

Cosmetics Quality and Safety Testing Technology

The issue of cosmetics quality and safety has always been the focus of public attention. Many well-known brands of cosmetics have successively detected banned or restricted substances. This has not only harmed consumers' rights, but has also caused significant economic losses to cosmetics companies. In order to reduce or avoid the emergence of cosmetic quality and safety incidents, researchers have adopted a variety of analytical testing techniques to establish a series of detection methods for chemical risk substances in cosmetics. This white paper summarizes the research progress of important academic journals in the field of cosmetic quality and safety testing technology in recent years, and introduces representative new technologies for cosmetic testing. It aims to provide theoretical support and technical reference for researchers in the field of cosmetics testing.

Pre-treatment Technology

In the process of cosmetic sample detection, pretreatment is usually the most critical and time-consuming step, and it is also an important step to concentrate the target components, improve the sensitivity of the method and remove interferers. The matrix of cosmetic samples is mainly oily raw materials and water. The former includes oils, waxes, hydrocarbons, and the higher fatty acids and fatty alcohols that make up these ingredients. Various components, such as spices, preservatives, colorants, water-soluble polymers, humectants, surfactants, metal ion adhesives and other components, are added to the matrix to make cosmetics. Therefore, the matrix in cosmetics is very complex, and there may be some interference effects in the analysis and detection of target compounds. The accuracy and reliability of the analysis results are directly determined by the sample pretreatment effect. At present, the extraction technologies reported in cosmetics testing include ultrasound-assisted extraction, mechanical vibration extraction, liquid-liquid extraction, dispersion-liquid microextraction, solid-phase

microextraction, and supercritical fluid extraction, *etc.* The purification technology is mainly solid-phase extraction, N-hexane degreasing, *etc.*

Extraction Technology

- **Ultrasound-assisted Extraction**

Ultrasound-assisted extraction (UAE) is easy to operate and can achieve high extraction efficiency in a short time. It is the most commonly used technical means for extracting organic components in cosmetics. As an extraction technology, the cavitation effect produced by ultrasonic waves occupies the main aspect. This effect can refine various substances and generate emulsions, accelerating the target components in the sample into the solvent. In addition, the thermal effects and mechanical effects produced by ultrasound are two important bases for its use as an extraction technology. Extraction technology is mostly used to detect hormones (glucocorticoids, androgens, estrogen, and progestogens), antibiotics (sulfonamides, nitrofurans, quinolones, and nitroimidazoles) and preservatives in cosmetics, and good recovery and precision are obtained.

- **Mechanical Vibration Extraction**

Mechanical vibration extraction technology can improve the turbulence degree of the solvent and reduce the thickness of the mass transfer interface between the sample and the solvent, so as to improve the extraction rate and efficiency. The main characteristics of this technology are high solvent utilization and extraction efficiency, but strong selectivity for extraction solvents. 9 kinds of glucocorticoids in cosmetics were extracted by acidification methanol oscillation, and then extracted twice with n-hexane. The recovery of all target analytes was 85.0% - 99.5%, and the RSD was less than 5.8%.

- **Liquid-liquid Extraction**

Liquid liquid extraction (LLE) is a separation process in which the components are transferred from one phase to another by the difference of solubility between two immiscible liquid phases. The technology has the characteristics of large capacity, good

separation effect, high recovery rate, continuous operation and automatic control.

Organic solvents were used to dissolve or disperse oily cosmetics such as lipstick and nail polish, and then seven colorants (including carmine, naphthol yellow, sunset yellow, acid red 33, allure red, acid red 13 and acid orange) were extracted by liquid liquid extraction using water as extraction solvent. The average recovery of the extraction method is 90.5%-100.6%, and the relative standard deviation is 3.2%-6.7%.

- Dispersive Liquid-liquid Microextraction

Dispersive liquid-liquid microextraction (DLLME) is a new type of microextraction technology. It uses a microsyringe to quickly inject the micro-upgraded extraction solvent into the sample liquid to form extractant beads in the dispersant-sample solution, which effectively increases the contact area between the two, makes the target analyte enter the extraction solvent quickly, and greatly improves the extraction efficiency and enrichment multiple. In the analysis of cosmetics, some researchers have also used this technique to extract and determine phthalate plasticizers and preservatives in cosmetics.

- Solid-phase Microextraction

Solid-phase microextraction (SPME) is a new type of extraction separation technology developed in recent years, which is based on the principle of chromatographic separation and achieves the purpose of separation by using the distribution balance of the target in two phases. Compared with traditional pretreatment methods, it is simple, does not require the use of solvents, and can directly desorb and inject samples while enriching the target, eliminating tedious operation steps, greatly reducing analysis time and improving analysis efficiency. In cosmetics testing, a solid-phase microextraction method was developed for 8 phthalate plasticizers in water-soluble cosmetics. After the samples were diluted with water and sealed with sodium chloride, they were stirred in the silicone oil bath for headspace extraction, and then the extraction fiber head was placed in the gas-phase chromatogram inlet for desorption. The recoveries of all analytes were 83% - 97% and RSD was 2.5% - 10.0%

- Supercritical Fluid Extraction

Supercritical fluid extraction(SFE) uses supercritical fluid as extractant to extract target analytes from liquids or solids so as to achieve the purpose of separation. The technology has the following advantages: easy separation from the product, safe and non-toxic, mild operating conditions, not easy to destroy the active components and so on. In recent years, some analysts have applied supercritical fluid technology to the field of cosmetic detection. This technique has been used to extract four Nipagin ester preservatives and four antioxidants from cosmetics.

Purification Technology

- Solid-phase Extraction

Solid-phase extraction (SPE) is a technology developed from the combination of liquid-solid extraction and liquid chromatography. It separates, purifies and enriches samples through selective adsorption and desorption of sample components by fillers. The main purpose is to reduce matrix interference in samples and improve detection sensitivity. Because of its simple operation, high recovery rate and good repeatability, this technique is widely used in the field of cosmetic detection. For example, solid phase extraction technology is used to remove the matrix components in the samples in the purification process of hormones, surfactants, halogenated phenols, prohibited drugs and coumarins, which can effectively reduce the interference to the target analytes.

- N-hexane Degreasing

As most of the matrix in oily cosmetics are non-polar fatty components, it is a simple and easy method to remove most of the non-polar matrix by adding n-hexane.

Instrumental Analysis Technology

HPLC and HPLC-MS

High-performance liquid chromatography (HPLC) is suitable for the separation of 80% known organic compounds because it is not restricted by the volatility and thermal stability of the substances to be determined. It has the characteristics of fast analysis speed, strong separation ability, high sensitivity and a wide range of applications. In recent years, analysts have established a method for the detection of a variety of prohibited components in cosmetics by using HPLC, such as hormones, dyes, preservatives, sulfonamides, nitrofurans, antibiotics, catecholamines, allergens, coumarins, phthalates, ultraviolet absorbers, zinc phenol sulfonate, benzalkonium chloride, antihistamines, naphthalene diphenols, formaldehyde, vitamins, halogenated phenols, quinolones antibiotics, colorants and so on.

High-performance liquid chromatography-mass spectrometry (HPLC-MS) technology is used for cosmetics sample detection. Multiple reaction monitoring (MRM) mode of tandem quadrupole mass spectrometry is favored. In this model, the characteristic parent ions are detected first, and the interference of other fragment ions can be removed after further collision induced dissociation (CID) of the selected parent ions. Only the selected characteristic ions are collected by mass spectrometry. Through the selective monitoring of secondary ions, the chemical background of mass spectrometry detection can be reduced, and the signal-to-noise ratio of the target can be significantly improved, thus the detection sensitivity can be improved.

GC and GC-MS

In cosmetics detection, gas chromatography (GC) is mostly used for the separation and analysis of low molecular weight compounds with strong volatility and weak polarity, as well as the detection and analysis of solvent residues. In recent years, researchers have established GC methods for the detection of a variety of volatile substances in cosmetics, including preservatives, volatile organic solvents, undecenoic acid and its salts, phthalates and so on. As a separation technology, GC has strong separation ability, but its application is limited by its inherent characteristics. GC-MS not only plays the role of high-speed separation of GC, but also combines the advantages of high sensitivity and high selectivity of MS. It is especially suitable for the qualitative and quantitative analysis

of unknown compounds in multi-component, as well as the determination of molecular weight and structure of compounds. In recent years, GC-MS is widely used in cosmetics detection, such as phthalate, allergen, preservative, methanol, acrylonitrile, dioxane, plasticizer, UV absorber, *etc.*

Capillary Electrophoresis

Capillary electrophoresis (CE) is a liquid phase separation technology that uses a capillary tube as a separation channel and a high-voltage electric field as a driving force to achieve separation based on the difference in electrophoretic mobility or distribution behavior between sample components. Because this technology has the characteristics of high efficiency, fast speed, small amount, and multiple modes, it is also widely used in cosmetics analysis and detection.

Ion Chromatography

Ion chromatography is a kind of high performance liquid chromatography, which is mainly used for the determination of anions and cations in samples. At present, ion chromatography is mainly applied to substances that are difficult to be separated by traditional analytical methods, and can ionize and analyze compounds without ultraviolet absorption. In the field of cosmetics testing, relevant scholars have adopted this technology to establish detection methods for certain prohibited substances. For example, an ion chromatography-post-column derivatized ultraviolet detector was used to establish an analytical method for iodate and bromate in cosmetics, and an ion chromatography was used to detect ammonium and six alkylamines in cosmetics.

Ion Mobility Spectrometry

The basic principle of ion mobility spectrometry (IMS) is: after the sample is converted into ions at the ion source, it is driven by the electric field, and the ions enter the weak electric field migration region through the ion gate that is periodically opened. During the continuous collision of molecules, due to the differences in the respective migration rates in the electric field, different ions are separated and reach the detector one after the other. At present, trace detection methods based on ion mobility spectrometry are mostly

used in the fields of drug detection, explosives detection, and food safety. There are relatively few applications in the field of cosmetic detection.

Summary

To sum up, there are a variety of extraction and purification technologies available in the process of cosmetic pretreatment. According to the literature reports in the past decade, ultrasound-assisted extraction has become the most widely used extraction technology because of its high efficiency, easy operation and universality in the extraction of restricted substances in cosmetics. However, according to actual requirements, continuous operation is required if the sample processing volume is large. According to the specific physical and chemical properties of the target substance, liquid-liquid extraction technology is also one of the commonly used extraction methods. In recent years, with the rise of green extraction technology, disperse liquid-liquid microextraction, solid-phase microextraction and supercritical fluid extraction are more and more used in the field of cosmetics. These emerging technologies use only a small amount of organic solvent or no solvent, and the detection is fast and reliable. It can be predicted that in the cosmetic pretreatment technology, high efficiency, high speed and environmental protection is an important development direction in the future.

In terms of instrumental analysis technology, the current mainstream detection technology for cosmetics is still HPLC, GC, or a combination of both and MS. Especially in the simultaneous qualitative and quantitative analysis of multiple components, the combination of chromatography and MS shows powerful separation and detection capabilities. However, any technology has its specific advantages and disadvantages. The traditional GC-MS technology requires the use of large desktop instruments, which is expensive, high maintenance cost and high consumption of organic solvents. In recent years, the emerging ion mobility spectrum overcomes the above shortcomings of the traditional chromatography-mass spectrometry technology, does not need complex sample pretreatment process, and the analysis speed is fast, which belongs to the

environment-friendly analysis technology. At the same time, the equipment is small and easy to carry, which makes the real-time detection on site possible. As people pay more and more attention to the concept of healthy consumption, it can be predicted that rapid, sensitive, high-throughput, low-cost and environment-friendly analysis and testing methods will become an important development direction of cosmetic quality and safety testing technology in the future.



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