Trivalent Chrome Passivation Bath Recycling

1. **Introduction / Task Description**
   Trivalent Chrome acid baths are used in metal working plants for plating and passivation of metal surfaces. In galvanic operation these chrome (III) baths are contaminated by processed working piece. Iron, brass or other pre-electroplated metal layer liberate ions like Fe$^{3+}$, or Zn$^{2+}$. Since the presence of Cr$^{3+}$ and other metals have an influence on the quality of the displaced chrome layer, the concentration of the impurities has to be controlled in a way that concentrations stay within a specific range. The impurities need to be removed without taking out the Cr(III). Normally the critical limits for the impurities are:

   120 - 150 mg/L for Fe
   12 - 15 g/L for Zn

2. **Removed Species; Feed and Outlet Concentrations**
   Depending on the type of working piece used in the process metal ions like Zinc, Nickel, and Iron may be present. The purpose of the treatment is to reduce the metal impurities concentrations down to ppm levels and recycle the Cr (III) to the Plating bath.

3. **Typical Composition and Physical Properties of the Treated Process Stream**
   The typical chromic acid bath contains 100 g/L Cr$^{3+}$, and pH is around 1-3. The temperature of the bath is ranging from 25°C to 60°C. The high acidity of the solution prevents / retards the adsorption of the ions on the resin. Furthermore, due to the high oxidation potential of chromic acid a dilution of the process stream to 30 g/L CrO$_3$ is recommended. A higher concentration might increase the degradation of the resin. The process stream should be cooled down to room temperature when carrying out the purification.

4. **Technical Concept of the Ion Exchange Unit**
   The ion exchange column is arranged in a bypass to the chromic acid bath. Usually the chrome electrolyte has to be diluted down to a 30% before entering into the ion exchanger. The process is started, when the metal concentration in the bath has reached a certain level. Several cycles are run, until the bath concentration has been decreased to the targeted level. The Typical Operating conditions and the resin characteristics of the resin are noted below.
Temperature resistance: max 80°C, optimum < 50°C
Operating pH-range: 1 – 6
Operating mode at exhaustion: Upflow
Throughput at exhaustion: 1 – 15 BV/hr (max 10 m/h)
Maximum allowed pressure loss: 2.5 bar
Volume change: max. 3%

D2EHPA content: ca. 150 g/L
Zinc-Total Capacity: > 15 g Zn / Liter resin
Zinc-Operating Capacity: up to 10 g Zn / Liter resin
Life Time(standard conditions) up to 1 year

Bead size ( > 90%) 0,315 – 1,6 mm
Density (true density) 0,97 g/L
Bulk density 600 g/L
Storability of the product 2 years
Storability (temperature range) -20 to 40°C

Acid requirement for regeneration 2 - 5 BV of a 5 – 15%ic H2SO4 or HCl
Rinse water requirement 3 - 5 BV
Linear velocity at regeneration 4 - 5 BV/h

Recycling of Trivalent Chrome Passivation Bath

Electrolytic Cell

Regenerants: HCl 15% (2 BV)

Recycled Cr³⁺ electrolyte, ca. 100 g/L Cr³⁺, pH 1 - 2

Selective IX

Fe³⁺

Zn²⁺
waste water
Levextrel Resin*: The functional group is not chemically fixed. The active ingredient is directly incorporated during the formation of the copolymer and is fixed by adsorption.
D2EHPA can be washed out if the resin is handled improperly, in particular if neutral to alkaline solutions are used.
→ for backwashing and rinsing always use weakly acidic solutions!
The density of the resin is lower than that of water: Therefore the resin is swimming!
Due to the special production method the resin contains a relatively high fraction of fine grain and is also relatively difficult wettable with water.

5. **Properties and Performance of the Applied Ion Exchange Resin**

6. **Regeneration Procedure**

Typically, the exhausted resin is regenerated with 3-5 BV of 15% HCl

Prior to the regeneration the column is to be backwashed using Weakly acidic Solutions (4 % salts, pH= 4) to remove the suspended particles.

After regeneration with acid the column needs to be washed with water to remove rest traces of the acid as well as the desorbed metal ions. 4 BV of water are required in minimum. Again the rinsing by using weakly acidic Solutions (4 % salts, pH= 4) to remove the suspended particles.

7. **Lifetime of Resin and Spent Resin Disposal**

Due to its increased grade of cross linking resin has a sufficient stability in chromic acid with a concentration of 30 g/L. Keeping temperature around 25-30°C, and free chlorine below 0.1 ppm, the resin lifetime is expected to be around 1 year.

Within this time the resin will continuously decompose by oxidative attack to the heavy metals.