level sensors										6.3.2.3	
Cleaning of flow meters										6.3.2.5	
Cleaning of conductivity sensors										6.3.2.6	
Month	1	2	3	4	5	6	7	8	9	10	11	12
Date												
Signature												

Every 2 months:

						Chapter
Calibration of dosing pumps						6.3.2.4
Month	1 - 2	3 -4	5 - 6	7 - 8	9 - 10	11 – 12
Date						
Signature						

Twice a year:

		Chapter
Cleaning the rectifier		6.3.2.14
Month	1 - 6	7 – 12
Date		
Signature		

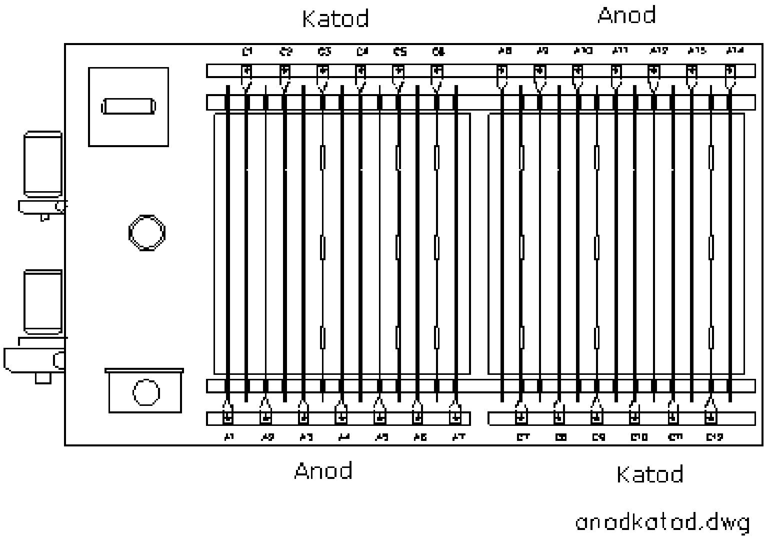
Annually:

	Chapter
Emptying and cleaning of equipment	6.3.7
Emptying and cleaning of intermediate tanks	
Month	1 - 12
Date	
Signature	

Notes:

Figure 6-8. Example of a maintenance log - annual

Measurement of the EE current weekly..........



	A		A
Anode 1		Anode 8	
Anode 2		Anode 9	
Anode 3		Anode 10	
Anode 4		Anode 11	
Anode 5		Anode 12	
Anode 6		Anode 13	
Anode 7		Anode 14	
Cathode 1		Cathode 7	
Cathode 2		Cathode 8	
Cathode 3		Cathode 9	
Cathode 4		Cathode 10	
Cathode 5		Cathode 11	
Cathode 6		Cathode 12	
Date		Date	
Signature		Signature	

Figure 6-9. Electroextraction - an example of a current measurement log - weekly

6.3.2.1 Density control unit

Perform the "calibration" of the hydrometer in the etching process line and the EE density control unit once a week using the analytical results obtained from the weekly analysis of the etching solution and the concentration of copper. Compare the height position of the hydrometer and the analytical results on the position of the inductive level sensors. (The data on the height of the sensors on the EE density control unit, read from above, should correspond to approximately 32, 25, and 20 g/l of copper, respectively).

Scenario: The electrolyte sample is analyzed for the concentration of copper in it. The result was 28 g/l of copper. The metal part, which is enclosed in the upper part of the hydrometer, should then be in the range of medium (25 g/l) and upper (32 g/l) inductive level sensors.

If the position of the hydrometer, the result of the analysis and the height level of the sensor do not match, then contact a representative of Sigma.

note

When servicing the inductive level sensors in the etching system density control unit, turn off the Mecer installation. This will prevent uncontrolled liquid entry into the etching plant.

6.3.2.2 pH meter/ORP tool on the etching plant

Accurate pH values and redox potential values are very important for the effective functioning of the etching process. To maintain accuracy, the pH values measured during operation (48-50°C) and in the laboratory at 25 °C, subject to comparative analysis on a daily basis. If the difference is greater than 0.4 pH, it is necessary to calibrate the pH electrodes.

If this is not required more often (as described above), the pH electrodes and redox electrodes should be checked and calibrated once a week. See the separate appendix for the pH meter calibration instructions.

Maintenance of the electrode

1. Inspect the electrode for scratches, cracks, formation of salt crystals or deposits on the membrane/interface nodes.

Rinse any salt formation with distilled water and remove any deposits on the membranes/junctions.

6.3.2.3 *Tank level sensors*

All level sensors should be removed and cleaned monthly. Rinse thoroughly before replacing.

note

It is important that each sensor is installed on the same tank from where it was removed, as they have been calibrated for each specific solution.

6.3.2.4 *Calibration of metering pumps and flow rate*

It is necessary to calibrate and check the flow rate from all dosing and centrifugal pumps every two months.

6.3.2.5 *Cleaning of flow meters*

Once a day, all flow meters of the installation should be checked visually for precipitation and debris that can block the movement of the float. Once a month, the flow meters must be disassembled and cleaned.

6.3.2.6 *Cleaning of conductivity sensors*

Once a month, the conductivity sensors must be removed from the housing unit and cleaned. At the same time, they must be tested for normal operation by closing two electrodes and thereby triggering an alarm.

6.3.2.7 *Not used*

6.3.2.8 *Replacement of cartridge filters*

All cartridge filters on the MECER installation must be replaced on a regular basis. The time for replacement should be determined based on pressure sensor readings and operational experience.

6.3.2.9 Not used

6.3.2.10 Not used

6.3.2.11 Formation of dendrites

The unit on the Maker installation has a built-in short circuit detector that monitors the presence of dendrites in the electrolyzers in order to prevent damage to the anodes from a short circuit. In order to reduce the risk of dendrite formation in electrolyzers, electrolytic cells should be checked for possible formation of copper dendrites on the cathodes. This is done by stretching a piece of plastic pipe between the anodes and cathodes in order to detect possible formation. If there is a risk of a short circuit, the cathode must be replaced with a new starter. This test should be performed once a week.

6.3.2.12 Anodes

The anodes are coated to provide low transient resistance.

Avoid mechanical cleaning of anodes.

The recommended cleaning solution is water and 10% hydrochloric acid.
The current consumption should not be higher than 5.0 A/dm ^{2}.
The temperature should not be higher 45°C.
The normal voltage in the cell during the rated current should be 2.0 - 2.5 V.
If the voltage is higher than 2.5 V / per cell compartment, it is necessary to stop precipitation and consult with a representative of Sigma.

6.3.2.13 Measurement of the current in the electrolyzer

After each cathode replacement (for 2 days) and once a week, it is necessary to measure the current passing through each cathode and anode using a clamp-type ammeter. If the measurement is outside the parameters, it is necessary to clean, correspondingly, replace the cathode/anode.
The current at the first and last anode in the cell compartment will be less than for other anodes, which is a normal situation. It can be 50% less.

6.3.2.14 Cleaning the rectifier

Every six months, the straightener should be cleaned with a vacuum cleaner. This should reduce the risk of overheating of electronic components.

6.3.2.15 Service/Equipment replacement

When servicing electrical equipment, turn off the power to the switch cabinet and the rectifier using the switch. It shuts down all hardware.

6.3.2.16 Interphase level of settling tanks

Check the interfacial level of the settling tanks once a day to ensure that the level is located at half the height inside the settling tanks.

6.3.3 REPLACEMENT OF COPPER CATHODES

Remove and replace the copper cathodes in the electrolyzer when their thickness is approx. 20 mm. A warning message will appear on the PLC when it's time to replace the cathodes.

carefully

WHEN THE CATHODE THICKNESS IS 20 mm, DO NOT DELAY THEIR REPLACEMENT. THERE IS A POTENTIAL FOR THE FORMATION OF COPPER SPIKES OR DENDRITES ON THE CATHODES, WHICH CAN REACH THE ADJACENT ANODE AND CAUSE SHORT CIRCUIT, WHICH CAN LEAD TO THE DESTRUCTION OF THE ANODE.

To remove the cathode, unscrew the suspension mount from the tire. Hang the cathode lifting frame on the suspension bracket and attach it to the lifter. Lift the frame and cathode vertically up and move them to the cathode flushing area. Rinse the cathode, then detach it from the lifting frame and disconnect the cathode from the suspension bracket.

warning

ELECTROEXTRACTION GENERATES ACID VAPORS OVER LECTROLYZERS. TO AVOID EXPOSURE TO ACID VAPORS WHEN REPLACING CATHODES, TURN OFF THE RECTIFIER.

SULFURIC ACID CAN CAUSE BURNS TO THE SKIN AND EYES, AS WELL AS CAUSE IRRITATION OF THE MUCOUS MEMBRANE. WEAR PROTECTIVE CLOTHING WHEN REMOVING AND RINSING CATHODES.

Clean the contact points on the suspension mount with sandpaper and install a new starter. Place the starter into the electrolyzer manually or using the lifting frame of the lifting device. Use contact grease and re-bolt the suspension mount to the tire, making sure that the contact surfaces are clean. Do not use contact grease between the suspension mount and the starter, as this may lead to acid contamination.

Turn on the rectifier again.

note

Do not allow contamination of the electrolyte from contact lubrication.

6.3.4 REPLACEMENT OF ANODES

Stable-sized anodes should be dismantled and replaced every five years.

6.3.5 NOT USED

6.3.6 NOT USED

6.3.7 CLEANING THE INSTALLATION

Daily - traces of drops and spills should be cleaned immediately when they appear. This will facilitate the production of maintenance, and extend the service life of the equipment.

Annually - Mixing/settling units, pumping stations and intermediate tanks must be completely emptied into separate storage tanks, rinsed with water and wiped with a cloth to eliminate the formation of sediment.

When emptying and refilling the tanks, mixing solutions or cross-contamination of the tanks should be avoided. Each container must be filled with the initial contents.

carefully

DO NOT USE LOW MOLECULAR WEIGHT ORGANIC SOLVENTS FOR CLEANING PLASTIC PARTS OF THE INSTALLATION. this IT CAN LEAD TO THE FRAGILITY OF PVC PARTS AND THE "CLOUDING" OF THE PLASTIC SURFACE. TO REMOVE SPILLS SUCH AS GREASE OR OIL, WE RECOMMEND USING ISOPROPANOL OR METHANOL AND THEN RINSING WITH WATER.

SOAPS, DETERGENTS, AND OTHER CLEANERS THAT CONTAIN SURFACTANTS, AFFECT THE SURFACE THE TENSION OF THE ORGANIC EXTRACTANT AND PREVENT THE SEPARATION OF PHASES IN THE LIQUID EXTRACTION UNIT.IF THESE SUBSTANCES ARE USED FOR PURIFICATION, DO NOT AVOID CONTACT WITH PROCESS FLUIDS OR CONTAMINATION OF CONTAINERS. ALWAYS RINSE THOROUGHLY WITH WATER.

7 TROUBLESHOOTING

This section discusses the problematic situations that may arise during the operation of the MECER installation and ways to solve them. The following topics are considered:

Emergency messages

Alarm level alarms

Alarm density alarms

Conductivity alarms

Other alarms and messages

Short circuits in the EE unit

Chemical problems

7.1 EMERGENCY MESSAGES

The operation of the MAKER plant together with the etching plant is a balanced process, including the output of technological operations, copper concentration, chlorine concentration, pH, etc. They depend on each other, ensuring the efficient operation of the installation. To ensure this, most of the monitored parameters on the installation have an alarm function.
In the event of any emergency situation, the alarm light is lit and a sound signal is heard, and in some cases, the equipment is automatically stopped.

The beep will continue until the reset button on the operator interface panel is pressed. The light bulb will continue to shine until the emergency situation is resolved. The light bulb, the sound signal and the operator interface panel are located on the main control panel. Simultaneously with the activation of the alarm, a message is displayed on the operator interface panel.

Possible emergency messages and corresponding section numbers for troubleshooting assistance are listed below:

Emergency messages	**Section number**	**Emergency messages**	**Section number**
TANK T101 IS FULL Used pickling solution Intermediate tank INCL. SAME, STOP INSTALLATION FOR ETCHING	7.2.1	TANK T102 EMPTIED Reg. pickling solution Intermediate tank ON, STOP THE ETCHING INSTALLATION	7.2.2
TANK T102 Reg. etching solution Intermediate tank is FILLED ON, STOP THE ETCHING INSTALLATION	7.2.2	TANK T103 IS EMPTIED Waste water Intermediate tank ON, STOP THE ETCHING INSTALLATION	7.2.3
TANK T103 IS FULL Waste water Intermediate tank ON, SAME, STOP INSTALLATION FOR ETCHING	7.2.3		

The emergency message will be displayed until the emergency situation is eliminated and until the “Reset” button is pressed on the operator interface panel.

If several emergency situations occur simultaneously, messages will be sent to the PLC. The emergency with the highest priority will be displayed first.

7.2 LEVEL ALARMS

The level in each process tank depends on both pumps and overflows to the tank and pumps diverting liquid from the tank. The levels also vary depending on the synchronicity of the etching unit with the MECER installation. Therefore, when investigating level alarms, several causes of malfunction can be detected.

When the level sensor gives an alarm signal, you should first make sure that the level sensor itself is working properly. If this is the case, then the following sections discuss some of the possible causes of problems with respect to the level.

7.2.1 INTERMEDIATE TANK OF SPENT PICKLING SOLUTION (T101)

TANK T101 IS FULL (T101 FULL)

note

If this situation occurs, the supply of regenerated etching solution to the etching plant will be stopped. Therefore, the malfunction must be eliminated immediately.

There are two reasons for the occurrence of this situation:

1. The total volume of the pickling solution on the installation is too large.

The total turnover of the pickling solution in a closed circuit should have such a limited volume that one of the intermediate tanks with the pickling solution is emptied (before filling (the other tank. For further recommendations on how to resolve this situation, go to the section below 7.2.2.

The ability to regenerate the installation may have been temporarily overloaded. The size of your installation MECER was designed to correspond to the normal conditions of the etching process at your company (for example, the average number of hours etching the day, the size of the production line of etching, etc.). If the production line of etching is operated on a much larger regime than had been anticipated, possible that installing MECER can not cope with the load.

It is necessary to stop or reduce the volume of etching and give the installation time to cope with the specified volume.

7.2.2 INTERMEDIATE TANK OF REGENERATED PICKLING SOLUTION (T102)

TANK T102 IS EMPTIED (T102 EMPTY)

note

If this situation occurs, the supply of regenerated etching solution to the etching plant will be stopped. Therefore, the malfunction must be eliminated immediately.

The reason for this situation may be the following:

1. exceeding the power of the installation (see Section 7.2.1) or
2. loss of effective functioning of the MECER installation.
   Contact a Sigma representative.

TANK T102 IS FULL (T102 FULL)

note

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

This situation is caused by an excessive amount of the total circulating volume of the etching solution in the etching solution circuit. Under normal conditions, the intermediate tank T101 for the spent pickling solution should have been emptied (, and then the RE unit should stop the production of the regenerated pickling solution before the tank T102 reaches the fullness level .

The reason for the situation with filling the tank (related to the conditions of the water balance in the circuit. A certain amount of water is produced "chemically" at the etching plant (about 0.28 kg per 1 kg of etched copper). On the other hand, some water is lost due to evaporation. Depending on the ventilation and entrainment conditions at the etching plant, some total volume may accumulate during long-term operation.

To restore proper conditions in relation to the total circulating volume, a certain volume of pickling solution can be pumped out of the tank for temporary storage. In the future, this volume can be added to the contour of the etching solution when it is "in volumetric equilibrium».

7.2.3 INTERMEDIATE TANK WITH WASTE WASHING WATER (T103)

TANK T103 IS EMPTIED (T103 EMPTY)

ПРИМЕЧАНИЕ

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

This situation will take place when the water turnover is out of balance. To restore normal conditions, it is necessary to increase the supply of deionized water to the etching process line at the stage of fresh washing water.

TANK T103 IS FULL (T103 FULL)

If the flow rate of deionized water is too high, then too much waste flushing water is supplied to the tank T103.

Possible reasons in this situation may be the following:

1. The flow rate of deionized water is set to too high a level. The speed of the augmented deionized water must be set to ensure appropriate flushing. The flow rate should be checked.

7.2.4 REAGENT TANK MX 90 (T105)

TANK T105 IS EMPTIED (T105 EMPTY)

note

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

The low level in the T105 tank is ultimately a consequence of losses due to entrainment and ventilation. In this case, fresh MX 90 reagent should be added to the tank in accordance with the instructions in section 6.3.1.1 "Adding MX 90 reagent to the unit LE».

A low level can also be caused by blocking one of the terminals of the sump mixer. Check the volume of the organic phase in the settling mixers to determine the increase in the turnover of the organic phase. The organic phase should be about half of the level.

TANK T105 IS FUL (T105 FULL)

note

Under these conditions, the installation stops immediately (Block LE and EE)

Check the tank T105 to make sure that a significant volume of aqueous solution is not available.
Drain some of the extractant from the T105 tank to provide a larger volume of "intermediate space».

7.2.5 PUMPING STATION OF REGENERATED PICKLING SOLUTION (T106-2)

CAPACITY T106-2 IS FULL (T106-2 FULL)

note

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

In this situation, the flow capacity from the tank T106-2 to the intermediate tank T102 is too low or limited. Check the level sensor LA106-2, filter F106-2, pump P106-2 and the corresponding pipe fittings for proper operation.

7.2.6 WASTE WASHING WATER PUMPING STATION (T107-2)

CAPACITY T107-2 IS FULL (T107-2 FULL)

note

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

In this situation, the flow power from the T107-2 tank is too low or limited. Check the level sensor LA107-2, filter F107-2, pump P107-2 and the corresponding pipe fittings for proper operation.

7.2.7 SATURATED ACID PUMPING STATION (T111)

CAPACITY T101 IS FULL (T111 FULL)

note

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

In this situation, the flow power from the T111 tank to the EE unit is too low or limited. Check the level sensor LA 111, filter F111, pump P111 and the corresponding pipe fittings for proper operation.

7.2.8 SATURATED ACID PUMPING STATION (T112)

CAPACITY T112 IS FULL (T112 FULL)

note

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

In this situation, the flow power from the tank T112 to the EE unit is too low or limited. Check the level sensor LA 112, filter F112, pump P112 and the corresponding pipe fittings for proper operation.

7.2.9 EE CIRCULATION CAPACITY (T108)

LOW LEVEL IN T108 CAPACITY (T108 LOW)

A sudden leakage or loss of acid in the SAME or EE unit eventually leads to a decrease in the level in the T108 tank. Losses due to evaporation and acidic vapors in the E block can also cause a reduced level if these losses are not compensated by water, respectively. sulfuric acid, as described in the section 6.3.1.2.

HIGH LEVEL IN CAPACITY T108 (T108 HIGH)

A high level in T108 may occur if water due to a malfunction in the block goes to the stage of re-extraction and is transferred to the container T108. Another reason may be the excessive addition of deionized water or acid to the container T108.

7.2.10 SODIUM HYDROXIDE TANK

TANK T113 IS EMPTIED (T113 EMPTY)

This alarm is intended only to notify the operator that it is time to replenish the tank with sodium hydroxide.

7.2.11 PUMPING STATION FOR SPENT PICKLING SOLUTION FROM THE PICKLING PLANT

CAPACITY T109 IS FULL (T109 FULL)

A high level in this tank will be caused by a low flow rate from the pump or a restriction in the line. Check the level sensor, filter, pump and pipe fittings for proper operation.

7.2.12 PUMPING STATION FOR WASTE FLUSHING WATER FROM THE ETCHING LINE

CAPACITY T110 IS FULL (T110 FULL)

A high level in this tank will be caused by a low flow rate from the pump or a restriction in the line. Check the level sensor, filter, pump and pipe fittings for proper operation.

7.2.13 HYDROCHLORIC ACID TANK

TANK T602 IS EMPTIED (T602 EMPTY)

This alarm is intended only to notify the operator that the time has come to replenish the tank with hydrochloric acid.

7.2.14 EMERGENCY DENSITY DETECTORS

The density or concentration of copper is carefully monitored on the etching unit and the EE unit. The emergency situation at the EE unit is a manifestation of industrial incompetence and must be eliminated immediately.

7.2.15 HIGH CONCENTRATION OF COPPER IN THE EE ELECTROLYTE

If such a situation occurs, the block is automatically disabled. In this situation, the following actions should be performed:

1. Make sure that there is no short circuit between the anodes and cathodes in the electrolyzer. (See Section 7.5, "Short circuits in the EE unit»).
2. Make sure that the rectifier outputs the desired current.

3. Make sure that the hydrometer in the EE density control unit is functioning normally.
4. Check the copper, sulfuric acid and ammonia concentration in the electrolyte by chemical analysis (see section 6.2).

The situation may also happen if the total power of the electroextraction unit is less than the power set for the extraction unit. For further recommendations, contact a Sigma representative.

7.3 CONDUCTIVITY DETECTORS

7.3.1 HIGH CONDUCTIVITY IN MIXERS S101, S102 OR S103

note

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

If the conductivity sensors detect high conductivity in the mixer S101, S102 or S103, they will give an alarm signal and the LE unit will automatically stop. The most likely cause of this failure is that the flow sensors report incorrect flow data, which can lead to an increase in the proportion of the aqueous solution in the mixers more than the desired proportion. The proportion of the aqueous solution in the mixer S101, S102 and S103 during normal operation is approximately 25%.
The proportion of the aqueous solution is usually automatically reduced during the normal shutdown mode to ensure that the matching phase is maintained at startup.

After the conductivity alarms in S101, S102 or S103 are triggered, all active flow sensors must be calibrated before the unit is restarted. After restarting, the unit can again send a high-conductivity signal in the mixer S101, S102 or S103. This is a normal situation and it is important to allow the device to complete each shutdown cycle before it is restarted. It may be necessary to operate the unit one or two additional shutdown cycles until the necessary proportion between the aqueous phase and the organic phase is reached.

Contact a Sigma representative if the high conductivity problem is not resolved after the unit has passed three normal shutdown cycles.

7.4 OTHER ALARMS AND MESSAGES

7.4.1 OVERLOAD OF PUMPS AND ELECTRIC MOTORS OF MIXERS

note

Under these conditions, the installation stops immediately (block LE and EE)

Electric motors of mixers and centrifugal pumps can be overloaded as a result of excessive mechanical load on the motor shaft. If such a situation occurs, the appropriate motor current protection is triggered and all work on the MECER installation is stopped.

After the error is found and corrected, you can press the reset button on the motor protection device. You can start the engine or pump manually for testing purposes. If the motor protection is triggered again, the protective device should be checked and compared with the specification of the electric power consumption on the engine. Increase the setpoint values if they are too low. Restart the motor and measure the current load to compare the actual value with the current specification. Consult with a Sigma representative in case of non-compliance.

7.4.2 RECTIFIER ALARM

EMERGENCY SIGNAL (R101 ALARM)

If this alarm is triggered, it means that the rectifier has been blocked for some reason. In most cases, this is due either to a malfunction in the power supply or overheating inside the rectifier.

Make sure that when the power is supplied, the current is applied to all three phases. Also check that the ventilation of the rectifier is not blocked in any place. If the electronic components inside the rectifier are covered with dust, this can lead to overheating. In this case, you should use a vacuum cleaner to clean it, but do not forget to turn off the power before opening the straightener.

7.4.3 NOT USED

7.4.4 NOT USED

7.4.5 NOT USED

7.4.6 LONGER DOSAGE TIME OF THE REG. PICKLING SOLUTION

EMERGENCY SIGNAL (P102 ALARM)

If this alarm is triggered, it means the activation of the signal "High pH in etch module" (High pH in etch module) for a longer time than is considered normal.

Это может быть:

- The pump of the regenerated pickling solution P102 does not work properly.
- The flow of the regenerated pickling solution is too low (the filter is blocked).
- The copper content in the regenerated pickling solution is too high.
- The intermediate tank with regenerated pickling solution is empty.
- The dosing capacity is overloaded (too much copper for etching).

7.4.7 THE ELECTRIC EXTRACTION CIRCULATION PUMP HAS STOPPED

STOPPING ACIDIC ABOUT THE PUMP (ACID PUMP STOP)

note

In these conditions, the installation will switch to shutdown mode «Shutdown Mode».

Correct the alarm that caused the acid circulation pump to stop.

After starting the pump (the acid pump switch is on "ACID PUMP ON"), the extraction unit must be restarted.

7.5 SHORT CIRCUITS IN THE EE UNIT

An electrical short circuit between the anodes and cathodes in electrolyzers can damage the anodes. A short circuit is usually caused by one of the following:

1. Formation of copper dendrites or localized copper spikes on the cathode. When the dendrites become large enough to reach the adjacent anode, a short circuit occurs. The risk of dendrite formation is significantly higher when the rectifier is operating at rated load and low copper concentration. Therefore, it is important to maintain the concentration of copper within the specified interval.

2. Bending of the cathode or anode. A curved corner or a curved edge will lead to faster deposition of copper in this area, increasing the risk of dendrite growth.

The MAKER installation is equipped with short circuit detection equipment, which automatically sends an alarm signal in case of a short circuit detection. A short circuit can also be prevented and detected using the following methods:

1. Stretch a piece of PVC pipe between the anodes and cathodes in order to detect dendritic growths.

2. Monitor the temperature of the electrolyte in the electrolyzers, as the temperature in the cells increases when a short circuit occurs.

3. Monitor the deposition performance of the installation. A short circuit ultimately leads to a decrease in deposition performance compared to extraction performance..

4. Measure the current passing through each cathode using a clamp-type ammeter. A shorted cathode will have a much higher current reading.

carefully

It is very important that short circuits are detected and corrected as soon as possible, as they can lead to serious damage to the anodes.

7.6 CHEMICAL PROBLEMS

The selective control method and the analysis program are described in Section 6.2, which provide for regular monitoring of the operation of the installation. If the program is executed properly, many chemical problems can be quickly detected and eliminated using the measures described in Section 7.6.1. Some additional problems related to chemical processes may occur, however, cannot be detected by the program. These situations are described in Sections 7.6.2, 7.6.3 and 7.6.4.

7.6.1 ANALYTICAL TROUBLESHOOTING

The sampling method and analysis program described in Section 6.2 list the normal operating concentrations for some components of the installation. If the results of the tests go beyond these normal values, then measures must be taken.

7.6.1.1 COPPER CONCENTRATION IN THE SPENT ETCHING SOLUTION

Correct deviations by adjusting the ratio between the addition of regenerated pickling solution and hydrochloric acid to the pickling plant.

7.6.1.2 Chlorine concentration in the spent pickling solution

Low concentration «LOW »:
Low chlorine concentration negatively affects the etching rate and increases the risk of sediment formation in containers with spent etching solution.

The supply of too much water to the etching plant, resulting in dilution of the etching solution or reduction of sodium chloride salt, is added to the solvent station.

There are several possible sources for water:

- The printed circuit boards included in the etching unit are too wet.
- The flushing water from the flushing stage somehow flows back into the etching plant.

- Water from the cooling jacket flows into the etching chamber.
- Condensate from ventilation.

note

High concentration «HIGH Concentration»:
A high concentration of chlorine can lead to the formation of crystallization and precipitation throughout the system.

Too much water evaporates into the etching plant.

Corrective actions:

- Step by step add deionized water to the etching plant and intermediate tanks T101 and T102. Analyze chlorine between steps.
- Reduce evaporation – by reducing the ventilation level and stopping spraying during production stops.

7.6.1.3 pH value in the spent pickling solution

Recalibrate and check the setpoint value on the pH meter. If you get a slow reaction or an unstable reading, replace the electrode.

7.6.1.4 NOT USED

7.6.1.5 COPPER CONCENTRATION IN THE REGENERATED PICKLING SOLUTION

High concentration «HIGH Concentration»:

- Check the flow of MX 90 organic reagent and spent pickling solution.
- The MX 90 reagent may not work properly. Conduct a performance test of the MX 90 reagent and consult with a representative of the company Sigma.

7.6.1.6 Concentration of copper in the waste washing water

High concentration «HIGH Concentration»:

- Check the addition of deionized water to the washing water.
- If possible, increase the addition of deionized water to the washing water.
- Try to reduce entrainment from the etching plant.
- Consult with a Sigma representative.

7.6.1.7 Chlorine concentration in the waste washing water

High concentration «HIGH Concentration»:

- Check the addition of deionized water to the washing water.
- Increase the addition of deionized water.
- Try to reduce entrainment from the etching plant.
- Consult with a representative of the company Sigma.

7.6.1.8 CONCENTRATION OF COPPER IN ACIDIC ELECTROLYTE Deviations can be eliminated by adjusting the inductive sensors in the EE density control unit.

High concentration «HIGH Concentration»:
Checking the density regulator

- Does the electrolyte flow through the density regulator?
- Does the hydrometer have the ability to move freely or is it blocked by crystals?
- Is the sensor location correct?

Too high concentration of copper in the electrolyte can negatively affect the efficiency of organic phase extraction and, consequently, the ability to LE.

note

If the concentration of copper is high (Cu>32 g/l), then there is a risk of crystallization in the fourth and fifth blocks of sediment. It can even destroy the sump due to crystal growth.

Low concentration «LOW Concentration»:

Checking the density regulator

- Does the electrolyte flow through the density regulator?
- Does the hydrometer have the ability to move freely or is it blocked by crystals?
- Is the sensor location correct?

Low copper concentration increases the risk of dendrite formation.

7.6.1.9 CONCENTRATION OF SULFURIC ACID IN AN ACIDIC ELECTROLYTE

Low Concentration - If the concentration is too low, sulfuric acid is added to the electrolyte (see Section 6.3.1.2 "Adding sulfuric acid to the EE unit »).

note

A low acid concentration will reduce the reextraction of the saturated organic phase.

7.6.1.10 CONCENTRATION OF CHLORINE IN ACIDIC ELECTROLYTE

High Concentration - Too many components of the pickling solution are involved in the MX 90 reagent and transferred to the electrolyte. Consult with a Sigma representative

.

note

High chlorine content can cause damage to the anodes.

7.6.1.11 NOT USED

7.6.1.12 Temperature in the electrolyte

Too high a temperature in the electrolyzer can worsen the deposition of copper and lead to damage to the anodes and all equipment (parts from PCBs!).

Corrective actions:

- Check the operation of the heat exchanger.
- Check the copper deposition (Short circuits? See the section 7.5).
- Consult with a Sigma representative.

7.6.1.13 *Checking the performance of the organic phase*

Part A: High concentration of copper "HIGH Copper Concentration": The organic phase is not subjected to re-extraction at the stage of re-extraction in the mixing-settling unit. You can take the following actions:

- Check the operation of the faucets themselves. If the organic phase and the acid are not mixed well, then less copper will pass from the organic phase to the acid.
- Check the speed setter (frequency converter) of the mixer motors.
- Check the flow rate in the sump mixers.
- Check the concentration of sulfuric acid in the acidic electrolyte. If the acid is not strong enough, it will not efficiently re-extract copper from the organic phase.
- Check the concentration of copper in the acidic electrolyte. If the concentration is too high, the amount of copper that the acid can re-extract is reduced.
- Consult with a Sigma representative.

Part B: High concentration of copper "HIGH Copper Concentration": Too high a load will lead to the formation of sediment in the mixer tanks. Ventilation evaporated too many additives in the organic phase. Add the complementary solution (MX 90) to the storage tank and contact a Sigma representative.

Part B: Low Concentration "LOW Concentration": The organic phase is probably contaminated and may need to be replaced.
Consult with a Sigma representative.

7.6.2 LOSS OF ETCHING SPEED AT THE ETCHING PLANT

The following parameters will affect the etching:

Etching module:

- Spray pressure (top/bottom).
- Nozzles (flat - round).
- Ventilation/bubbling air
- Temperature

Composition of the pickling solution:

- Copper content
- Chlorine content
- pH

From the printed circuit board:

- Foil thickness
- Deposition constants
- Layout

Changing the parameters will have the following result:

Copper content:

A higher copper content increases the etching rate, but also increases the distortion of the elements during poisoning.

Chlorine content:
A higher concentration of chlorine will improve the solubility of copper and increase the rate of.

In case of loss of etching characteristics at the etching plant, the following measures should be taken:

7.6.2.1 *Significant drop in etching rate*

If the etching rate drops significantly.

Take a sample of the pickling solution for analysis for the concentration of copper and chlorine (use the procedures in the Section 6.2 for spent pickling solution). Compare the results with normal working intervals. If the results are normal, go to step 2.

1. Calibrate the ORP and pH electrodes to check their function.

7.6.3 PRECIPITATION OF SODIUM CHLORIDE IN PICKLING SOLUTION

If sodium chloride is deposited in an etching solution, it can be recognized by taking a sample of the etching solution. Precipitation can be caused by:

1. Disproportionate ratio of copper to chlorine in the etching solution. If the concentration of chloride is too high, then sodium chloride will precipitate.

Take a sample of the pickling solution for analysis for copper and chlorine. Compare the results with normal working intervals.

7.6.4 CLOGGING IN THE MIXER/SETTLING TANKS

During the etching process, the etching solution can dissolve a small amount of unwanted metals. The impurities of the particles will be filtered out on the filtering equipment with the spent pickling solution and the spent washing water. But dissolved metals and very fine particles will be transferred with an aqueous solution to the extraction unit.

Particles and sediment that are not removed by filters will accumulate on the walls and at the bottom of the mixing/settling blocks. Particles and sediment can also accumulate on the interface between aqueous and organic solutions.

7.6.4.1 *Sludge reduction*

The formation of sediment in the mixing/settling unit can be reduced as follows:

- Filtration of process fluids.
- Use of deionized water in the washing process

7.6.4.2 *Sludge removal*

The sediment from the mixing/settling unit can be removed as follows:

Chemical removal:

- The installation must be in shutdown mode.
- Disconnect the electrical connection (plug).
- Unscrew the bolts on the mixer body.
- Remove the mixer (together with the motor).
- Place the mixer in the cleaning solution for one hour.
- Rinse the mixer with water.
- Put the mixer back into the mixer housing, tighten the bolts and connect to the mains.

Mechanical removal:

- The installation must be in shutdown mode.
- Disconnect the electrical connection (plug).
- Unscrew the bolts on the mixer body.
- Remove the mixer (together with the motor).
- Clean the mixer (use a soft brush) and rinse with water.
- Empty the mixer chamber on each mixing/settling unit into five separate containers using drain valves. Empty the sump chamber on each mixing/settling unit into five separate containers using drain valves. The liquid must be returned to the same chambers from where they were drained when the operation ended.
- Clean the interior (use a soft brush) and rinse with water.
- Fill the mixer/settling tanks. Pay attention not to mix up the containers and fill them with the wrong liquid by mistake.
- Put the mixer back into the mixer housing, tighten the bolts and connect to the mains.

8 SAFETY PREVENTION

8.1 INSTRUCTIONS

8.1.1 RESPONSIBILITIES OF THE OPERATING ORGANIZATION/PERSONNEL

The operating organization is responsible for ensuring that operation and maintenance is provided only by trained specialists.
These personnel should be trained in all the functions of the equipment, environmental issues and hygiene and safety rules.
Regular monitoring and, if necessary, retraining of new personnel should be carried out.
Eye, respiratory and body protection must be worn at all times.

8.1.2 WARRANTY

The equipment of the Maker installation is intended only for the disposal of acid pickling solution and must be operated and maintained in accordance with the provisions of Section 6.
Any claims or warranty issues will be rejected if the equipment is not used for its intended purpose. the following reasons will be the basis for any claims:

- Misuse of MECER installation equipment
- Do not strictly adhere to the provisions of the Operating Instructions
- Service and maintenance procedures are not followed
- Use of equipment in the presence of pronounced and existing malfunctions, electrical, mechanical, etc.
- Disregard for the Operating Instructions and Equipment Manual
- Independent changes or changes without SIGMA approval.
- Lack of maintenance/control on spare parts and consumables
- Unapproved maintenance and repair
- Force majeure.

8.2 SAFETY MEASUREMENTS

The equipment of the MECER installation is made according to the latest level of technology and safety regulations. Despite this, if there is improper use of the equipment or any unforeseen circumstance, the maintenance and operation of the equipment can be dangerous, especially for third parties.

The equipment of the MECER installation is intended only for the recovery of copper from acid pickling solution.
In addition, the user must comply with and follow local environmental, health and safety regulations.

8.2.1 SAFETY MEASURES DURING OPERATION

Before operating the equipment, it is necessary to regularly check the functionality of tanks, pumps, filters and exhaust systems in order to ensure safe working conditions.

8.2.2 ELECTRICAL SAFETY

All electrical work on the equipment must be carried out by trained and licensed personnel.
Wiring, electrical equipment and control systems should be regularly inspected to avoid technical problems and violations.
Before checking the electrical system, the main switch must be turned off.
The power/control cabinet must be closed at any time and can only be opened by qualified personnel.

8.2.3 HAZARDOUS CHEMICALS

Hazardous chemicals are used to work on the Mecer installation.
Please strictly follow the Material Safety Data Sheet and the Operating Instructions.
Only trained personnel with protective clothing can perform maintenance.

8.3 MAINTENANCE / INSPECTION

Recommended maintenance and inspection work should be carried out strictly in accordance with the proposed schedule.
Only original spare parts should be installed. Disposal of any parts according to chemicals must be produced in accordance with local regulations. If you have any questions in this regard, please contact your local SIGMA representative.