ALPHA CHEMICALS PTY LTD

SUPPLIERS OF INDUSTRIAL CHEMICALS

A.B.N. 29 001 174 741

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Product Information Sheet

ALPHACOL IG

Alphacol IG brand of Ferric Ammonium Oxalate is used in aqueous solution to product light fast gold shades on anodised aluminium.

Properties:

Appearance:	Emerald green crystals
pH 3% solution:	4.5 +/- 1
Iron as Fe:	13.0%
Oxalate as (C_2O_4):	61.7%

Method of Application:

Preparation and operation of the bath.

The tanks for the immersion bath are best constructed from 316 Mo stainless steel. However tanks with suitable acid resistant claddings may be used but it is essential the cladding does not develop any leaks.

The concentration of the bath depends on the shade of gold required.

Alphacol IG	
Medium to dark shades	20 – 30 gpl
Light shades	10 – 20 gpl

Colour is also affected by the temperature of the bath with a higher temperature favouring the darker shades. As a guide the following temperature ranges can be used for the concentration given above.

Medium to dark shades	40 – 55 °C
Light shades	30 – 40 °C

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The immersion time will also vary with the shade required. For practical considerations the factors mentioned above should be controlled to give an immersion time of 2 – 5 minutes.

In preparing the bath de-ionised or very soft water is preferable. However if hard water is used an addition of oxalic acid should be made to compensate for the precipitation of alkaline earth oxalates. This addition should be of the order of 2 – 5 gpl of oxalic acid.

When preparing a fresh bath the water should be heated to operating temperature. Then oxalic acid should be added with agitation followed by the required amount of Alphacol IG. Agitation can be achieved by bubbling air through the solution by stirring or by circulation.

Agitation is also desirable to maintain uniformity of bath composition and also to mix in additions to the bath. For this purpose oxalic acid and Alphacol IG should be dissolved in hot water prior to the addition.

The pH of the bath should be adjusted to be in the range 4.0 - 5.0.

Control of the bath

Ferric oxalate is photosensitive and under the effect of light is reduced to Ferrous oxalate. This reduces the colouring power of the bath which will become turbid and yellowish as ferrous oxalate is insoluble and yellow. When the bath is not in operation it should be covered to prevent the access of light. However if this photoreaction occurs it can be reversed by the addition of hydrogen peroxide.

During the operation the pH can drift upwards and at pH6 or over the ferric oxalate is hydrolysed to produce a gelatinous precipitant of ferric hydroxide. The effect of this hydrolysis can be reversed by the addition of oxalic acid.

The methods for analysing the bath are given below and if the bath is maintained as recommended it should last for at least 12 months.

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ANALYTICAL METHODS – 30 gms per litre

TYPICAL CONCENTRATION:

A. REAGENTS.

1) 0.1N Potassium Permanganate.
2) 20% w/w Sulphuric acid or Concentrated Shlphuric Acid (98%).
3) 0.1 M E.D.T.A. (disodium ethylene diamine tetra-acetic acid).
4) 1:1 Ammonia Solution (1 vol conc. NH_3 mixed with 1 vol water).
5) Indicator solution: 5% Sulphosalicylic acid in water.

B. DETERMINATION OF OXALATE

Pipette exactly 10 ml of the bath solution into 500ml flask containing approx. 100ml distilled water and 10ml of 20% sulphuric acid (or 2ml Conc. Sulphuric Acid). Heat the solution to 70 – 90°C and titrate with 0.1N potassium permanganate until a permanent faint pink end point persists.

Let the titre be "**a**" mls.

C. DETERMINATION OF IRON

Cool the solution from B to room temperature and, using 1.1 ammonia adjust the pH to 2.5 (2.2 to 2.7) using a pH meter.

Add approx. 2ml of indicator solution (turns red) and titrate with 0.1M EDTA until the dull red colour disappears (the solution becomes yellow). Add 1ml of indicator solution and if the red colour reappears continue the titration until it disappears.

Let the titre be "**b**" mls.

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D. CALCULATIONS

1) Ferric Ammonium Oxalate concentration is:

b x 4.28 (g/l)

2) Oxalate concentration is:

a x 0.7133 (g/l)

3) Solution balance:

For a stable solution the ratio (R) must be in the range

$$R = \frac{\mathbf{a}}{\mathbf{b}} = 3.0 \text{ to } 4.2$$

and preferrably R = 3.3

4) Solution adjustment:

4.1 The concentration of ferric ammonium oxalate is adjusted by additions as required by (1) add (30-b) x 4.28 (g/l)
This adjustment must precede 4.2

4.2 The oxalate balance is adjusted by adding oxalic acid dihydrate (as a solution in water) to the bath according to the table:

R	Addition of Oxalic Acid dihydrate
$\frac{\mathbf{a}}{\mathbf{b}}$	g/l
2.8	4.4
2.9	3.5
3.0	2.7
3.1	1.8
3.2	0.9
3.3	nil
3.3	nil

4.3 Adjust the pH of the bath into the range 4.0 to 5.0 with ammonia or acetic acid as required. (It is best to estimate the amount of ammonia or acetic acid required by adjusting 2L of bath solution in the laboratory).

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E. TEST FOR FERROUS OXALATE

REAGENTS.

Potassium ferricyanide 5%.

Prepare from distilled/deionised water and store in opaque container.

Sulphuric Acid 20%

TEST METHOD

Add 2 drops of sulphuric acid 20% to 2ml approx. of ulfiltered dye solution and boil briefly.

The rapid appearance of a blue green colour indicates the presence of ferrous oxalate.

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F. TEST FOR HYDROGEN PEROXIDE

REAGENTS

Starch-potassium iodide paper.

TEST METHOD

If hydrogen peroxide has been added to the dye bath to regenerate ferrous oxalate a drop of the dye solution is placed on the indicator paper. A blue/violet colour indicates the presence of hydrogen peroxide.

REGENERATION OF FERROUS OXALATE WITH HYDROGEN PEROXIDE

If the appearance of the bath and the test for ferrous oxalate indicates the presence of ferrous oxalate this can be regenerated by the addition of hydrogen peroxide and oxalic acid. In theory the oxidation of ferrous oxalate with hydrogen peroxide preceeds as follows:

$$2\ Fe\ (C_2O_4) + H_2C_2O_4 \quad => \quad Fe_2\ (C_2O_4)_3 + 2\ H_2O$$

However the oxidation with hydrogen peroxide is not 100% efficient as some will be decomposed. Also the addition of oxalic acid should be made bearing in mind the comments made under item 4.2 and the pH adjusted as required with ammonia or acetic acid.

A practical method of assessing the quantity of hydrogen peroxide to add is to take a 5 litre aliquot of the thoroughly mixed dye solution and add hydrogen peroxide diluted to approx 3 - 5% w/w strength with stirring until a slight excess of hydrogen peroxide exists.

From this calculate the quantity required for the bath. When adding hydrogen peroxide to the bath dilute to approx 3 - 5% w/w strength and add over the bath surface with maximum agitation.

Analyse the solution and make any adjustments to Alphacol IG or oxalate concentration as required.