APPLICATION BRIEF

Chemical/Industrial

# QA/QC Analysis of Parabens in Hand Lotion Using UHPLC/SQ MS

Parabens are esters of *p*-hydroxybenzoic acid which are often added to cosmetics and other personal care products including shampoos, moisturizers, etc. Parabens are considered good preservatives for their broad antimicrobial spectrum but also for their relatively low toxicity, low volatility and high stability.

Recent studies suggest parabens may be carcinogenic and can cause estrogenic disrupting activity, thereby disputing the notion of low toxicity for these compounds. We present an LC/MS assay to identify several of the parabens found in hand lotion.

## Experimental Conditions

Target Analytes: Methyl paraben, ethyl paraben, *n*-propyl paraben, isopropyl paraben, *n*-butyl paraben, isobutyl paraben

### Sample Preparation Conditions

Hand lotion (100 mg) obtained from a grocery store was spiked with isopropyl paraben as internal standard (280 ng) and extracted in two different solvents of varying hydrophobicity (1 mL of methanol or ethyl acetate). The samples were vortexed and centrifuged for 10 min at 4000 RPM to separate the undissolved solids from the liquid. The extracts were then diluted in methanol (1:25) and analyzed by UHPLC/SQ MS.

### Liquid Chromatography Conditions

| | |
|---|---|
| Pump Type: | PerkinElmer® Flexar™ FX-15 |
| Column: | PerkinElmer Brownlee™ HRes C18 column (2.1 mm x 50 mm, 1.9 µm) |
| Mobile Phase: | A: water containing 0.1% formic acid<br>B: 50/50 acetonitrile/methanol containing 0.1% formic acid |
| Flow Rate: | 0.5 mL/min |
| Injection Volume: | 2 µL in partial fill mode |

Gradient:

| Time (min) | %A | %B |
|---|---|---|
| 0 | 70 | 30 |
| 2 | 50 | 50 |
| 2.5 | 43 | 57 |

### Mass Spectrometer Conditions

Ionization: Ultraspray™ ESI – Negative mode

$[M+Na]^-$ ions of each of the analytes were monitored in two different time periods:

Time Period 1: (0-3 min) SIM ions 151.0, 165.0, 179.1 for methyl, ethyl, *n*-propyl and isopropyl paraben, respectively; dwell time of 120 ms each

Time Period 2: (3-4.5 min) SIM ion 193.0 for *n*-butyl and isobutyl paraben; dwell time of 120 ms

Capillary Exit Voltage: -50 V

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## Results

Figure 1 shows the separation and detection of parabens in SIM mode by UHPLC/SQ MS. Calibration curves for each of the parabens were developed using isopropyl paraben (11 ng/mL) as internal standard (Figure 2 shows the calibration curve for methyl paraben).

Hand lotion extracted for parabens showed slightly better recoveries (not significantly different) in ethyl acetate than methanol. The recovery of internal standard taken through the extraction procedure showed 110% ±0.9% (n=3) recovery.

Figure 3 shows that methyl paraben was the predominant paraben found in the ethyl acetate extract of hand lotion, at 7.8 µg/g (%RSD=10.5, n=3). Very low concentrations of *n*-propyl paraben and *n*-butyl paraben were also observed in the lotion.

These results were consistent with the ingredient methyl paraben listed on the bottle. The European Directive 76/768/EEC and its amendments limit the use of a single ester of paraben to 0.4% (w/w) in a cosmetic product. We detected 0.001% of methyl paraben in hand lotion by UHPLC/SQ MS.

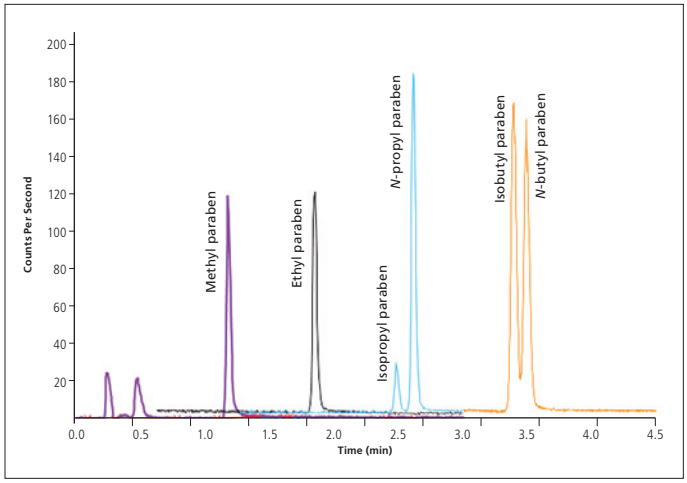


*Figure 1.* Overlaid chromatograms of the $[M-H]^-$ SIM ions of the various parabens analyzed by UHPLC/SQ MS.

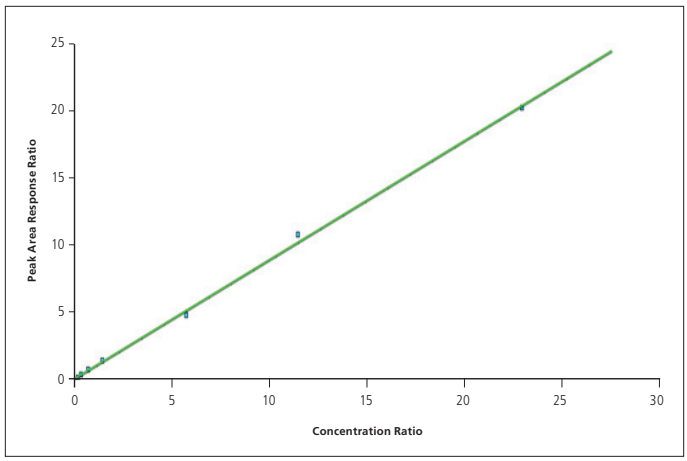


*Figure 2.* Calibration curve for methyl paraben with isopropyl paraben used as the internal standard (concentration range of methyl paraben 1.95-250 ng/mL, $r^2$=0.9987).

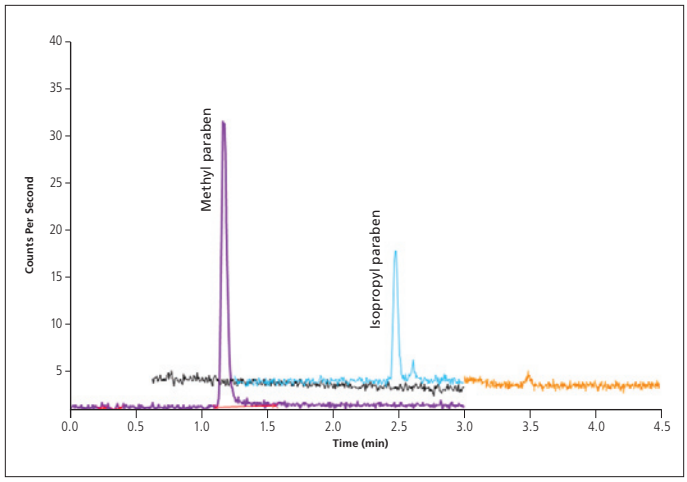


*Figure 3.* Hand lotion extracted in ethyl acetate and spiked with isopropyl paraben as the internal standard shows the majority of parabens in the sample to be methyl paraben.

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