SHIMADZU
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Gas Chromatography Analytical Methods for Teaching Laboratories

Identification of organic aromatic compounds in personal care products using GCMS and mass spectral libraries.

Introduction

GCMS is an important analytical technique used by the perfume industry as it allows aromatic compounds within the sample to be separated and identified. This helps perfume makers to carry out quality control checks on raw materials, ingredients, and final products. It can also used for product development, troubleshooting when ‘unknown smells’ are detected and to reverse engineering of competitor products.

This application utilizes a GCMS with a standard inlet to analyze one or more commercially available perfumes or personal care products. The samples will be analyzed with the mass spectrum detector in scan mode and components found in the sample identified by comparing their mass spectra to those in a mass spectral library, such as NIST.

Reagents

Methyl tert-butyl ether – analytical grade

Deionized water

Anhydrous sodium sulphate

Sample material – any commercially available perfume or personal care product.

Safety

Lab coats, safety glasses and gloves should be worn when handling reagents.

Follow all appropriate safe operating procedures and beware of any specific actions raised in the associated risk assessments.

Apparatus

- Gas chromatograph fitted with a split/splitless injection port and mass spectral detector (MS).
- NIST Mass Spectral Library or suitable equivalent.
- Autoinjector and autosampler capable of handling liquid samples in 1.5 mL vials
- SH-PolarWax column, 30 m x 0.25 mm I.D. x 0.25 µm, p/n 227-36322-01 (or suitable equivalent).
- Centrifuge
- 50 mL centrifuge tubes
- Syringes and filters
- 1.5 mL autosampler vials and caps.
- Appropriate laboratory glassware for the preparation of the samples.

Instrument Conditions

GCMS Parameters	
Injections Temperature	230°C
Injection Mode	Split
Split Ratio	1:40
Purge flow	6 mL/min
Column	SH-PolarWax column, 30 m x 0.25 mm I.D. x 0.25 µm
Oven Program	50°C initial temperature, hold 0 minute 15°C/min to 100°C and hold for 0 minutes 5°C/min to 250°C and hold for 10 minutes
Run Time	43.33 minutes
Carrier Gas	Helium, linear velocity 45.0 cm/s
Injection Volume	1.0 µL
Transferline Temperature	230°C
Source Temperature	200°C
MS Scan range	35 to 500 m/z

Preparation of test sample

Perfumes - Dilute the test sample 1 in 10 with methyl tert-butyl ether and transfer 1 mL to a 1.5 mL vial for analysis.

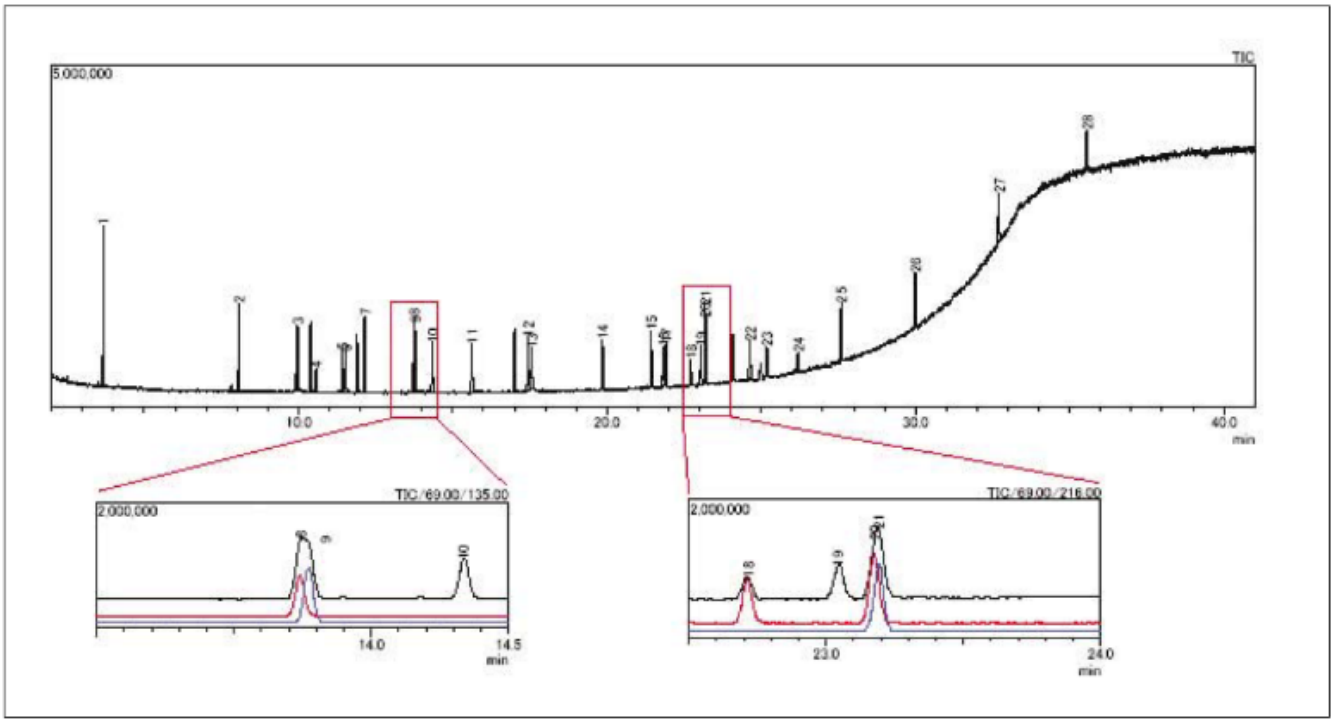
Creams/lotions – weigh 0.5 g of sample into a 50 mL centrifuge tube. Add 5 mL of deionized water and 5 mL of methyl tert-butyl ether then shake for 30 minutes. Add 5 g of sodium sulphate to remove the water and centrifuged for 30 minutes. Filter 1 mL of the supernatant with a syringe filter into a 1.5 mL vial.

Interpretation of results

The components of the test sample should be adequately separated to allow them to be tentatively identified by comparing their mass spectra to a commercially available database, such as the NIST mass spectral library. Some of the more complex matrices, such as cream based products, may be more challenging to interpret as the number of individual components may increase.

The image below shows an example total ion chromatogram (TIC) with 26 individual fragrance components detected.

Note: compounds 5 and 27 are internal standards not required for this application.



Peak number	Compound name	Peak number	Compound name
1	Limonene	15	Amyl cinnamal
2	Linalool	16	Anisyl alcohol
3	Methyl heptin carbonate	17	Cinnamyl alcohol
4	Citral 1	18	Farnesol 1
6	Citral 2	19	Isoeugenol
7	Citronellol	20	Farnesol 2
8	Geraniol	21	Hexyl cinnam-aldehyde
9	3-Methyl-4-(2,6,6rimethyl-2-cyclohexen-1-yl)-3-buten-2-one	22	Coumarin
10	Benzyl alcohol	23	2-(4-tert-Butylbenzyl) propionaldehyde
11	Hydroxy-citronellal	24	Amylcin namyl alcohol
12	Cinnamal	25	Benzyl benzoate
13	Hydroxy-methylpenthyl-cyclohexenecarboxaldehyde	26	Benzyl salicylate
14	Eugenol	28	Benzyl cinnamate

Key learning outcomes

- Understanding that the fragrance of a perfume is produced by a complex mixture of compounds, and that the concentration of each component needs to be maintained to ensure a consistent scent is produced every time a new batch of product is manufactured.
- How compounds in a complex matrix can be identified by comparing their mass spectrum to those held in a database such as the NIST mass spectral library.

Notes

The instrument conditions are based on the Shimadzu application note M228 ‘Analysis of Fragrances of Cosmetics using GCMS’. If an alternative column or carrier gas is used, these conditions will need to be adapted appropriately.