Rapid analysis of additives in food packaging

Using an Agilent 6120 LC/MSD with an atmospheric pressure solid analysis probe (ASAP) and accurate mass identification by MassWorks

Abstract

This Application Note demonstrates a rapid analysis of food packaging using a single quadrupole mass spectrometer with a direct probe ion source attached, without chromatographic separation. The formula of an additive component was identified from the profile mass spectral data using Cerno MassWorks software. By searching a compound database with the formula determined, we confirmed the presence of an additive in a food wrapper.
Introduction

The prevalence of liquid chromatographic mass spectrometry (LC/MS) has increased significantly. The advent of a system for rapid sample analysis by simplifying sample preparation is desirable. Direct sample introduction into a mass spectrometer without separation by chromatography has recently received more attention.

One approach for direct ionization without chromatography is an atmospheric pressure solid analysis probe (ASAP) (Figure 1). ASAP is an effective tool to rapidly make a direct analysis of volatile or semivolatile substances in solid and liquid samples by atmospheric pressure ionization. It also allows for the ionization of low polarity compounds unsuitable to electrospray ionization (ESI). In addition, the direct ionization of samples makes sample pretreatment or separation by chromatography unnecessary.

A single quadrupole mass spectrometer is powerful, inexpensive, and simple to operate, providing information related to the molecular weight of the target component. However, quadrupole mass spectrometry typically does not provide accurate mass data for identifying unknown compounds. To overcome this limitation, MassWorks was used to first calibrate the profile mode MS data, through the introduction of known calibration standards, into accurate mass data. The accurate mass data obtained from the quadrupole MS system could then be used to economically determine elemental compositions for unknowns found in a sample.

The ASAP was connected to the Agilent 6120 LC/MSD to study the feasibility for rapid analysis of food packagings. To determine the elemental compositions of observed ions, the MS data were calibrated into accurate mass data through MassWorks, then analyzed with Calibrated Lineshape Isotope Profile Search (CLIPS).

Experimental conditions

Approximately 300 mg of a commercial food wrapper was weighed and dissolved in 5 mL of tetrahydrofuran (THF). The dissolved sample was applied to a glass capillary. After wiping the excess, the sample was introduced into the ionization source by inserting the capillary into the probe. The sample was vaporized by heated nitrogen gas in the ionization source, and ionized by corona discharge through atmospheric pressure chemical ionization (APCI). Figure 2 shows the complete sample introduction procedure.
Figure 3 shows an infusion pump connected to the ASAP ionization source. For the calibration required by MassWorks and the subsequent elemental composition determination, a calibration standard was infused using the syringe pump. Vitamin D3 solution (100 mg/L) was used as the standard, and introduced at a low flow rate (approximately 50 µL/min).

Figure 3. Overview of the ASAP/MS system with an infusion pump installed to introduce the calibrant used for accurate mass calibration by MassWorks.

Results and discussion

Figure 4A shows a total ion chromatogram obtained from an analysis of the THF extraction of a food wrapper. Figure 4B shows a mass spectrum after accurate mass calibration by MassWorks. After the introduction and data acquisition of the THF extracted food wrapper, the Vitamin D3 (C_{27}H_{45}O_{2}, m/z 385) standard solution was introduced from the infusion pump. A characteristic ion was found at m/z 403.2463 in the food wrapper sample by direct rapid analysis without chromatographic separation. A CLIPS search within MassWorks revealed the most likely elemental composition as C_{20}H_{35}O_{8}+ (proton adduct) with the highest spectral accuracy of 99.5 %.

Figure 5 shows the top six hits arranged in descending order of spectral accuracy, which measures the goodness of fit between the measured MS profile data after calibration and that calculated for the given formula candidate. This combination of the single quadrupole mass spectrometer with an ASAP installed and MassWorks software allowed rapid analysis and economical formula determination for unknown substances contained in a food wrapper.

Figure 4. ASAP analysis of a food wrapper.

Figure 5. MassWorks CLIPS elemental composition determination results table for the unknown m/z 403.2463 ion.
Various public databases can be searched for the structure of the result obtained here. Figure 6 shows a hit from an online search in ChemSpider, one of the chemical databases, using the elemental composition obtained from this analysis. This hit points to acetyl tributyl citrate, a frequently used plasticizer in food wrappers.

Conclusions

We tested a rapid analysis approach for food wrappers using the Agilent 6120 LC/MSD with an ASAP installed. This rapid analysis, including the calibration standard introduction required by MassWorks, was accomplished in minutes. With this calibration standard data from the rapid analysis, the MassWorks software can calibrate the quadrupole mass spectrometer data into accurate mass, allowing elemental composition determination of unknowns. With the obtained elemental composition, we searched a compound database and found an additive contained in a food wrapper. This study confirmed that this system configuration is applicable to the rapid and accurate mass analysis of additives.

The ASAP probe is an ionization source for direct analysis provided by IonSense, Inc.

MassWorks is a post processing MS calibration and analysis software provided by Cerno Bioscience.

Figure 6. One example of ChemSpider search results for \( \text{C}_{20}\text{H}_{34}\text{O}_8 \).