

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

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Overview

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

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.

Method

Common food storage products purchased from local grocery store including plastic bag, plastic cup, and aluminum foil 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 70 °C for 24 hours. Samples in 95% ethanol were extracted under 60°C 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; 3µm) 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 °C.

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 °C and the heat block temperature was set at 400 °C.



LCMS-9030 Q-TOF MS

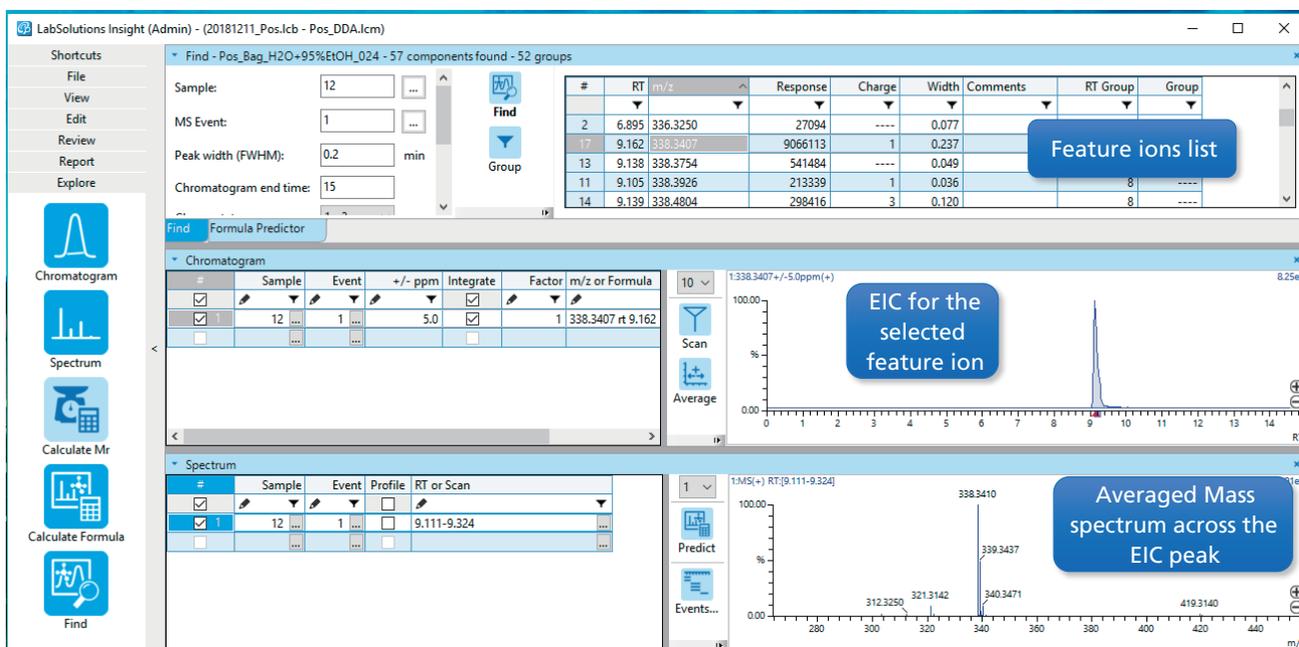
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Results

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.

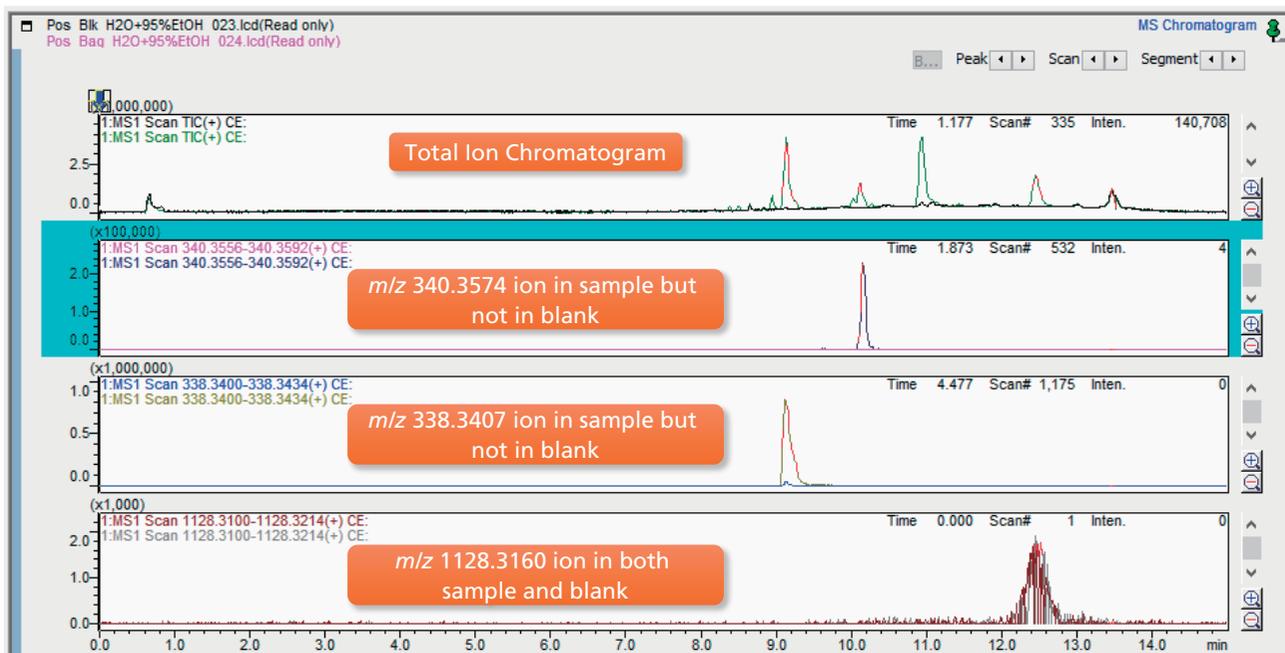
Step 1:
Find the feature compounds in sample by Find Algorithm.



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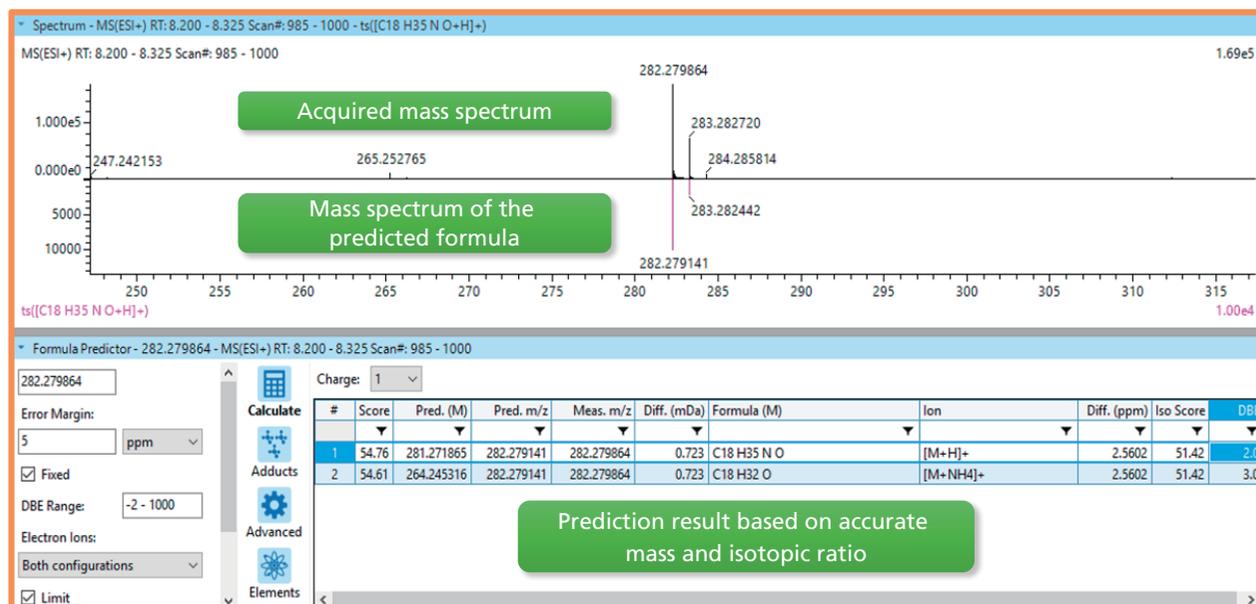
Step 2:

Compare features of blank and sample chromatograms in Data Browser.



Step 3:

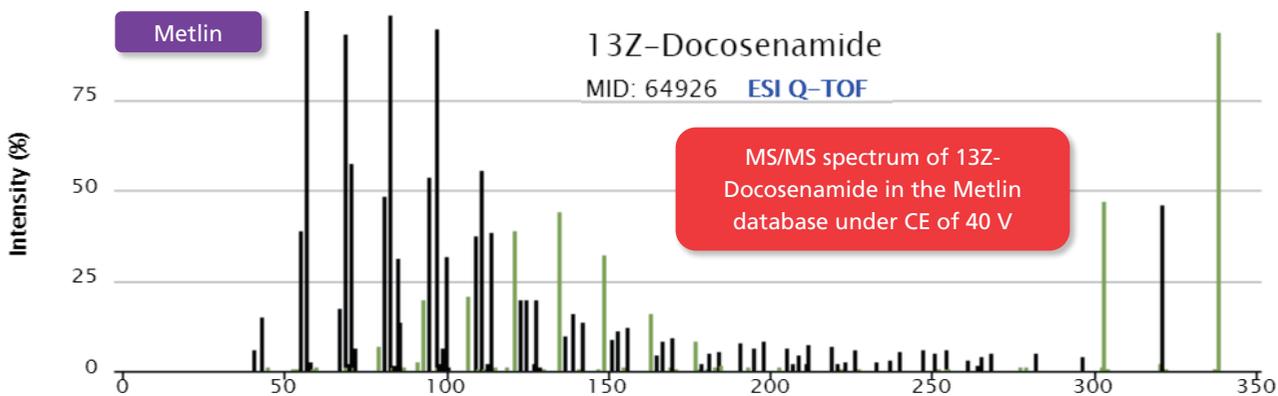
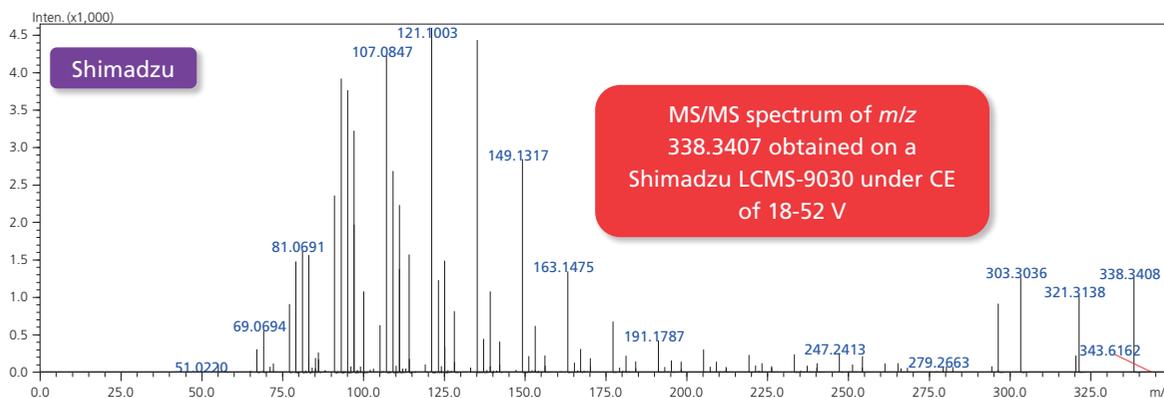
Use Formula Predictor function to identify compounds according to the accurate mass and isotopic pattern.



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Step 4 (optional):

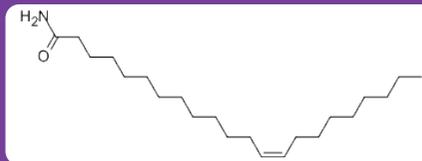
Confirm the formula by comparing MS/MS spectrum with open source databases.



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



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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

		Food Storage Product		
		Plastic Sandwich Bag	Plastic Cup	Aluminum Foil
Extraction Solvent	H2O	2{2-2-(hexanoyloxy)ethoxy}ethyl octanoate; Docosanamide; Erucamide; Octanoic acid, 1,1'-(1,2-ethanediyl-bis(oxy-2,1-ethanediyl)) ester; Oleamide	2{2-2-(hexanoyloxy)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-ethanediyl-bis(oxy-2,1-ethanediyl)) ester; Octylated diphenylamine; Oleamide	2{2-2-(hexanoyloxy)ethoxy}ethyl octanoate; Dicumyl peroxide;Irganox 1010;N,N-bis(2-hydroxyethyl) dodecanamide; Palmitic acid;Dodecanamide; Irganox 1310	2{2-2-(hexanoyloxy)ethoxy}ethyl octanoate; Octadecyl-m-t-butyl-p-hydroxyphenylpropionate (OTHP); Oleamide
	95% EtOH	Dilauryl thiodipropionate DLTDP; Dimethyl Terephthalate; Docosanamide; Erucamide; Ethylidiphenylphosphine 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

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Conclusions

A Shimadzu high resolution accurate mass LCMS-9030 Q-TOF mass spectrometer was used to identify extractables and leachable in plastic sandwich bag, plastic cup, and 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 aluminum foil. Docosanamide is a smoothing and releasing agent for PVC, polysterene, and other plastic. It was detected in 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.

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