Mass spectrometry methods for pharmaceutical analysis.

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Perspective

Numerous techniques have been developed to ionize, scan, focus, fragment, and detect chemical structures. In the early days of MS, the detection of a single precursor-product-ion pair allowed the identification of different structures, primarily by fragmenting the molecule into smaller building blocks. Objects to be analyzed can now also be identified from the highly accurate mass obtained by high resolution mass spectrometry (HRMS). New developments allow for more sensitive, more selective, more robust and more reproducible analysis. The improved accuracy and reproducibility of the new generation of mass spectrometers has made it possible to use these instruments not only for identification but also for quantification of objects to be analyzed. In this way, MS has become an established value in pharmaceuticals, (bio) chemistry, (bio) medicine, biology, environmental science, and more.

Mass spectrometers are often mistakenly seen only as an alternative to UV / VIS, hydrogen flame ionization, fluorescence and electrochemical detection. Due to the detection principle based on the analysis of a specific mass-to-charge ratio, the mass spectrometer can also be used as an independent separation technique. However, sample complexity in new areas such as environment, toxicology, and biomarker discovery studies requires sensitive and selective measurements to avoid interference, so mass spectrometers are used alone. That is still very difficult. For example, the analysis of trace elements in complex matrices such as wastewater, blood and urine remains a major challenge.

It is recommended to perform a sample preparation step prior to analysis to achieve high sensitivity or to avoid injecting highly contaminated samples. Examples of commonly used sample preparation strategies are liquid-liquid extraction (LLE), protein precipitation, solid-phase extraction (SPE), and all forms derived from them. As an alternative, or in addition to sample preprocessing, additional separation steps are typically added to the methodology. Gas chromatography (GC), capillary electrophoresis (CE), and liquid chromatography (LC) are the most common techniques used to keep all molecules out of the ionization source at the same time. This means that the signal receives less matrix effect (ME) and is more sensitive. The main challenge is to develop a reproducible sample pretreatment method that provides an acceptable and reliable recovery rate in order to accurately represent the results obtained. For this reason, you should consider using internal standards. Equipment progress a) Mass spectrometer, the photomultiplier tube is made up of many dynodes. By dividing the high voltage applied to the first dynode by a resistor, a gradually decreasing negative voltage (more positive voltage) is applied to each dynode in series. Some secondary electrons are emitted from the first dynode by the impact of the ions. The photomultiplier tube is moved off-axis to reduce the noise signal caused by the direct impact of the neutral particles.

Ionization method

Modern Atmospheric Cyclonic Ionization (APCI), Electrospray Ionization (ESI), Matrix Assisted Laser Desorption Ionization (MALDI), and other derivative methods have been adopted in mass spectrometry laboratories. The reason for APCI over EI is that APCI forms protonated molecules and is fully compatible with liquid chromatography (LC), whereas EI tends to fragment ions, resulting in ambiguous molecular weight identification. This is because it may not be compatible with LC. ESI, along with matrix-assisted laser desorption / ionization (MALDI), has made FAB very popular, but has essentially eliminated the use of FAB because it produces much more sensitive protonated molecules. .. In addition, MALDI, along with ESI, has enabled ionization and measurement of high molecular weights that was previously impossible with FAB. The ESI has the advantage of being easily compatible with LC. MALDI offers advantages for imaging mass spectrometry.

Ion formation and guidance

Device manufacturers recognize that increased sensitivity can be achieved primarily by optimizing ion formation and processing. The evaporation efficiency of the newly developed ion source is high, especially in the mobile phase with a high proportion of organic modifiers, allowing much higher flow rates to be used compared to previous generation ESI sources.

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