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Extractables Analysis of Food Contact Materials Using High-Resolution GC/MS and LC/MS

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Introduction

Chemicals residing in food contact materials (FCM) can migrate from packaging and containers into food. The FCM include a variety of materials including paper and plastic among others. The substances that migrate from FCM include volatile, semi-volatile and non-volatile compounds and therefore a combination of GC/MS and LC/MS techniques is needed for comprehensive analysis of potential migrants. In this study we evaluated different types of FCM including food vacuum sealer bags, heavy duty plastic food containers as well as lined paperboard containers. Various solvents were assessed for their ability to extract different classes of food packaging-derived chemicals. For increased confidence in compound annotation, high-resolution GC and LC MS platforms were used in this study in combination with application-specific accurate mass libraries.

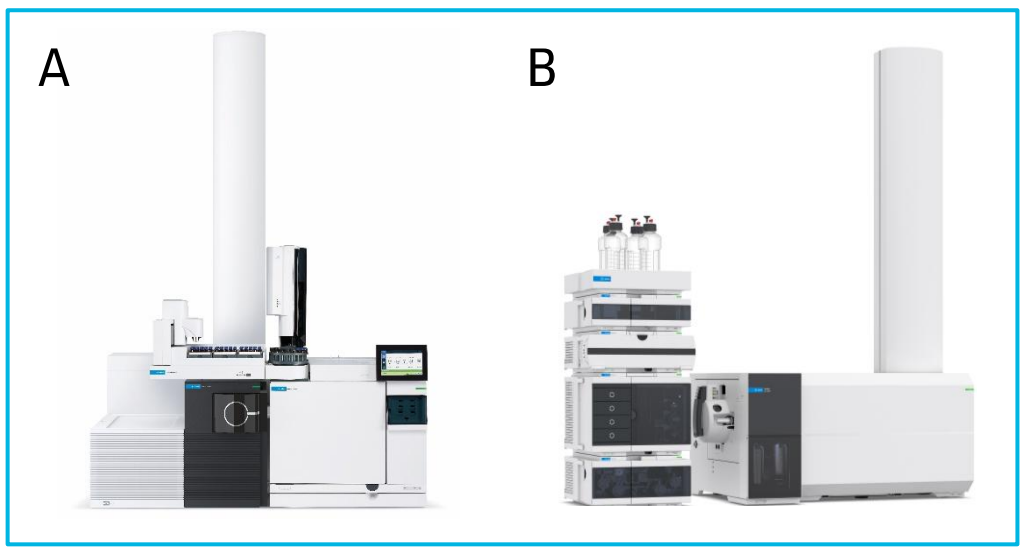

Figure 1.A) 7250 GC/Q-TOF and B) Revident LC/Q-TOF

Experimental

Different types of FCM were extracted with either ethanol, ethanol:hexane (1:1), hexane or isooctane at 70˚C for 2 hours. The extracts were centrifuged to eliminate any debris, and the supernatants were analyzed using both GC/Q-TOF and LC/Q-TOF (ethanol extyracts only). Thermal extractions (TE) of these FCM was performed using Thermal Desorption Unit.

The GC/MS data processing was performed in MassHunter Qualitative Analysis and Unknowns Analysis software. Compound identification leveraged an expanded accurate mass GC/Q-TOF E&L library, NIST23, and for LC/MS a new FCM focused database and MS/MS library. Statistical analysis for the GC/MS data was performed using Mass Profiler Professional (MPP, 15.1) and with Explorer (2.0) software for the LC/MS data.

Experimental

The instrumental parameters are shown in Tables 1 and 2.

Table 1. GC/Q-TOF method parameters

Parameter	Value
MS	Agilent 7250 GC/Q-TOF
GC	Agilent 8890 GC
Column	Agilent DB-5Q, 30 m, 0.25 mm, 0.25 µm
Inlet	MMI, 4 mm Ultra Inert liner, single taper with wool
Injection volume	1 µL
Injection mode	Splitless
Inlet temperature program	65 °C for 0.01 min, 300 °C/min to 280 °C
Oven temperature program	45 °C for 2 min; 12 °C/min to 325 °C, 11 min hold
Carrier gas	Helium
Column flow	1 mL/min constant flow
Transfer line temperature	325 °C
Quadrupole temperature	150 °C
Source temperature	200 °C
Electron energy	70 eV; low energy EI (9 – 15 eV)
Emission current	5 µA for 70 eV; 0.3 – 0.7 µA (low energy EI)
Spectral acquisition rate	5 Hz
Mass range	50 to 1000 *m/z*

Table 2. LC/Q-TOF method parameters

Parameter	Value
LC	Agilent 1290 Infinity II
MS	Agilent Revident LC/Q-TOF
Analytical column	Agilent Poroshell Aq-C18 2.1 x 150 mm, 2.7µm column
Column temperature	40 °C
Solvent delay column	Agilent Poroshell EC-C18 4.6 x 50 mm, 2.7µm column
Injection volume	5 µL
Flow rate	0.35 mL/min
Mobile phase A	Water w/ 2.5mM NH_4Formate, 0.05% FA, 100 µM NH_4F
Mobile phase B	Methanol w/ 2.5 mM NH_4Formate, 0.05% FA, 100 µM NH_4F
LC gradient	2% B for 1.0 min; to 100% B at 16.0; hold 100% B to 26.0 min; to 2% at 26.1 min post time 1 min
MS source	Dual AJS ESI
MS mode	Auto MS/MS
Polarity	Positive
Collison energy	10, 20, 40 eV
MS range	40-1700 *m/z*
Acquisition rate	4 spectra/sec for MS; 6 spectra/sec for MS/MS
Drying gas	250 °C, 12 L/min
Sheath gas	300 °C, 11 L/min
Nebulizer pressure	35 psi
Capillary voltage	4000 V

Results and Discussion

Expanded Accurate Mass E&L Library for GC/Q-TOF

To enhance compound annotations, the GC/MS accurate mass extractables and leachables (E&L) library was expanded and now contains over 500 compounds (Figure 2). The new entries included additional packaging contaminants: antioxidants and UV stabilizers, a wide range of phthalates, ink components, pigments, accelerators and other compounds from FDA's Chemical List for Analytical Performance (CLAP). The standards for building the E&L PCDL were from the CLAP kit and the Agilent Food Contact E/L Standards Kits obtained from AChemTek.

Results and Discussion

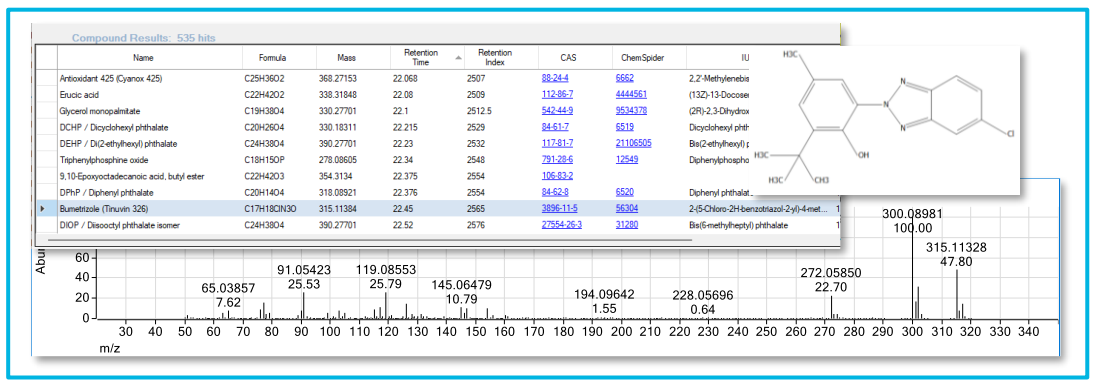

Figure 2. Accurate mass E&L GC/Q-TOF PCDL contains EI spectra as well as the metadata, including molecular structure and database identifiers

Comparison of Extractions and Different Types of Food Containers Using GC/Q-TOF

For plastic bags and paper containers ethanol extracts covered a wider range of compound classes and provided the largest number of unique compounds as compared to other solvents, while for plastic containers hexane:ethanol (1:1) extractions were most efficient (Figure 3).

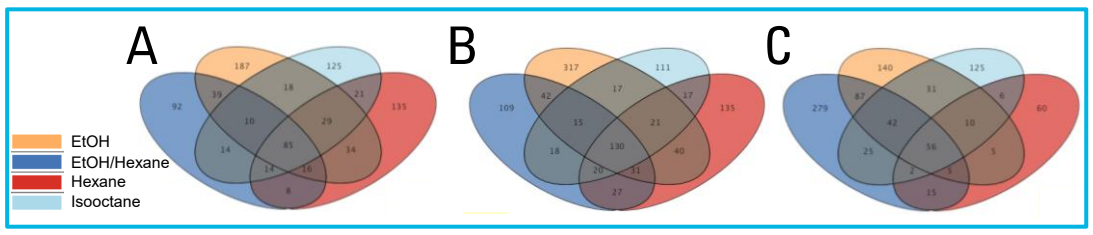

Figure 3. Comparison of plastic bags (A), paper (B) and plastic containers (C) extractions with different solvents.

The isooctane extracts exhibited higher extraction efficiency for many types of hydrocarbons, compounds with tert-butyl groups as well as branched secondary and tertial alcohols, such as Irgafos 168 phosphate (a triaryl phosphite), 2,6-di-tert-butylbenzoquinone and 2,2,4-trimethyl-3-pentanol (Table 3).

Table 3. Compounds preferentially extracted by isooctane as compared to ethanol.

Compound Name	Retention Time	Formula	Fold Change (FC) Plastic bag	Fold Change (FC) Plastic container	Compound Name	Retention Time	Formula	Fold Change (FC) Plastic bag	Fold Change (FC) Plastic container
2,3-Dimethylhexane	4.36	C8H18	1.74E+05		2,5,6-Trimethyloctane	7.66	C11H24		19.0
4,4-Dimethyl-2-pentanone	4.49	C7H14O	9.43E+06		tert-Butyl 3-hydroxybutanoate	7.82	C8H16O3	6.58E+05	
Tetrachloroethylene	4.80	C2Cl4	3.99E+04		2,4-Dimethyldecane	8.63	C12H26	4.54E+05	
2-tert-Butyl-3-methyloxetane	4.95	C8H16O	3.35E+07		6-Methylheptane-1,6-diol	9.10	C8H18O2		21.2
2,2,5,5-Tetramethylhexane	5.04	C10H22	6.03E+05		2,9-Dimethyldecane	9.18	C12H26		17.4
2,2,4-Trimethyl-3-pentanone	5.16	C8H16O	1.51E+07	23.1	2-Butyl-3-methyl-1-heptene	9.68	C12H24	4.62E+05	
4-Methyloctane	5.33	C9H20		18.2	Texanol	12.20	C12H24O3	1.19E+05	
1,3-Dimethylbenzene	5.45	C8H10		18.8	Surfynol 104	12.53	C14H26O2		16.5
2-Methyl-2-heptanol	5.48	C8H18O	1.07E+08	9.06E+07	(E)-3-Tetradecene	12.63	C14H28	1.09E+06	
5-Methyl-2-hexanol	5.57	C7H16O	1.30E+06	18.9	2,6-Di-tert-butylbenzoquinone	13.21	C14H20O2	12.6	
5,5-Dimethylhexanal	5.66	C8H16O	3.81E+06		TBP / Tributylphosphate	14.95	C12H27O4P	4.82E+04	
2,2,4-Trimethyl-3-pentanol	5.69	C8H18O	48.1	3.9	Cyclotetradecane	15.04	C14H28	8.5	
2,7-Dimethyloctane	6.35	C10H22		17.2	3,5-di-tert-Butyl-4-hydroxybenzaldehyde	16.13	C15H22O2	3.8	
2,4,4-Trimethyl-1-pentanol	6.69	C8H18O	1.69E+06	19.8	Irgafos 168 Phosphate	28.67	C42H63O4P		4.3

The extracts of different packaging materials showed distinct profiles (examples are shown in Table 4 and Figure 4). Plastic container extracts showed hydrocarbons as their primary constituents. Antioxidants, such as Irganox 1076, Irgafos 168 and Irgafos 168 phosphate prevailed in both plastic bags and plastic containers. Plastic bag extracts, apart from hydrocarbons and antioxidants, contained caprolactam, erucamide, and 1,8-diazacyclotetradecane-2,9-dione as some of their major components. Paperboard container extracts displayed the most complex profile. In addition to high diversity of hydrocarbons they also contained numerous plant secondary metabolites, PAH and other trace levels contaminants. Interestingly, Bisphenol AF was detected in all types of FCM, and trace amounts of Bisphenol A were present in plastic container and plastic bag.

Table 4. Selected compounds present at significantly different levels in plastic bag, and paper and plastic containers.

Compound	Retention Time	Formula	FC ([P] vs [PB])	Regulation ([P] vs [PB])	FC ([PC] vs [PB])	Regulation ([PC] vs [PB])	Frequency	Primary Hit Source
Diethylene glycol ethyl ether	7.51	C6H14O3	-4.92E+06	down	-4.92E+06	down	3	PCDL
Caprolactam	10.88	C6H11NO	-9.90E+08	down	-9.90E+08	down	3	NIST23
PTBP / 4-tert-Butylphenol	11.22	C10H14O	2.8	up	-9.53E+05	down	6	PCDL
Surfynol 104	12.54	C14H26O2	-1.44E+06	down	-1.04E+08	down	4	PCDL
2,4-Di-tert-butylphenol	13.61	C14H22O	-7.4	down	-2.7	down	9	NIST23
Irgacure 184 (1-Hydroxycyclohexyl phenyl ketone)	15.59	C13H16O2	-2.18E+06	down	-2.18E+06	down	3	PCDL
3,5-di-tert-Butyl-4-hydroxyacetophenone	16.56	C16H24O2	-7.67E+04	down	2.6	up	6	NIST23
Methyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate	17.58	C18H28O3	-2.21E+06	down	-1.1	down	6	NIST23
Irgafos 168 (Antioxidant 168)	27.17	C42H63O3P	-3.04E+07	down	11.7	up	6	PCDL
Irgafos 168 Phosphate	28.68	C42H63O4P	-93.7	down	-21.5	down	9	PCDL
Irganox 1076	28.92	C35H62O3	-5.05E+08	down	-7.6	down	6	PCDL
TEP / Triethyl phosphate	9.07	C6H15O4P	9.09E+03	up	-22.1	down	4	PCDL
5-Chloro-2-methyl-3(2H)-isothiazolone	10.46	C4H4ClNOS	2.70E+05	up	-1	down	3	NIST23
Benzo[a]pyrene	24.32	C20H12	5.63E+05	up	-1	down	3	PCDL

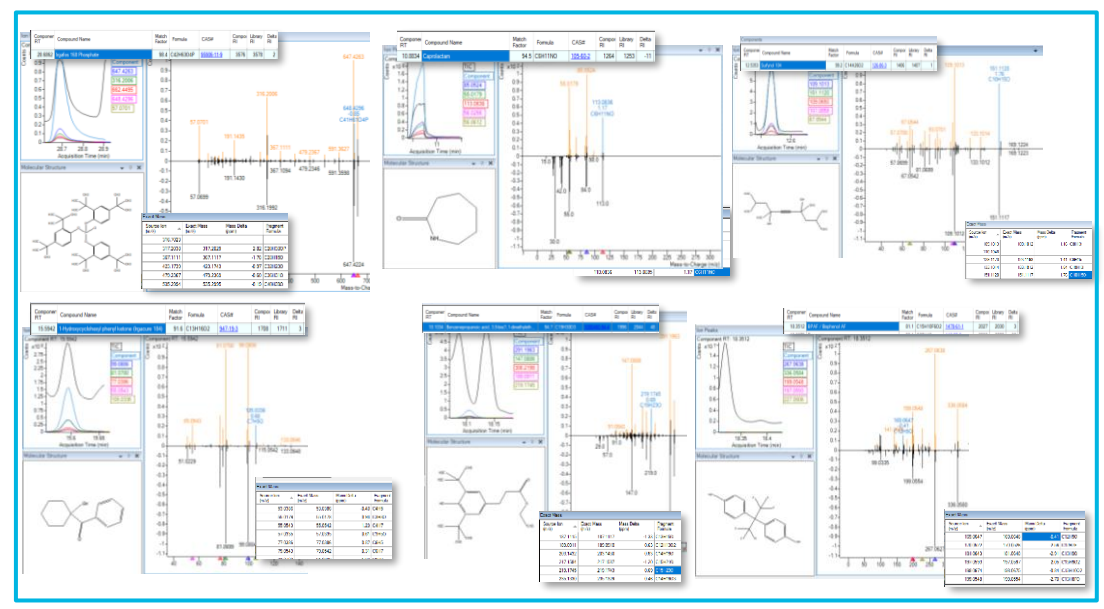

Figure 4. Examples of spectral and accurate mass matches for selected compounds from the extracts.

Direct Thermal Extraction of the Wax Liner and Paper

Coatings for certain FCM provide a “protective” barrier between the food and main material used for the container. This creates a more complex sampling requirement to identify the origin of E&L components of interest. A small amount (~5mg) of the wax coating and paper container were added to a GERSTEL TD tube and directly heated on the instrument (Figure 5).

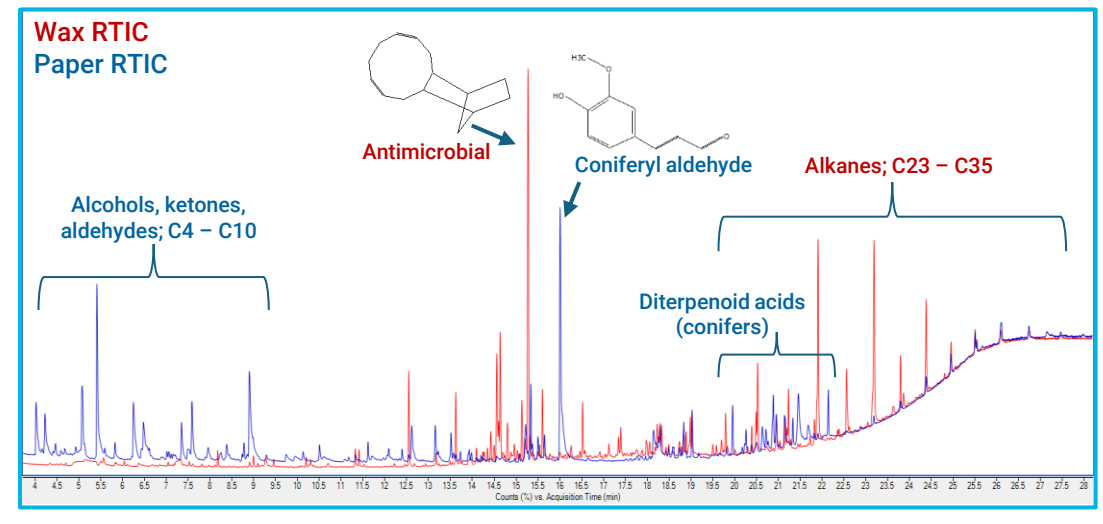

Figure 5. An overlaid chromatogram showing the difference in TE compounds with tentative IDs using LEEI and PCI.

Results and Discussion

Comparison of EtOH Extractions of Plastic Bags of Different Brands by GCMS

Ethanol extracts of plastic bags B and C compared to A showed prevalence of fatty esters (typically used as lubricants and slip agents, Figure 6). Surfactants, solvents, plasticizers and plastic degradation products dominated in extract A. Interestingly, antioxidants were present at similar levels in all plastic bag extracts.

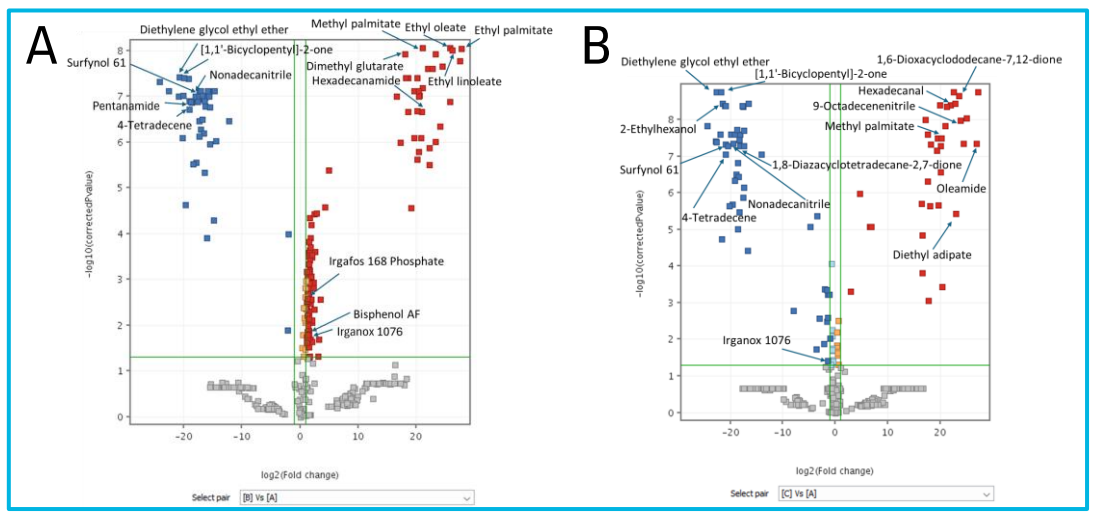

Figure 6. Volcano plot comparing B vs A (A) and C vs A (B) plastic bag extracts. Selected compounds are labeled.

Structure Elucidation of Unknown EtOH Extractables from plastic bags

To predict molecular ion of unknowns, data were acquired using low energy EI (LEEI), followed by EI MS/MS for further structure elucidation in MSC (Figure 7).

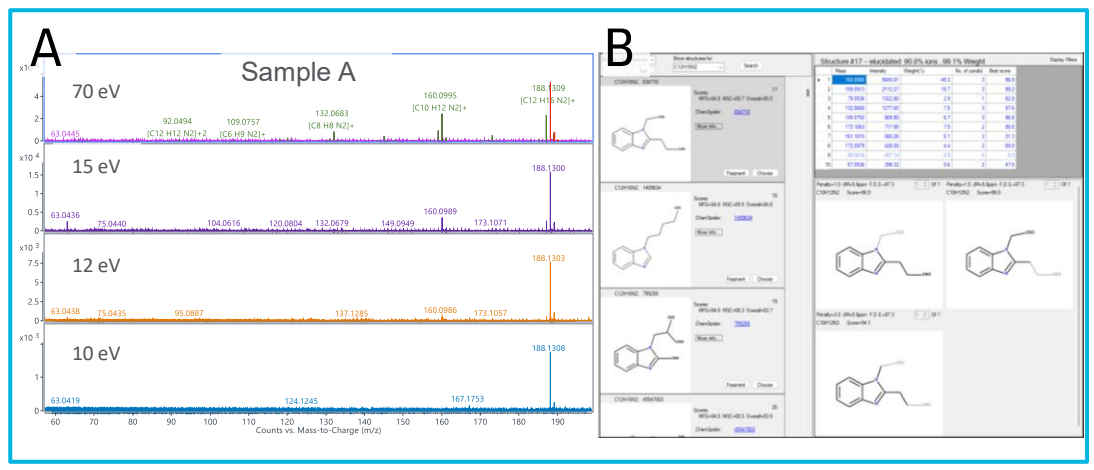

Figure 7. 70 eV and LEEI spectra of an unknown in sample A (A); one of the possible structures for the same compound based on MS/MS experiments (B).

LC/MS Compatible Compound Identification Database

The Agilent ChemVista Library management software provides means to build application focused databases that merge online content from sources such as the EPA Comptox Dashboard, the Food Packaging Migration and Food Contact Material databases with in-house collected MS/MS spectral data or spectra downloaded from Mass Bank of North America. A more detailed description of the building of this application focused database is described in Poster WP-307.

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Comparison of EtOH Extractions of Plastic Bags of Different Brands by LC/MS/MS

MassHunter Explorer 2.0 software was utilized to batch process the positive-ion and negative-ion iterative MS/MS data from EtOH extracts, generating list of features by retention time, neutral mass and abundances. Explorer allows data to be reviewed by conditions: sample (A,B,C), duration time (10, 20, 60 min.), Sample type (blank/ex) all filtered by an abundance threshold of 10K. Compounds present in Blank and all the extracts are easily removed by filtering by 4X fold change. As shown in the GC/MS work, the antioxidants (Irganox 1010, 1076, 1135, 1141, 565 and Irgafos Phosphate) were present in about the same level in all the extracts. The C18-C24 Amides and alkyl ethanol amines higher in the C sample. Cyclic nylon dimers, trimers, tetramers were only detected in A.

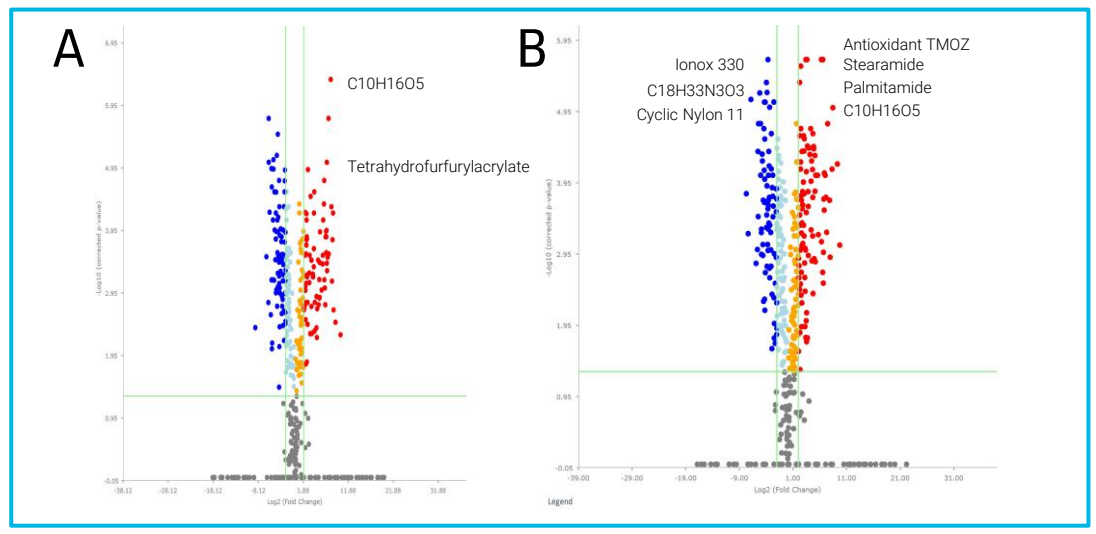

Figure 8. Volcano plot comparting B vs C (A) and C vs A (B) plastic bag extracts.

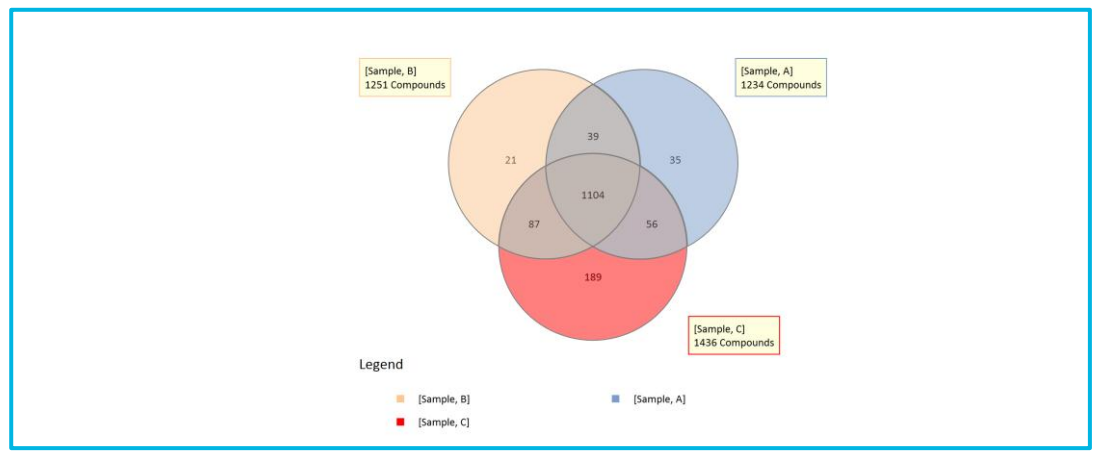

Figure 9: Unique Compounds by LCMS of Extracts

Conclusions

- FCM extractions were optimized and successfully analyzed using a variety techniques based on high-resolution GC/MS and LC/MS.
- Combination of different high resolution MS techniques helped to ensure a comprehensive E&L compound coverage.
- E&L focused accurate mass libraries were key to efficient data analysis.

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