In-vitro Method for Quantification of Fragrance Capsules Leakage in Personal Care Products

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Abstract Summary:
An in-vitro method was developed to quantify the leakage of fragrance from capsules in personal care products using GC Headspace. This method is currently being used to evaluate the stability of different fragrance capsules in personal care products.

Introduction:
Today the use of encapsulates in the household and personal care products industry is widespread.¹ ² Long lasting fragrance is especially important in underarm products where it signals to the consumer that the product is providing the desired odor and wetness protection. An example of these capsules being used in Antiperspirant Sticks is starch capsules³ that are present in several commercial products.

Therefore, encapsulation technologies that provide enhanced fragrance longevity are highly desirable in underarm products. In order to evaluate the stability of these new technologies, a project was initiated to develop an in-vitro method with minimum sample preparation and time.

Experimental Methods:
Samples of capsules were obtained from different suppliers and incorporated in the underarm product formulations to be evaluated. Stability experiments were carried out for 3 months in ovens set at room temperature and 40 °C with measurements made at 4, 8 and 13 weeks.

The underarm product samples were evaluated using a Perkin Elmer Clarus 500 GC with an FID detector using an automated sample headspace injector Perkin Elmer Turbo Matrix Headspace. The GC is equipped with a Zebron-Phase ZB-WAX column (L=30m x I.D. = 0.32mm x df=0.50um) with an oven starting temperature of 50 °C and using a ramp of 5 degrees per minute up to 220 °C.

The underarm product sample (2g of sample as is, no preparation needed) is weighed in a GC vial (20 ml) and equilibrated in the headspace instrument for 1 hour at 50 °C before injection of the headspace into the GC. All measurements are done in triplicate.

Standard samples of the product are prepared at the same time as the samples with capsules with different amounts of fragrance (same fragrance used in the capsules). Three concentrations (1.00, 0.50 and 0.25%) of fragrance in the product were used. A set of 4 formulations is made for every capsule and divided in several containers to be placed in the stability ovens at RT and 40 °C. The set of 4 formulations consists of the test formulation with 1% fragrance in the capsules (example: if capsule contains 50% fragrance then 2% of fragrance capsules is used).

It is important to note that the standards are placed in the ovens together with the test sample and evaluated on the GC headspace at the same time in order to minimize any issues with stability of the fragrance in the product.

Fragrance is normally composed of 20-100 ingredients and it is not possible to quantify each and every one of them. The main components (5-20) are quantified and taken as representation of the total fragrance quantification. The areas for the main peaks present in the fragrance are quantified for the set of 4 formulations. Using the standards with the different amounts of fragrance (1, 0.5 and 0.25%) a linear regression is calculated and used to quantify the amount of fragrance in the test formulation with the capsules at each time point.

The software (Totalchrome) used to control the instrument (GC injections) has the capability of being programmed to do the calculations automatically so that you can obtain the % leakage of each peak directly from the software.
We prefer to quantify each of the GC peaks first then add them up and calculate the percent leakage.

**Results and Discussion:**
This method has been used to evaluate the stability of different fragrance capsules in several Personal Care products:

- Antiperspirant Sticks
- Antiperspirant Aerosols
- Antiperspirant Roll-ons

Here is one example of the application of this method in Antiperpirant Aerosols, but other examples will be illustrated in the final presentation.

As can be seen in figures 1 and 2 below, the fragrance capsules, as expected, are more stable at Room Temperature (RT, 25°C) than at 40°C. The graphs also show that the capsules leak more fragrance with time.

The final result is that after 3 months (13 weeks) there is 15% leakage of fragrance at RT and 30% leakage at 40°C. This result is acceptable since there is still 85 and 70% respectively encapsulated fragrance left in the product that can perform when used by the consumer.

**Conclusions:**
A very simple in-vitro method has been developed to quantify the leakage of fragrance from capsules in personal care products using GC Headspace.

The advantages of the method are:
- No sample preparation is needed
  - Product samples are used “as is” in the GC vial
  - Saves time and resources
- Reliable method
- Very simple method that can be used with any other capsule. The only requirement is that the encapsulated material is volatile enough to be quantified by GC

**References:**
1.- Microcapsules for Fragrances and Cosmetics S. Park, R. Arshady Chapter in Microspheres, Microcapsules & Liposomes 2003, 6.
3.- Controlled release composition for anhydrous consumer and cosmetic products, has fragrance burst upon contact of micro-spheres with moisture US2002122549A Salvona IP, LLC