COSMETICS TESTING ARTICLES

Determination of benzyl alcohol in cosmetics by gas chromatography-mass spectrometry

Ren Min   Wang Yu

(Beijing Physical and Chemical Analysis and Testing Center,
Beijing, 100089)

Abstract: The benzyl alcohol in cosmetics was determined by gas chromatography/mass spectrometry in this paper.   This experiment mainly optimized instrumental conditions, and the recovery of benzyl alcohol in solid and liquid samples were between 96-101%.   This method has the characteristics of good stability, resistant against interference, high sensitivity, and it allows simple and quick determination of benzyl alcohol in cosmetics.

Key words: gas chromatography/mass spectrometry; cosmetics; benzyl alcohol

1   Introduction

Benzyl alcohol is one of the simplest aromatic alcohols, and in nature, most of them exist in the form of esters in essential oils[1].   Benzyl alcohol is used as solvent and viscosity reducer in various cosmetics, and it is also a new preservative in recent years[2].   As benzyl alcohol is more toxic, it has been restricted by some countries in some aspects[3].   Foreign toxicological data indicates that benzyl alcohol has anesthetic effect, and it's intensely irritating to eyes, skin and respiratory system.   It is harmful to the body through ingestion, inhalation or skin contact; it also has certain effect in skin irritation and causing allergy[4] .

With continuous improvement of living standards, cosmetics have gradually become an important daily consumer goods.   For their frequent and close contact with human body, their safety performance is closely related to human health.   As cosmetics contain nutrients that are beneficial to microbial reproduction, odor or change in appearance may occur due to bacterial contamination during production, use, and storage of cosmetics.   Addition of certain amount of preservative to the product effectively eliminates microbial growth, but frequent exposure can adversely affect human health.   Benzyl alcohol is quite widely used in cosmetics for its low toxicity and good sterilization effect[5].   China's Cosmetic Hygiene Code 2007 edition lists benzyl alcohol

as restricted preservative[6].

At present, instrumental methods commonly used for determination of benzyl alcohol are high performance liquid chromatography, gas chromatography and gas chromatography-mass spectrometry[7-9]. In this experiment, ultrasonic assisted extraction and gas chromatography-mass spectrometry were used to determine the benzyl alcohol content in various cosmetics.

2 Experiment

2.1 Instruments and reagents

GC-MS3200 (EAST & WEST ANALYTICAL INSTRUMENTS, INC);

AC-2546 centrifuge (HKUST Innovation Co., Ltd. Zhongjia Branch);

KQ5200E ultrasonic cleaning instrument (Kunshan Chaosheng Instrument Limited Company)；

benzyl alcohol standard solution (J & K SCIENTIFIC LTD);

ethyl alcohol absolute (LC grade, Fisher);

cosmetic samples purchased from the market.

2.2 Instrument conditions

Gas chromatography/mass spectrometry conditions: Injection port temperature: 260℃; Column flow rate: 1mL/min; Interface temperature: 250℃; Column oven temperature program: hold at 60ºC for 5 minutes, ramp to 270ºC at 35ºC/min, hold for 1 min; Column: DB-5MS (30 m×0.25 mm×0.25μm)

Quantitative analysis using selective ion monitoring mode (SIM), quantitative ion is m/z 79, reference qualitative ions are m/z 77, m/z 107, m/z 108.

Under this instrumental condition, the total ion chromatogram (TIC diagram) of benzyl alcohol by gas chromatography/mass spectrometry is shown in Figure 1.
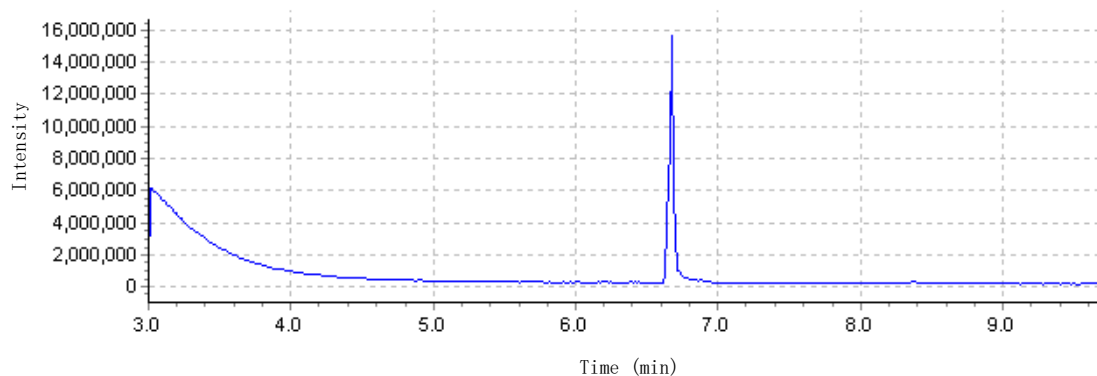
Figure 1 Thotal ion current diagram (TIC diagram) of benzyl alcohol

2.3 Sample treatment

1.0 g cosmetics (precision to 0.0001 g) is weighed, placed in a 15 mL centrifuge tube. 5 mL absolute ethanol is added, sonicated for 30 minutes, then made up to 10 mL with absolute ethanol, and centrifuged for 10 minutes at 3000 r/min. The supernatant was taken for testing.

3 Results and discussion

3.1 Linear range and calibration curve

Under gas chromatography/mass spectroscopy conditions stated above, the linear range of benzyl alcohol standard solution was determined experimentally, and the calibration curve was plotted, as shown in Figure 2. This figure showed that benzyl alcohol exhibited good linearity in the range of 0.0625 - 100 μg/mL in the linear range.
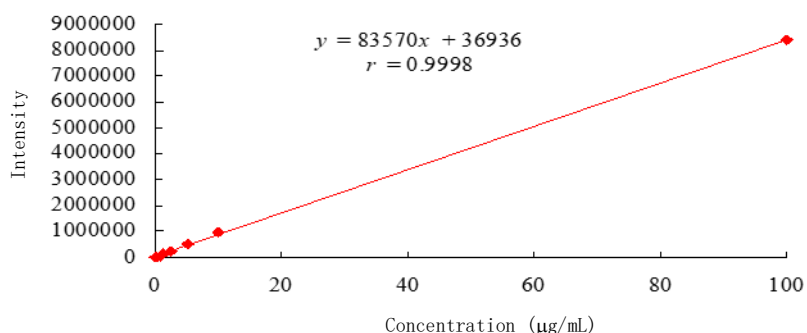

$$y = 83570x + 36936$$
$$r = 0.9998$$

Figure 2 Linear range and calibration curve of benzyl alcohol

3.2 Accuracy and precision

Accuracy and precision of the method were determined. Recovery test of hand cream and lotion samples were carried out (added concentration was 10.0μg·mL^{-1}, repeated 7 times), and recovery rate of the method was determined. At the same time, the detection limit and reproducibility were measured. The results are shown in Table 1.

Table 1 Method accuracy and precision

Sample	Added concentration, μg/mL	Percent recovery, %	Reproducibility, RSD%	Detection limit, mg/kg
Lotion	10	96	3.31	5.4
Hand cream	10	101	3.74	2.1

3.3 Sample measurement

Using the experimental method established in this paper, the benzyl alcohol in 6 types of

cosmetics samples, such as face cream, hand cream -1#, perfume, facial cleanser, lotion, and hand cream -2#, are determined.　The results are as shown in Table 2.

Table 2 Cosmetic sample measurement results

Sample	Concentration[†], mg/kg
Perfume	27
Face cream	118
Hand cream - 1#	ND
Facial cleanser	ND
Hand cream - 2#	ND
Lotion	ND

Note: "ND" indicates that benzyl alcohol is not detected

4　Conclusion

Benzyl alcohol in cosmetics was determined by ultrasonic extraction and GC-MS 3200.　The recovery was between 96-101% in this experiement.　GC-MS3200 is a very suitable instrument for the determination of benzyl alcohol in cosmetics, and it can be used for qualitative and quantitative analysis.