INTRODUCTION

TURPINAL® products are high purity grades of etidronic acid and sodium etidronate which are suitable for use in cosmetics or any applications requiring a very low metal content. The high purity of TURPINAL® is guaranteed by thorough quality control procedures (including testing for microbiological contamination). Moreover, the certificate of analysis accompanying each batch contains detailed information covering chemical purity and trace metal content.

TURPINAL® sl and TURPINAL® 4NL are respectively the acid form and the tetra sodium salt of etidronic acid. They are colourless to slightly yellow liquids with a neutral odour. TURPINAL® 4NP is the tetrasodium powder form of etidronic acid.

These products can be mixed with water in all ratios and have a strong complexing capacity in relation to polyvalent cations. The excellent complexing capacity for metals is exploited for the stabilization of H₂O₂-containing preparations and other preparations with active oxygen. The product is also suited for application in mercaptan-containing formulations and enhances the colour stability of bar soaps. It is recommended that TURPINAL® be added during production with a concentration of 0.1 – 2% with reference to the finished preparation.

Properties and Applications

The chelating properties of TURPINAL® products make them outstanding candidates for the chelation of metal ions, in particular the transition metals (Fe, Cu, Mn, Zn) and the water hardness ions (Ca, Mg).

Stability constants of etidronic chelates have been measured and the approximate amount of free ion have been calculated:

<table>
<thead>
<tr>
<th>METAL</th>
<th>LOG K</th>
<th>APPROXIMATE % CHELATED</th>
<th>FREE METAL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca**</td>
<td>6.5</td>
<td>99.99997</td>
<td>3 x 10⁻⁵</td>
</tr>
<tr>
<td>Mg**</td>
<td>4.5</td>
<td>99.997</td>
<td>3 x 10⁻⁵</td>
</tr>
<tr>
<td>Fe**</td>
<td>21.6</td>
<td></td>
<td>2.5 x 10⁻⁹</td>
</tr>
<tr>
<td>Cu**</td>
<td>6.4</td>
<td>99.9996</td>
<td>4 x 10⁻¹⁰</td>
</tr>
<tr>
<td>Mn**</td>
<td>6.9</td>
<td>99.9999</td>
<td>1.2 x 10⁻⁶</td>
</tr>
<tr>
<td>Zn**</td>
<td>10.6</td>
<td>99.9999</td>
<td>2.5 x 10⁻⁹</td>
</tr>
</tbody>
</table>

In most cases, stability constants greater than 5 to 6 are not needed for practical removal of unwanted metal ions. The high chelation strength of TURPINAL® allows to limit, or even prevent, the precipitation of Iron hydroxide. Copper also is very strongly chelated by TURPINAL® and the formation and precipitation of copper oxide can often be significantly reduced or sometimes totally prevented.

To prevent calcium scaling and precipitation, it is not always necessary to work with stoechiometric chelant concentrations. Indeed, in many instances, calcium scale formation can also be prevented with sub-stoechiometric amounts of TURPINAL®. This property is known as “threshold effect”.

Owing to these chelating properties and threshold effect, and added to its chemical stability in oxidizing environment, TURPINAL® can bring substantial benefits in many application fields and more specifically in the cosmetics industry.

The toxicological properties of etidronic acid have thoroughly been tested, and the products are allowed by the EU in hair preparations up to 1.5% active acid, and in bar soaps up to 0.2% active acid concentration.

These properties make TURPINAL® products ideal chelants or additives in the following applications:

- In the stabilization of peroxide bonds, in alkaline media specifically.
- As a chelant in hair cosmetics containing mercaptan groups, i.e. thiglycolic acid, thiocetic acid.
- As a means of limiting hair damage caused by bleaching or colouring.
- In the stabilization of bar soaps, where TURPINAL® is used as antioxidant and prevents discolouration and rancidity, in particular under the influence of day light.
- In the stabilization of fragrances, which would otherwise lose their strength over time during storage of cosmetic formulations.

**Peroxide stabilization**

Aqueous solutions of hydrogen peroxide were prepared at pH 10 and the stability studied at 20°C for 50 days. The solutions were made of:

<table>
<thead>
<tr>
<th>Component</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>H₂O₂ (100%)</td>
<td>10</td>
</tr>
<tr>
<td>Turpinal® 4NL (active acid)</td>
<td>1.5</td>
</tr>
<tr>
<td>Potassium pyrophosphate (buffer)</td>
<td>10</td>
</tr>
<tr>
<td>Water</td>
<td>up to 1000 ml</td>
</tr>
</tbody>
</table>

The loss of H₂O₂ was measured over time and the results are reported hereafter:

<table>
<thead>
<tr>
<th>Time</th>
<th>Blank</th>
<th>EDTA</th>
<th>Turpinal® 4NL + EDTA</th>
<th>Turpinal® 4NL</th>
</tr>
</thead>
<tbody>
<tr>
<td>11 days</td>
<td>25%</td>
<td>3%</td>
<td>5%</td>
<td>5%</td>
</tr>
<tr>
<td>25 days</td>
<td>53%</td>
<td>5%</td>
<td>10%</td>
<td>10%</td>
</tr>
<tr>
<td>50 days</td>
<td>93%</td>
<td>9%</td>
<td>90%</td>
<td>90%</td>
</tr>
</tbody>
</table>

The amount of TURPINAL® required to stabilize peroxide bonds typically varies between 0.05 and 0.5%.

The key advantage of TURPINAL® is that the oxidative environment does not alter the chelation properties of TURPINAL®. This contrasts with other chelants like EDTA, NTA or DTPA which lose their chelation power once oxidized into their N-Oxide form under the action of H₂O₂.

**Hair cold weaving and depilatory products**

Mercaptans, such as thioglycolic acid or thiolactic acid and their salts, are incorporated in preparations designed for cold waving of hair and in preparations for depilatory creams. Metal ions make the colour of these preparations unstable and adversely influence the aspect of the products.

TURPINAL®, however, can eliminate such inconveniences, thanks to its metal chelation power.

Although the addition of TURPINAL® can be carried out at any time, it is preferred to add it at an early stage of the process. The concentration of TURPINAL® should be 0.1 to 1.5% in the formulation.

**Prevention of hair damage during bleaching or dyeing**

In hair bleaching, dyeing or cold waving processes, several types of peroxides are typically used. Iron and other transition metals are known to bind on the hair protein structure. The presence of such ions catalyse the decomposition of hydrogen peroxide used in hair bleaching or dyeing. This results in local reinforcing of the oxidation effect of H₂O₂ or other peroxides and subsequently weakening of the hair structure.

This type of damage can easily be avoided if TURPINAL® is added to the formulation in order to chelate the transition metals. The recommended level of TURPINAL® is between 0.1 and 1.5%.

**Stabilization of bar soaps**

Soaps are alkaline salts of fatty acids, mainly oleic, stearic, palmitic, lauric and myristic acids. These acids are obtained from oils and fats of both vegetable and animal origins. The final properties are determined by the relative ratios of these acids and by the ratio of saturated to unsaturated chains. These soaps are formulated with a number of additives to optimize the final properties and ensure the stability of the product.

The main concern in formulating bar soaps is to prevent oxidation of the soap base, additives and the fragrance. Transition metals are known to catalyse oxidation of fatty acids and subsequently degrade the finished product.

It has been established that a level of 300 to 700 ppm of an appropriate chelant is optimum to produce a good whiteness in the soap. TURPINAL® 4NL is the product of choice for this application.

Figure 1 illustrates this benefit. The graph shows the reduction of the soap whiteness over time at a storage temperature of 45°C, with respectively 300ppm TURPINAL® 4NL, EDTA and a 1:1 blend of TURPINAL® 4NL and EDTA. The latter giving the best balance of cost/benefit.

**FIGURE 1: EFFECT OF TURPINAL® ON BAR SOAP STABILITY**

A visual evaluation of the benefits of TURPINAL® in the stabilization of bar soaps is further illustrated by the following pictures. The results show the discoloration of the soaps after 13 weeks storage at 45°C.
### Storage and transportation

In sealed original containers and at temperatures below 30°C and above 5°C, TURPINAL® 4NL and TURPINAL® SL remain stable for at least one year. For transport and storage, only containers made of V4 A-steel and polyethylene are suitable on account of the corrosive effect. Pipes and pumps for company internal supply should also be made of one of these materials. Solutions of TURPINAL® 4NL and TURPINAL® SL are insensitive to temperature variations. However, the solutions should be protected against frost in order to prevent crystallization of the product. In the event of crystallization TURPINAL® 4NL and TURPINAL® SL are redissolved at higher temperatures with strong mixing without a change in the product characteristics.

### Toxicological profile

The human health safety properties of TURPINAL® have been extensively studied. The product showed low toxicity in both acute and chronic testing. No mutagenic or sensitizing properties have been reported. Etidronic acid and its sodium salt are cleared by the EU Scientific Committee on Cosmetics and Non-Food consumer products.

The conditions of use are specified in the Cosmetics Directive 76/768/EEC, Annex III, Part I, item 53. The concentrated products (as supplied) should be handled with care as recommended in Italmatch Chemicals S.p.A. (MSDS). Detailed information can be obtained upon request from Italmatch Chemicals S.p.A.

### Packaging

- **TURPINAL® SL**: 25 kg, 75 kg, 290 kg drums
- **TURPINAL® 4NL**: 25 kg, 75 kg, 270 kg drums
- **TURPINAL® 4NP**: 25 kg paper bags

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**Table 1: Chemical and Physical Properties**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>TURPINAL® 4NL</th>
<th>TURPINAL® SL</th>
<th>TURPINAL® 4NP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular weight</td>
<td>294 g/mol</td>
<td>206 g/mol</td>
<td>294 g/mol</td>
</tr>
<tr>
<td>Density at 20°C in g/cm³</td>
<td>1.29-1.33</td>
<td>1.4-1.5</td>
<td>-</td>
</tr>
<tr>
<td>Bulk density in g/cm³</td>
<td>-</td>
<td>-</td>
<td>0.7-0.9</td>
</tr>
</tbody>
</table>

**Table 2: Microbiological Analysis**

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>TURPINAL® 4NL</th>
<th>TURPINAL® SL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bacteria</td>
<td>&lt; 1 cfu/10 ml</td>
<td>&lt; 1 cfu/10 ml</td>
</tr>
<tr>
<td>Yeasts</td>
<td>&lt; 1 cfu/10 ml</td>
<td>&lt; 1 cfu/10 ml</td>
</tr>
<tr>
<td>Molds</td>
<td>&lt; 1 cfu/10 ml</td>
<td>&lt; 1 cfu/10 ml</td>
</tr>
<tr>
<td>E. Coli</td>
<td>Absent in 10 ml</td>
<td>Absent in 10 ml</td>
</tr>
<tr>
<td>Ps. Aeruginosa</td>
<td>Absent in 10 ml</td>
<td>Absent in 10 ml</td>
</tr>
<tr>
<td>Staph. aureus</td>
<td>Absent in 10 ml</td>
<td>Absent in 10 ml</td>
</tr>
</tbody>
</table>

**Table 3: Products Specifications**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>TURPINAL® 4NL</th>
<th>TURPINAL® SL</th>
<th>TURPINAL® 4NP</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Description</strong></td>
<td>Clear, colourless to pale yellow solution, free from solids</td>
<td>Clear, colourless to pale yellow solution, free from solids</td>
<td>White powder</td>
</tr>
<tr>
<td><strong>Active Substance</strong></td>
<td>29 – 31 % Tetrasodium etidronate</td>
<td>58 – 61 % Etidronic acid</td>
<td>83 – 88 % Tetrasodium etidronate</td>
</tr>
<tr>
<td><strong>pH Value</strong></td>
<td>10-12</td>
<td>&lt; 2*</td>
<td>-</td>
</tr>
<tr>
<td><strong>Phosphorous acid</strong></td>
<td>1 % max</td>
<td>2 % max</td>
<td>2.5 % max</td>
</tr>
<tr>
<td><strong>Arsenic</strong></td>
<td>&lt; 2 ppm</td>
<td>&lt; 2 ppm</td>
<td>&lt; 3 ppm</td>
</tr>
<tr>
<td><strong>Cobalt</strong></td>
<td>&lt; 1 ppm</td>
<td>&lt; 1 ppm</td>
<td>&lt; 2 ppm</td>
</tr>
<tr>
<td><strong>Chromium</strong></td>
<td>&lt; 1 ppm</td>
<td>&lt; 1 ppm</td>
<td>&lt; 2 ppm</td>
</tr>
<tr>
<td><strong>Cadmium</strong></td>
<td>&lt; 1 ppm</td>
<td>&lt; 1 ppm</td>
<td>&lt; 2 ppm</td>
</tr>
<tr>
<td><strong>Manganese</strong></td>
<td>&lt; 1 ppm</td>
<td>&lt; 1 ppm</td>
<td>&lt; 2 ppm</td>
</tr>
<tr>
<td><strong>Copper</strong></td>
<td>&lt; 1 ppm</td>
<td>&lt; 1 ppm</td>
<td>&lt; 2 ppm</td>
</tr>
<tr>
<td><strong>Iron</strong></td>
<td>&lt; 2 ppm</td>
<td>&lt; 2 ppm</td>
<td>&lt; 6 ppm</td>
</tr>
</tbody>
</table>

*1 % solution

1 Item available on any COA
More information

If you would like to obtain more detailed information about DEQUEST® products or are interested in obtaining a sample for evaluation in your system, please contact your nearest Italmatch Chemicals S.p.A. representative or visit our website: http://www.dequest.com

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