



## Sealing Salt AS

A nickel acetate preparation for high grade, deposit-free sealing

Anodizing by electrolytic oxidation produces a hard, smooth aluminium surface with high resistance to aggressive mechanical action, weathering and corrosion. But to obtain these valuable properties, due care must be taken with sealing, which is an integral part of the finishing process as a whole.

Inadequate sealing of dyed anodic films greatly reduces the light and weather fastness, while with both dyed and undyed aluminium the corrosion resistance is decidedly poorer.

Sealing in **Sealing Salt AS** baths offers the following major advantages over water sealing:

- Minimal bleed-out of dyeings
- Buffered and therefore more stable sealing bath
- High grade of sealing, even when only poor quality water is available
- Superior sealing quality with higher light and weather fastness on films dyed with organic dyes.

**Sealing Salt AS** can be applied in both one-stage and two-stage processes.

## 1. General points

All the sealing methods employed in practice have to be conducted at the boil for specified times in order to produce the desired sealing effect. Lower temperatures and shorter sealing times lead to unsatisfactory results.

The methods commonly used in the industry are:

- nickel salt
- water
- steam and
- dichromate sealing

(For details see section 6).

## 2. Properties

Chemical character: **Sealing Salt AS** is a nickel acetate preparation containing both a smudge suppressant and a pH regulator.

Commercial form: Fine, greenish powder with a faint acetic acid odour

Bulk density: 570 g/l

Storage stability: In tightly closed containers the product is storable for more than 5 years

Solubility: Readily soluble in warm water.

### 2.1 Advantages

- Sealing of consistently high quality
- Virtual absence of smudging
- Minimal or no bleeding of dyed aluminium
- Sealing bath exercises a buffering action, making pH corrections less often necessary
- Suitable for both dyed and undyed aluminium
- Superior sealing quality with higher light and weather fastness on films dyed with organic dyes
- Low quality requirements for the sealing water.

### 2.2 Scope of application

Preferably for films dyed with organic dyes, but also for:

- Undyed films
- Films produced by integral colour processes
- Films coloured by electrolytic methods with metal salts
- Films inorganically coloured by chemical methods.

## 3. Application

**Sealing Salt AS** is applicable by the

One-stage process at the boil

and the

Two-stage process

comprising presealing with **Sealing Salt AS** at 70-80°C/158-176°F and final sealing in a normal boiling water bath or with steam.

### 3.1 Sealing tanks

With respect to corrosive action **Sealing Salt AS** solutions behave the same as normal nickel/cobalt acetate baths, so that when using **Sealing Salt AS**, the solutions must not be allowed to come into contact simultaneously with aluminium and iron, e.g. with iron pipes in aluminium tanks, as otherwise the aluminium will gradually be corroded.

Stainless steel and metals which are enamelled or clad with suitable plastic are the most suitable materials for tanks used in aluminium finishing.

### 3.2 Application guidelines

#### **One-stage sealing**

- Concentration : 4 – 5 g/l **Sealing Salt AS**
- Water quality : tap or deionized water
- pH value : 5,7 (± 0,3) corrected as necessary with acetic acid or ammonia
- Temperature : minimum 96°C/205°F (check after 10 min)
- Time : 2 – 3 min/μm (but at least 10 min).

#### **Two-stage sealing**

Since two-stage sealing is regarded by industry as very important, we recommend the following working methods:

##### **METHOD A**

Stage I – Presealing (nickel impregnation) with **Sealing Salt AS**

Temperature	Time	Sealing Salt AS	Sealing medium	pH*
70°C/158°F	5 min 3 min	5 g/l 10 g/l	deionized or not too hard Water	5.7 ( ± 0,3)
80°C/176°F	2 min 1 min	5 g/l 10 g/l		

Intermediate rinsing is not absolutely necessary but it is advisable as it prolongs the stability of the Stage II solution. (As a result the smudge suppressing action of the solution remains unimpaired over a longer period of time).

Stage II – Main sealing with water + **Anodal SH-1**

Temperature	Time	Sealing medium	pH*
minimum 96°C/205°F	2 - 3 min/μm	deionized water + 2 ml/l <b>Anodal SH-1</b>	5.7 ( ± 0.3)

\* Adjustment with ammonia or acetic acid.

## METHOD B

Stage I – Presealing (nickel impregnation) with **Sealing Salt AS**

Temperature	Time	<b>Sealing Salt AS</b>	Sealing medium	pH*
70°C/158°F or 80°C/176°F	1 min/μm 0.5 min/μm	5 g/l	deionized <u>or</u> not too hard water	5.7 ( ± 0.3)

Intermediate rinsing is not essential but it is advisable as it prolongs the stability of the Stage II solution. (The entrainment of **Sealing Salt AS** into main sealing solution would encourage the formation of drying films).

Stage II – Main Sealing with boiling water or with steam

Temperature	Time	Sealing medium	pH*
minimum 96°C/205°F	3 min/μm	- <i>deionized water</i> (preferably buffered with ammonium acetate <u>or</u> - <i>steam</i>	5.7 ( ± 0.3)

\* Adjustment with ammonia or acetic acid.

*Explanatory notes on two-stage sealing*

*Functions of **Sealing Salt AS***

**Sealing Salt AS** contains nickel acetate, a dispersing agent and a buffer substance. It performs the following functions:

- 1) Impregnation of the coating with nickel (precipitation of mixed aluminium/nickel hydroxides in the pores)
- 2) Prevention (by the dispersing component) of surface film formation
- 3) Regulation of the pH of the sealing solution

Functions 1 and 2 are dependent on the treatment time, the temperature and the **Sealing Salt AS** concentration of the solution.

*Method A*

In this method **Sealing Salt AS** does not have to exercise smudge suppressing action because an antismudge agent is present in the main sealing solution. The sole purpose of presealing is to deposit the nickel uniformly in the pores so as to ensure that the quality of seal will be satisfactory. Experimental applications have shown that optimum nickel salt impregnation is reached after only a short treatment time.

*Method B*

In method B, however, the treatment time is longer (0.5 – 1 min/m) in order to get an adequate smudge suppressing action because the main sealing phase is effected with water only, without any addition. This has no influence on the quality of the sealed coating.

Method B has one disadvantage: depending on the state of the two solutions the surface may not be left so clean as it is in Method A.

The great advantage of Method B is that it allows steam to be used as an alternated medium in the main sealing phase.

### 3.3 **Aftertreatment**

After one stage sealing the work should be thoroughly rinsed at once, as is normal in nickelacetate sealing, before it is dried.

On parts with a bright, specular finish sealed with **Sealing Salt AS** a faint deposit may be visible after drying. If this is so a short acid aftertreatment is advisable, e.g. with dilute nitric acid (1:1 with water), for a few seconds at about 20°C/68°F. This is followed by a further rinse.

Two stage sealing dispenses with the need for an aftertreatment as it precludes deposit formation in drying.

## 4. **Sealing bath maintenance**

### 4.1 **Bath analysis**

The following method permits rapid control of the **Sealing Salt AS** concentration in the bath.

#### ***Principle***

Determination of the **Sealing Salt AS** concentration by complexometric titration.

#### ***Reagents***

- *0,1 M-EDTA (ethylenediamintetraacetic acid)*  
Dissolve 37.2 g ethylenediamintetraacetic acid-disodium salt (MW 372) in water and dilute to 1000 ml in a measuring flask. Instead of the solid EDTA a ready-made concentrate can be used which is adjusted for the preparation of 1 litre of a 0.1 M-solution, e.g..Titriplex III, No. 9992 (Merck)
- *Indicator*  
1 g Murexid (purpuric acid, ammonium salt) and  
100 g sodium chlorid. Mix and grind as finely as possible.
- *Ammonia solution, pure, approximately 25 %.*

#### ***Titration***

Pipette 100 ml of the **Sealing Salt AS** solution clarified by filtration into a 250 ml glass beaker with a stirrer. Add a pinch of indicator (about 50 mg) and 10 ml of ammonia solution. Drop into the initially slightly turbid, brown-yellowish solution 0.1 M-EDTA from the burette until the now clear solution shows a purple colour (with an excessive amount of EDTA the colour will be blue-violet).

Consumption: a ml of 0.1 M-EDTA.

#### ***Calculation***

Concentration of **Sealing Salt AS** in g/l = 0.44 a

## 4.2 Strengthening the baths

Cost considerations mean that sealing baths remain in use over a period of several weeks or months. For consistent sealing results the concentration must be checked at regular intervals and the bath strengthened as indicated by the analysis.

### ***Bath life/Bath replacement:***

Owing to the gradual impoverishment of the sealing bath in active substances and the entrainment of foreign ions with interfering action, after a longer spell in use the concentration ratio is shifted. As a result the effectiveness of the bath in preventing smudging diminishes, even when its concentration is checked and strengthened at regular intervals. **Sealing Salt AS** baths must therefore be replaced from time to time.

An optimized process technology together with efficient bath control will, however, prolong the serviceability quite considerably and necessitate less frequent bath replacement.

In the two stage sealing process the lower temperature (70°C/158°F instead of 96°C/205°F) prolongs the working life of the **Sealing Salt AS** bath.

### ***Consumption***

The consumption of **Sealing Salt AS** is calculated from the amount absorbed by the anodic film, the amount carried out of the bath, and the amount of active ingredients inactivated by the introduction of foreign substances. The results of our tests show that the average consumption of **Sealing Salt AS** in the one-stage process per unit area of **Sealing Salt AS** of sealed surface is as follows:

- adsorption by the film	0,4	g/m <sup>2</sup>
- amount carried off	0,3	g/m <sup>2</sup>
- consumption for strengthening	<u>7</u>	<u>g/m<sup>2</sup></u>
additions	8	g/m <sup>2</sup>

## 4.3 Measures for clean working

A haze or solid deposit on sealed aluminium is generally due to bath turbidity or drying residues.

### ***Bath turbidity***

Freshly prepared **Sealing Salt AS** baths are clear, pure solutions. In use they become contaminated by precipitates and grow turbid. If not removed these contaminants form undesirable deposits on the sealed surface.

The effect can be due to the following factors:

- an over-high pH of the sealing bath; at pH values above 6 nickel acetate may be converted into insoluble nickel hydroxide
- hard water
- entrained impurities

The following precautions minimize this danger:

- maintenance of the pH
- thorough rinsing of the anodized, dyed or undyed work prior to sealing to prevent as far as possible the introduction of foreign substances; this also helps to delay turbidity
- filtration to clarify the bath and so prevent surface deposit formation.

### **Surface deposits**

Sealing baths which like those of **Sealing Salt AS** are of relatively high solids content tend to form a deposit on dried parts, especially if these are of large size and the holding and carry-over time are lengthy. These deposits cannot be completely removed in the subsequent rinsing bath. In two-stage sealing no such deposits are formed on drying. The following measures prevent deposit formation:

- wet-on-wet rinsing immediately after sealing
- washing down with fan jets, spurt or spray pipes using a minimum of water (preferably deionised) directly above the **Sealing Salt AS** bath; the resulting increase in the bath volume is offset by evaporation.

### **Filtration of sealing baths**

Filtration clears the bath of precipitates with interfering action, which consist of

- aluminium and nickel compounds and
- salts causing hardness from the plant water supply.

A bath set at the normal concentration of 4 g/l **Sealing Salt AS** and used for 4 weeks under production conditions contains 0.1 to 1 g/l of precipitates with particles greater than 15 µm in size.

### **Filter types and systems**

Filtration can be carried out batchwise or, preferably, continuously (filter pore size 8 – 15 µm).

#### *Discontinuous (batchwise)*

In this method filter aids, for example commercial products based on kieselguhr such as "Hyflo", can be used to facilitate filtration and counteract blocking of the filters.

#### *Continuous*

In the industry, compact pressure filters consisting of a filter pump with an electric motor, a filtering vessel and connecting pipes between pump, filter and vessel are giving good satisfaction. For a **Sealing Salt AS** bath of, say, 10,000 l volume, filtration rates of about 500–1000 l/h are necessary. It has been found advantageous to start operating the filter apparatus from the moment a fresh bath is put into use. The service life of the filter is limited and the filtering elements must be replaced from time to time. Deep-bed filtration, e.g. with wound candle filters, prolongs the service life.

## **5. Properties of the coating**

A valuable property of **Sealing Salt AS** is that it largely prevents the sealing quality from being impaired by foreign ions in the bath.

The impurities present in normal industrial water (neutral salts and salts causing hardness), together with the foreign substances such as sulphates that are carried into the bath, can lead to serious deterioration in the sealing quality. Water sealing therefore requires water of high purity, failing which the water must be purified, e.g. by demineralization in ion exchangers, and checked regularly during use.

When **Sealing Salt AS** is used the requirements for the water quality are less severe than for water sealing. Sealing with this nickel salt products works out more expensive than sealing in water alone.

But the use of **Sealing Salt AS** invariably results in excellent sealing quality even when the water is deficient in quality.

## 6. Sealing Salt AS sealing compared with other sealing processes

### **Nickel salts**

Nickel acetate and nickel formate produce the same quality of sealing as **Sealing Salt AS**. However, a serious disadvantage is the sealing deposit ("bloom"), which often can only be rubbed off with great difficulty. Nickel sulphate sealing baths cause many aluminium dyestuffs to bleed heavily; they lower pH stability and the sealing quality is poorer than with nickel acetate or **Sealing Salt AS**.

### **Water**

This is a simple, economic method, but the disadvantage is that many aluminium dyestuffs bleed more or less heavily, even when the bath is held at the boil. For fully satisfactory sealing quality and corrosion resistance, deionized water must be used. Hard or otherwise unsuitable water impairs the sealing quality and promotes chalking formation. This sealing medium must be buffered to avoid severe pH fluctuations.

**Sealing Salt AS** is buffered, resulting in a more stable bath pH.

### **Steam**

In this method the aluminium is treated with steam in an open or closed vessel. Dyeings bleed less than in water, but a pronounced sealing deposit forms which is almost impossible to suppress.

### **Dichromate**

Dichromate sealing produces films with particularly high corrosion resistance. However, dichromate causes a marked yellow-greenish discoloration.

## 7. Treatment of spent baths

Spent **Sealing Salt AS** baths contain metals, notably nickel, which pollute waste waters. Ecological considerations have led to increasingly stringent regulations for the control of industrial effluents which make it necessary to remove these metals from spent **Sealing Salt AS** baths before they are conducted into mechanical/biological purification plants or the drainage system. Chemical precipitation and mechanical separation of the metals before the bath flows into other waste waters can best be accomplished on the lines recommended in the technical information bulletin "**The removal of noxious metals from spent baths**".

Many of their dyestuffs, pigments and chemicals are patented by Clariant in numerous industrial countries.

® Trademark of Clariant registered in numerous countries.

®\* Trademark registered in numerous countries and licensed to Clariant.

®+ Other Manufacturer's registered trade mark

The signs ®, ®\* and ®+ appear only at the first mention of the product.

The information and recommendations presented here were compiled with the utmost care, but cannot be extended to cover every possible case. They are intended to serve as non-binding guidelines and must be adapted to the prevailing conditions.