Cosmetics and grapefruit seed extracts / quaternary ammonium compounds

Privately commissioned investigations

Number of samples tested: 3 *Of which were objected to: 3*
Grounds for objection: *Non-permitted preservative*

Introduction and statutory basis

The demand for natural biocides and medicines continues to lead consumers and manufacturers to grapefruit seed extract (GSE). Even though it has been known for years that pure grapefruit seed extract is essentially free of biocidal activity (for example refer to von Woedtke et al. Pharmazie 54 (1999) 6, 452-456) advertisements for such products continue to appeal to this property. These claims are nevertheless justifiable due to the addition of technological preservatives, which in itself achieves the biocidal activity. In recent years we have regularly found these products to contain up to 20% benzethonium chloride. This chemical, which is classified in Switzerland as a category 2 poison and teratogen must not exceed 0.1% in rinse-off cosmetics. Other investigations have also reported the presence of benzalkonium chloride or triclosan.

Samples

Following a private commission, we investigated one cosmetic and two grapefruit seed extracts.

Test methods

Screening for and quantitative detection of quaternary ammonium compounds (Quats) was carried out using HPTLC and postchromatographic derivation. Confirmation was via reverse phase HPLC and ESI-MS/MS detection. Triclosan and other UV-active preservatives were investigated using HPLC/DAD.

Results and opinion

Grapefruit seed extract was declared as "Citrus grandis" on a cosmetic packaging. A quality assurance agent noticed this while appraising the packaging of a "natural" skin lotion. We investigated this product at his instigation and found a content of 0.2% benzethonium chloride, which is not permitted in skin lotions. The product was promptly removed from the product range and the recipe adjusted. It is a shame that the product developer was clearly not knowledgeable about GSE. This was the first Citrus grandis-containing cosmetic found positive for benzethonium in our laboratory. The „highly concentrated“, and hence expensive GSE contained 20% benzethonium, and was therefore little more than a highly concentrated solution of this critical chemical.

We were in for a surprise with the second privately-commissioned sample. The extract did not contain the known substances benzethonium, benzalkonium or triclosan, but the group-specific HPTLC signal nevertheless indicated the presence of a quat. Evaluation of the LC/MS experiments showed the product to contain didecyldimonium chloride. This chemical was then quantified using HPTLC and was present at a concentration of 0.25%. This biocide is not permitted for use in cosmetics, but is often found in technological preservatives, for example for wood preservation or disinfection of swimming pools. The concomitant component spectrum largely corresponded to that for a technological product based on didecyldimonium, which we investigated some years ago. A 0.3% concentration of didecyldimonium chloride was found to inhibit the growth of several varieties of bacteria in inhibition-zone tests; this result was practically identical to that for „grapefruit seed extract“.

Continued findings of technological preservatives in GSE products show that our investigations are necessary. In contrast to 1997/1998, when the market was overwhelmed with these products, only a few such products are found on the market today, although these products are admittedly easily available to customers via the internet. What is new is that a cosmetics manufacturer was taken in by the spiel and used grapefruit seed extract as a "natural" preservative. The fact that disinfectant-containing GSE products continue to pop up on the market in Switzerland is possibly due to the inability of the US authorities to prevent improper manufacturing and aggressive marketing (via the internet) and the continuing loss of knowledge regarding these illegal products in Switzerland.