Extractables and Leachables Analysis of Common Household Food Storage Products using a Quadrupole Time-of-Flight (Q-TOF) Mass Spectrometer

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1. Overview

Using a high-resolution mass spectrometer to investigate extractables and leachables in plastic sandwich bag, clear plastic cup, and aluminum foil.

2. Introduction

Since food contact materials (FCMs) are in direct contact in consumer products, the existence of extractables and leachables (E&L) in the packaging and storage materials can raise health and safety concerns. To address these concerns and comply to the regulations, companies demand methods and techniques to analyze their products for research and development as well as quality assurance purposes. In response to this increasing demand, we developed a method to analyze extractables and leachables in FCMs using a liquid chromatography high accuracy mass spectrometer.

3. Methods

Common food storage products including plastic bag (Ziploc), plastic cup, and aluminum foil (Reynolds) were selected to be analyzed. Equal size strips (3 x 5 cm) from each sample were extracted in water, 3% acetic acid, 10% ethanol, and 95% ethanol. Samples extraction was done in an oven at 70ºC for 24 hours. Samples in 95% ethanol were extracted under 60ºC for 6 hours due to the high alcohol content. Blank was collected in every matching conditions for comparisons.

A Restek Viva C18 column (100 x 2.1 mm; 3um) was used for chromatography separation. Mobile phase A was water with 0.05% formic acid and 5 mM ammonium formate. Mobile phase B was acetonitrile. A generic gradient from 30% to 100% B in 7 minutes was used and the total run time was 15 min. The injection volume was 3 µL and the column oven temperature was set to 40 ºC.

A Shimadzu LCMS-9030 Quadrupole time-of-flight mass spectrometer was used in scan mode (*m/z* 100-2000) for the potential extractable and leachable ions identification. Data dependent acquisition (DDA) was employed to automatically trigger MS/MS scan (*m/z* 50-2000) when a threshold of ion intensity was reached. Nebulizing gas, heating gas and drying gas were set at 3 L/min, 10 L/min, and 10 L/min, respectively. DL temperature was set at 250 ºC and the heat block temperature was set at 400 ºC.

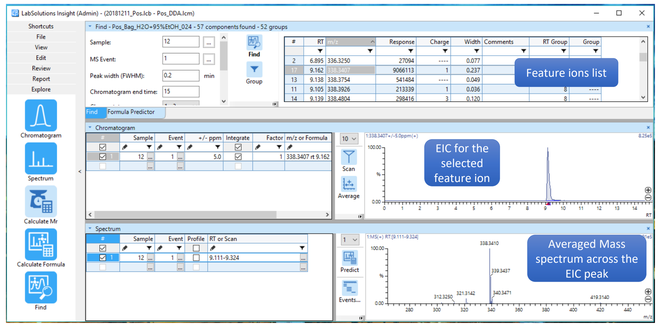
LCMS-9030 Q-TOF MS

4. Results

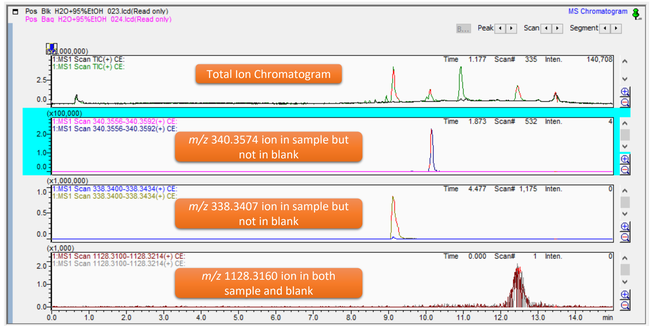
4-1. Extractables and leachables analysis workflow

The overall workflow for identifying unknown extractables and leachables in polymers can be broken down into four steps outlined in figures below. Plastic bag extracted in 95% ethanol was used as an example.

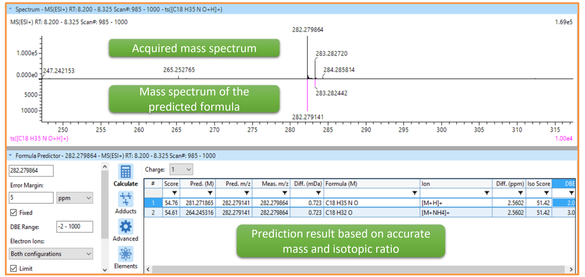
Step 1:
Find the feature compounds in sample by **Find Algorithm.**



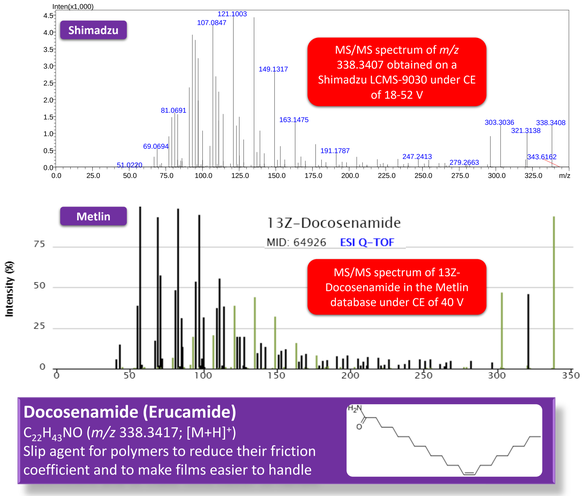
Step 2:
Compare features of blank and sample chromatograms in Data Browser.



Step 3:
Use **Formula Predictor function** to identify compounds according to the accurate mass and isotopic pattern.



Step 4 (optional):
Confirm the formula by comparing MS/MS spectrum with open source databases.



Docosenamide (Erucamide)
$C_{22}H_{43}NO$ (*m/z* 338.3417; $[M+H]^+$)
Slip agent for polymers to reduce their friction coefficient and to make films easier to handle

4-2. Potential extractable and leachable ions detected in food storage product

Table1 Suspected extractable and leachable ions detected in different food storage products using various extraction solvents

Extraction Solvent	Food Storage Product: Ziploc sandwich bag	Food Storage Product: Plastic cup	Food Storage Product: Reynolds Wrap Aluminum foil
H_2O	2(2-2-(hexanoyloxyl)ethoxy)ethyl octanoate; Docosanamide; Erucamide; Octanoic acid, 1,1'-(1,2-ethanediylbis(oxy-2,1-ethanediyl)) ester; Oleamide	2(2-2-(hexanoyloxyl)ethoxy)ethyl octanoate; Dicumyl peroxide; N,N-bis(2-hydroxyethyl) dodecanamide; Palmitic acid; Irganox 1310	Oleamide
3% AA	Dimethyl Terephthalate;Docosanamide; Erucamide; Octanoic acid, 1,1'-(1,2-ethanediylbis(oxy-2,1-ethanediyl)) ester; Octylated diphenylamine; Oleamide	N,N-bis(2-hydroxyethyl) dodecanamide	2,6-di-t-butyl-4-hydroxy-4-methyl-2,5-cyclohexadienone
10% EtOH	Dimethyl Terephthalate;Docosanamide; Erucamide; Octanoic acid, 1,1'-(1,2-ethanediylbis(oxy-2,1-ethanediyl)) ester; Octylated diphenylamine; Oleamide	2(2-2-(hexanoyloxyl)ethoxy)ethyl octanoate; Dicumyl peroxide; Irganox 1010; N,N-bis(2-hydroxyethyl) dodecanamide; Palmitic acid; Dodecanamide; Irganox 1310	2(2-2-(hexanoyloxyl)ethoxy)ethyl octanoate; Octadecyl-m-t-butyl-p-hydroxyphenylpropionate (OTHP); Oleamide
95% EtOH	Dilauryl thiodipropionate DLTDP; Dimethyl Terephthalate; Docosanamide; Erucamide; Ethyldiphenylphosphine oxide; Irganox 1076; Octanoic acid, 1,1'-(1,2-ethanediylbis(oxy-2,1-ethanediyl)) ester; Octylated diphenylamine; Oleamide; Triphenylphosphine oxide; Stearic acid	Dicumyl peroxide; Dilauryl thiodipropionate DLTDP;Erucamide; Irganox 1010; N,N-bis(2-hydroxyethyl) dodecanamide; Triphenylphosphine oxide; Dicumyl peroxide; Palmitic acid; Stearic acid	Octadecyl-m-t-butyl-p-hydroxyphenylpropionate (OTHP); Oleamide

5. Conclusions

A Shimadzu high resolution accurate mass LCMS-9030 Q-TOF mass spectrometer was used to identify extractables and leachable in Ziploc sandwich bag, plastic cup, and Reynolds Wrap aluminum foil. Base on the accurate mass information, 19 unknown ions from both positive and negative modes were tentatively identify and listed in Table 1. Irganox 1010 and 1310 were detected only in plastic cup and Octadecyl-m-t-butyl-p-hydroxyphenylpropionate (OTHP) were detected only in the Reynolds Wrap aluminum foil. Docosanamide is a smoothing and releasing agent for PVC, polysterene, and other plastic. It was detected in Ziploc plastic sandwich bag. Erucamide is a slip agent for polymers to reduce friction and was also detected in the plastic bag.

Shimadzu Insight Explore and LabSolutions Browser were used to screen feature ions. The identities of these unknown ions were investigated using formula predictor according to the accurate mass and the isotopic ratio. Further confirmation can be done by comparing the fragmentation data to a MS/MS library. In this case, Metlin library was used. Highly similar fragment patterns between experimental unknowns and database were observed which further supports the identification results.