Anodal® SH-1 Liquid

Sealing smudge suppressant

During the sealing of anodized aluminium in boiling water, the pores of the anodic coating swell at the outer surface while the pores are pressed together and close. On the surface a disturbing sealing deposit is thus formed called a smudge or smut.

Anodal SH-1 Liquid is used to obtain smudge-free hot water sealing of anodized aluminium and produces excellent sealing qualities.

Anodal SH-1 Liquid was developed by Clariant and patent application filed in the principal industrial countries.

1. Properties

Appearance	clear,aqueous solution
Storage stability	at least 3 years in closed original containers at 0-40°C. Due to the influence of light, the solution may turn slightly yellowish-brown, but its effectiveness is not impaired.
Dilutability	dilutable with water in any proportion
Ecotoxicological data	see Safety Data Sheet.

2. Scope of application

Anodal SH-1 Liquid is used as an additive in the hot water sealing of colourless, integral and electrolytically dyed anodized aluminium to prevent the formation of sealing deposits (smut).
Anodal SH-1 Liquid can also be used for the smudge-free sealing of coatings which are adsorptively dyed with organic dyes or combination dyeings such as the "Sandalor" (registered trademark of Interoxyd AG, Altenrhein, Switzerland) dyeings produced with **Aluminium** and **Sanodal**® dyes. In the latter case, however, a two-stage sealing method is recommended, i.e. presealing with **Sealing Salt ASL** followed by the main sealing step with water and an addition of **Anodal SH-1 Liquid** (see Technical Information bulletin "**Sealing Salt ASL**").

3. Application conditions

Substrate

Oxide coatings - preferably anodic - on aluminium or its alloys.

Applicable amounts

The applicable amount depends on the water quality, deionized water is preferable, and the type of oxide coating.
The following amounts are recommended as a guideline:

- for undyed coatings 1-2 ml/l **Anodal SH-1 Liquid**
- for dyed coatings 2-3 ml/l **Anodal SH-1 Liquid**.

Sealing temperature

at least 96°C, preferably boiling.

Sealing time

2 min/µm
3 min/µm for highest requirements.

pH

5.5 - 6.0

Anodal SH-1 Liquid has a buffering effect. However, to ensure the best possible sealing conditions, the pH value should be checked when setting the sealing bath and during its use, and adjusted as required by adding diluted ammonia or acetic acid.

4. Preparation, service and maintenance of the sealing baths

Sealing bath tanks

Use acid-resistant materials, preferably stainless steel.

Water quality

It is preferable to use deionized water for sealing. This ensures that best results will be obtained with respect to sealing quality and freedom from smut. Softened tapwater can also be used. However, the water should be free of sealing contaminants such as phosphate, fluoride and silicate ions, as these delay or even obstruct the sealing process, i.e. the closing of the pores. With tap water, drying deposits may form, due to the dissolved mineral salts in the water, even though there may be no sealing smut on the surface.

Preparing the sealing baths with deionized water which is free of sealing contaminants counteracts the formation of smut. When the sealed articles are lifted out of the bath, the water quickly runs off, thereby preventing deposits during drying in air.

Reinforcement of sealing baths

To avoid excessive concentrations, the recommended usage levels should be adhered to as far as possible. Since it is not possible to monitor this by chemical means, it is proposed as a practicable alternative to determine the yield of the bath once it has been set by measuring the amount of dm^2/l of smut-free surface and assigning this figure a value of 100%.

The yield (in dm^2/l) is ascertained in the laboratory by determining the number of electrolytically dyed (Sn) dark bronze panels with a surface area of 1 dm^2 each that can be sealed free of deposits in a bath volume of 500 ml, and converting the results to 1 l. If the sealing activity should fall below 50%, the bath must be reinforced by the corresponding amount of smudge suppressant. After reinforcing the bath, the sealing quality should be determined according to ISO 3210 ("Determination of the sealing quality by measuring the weight loss after immersion in a phosphoric-chromic acid solution with nitric acid pre-dip").

The sealing bath must be totally or partially renewed only when the test results of the sealing quality go beyond the fixed tolerance limits. This method of determination is naturally also suitable, with appropriate modifications, for testing and adjusting smudge suppresssant-containing sealing baths which are continuously renewed, for example with constant partial reinforcement by metering in the suppressant. At a given rate of production, the metering of the smudge suppressant can be set in a very simple manner using the method described. However, as with batchwise operating, both the yield of the sealing bath and the sealing quality should be tested by ISO 3210 at regular intervals.
Concerning overdosage of the product, see also **section 7**.

Drag-in of foreign ions

Anodal SH-1 Liquid does not precipitate in the presence of small amounts of dragged-in foreign ions such as Al, Ca, Sn and Ni ions, nor is it decomposed by hot pipes or plates.

Standing times of sealing baths

The life of the sealing baths depends mainly on the amount of material treated in them. The amounts of entrained anodization acid, dyebath residues and rinsing water impurities also have an influence on the standing times of sealing baths.

The pH of the sealing baths may alter due to entrained sulphuric or nitric acid as well as alkalis coming, for example, from the neutralization bath. In such cases correction is made according to **section 3**.

For long standing times it is therefore recommended to rinse the aluminium articles as thoroughly as possible before sealing, preferably by immersion **and** spraying off.

A continuous oeprating procedure, i.e. continuous partial reinforcement by metering in the smudge suppressant with a metering pump, for example, has a particularly favourable effect on the freedom from smut and lifetime of the sealing bath.

5. Aftertreatment

If deionized water is used for setting the baths, it is not necessary to give an aftertreatment to aluminium parts sealed with **Anodal SH-1 Liquid** .

If tapwater is used, however, drying residue may form on the aluminium surface (see **section 4**), but this can be easily removed with water.

6. Sealing quality

The values from shortterm tests given below apply to the following conditions:

Material	AlMg 1
Anodization	normal D.C. sulphuric acid method 190(± 5) g/l H_2SO_4 5-10 g/l Al, 19°C, 15-16 V, 50 min Coating thickness 20 ± 2 µm
Dyeing	electrolytic, Sn sulphate, 15 V, 5 min, dark bronze
Sealing	**Anodal SH-1 Liquid** 2 ml/l 3 min/µm, boiling temperature, pH 5.5 - 6.0, deionized.

Admittance Y_{20} (according to ISO 2931)

Anodal SH-1 liquid	Substrate	Y_{20}, µS
without	undyed	15
	dyed	35
with	same values as above	

Weight loss after immersion in chromic-phosphoric acid with nitric acid pre-dip (ISO 3210)

Anodal SH-1 flüssig	Substrate	Weight loss, mg/dm^2
without	undyed	7
	dyed	8
with	same values as above	

Dye spot test (according to ISO 2143: Estimation of loss of absorptive power - Dye spot test with prior acid treatment)

Anodal SH-1 flüssig	Substrate	Intensity of the stain
without	undyed	0-1
with	same values as above	

Corrosion by SO_2 (Kesternich test according to DIN 50018, 2.0 S, 6 cycles)

Anodal SH-1 Liquid	Substrate	Corrosion
without	undyed	slight attack
	dyed	slight attack
with	same values as above	

Corrosion by spraying with common salt solution, (CASS test according to ISO 3770 "Metallic coatings - copper accelerated acetic acid salt spray test", 24h)

Anodal SH-1 flüssig	Substrate	Corrosion
without	undyed	Rate * 9-10
	dyed	Rate * 9-10
with	same values as above	

* according to ISO 4540: Metallic coatings - Coatings cathodic to the substrate - Rating of electroplated test specimens subjected to corrosion test"

7. Overdosage

As with all smudge suppressants for hot water sealing, excessive concentrations of **Anodal SH-1 Liquid** cause a slight increase in the weight loss values after immersion in chromic-phosphoric acid with nitric acid pre-dip (ISO 3210).

For example, if 5 times the recommended application concentration is present in the sealing bath, the following values are obtained:

Anodal SH-1 liquid	Substrate	Weight loss, mg/dm^2
10 ml/l	undyed	15
	dyed	17

Although most of these values are still within the tolerance limits of the ISO standard, overdosage of the product should be avoided.

8. Suitability for two stage sealing

Anodal SH-1 Liquid is added in the main sealing step (stage 2) in this method.

Details on the two stage sealing method are contained in the Technical Information bulletin "Sealing Salt ASL.

9. Methods for determining the amount of Anodal SH-1 Liquid in the sealing bath

The concentration of the smudge suppressant still present in the sealing bath can be determined by two methods.

Anodal SH-1 Liquid can be measured either **visually by colorimetry** or **spectrophotometrically**. The latter method is somewhat easier to use and produces slightly more precise results. However, the accuracy of the values determined by the visual colorimetric method are fully adequate.

9.1 Analysis specification for colorimetric determination

Principle

In the pH region of 2-3, trivalent iron ions react with the active component of smudge suppressant **Anodal SH-1 Liquid** to form a coloured complex, colouring the bath solution red-violet. The concentration of **Anodal SH-1 Liquid** in the sealing bath can be determined and the required further addition of the product caculated in a simple manner by visual colorimetric comparison of the colour intensity of the bath solution with that of a known concentration of **Anodal SH-1 Liquid** (comparative solution).

Reagents

Use only distilled or demineralized water to prepare the solutions. Mix the solutions thoroughly.

Solution A

Dilute 3.0 ml **Anodal SH-1 Liquid** with about 800 ml water, adjust to pH 2.5 -3.0 by dropping in 5% nitric acid and fill up with water to a volume of 1000 ml.

Solution B

Dissolve 3.0 g iron chloride ($FeCl_3$) and 0.8 g ammonium hydrogen fluoride ($NH_4HF_{2)}$ in about 80 ml 5% nitric acid and then fill up with 5% nitric acid to a volume of 100 ml. If the solution is not entirely clear, carry out a clarifying filtration through a folded filter.

Attention! Solution B etches glass and can be stored anly in plastic containers. Avoid contact with the skin and eyes!

Comparative solution

Place 45 ml *Solution A* in a 50 ml measuring cylinder, mix in 2 ml *Solution B* and fill up with water to a volume of 50 ml.

Analysis

Take about 100 ml of the sealing bath solution under test and adjust the pH to 2.5 -3.0 with a few drops of 5% nitric acid. Place 45 ml of this solution in a 50 ml measuring cylinder, mix with 2.0 ml *Solution B* (colour reagent) and fill up with demineralized water to a volume of 50 ml. Mix thoroughly. After allowing both the comparative and the bath solution to stand for at least 15 min for complete development of the violet colour, hold both measuring cylinders side by side over a white background (if possible) and make a visual comparison of the colour intensity of the two samples. Pour off the comparative solution until the colour intensity of both samples is the same. Read of the **ml** of this remaining comparative solution and multiply it by a factor of **0.06**; this directly gives the bath concentration of **Anodal SH-1 Liquid** (ml/l) to be determined.

This procedure should be repeated once or twice.

y = x . 0.06

x = ml comparative solution

y = ml/l **Anodal SH-1 Liquid** in the bath.

The colour intensity of the bath sample must not be greater than that of the comparative solution. However, if this is the case, the analysis should be carried out with less than 45 ml sealing bath solution. When using the volumes of sealing bath solution given in the table below, the bath concentration of **Anodal SH-1 Liquid** calculated must be multiplied by the corresponding correction factor given in the table.

Sealing bath solution	Correction factor
35	1.28
25	1.80
15	3.00
10	4.50

Strengthening the sealing bath

The amount of **Anodal SH-1 Liquid** which must ber added to strengthen the sealing bath to the desired working concentration is given by the difference between the nominal concentration and the bath concentration found.

Remarks

- Prolonged, simultaneous standing of the red-violet bath and comparative solutions (up to 2 h) does not affect the analysis results.
- With sealing baths that have been in use for longer periods it may happen that the colour of the bath sample turns out to be somewhat redder than the comparative solution. This change in hue is due to impurities present in the sealing bath. However, any resulting uncertainties in assessing the comparative and the bath solution can rapidly be resolved by using the method of analysis described.

9.2 Analysis specification for spectrophotometric determination

Principle

The extinction value (diminished light intensity) of a bath sample is compared with that of an **Anodal SH-1 Liquid** solution of known concentration (comparative solution). The comparison of these extinction values permits calculation of the concentration of **Anodal SH-1 Liquid** in the sealing bath and thus the amount of product necessary to strengthen the bath to working concentration.

Reagents

Use only distilled or demineralized water to prepared the solutions. Mix the solutions well.

Solution A

Place 2.0 ml Anodal SH-1 Liquid in a beaker and fill up with water to a volume of 1000 ml.

Solution B

Dissolve 8.0 g ammonium hydrogen fluoride (NH_4HF_2) in approx. 800 ml water. Then add 50 g L(+)-tartaric acid ($C_4H_6O_6$), dissolve and fill up with water to a volume of 1000 ml.

Caution: Solution B is corrosive to glass and can be stored only in plastic containers. Avoid contact with the skin and eyes!

Comparative solution C_0

Place 45 ml Solution A in a 50.0 ml measuring flask, add 2 ml Solution B and then fill up with water to a volume of 50 ml.

Bath solution C

Place 45.0 ml of the bath solution in a 50.0 ml measuring flask, Then add 2.0 ml of Solution B and fill up with water to 50.0 ml.

Analysis

Determine the respective extinction values (E_0 and E) of the comparative solution (C_0) and the bath solution (C) at a wave length of 319 nm.
Since at approximately the same dilution the extinction values vary in proportion to the concentration of **Anodal SH-1 Liquid,** the following equation is obtained:

$$C = \frac{C_0}{E_0} \cdot E \quad \text{or} \quad C = \frac{2}{E_0} \cdot E$$

This equation can be used to calculate the bath concentration C in ml/l **Anodal SH-1 Liquid.**

Strengthening the sealing bath

The amount of **Anodal SH-1 Liquid** necessary to strengthen the sealing bath to working concentration is given by the difference between the nominal concentration of the product and the bath concentration found by analysis.

Remarks

As compared with the colorimetric method, the spectrophotometric method is easier, quicker, and leads to more precise results.