Cosmetics with grapefruit seed extract / preservatives

Joint campaign of Basel City (specialist laboratory) and Aargau

Number of samples tested: 11

Objected to: 10 (90%)

Grounds for objection:
- Prohibited preservative (8), limit value exceeded (3), incomplete declaration of ingredients (6), health benefit claims (2), absence of warning notice in three official languages (1), no address in Switzerland (1)

Background and aims of the investigation

The presence of benzethonium in grapefruit seed extract (GSE) is a recurring theme in our laboratory and, as it is prohibited, regularly leads to objections. Further background information is available in our report of 2004 (http://www.kantonslabor.bs.ch/infos_berichte.cfm?Labor.Command=detail&Labor.Jahr=2004&Labor.ID=319). Last year we found benzethonium in a cosmetic containing GSE for the first time, and therefore decided to investigate the market for suspicious cosmetics and grapefruit seed extracts. Thanks to a clean-up operation in 1997 and 1998, only a handful of such products are still found in pharmacies and department stores. However, an almost limitless array of grapefruit seed extract products may be ordered over the internet, and we came across one supplier based in Basel.

Statutory basis

Cosmetics have to adhere to the legal requirements of the Ordinance on articles of daily use (GebrV) and the Ordinance on cosmetic products (Vkos). Benzethonium is only permitted as a preservative in rinse-off products, with a limit value of 0.1%.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Evaluation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Preservatives</td>
<td>VKos, Appendix II</td>
</tr>
<tr>
<td>Declaration</td>
<td>GebrV, Art. 23</td>
</tr>
</tbody>
</table>

Sample description

The majority of products were obtained from a company in Basel which offers cosmetics containing GSE from the USA and natural cosmetics preserved with GSE. One sample was a German grapefruit seed extract.

<table>
<thead>
<tr>
<th>Origin</th>
<th>Number of samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Switzerland</td>
<td>6</td>
</tr>
<tr>
<td>USA</td>
<td>3</td>
</tr>
<tr>
<td>Germany</td>
<td>2</td>
</tr>
<tr>
<td>Total</td>
<td>11</td>
</tr>
</tbody>
</table>

Test methods

HPTLC and postchromatographic derivation were used to screen for 15 quaternary ammonium compounds (quats, for example benzethonium). Reverse phase HPLC and DAD- as well as ESI-MS/MS detection were used for confirmation.

Four HPLC methods were used to investigate the samples for other preservatives, including over 40 permitted and 8 banned preservatives, and when necessary to quantify these preservatives. HPLC/UV-DAD was used in three methods (UV-active preservatives, 46 parameters), Isothiazolinone (3 parameters) and free formaldehyde (following addition of with 2,4-dinitrophenylhydrazine). Redox-active preservatives (3 parameters) were determined using HPLC and electrochemical detection.
Results

• The German grapefruit seed extract was the only sample in which we were unable to detect any preservatives.

• The three questionable American products (a deodorant, a toothpaste and a nasal spray) that were promoted with the phrase “contains GSE” all contained prohibited benzethonium. The toothpaste contained 0.02% benzethonium, the deodorant 0.15% and the nasal spray, which was also found to be an unregistered drug contained 0.04% benzethonium.

• Five of the six Swiss-made leave-on products contained benzethonium at concentrations between 0.05% and 0.6% (!), in some cases greatly in excess of the limit value of 0.1% for rinse-off products. None of the products declared this, either as benzethonium or as grapefruit seed extract. The benzethonium originated from grapefruit seed extract, which was used as a “natural” preservative. The source of GSE was the same as for the American cosmetics previously mentioned.

• Other undeclared preservatives were also found in these products, including methylparaben and propylparaben and benzyl alcohol. Two products contained, respectively, 0.47% and 0.54% methylparaben, i.e. above the limit value of 0.4%.

• The ingredients lists for all six Swiss-made products were incomplete – for example no preservatives were listed.

• Warning notices, when provided, were only in German.

• Two products contained the illegal health claims, “Acne swab”, “mobility cream” and “rub in well to all areas with limited mobility”.

• The acne swab declared essential oils as an ingredient. The manufacturer confirmed that 10% essential oil had been used, however only 3% essential oil is permitted in cosmetics.

• One eye liner which was also investigated as part of this campaign did not carry a swiss address.

Measures taken

• The sale of products containing benzethonium was prohibited. The company in question will no longer use grapefruit seed extract to preserve their own products.

• We requested an assessment of labelling for the entire product range, to ensure they conform to the legal requirements.

Conclusions

At the risk of repeating ourselves, if these problems are not dealt with at their roots, new companies will continue to discover the “natural” preservative grapefruit seed extract. Manufacturers sometimes contend that the active substance is merely related to benzethonium, but do not say what the substance is, and these claims have been rebutted scientifically: Gary Takeoka et al.: Identification of Benzethonium Chloride in Commercial Grapefruit Seed Extracts, J. Agric. Food. Chem. 2001, 49, 3316-3320.