# LC World Talk

SHIMADZU'S NEWSLETTER FOR THE HPLC GLOBAL COMMUNITY

- Analysis of Melamine in Foodstuffs
- LCMS-2020 Product Introduction
- On-line Identification of Flavones from Flos Chrysanthemi by LCMS-IT-TOF
- Analysis of Synthetic Polymers using Offline SEC-MALDI TOF MS





# Analysis of Melamine in Foodstuffs

#### Introduction

Melamine is a type of organic nitrogen compound that has a structure centered on a triazine ring (Figure 1). It is often combined with formaldehyde to produce melamine resin, a synthetic polymer.

In the spring of 2007, many dogs and cats died in North America due to the adulteration of wheat gluten with melamine in pet foods. It is possible that melamine was added to products to boost their apparent protein concentration, which was determined indirectly from nitrogen content measured using the Kjeldahl method. The deaths were caused due to the damage from crystals that formed in their kidneys by the combination of melamine and its metabolite cyanuric acid. In 2008, adulteration of milk products with melamine in China led to kidney problems in thousands of babies and infants.

Until the 2007 pet food incidents, melamine had not been routinely analyzed in food except in the context of plastic safety or insecticide residue. Here, we present some examples of the analysis of melamine in foodstuffs using HPLC and LCMS.

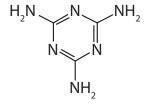
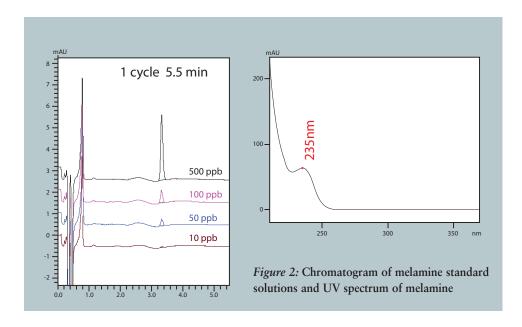


Figure 1: Structural formula of melamine



#### Quantitation of Melamine Using HPLC

Figure 2 shows chromatograms and a spectrum obtained by injecting  $10~\mu L$  of melamine standard solution. The analytical conditions are shown in Table 1. An SPD-M20A photodiode array UV-VIS detector was used for the detection. By using ultra-fast LC conditions with  $2.2~\mu m$  particle column technology, melamine could be eluted within 3.5~min.

Melamine could be analyzed with excellent linearity in the range of 10 to 500ppb (Figure 3).

#### Table 1: Analytical conditions

Column: Shim-pack XR-ODS

(75mmL.×3.0mmi.d. 2.2μm)

Guard Column: Security Guard kit ODS

4.0mmL×2.0mmi.d.

Mobile Phase: 10mmol/L Sodium

Octanesulfonate

10mmol/L Citric Acid (sodium)

Buffer (pH3.1)

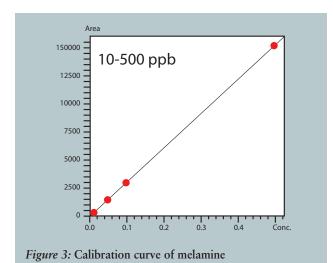
/ Acetonitrile = 92/8 (v/v)

Flow Rate: 1.2 mL/min

Temp:  $45^{\circ}$ C Inj. Volume:  $10 \mu$ L

Detection: PDA at 235nm

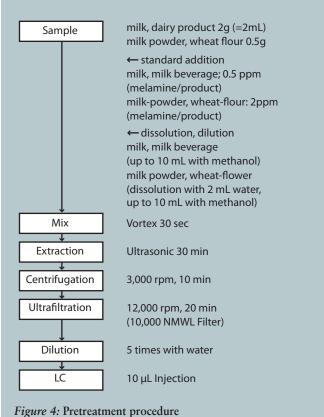
Conventional Cell



## Analysis of Commercial Milk Products and Wheat Flour

Melamine-spiked milk products and wheat flour were analyzed with LC-UV (Figure 5). After melamine was spiked into the commercial products at levels of 0.5ppm for milk and milk beverage and 2ppm for milk powder and wheat flour, the commercial products were pretreated according to the procedure shown in Figure 4.

Melamine spiked in each product could be determined well in the matrices.



1 181110 11. I Tetreatment procedure

#### Table 2: Analytical conditions

Column: Asahipak NH2P-50 2D

(2.0 mmI.D. x 150 mmL.)

Mobile Phase A: 5 mmol/L Ammonium formate

adjusted to pH 3 with formic acid

Mobile Phase B: Acetonitrile

Time Program: 90%B(0-10 min)-20%

B(10.01-20 min)-90%

B(20.01-35 min)

Flow Rate: 0.2 mL/min

Injection Volume: 1 μL Column Temp: 40°

Ionization Mode: ESI positive
Applied Voltage: 4.5 kV
Nebulizing Gas Flow: 1.5 L/min
Drying Gas Pressure: 0.1 MPa
HB Temp: 200°
CDL Temp: 250°

Monitor Ion: m/z 127.1 for melamine

### Analysis of Melamine in Milk Products by LCMS

LCMS can detect melamine directly without the derivatization requirements for GCMS analysis. In comparison with the LC-UV detection method, mass spectrometry offers superior selectivity, and LCMS is highly effective in both qualitative and quantitative analysis. Here, we present an example of LCMS analysis of melamine.

Figure 6 shows the SIM chromatograms of melamine standard solutions. The analytical conditions are shown in Table 2. An amino column, Asahipak NH2P-50 2D (2.0 mmI.D. x 150 mmL), was used for the analysis of melamine. Melamine can be analyzed quantitatively by LCMS.

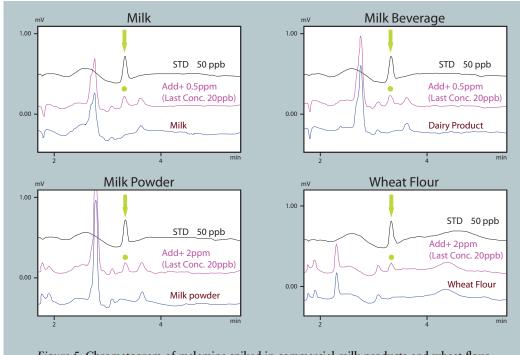
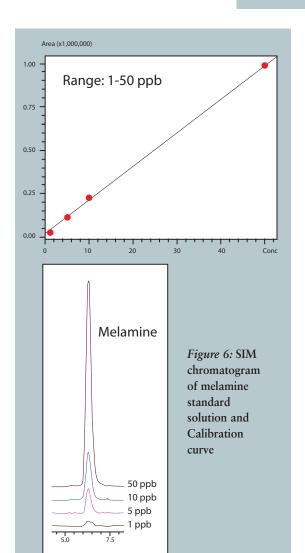


Figure 5: Chromatogram of melamine spiked in commercial milk products and wheat flour



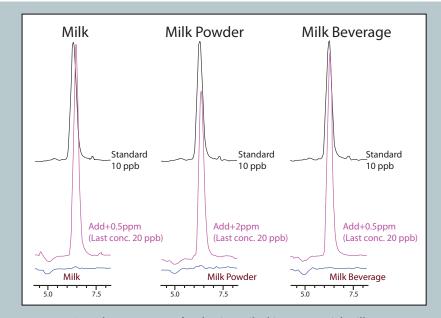


Figure 7: SIM chromatogram of melamine spiked in commercial milk products and wheat flour

Melamine-spiked milk products were analyzed with LCMS (Figure 7). The pretreatment of the commercial products were performed by the same procedure (Figure 4) as LC-UV. A melamine peak was observed after 6.2 minutes. Melamine spiked in the commercial milk products was detected well by LCMS.

#### Summary

Melamine in milk products can be quickly analyzed using HPLC with LCMS detection. These methods can be utilized as a simple screening and monitoring method for melamine detection.

## Introducing the New



## Shimadzu LCMS-2020

... the world's most sensitive, fastest scanning and fastest polarity switching single-quad mass analyzer ...

Utilizing patent-pending ultrafast (UF) technology, the LCMS-2020 has faster measurement speed and significantly higher sensitivity than any other single quadrupole analyzer. This provides more accurate detection of trace impurities in pharmaceuticals, environmental pollutants and other contaminants. Combine with a Prominence ultra-fast LC (UFLC/UFLCXR) to achieve the ultimate in separation performance and productivity.

Innovative features include:

#### **UFswitching Technology**

High-speed polarity switching between positive/negative ion modes

UFswitching technology (patent pending) enables switching between positive and negative ion modes in 15 milliseconds so even the fastest LC peaks can be analyzed in both modes, thus increasing productivity.

#### **UFscanning Technology**

Fast scanning speeds

By adopting new technology (patent pending), a scanning speed of 15,000 u/sec is achieved without sacrificing sensitivity or resolution, even during high-speed scans, obtaining the best chromatography for the fastest LC conditions.

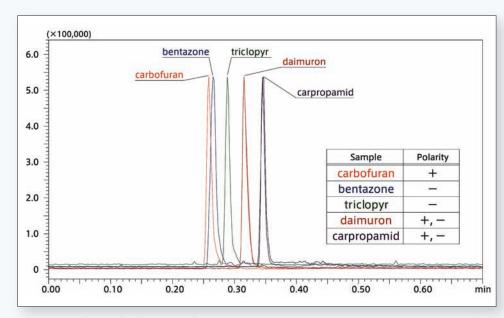


Figure 1: High-speed polarity switching

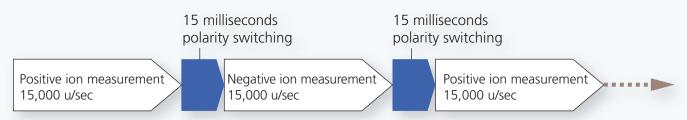
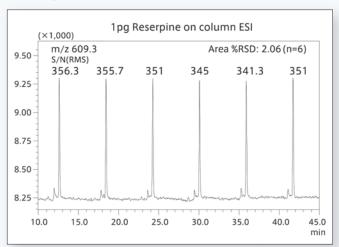


Figure 2: Fast scanning speed

#### **UFsensitivity**

Superior sensitivity, especially for ultra high-speed analysis

Innovative ion optics with the newly developed Qarray® ion optical system provide superior sensitivity, repeatability, and linearity, achieving 50% to 300% greater sensitivity than any other single quadrupole analyzer for substances most commonly measured.



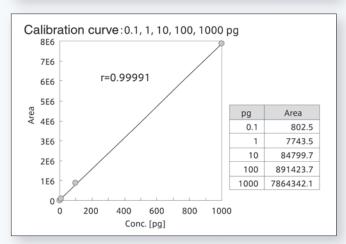
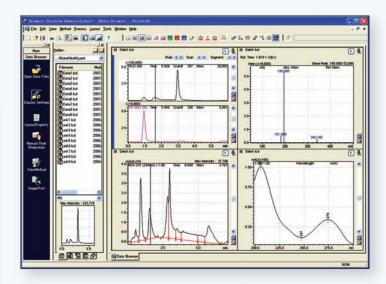


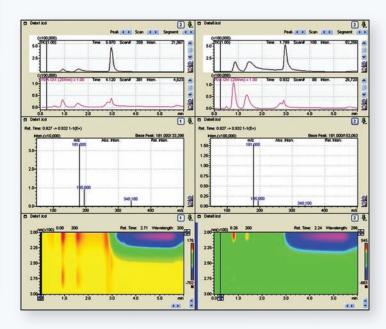
Figure 3: Superior sensitivity, repeatability and linearity

#### **Easy Operation**

The ESI method typically used for LCMS can be replaced with APCI, which is suitable for neutral compounds, or with a dual ionization source (DUIS) for simultaneous operation of both ionization modes, quickly and without tools. In addition, the desolvation line, where the ions enter the MS unit, can be replaced without breaking the vacuum.

LCMS-2020 control and data processing are handled by an updated version of LCMSsolution software. The new software includes an improved data browser function, which allows comparing multiple sets of data or confirming impurities by linking to LC or MS data. The new software controls the LCMS-2020 and is fully integrated with Shimadzu's Prominence Series of ultrafast and nano HPLCs.





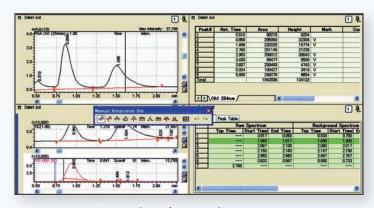


Figure 4: Operation window of LCMS solution

## On-line Identification of Flavones



## from Flos Chrysanthemi by LCMS-IT-TOF

#### **Abstract**

The aim of this study was to establish an LC-UV (post column derivatization) ESI-MS<sup>n</sup> method for rapid and exact analysis of flavones found in medicinal herbs used in Traditional Chinese Medicine. Based on the LCMS-IT-TOF and on-line post-column derivatization technique, the fingerprint of a methanolic extract of *Flos Chrysanthemi* was systemically investigated.

The data from derivatization and high-precision multi-stage mass experiments allowed the identification and determination of the structures of 18 flavones. This analytical method is of great value in the rapid identification of flavones, and can provide important information for further study of bioactive natural products in medicinal herbs.

#### Introduction

Nowadays, many herbal medicines have been proved to be safe and effective. As an alternative treatment method, medicinal herbs have attracted significant attention. *Flos Chrysanthemi* is a commonly used Traditional Chinese Medicine with diverse activity, such as anti-bacterial, anti-fungal, anti-viral, anti-inflammatory, and anti-oxidant activities. It also has been reported to reduce risks of certain cancers and cardiovascular diseases. According to previous chemical studies, the main chemical compositions of *Flos Chrysanthemi* are essential oil, flavonoids, caffeotannic acid, amino acids and microelements.

Mass spectrometry plays an increasingly important role in the field of natural product chemistry. Compared to other kinds of mass spectrometers, an ion trap combined with a time-of-flight mass spectrometer allows high-precision and rapid MS<sup>n</sup> experiments. It can provide precise formulae of product ions directly using both high accurate mass analysis and multistage mass fragmentation.

*Flos Chrysanthemi* has been cultivated for centuries, and there are numerous breeds, each of them possessing different chemical constituents. No systematical research concerning its chemical compositions has been reported previously. The aim of this study was to identify the main flavone compositions in one original breed of *Flos Chrysanthemi* (Hangbaiju) in China.

In addition, the UV data was not utilized much in identification work before, as it was thought only important for quantitative analysis. The addition of UV shift reagents induces a displacement of the absorption maxima that can be used to determine the positions of the free hydroxyl groups of flavone.

Therefore, a LC-UV (post-column derivatization) ESI-MS<sup>n</sup> method for rapid and exact analysis of chemical components found in herbal medicine was developed and applied for the identification and determination of flavones in *Flos Chrysanthemi*.

#### **Experimental**

Table 1: Structures of the compounds identified

$$R_7$$
  $R_4$   $R_5$   $R_4$ 

Name R <sub>5</sub>		R <sub>7</sub>	R₃	R <sub>4</sub>	MW(Da)	
Luteolin	ОН	ОН	ОН	ОН	286.045	
Apigenin	ОН	ОН	Н	ОН	270.053	
Acacetin	ОН	ОН	Н	OCH₃	284.066	

#### **HPLC-UV/PAD Analysis**

UV analysis of the methanolic extract of *Flos Chrysanthemi* was performed on a Shimadzu (Kyoto, Japan) Prominence LC-20A liquid chromatography system with PDA. The separation was achieved on a YMC C18 column (150 × 4.6 mm i.d., 5  $\mu$ m) combined with a Diamonsil<sup>TM</sup> C18 column (250mm×4.6mm i.d., 5 $\mu$ m). The components were eluted with a linear gradient of acetonitrile–water (containing 0.02% formic acid in positive ion mode), shown in Table 2. The oven temperature was 40° C, flow rate was 1.0 mL/min, UV detection was at 335 nm, and UV spectra (PAD) were recorded between 190 and 400 nm.

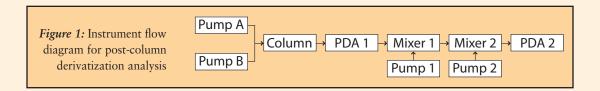
Time (min)	Acetonitrile (containing 0.08% formic acid) (%, v/v)	0.08% formic acid (%, v/v)
0	12	88
15	20	80
55	21	79
60	25	75
80	34	66
100	40	60
110	60	40
120	100	0
130	100	0

Table 2: Gradient conditions of HPLC mobile phase

#### **HPLC-MS<sup>n</sup> Analysis**

HPLC-MSn was performed directly after UV/PDA measurements. A Shimadzu LCMS-IT-TOF mass spectrometer (Kyoto, Japan) with an electrospray ionization (ESI) interface was used with the following conditions: Interface voltage and current were 4.50 kV and 1.6  $\mu$ A for positive ion mode, and -3.50 kV and 1.7  $\mu$ A for negative ion mode, respectively. Nebulizing gas was 1.5L min. CDL (Curved Desolvation Line) and heat block temperature were both 200°C. MSn experiments were performed by programming dependent scan events. All of the relative energy in collisions was 50%. Detector voltage of the TOF analyzer was 1.70 kV. Ultra-high purity argon was used as the collision gas for CID experiments.

Mass calibration was carried out using a trifluoroacetic acid sodium solution (2.5 mmol L-1) from 50 to 1000 Da. Data acquisition and processing were performed using LCMSsolution software supplied with the instrument. The ion accumulation time for each precursor ion was 50 ms.



The method used for post-column addition of UV shift reagents was based on a previously reported protocol (Wolfender and Hostettmann, 1993). For flavone analysis, the following reagents were used: at room temperature, sodium acetate (weak base), sodium hydroxide (strong base) and sodium acetate/ boric acid; and at 90°C (to ensure full derivatization): aluminum chloride under acidic and non-acidic conditions.

Shift Reagent	Pump 1	Flow 1 (ml/min)	Pump 2	Flow 2 (ml/min)	рН	Temp. (°C)
AICI <sub>3</sub>	0.02 M NaOH	1	0.3 M AICI <sub>3</sub>	0.3	8	90
AICI <sub>3</sub> / HCI	0.02 M NaOH	0	0.3 M AICI <sub>3</sub> / HCI	0.3	3.5	90
NaOAc	0.02 M NaOH	0.9	0.5 M NaOAc	0.5	8	90
NaOAc/H₃BO₃	0.02 M NaOH	0.5	0.1 M NaOAc- 0.7 M H <sub>3</sub> BO <sub>3</sub> = 1:1	0.6	5	90

Table 3: Experimental conditions for the post-column addition of UV shift reagents

#### **Results and Discussion**

#### LC-UV (post-column derivatization) ESI-MSn analytical results

With the optimized parameters,  $MS^n$  (n $\leq$ 5) data were obtained in both positive and negative ion modes, providing prolific structural information. A total of 25 compounds in the Flos Chrysanthemi HPLC fingerprint are identified and determined. 18 compounds presented typical UV spectra of flavones. Identified flavones with on-line UV and MS structural information are given in Table 5, in which the 18 identified flavones are divided into four groups.

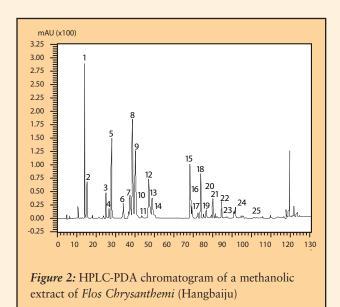
Table 5: On-line UV and MS(+) structural information of a methanolic extract of Flos Chrysanthemi (I-II=UV absorption maxima bands; Sh=shoulder)

										NaOAc/			
NO	Compound Rt (+)ESI-MSn		(+)ESI-MSn	UV spectra AICI₃/HCI		AICI <sub>3</sub>		NaOAc		Н₃ВО₃			
		, ,		ll ii	ı	II	I	II	I	II	I	II	I
			505 4007 440 4040(007 0540)	Luteolin	type					054.000			
3	luteolin-7-O- rutinoside	24.732	595.1667 → 449.1043(287.0540) → 287.0545 → 153.0190(135.0442)	254 266 (sh)	348	270	383	270	406	254 266 (sh)	345	258	362
5	luteolin-7-O- glucoside	27.865	449.1064 → 287.0557 → 153.0205 (135.0446) → 83.0170	254 266 (sh)	348	270	384	269	405	255 265 (sh)	348	258	363
13	luteolin-7- O-(malonyl) glucoside	48.493	535.1071 → 449.1055 → 287.0531 → 153.0181(135.0454)	253 266 (sh)	347	268	382	270	411	250 265 (sh)	343	258	364
19	Luteolin	76.119	287.0563 → 153.0204(135.0445)	253 266 (sh)	348	271	353 382	270	400	271	359	259	364
				Apigenin	type								
6	apigenin-7-O –rutinoside	33.588	579.1685 → 433.1096 → 271.0610 → 153.0178(119.0458)	267	336	273 295	337 374	273 294	331 374	266	339	266	339
9	apigenin-7-O- glucoside	39.93	433.1153 → 271.0605 → 153.0188(119.0496) → 97.0268	267	336	273 295	337 374	273 294	337 373	266	338	266	337
15	apigenin-7- O-(malonyl) glucoside	67.96	519.1168 → 433.1131 → 271.0565 → 153.0192(119.0463) → 97.0316	267	336	273 295	338 374	273 294	336 376	266	339	266	339
16	apigenin-7- O–(acetyl) glucoside	68.901	475.1242 → 271.0597 → 153.0203(119.0481)	267	336	273 295	337 374	273 294	337 373	266	340	266	338
20	apigenin-7- O–(acetyl) glucoside	78.504	475.1217 → 271.0586 → 153.0210- 119.0476	267	336	273 295	337 371	273 295	336 374	266	336	266	341
22	apigenin-7-O- (malonyl-acetyl) glucoside	83.845	561.1208 → 475.1210 → 271.0574 → 153.0197	266	336	273 295	337 374	273 295	336 376	266	336	266	342
23	Apigenin	90.328	271.0605 → 153.0211(119.0520)	267	336	273 295	337 374	274 296	338 374	273	356	266	337
				Diosmetin	type								
10	diosmetin-7-O- rutinoside	40.885	609.1831 → 463.1203 → 301.0722 → 286.0484 → 258.0517 → 229.0450	252,267	344	270	350 382	272	350 380	252,267	341	267	347
14	diosmetin-7-O- glucoside	49.564	463.1260 → 301.0669 → 286.0451 → 258.0505 → 229.0501	253,267	347	273	349 381	270	349 381	265	349	266	348
17	diosmetin-7- O-(malonyl) glucoside	71.707	549.1221 → 301.0684 → 286.0476 → 258.0532 → 229.0518(153.0142)	252,267	347	271	350 381	272	352 377	252,267	344	268	348
	Acacetin type												
18	Linarin	73.176	593.1861 → 447.1290 → 285.0780 → 270.0517 → 242.0579 → 153.0188	267	332	273 296	337 375	274 296	336 376	267	335	266	336
21	acacetin-7-O- glucoside	79.445	447.1273 → 285.0753 → 270.0497 → 242.0589 → 153.0196	267	332	274 296	337 374	274 295	336 374	266	335	266	335
24	acacetin-7- O-(malonyl) glucoside	90.807	533.1301 → 447.1310 → 285.0762 → 270.0503 → 242.0574 → 153.0160	267	332	274 296	337 375	274 296	336 377	267	335	267	335
25	acacetin-7-O- (malonyl -acetyl) glucoside	101.423	575.1390 → 489.1349 → 285.0803 → 270.0515 → 242.0538 → 153.0214	269	332	271 294	337 375	273 294	335 374	266	332	268	333

#### Results and Discussion (continued)

Table 4: LCMS<sup>n</sup> data of standards; data from the Flos Chrysanthemi HPLC fingerprint

No	Name	[M+H]+ (Calculated)	[M-H]- (Calculated)	MSn Data (Observed) in Positive	MSn Data (Observed) in Negative
1	Luteolin	287.056	285.040	287.0556 → 153.0180 → 111.006	285.0379 → 175.0399 → 131.0449
2	Apigenin	271.061	269.045	271.0606 → 153.0180 → 97.029	269.0455 → 149.0283 → 105.0379
3	Acacetin	285.076	283.061	285.0842 → 270.0538 → 242.0595 → 153.0204 → 97.0276	283.0623 → 268.0369 → 240.0409 → 211.0385 → 183.0446



Based on the (±)ESI-MSn data, UV shift information, the

standards and many related references, the structures of 18 flavones were identified.

#### **Novel Aspects**

It has been widely accepted that the medicinal effects of herbal medicines are dependent on the interaction of their multiple constituents. The HPLC fingerprint technique is used as a quality control method in order to ensure the consistency of herbal medicines. However, the HPLC fingerprint can only provide peak retention time without information about chemical structures. Even when chemical standards were used in this kind of fingerprint analysis, identification results were obtained based on retention times, which are not always conclusive.

Therefore, the LC-UV-ESI-MSn method to identify and determine the constituents in herbal medicines was developed. This method can provide multiple and accurate chemical structural information based on high-precision data and

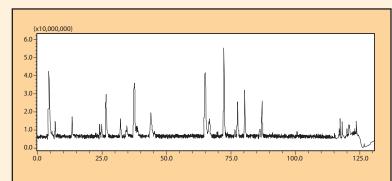


Figure 3: The total ion chromatogram of a metholic extract of Flos Chrysanthemi (Hangbaiju) in positive ion mode

multiple-stage mass fragment information. In addition, the application of on-line post derivatization helps to confirm exact positions of free hydroxyl groups in flavone, which guarantees the accuracy of identified results. Also, it is a facile method to obtain the UV shift results of numerous constituents simultaneously. As a result, the application of the LC-UV-MS<sup>n</sup> technique with on-line post derivatization will increase the reliability and accuracy of quality control work and also improve work efficiency.

Additionally, with the multiple structural information acquired, it can become a guide for chemistry study in herbal medicine and solve numerous problems in this area.

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## Analysis of Synthetic Polymers using Offline SEC-MALDI TOF MS



#### **INTRODUCTION**

Size exclusion chromatography (SEC) is a chromatographic method commonly used to separate large molecules or macromolecular complexes such as proteins and industrial polymers. It operates on the principle of separation of components according to their size or hydrodynamic volume. Particles of differing size will elute through the column's stationary phase at varying rates.

SEC can be used as a measure of both the size and the polydispersity of a synthesized polymer—i.e. the distribution of polymer molecules of varying size. If standards of a known size are analyzed previously, a calibration curve can be created to determine the sizes of polymer molecules of interest. Alternatively, techniques such as light scattering and/or viscometry can be used online with SEC to yield approximate molecular weights without reliance on calibration with standards of known molecular weight. However, these techniques are not particularly accurate and, to complicate matters further, two polymers can have the same molecular weight but differing structures—this added complication can be investigated using mass spectrometry.

Recently, the general applicability of offline-coupled SEC-MALDI TOF MS has been demonstrated by the characterization of a wide variety of synthetic polymers having polydispersities in the range of 1.7-3.0, and extended with end-group analysis from isotopically resolved mass spectra of oligomers.

However, offline SEC-MALDI TOF MS involves fraction collection, evaporation, pipetting, etc. and is laborious and time-consuming. Consequently, direct deposition methods in which SEC fractions and MALDI matrix are directly deposited onto the MALDI target are preferred. The following examples show the union of SEC and MALDI via an automated robotic spotting device, the AccuSpot<sup>TM</sup>. The configuration of the whole system is shown in Figure 1.

#### **METHODS**

#### **SEC Conditions**

The following typical SEC conditions are utilized to perform the separation component of the analysis. An SEC chromatogram is shown in Figure 2.

A standard AccuSpot automated deposition device is modified slightly to accommodate the organic mobile phase used (THF). This modification consists mainly of changing the polyimide coated fused silica delivery capillary to a teflon capillary (0.375 mm OD x 0.05mm ID).

Column:	Shodex GF310A-1E (1.0 mmID×250 mm)
Flow rate:	10 μL/min
Mobile phase:	THF
Detector:	UV (λ=220/254nm)
Sample amount:	1 μL (40μg)

Following SEC separation, the column eluent is delivered directly to the AccuSpot. Here, it is mixed with a MALDI matrix and spotted automatically onto a MALDI target.

#### **AccuSpot Conditions**

Spot interval: 6 seconds Loading: 1  $\mu$ L/well Total loading (mixture of matrix and cationizing reagent): 0.2  $\mu$ L/well

The resultant samples were then analyzed by MALDI TOF MS to generate a profile of each well.

#### **MALDI TOF MS Conditions**

Matrix: Dithranol-20 mg/ml
Cationizing reagent: Ag-TFA-10 mg/mL
Instrument: AXIMA Performance<sup>TM</sup>

Co-polymer used in this study: poly(styrene-b-ethyleneoxide): poly(styrene-b-EO)

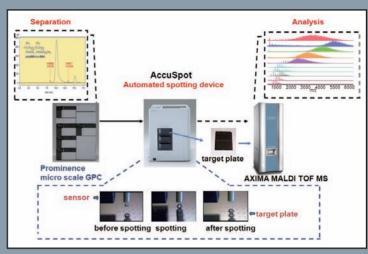


Figure 1: SEC-MALDI TOF MS configuration

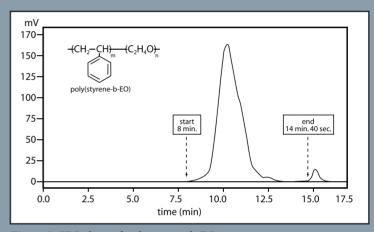


Figure 2: SEC chart of poly(styrene-b-EO)

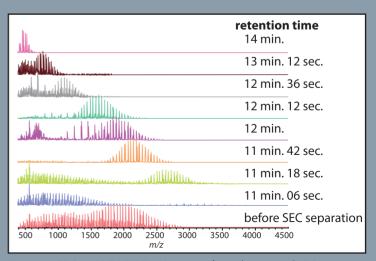


Figure 3: SEC MALDI TOF MS spectra for poly(styrene-b-EO)

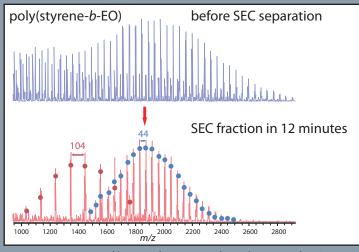


Figure 4: Detection of homopolymer units for poly(styrene-b-EO)

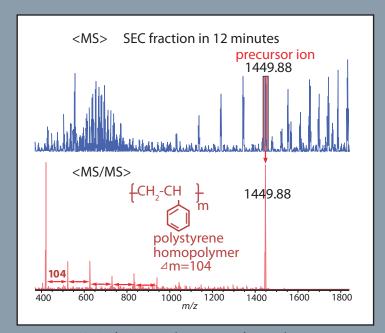


Figure 5: MS/MS of m/z 1449 showing  $\Delta m$  of 104 indicating a polystyrene structure

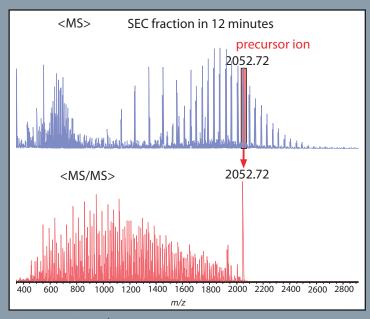


Figure 6: MS/MS of m/z 2052

#### **RESULTS**

The MALDI TOF MS spectra displayed in Figure 3 represent the SEC separated fractions for poly(styrene-b-EO) with time intervals indicated, in addition to the complex un-separated sample. The detection of homopolymer units is shown in Figure 4.

Individual precursor ions may be easily selected from the MS spectra as candidates for MS/MS. The spectra in Figures 5 and 6 show the valuable structural information that may be gained from MS/MS analysis.

#### **CONCLUSIONS**

By combining micro-LC (SEC) with the AccuSpot and AXIMA Performance, an offline SEC-MALDI TOF MS analytical system has been developed. Using the SEC-MALDI TOF MS analytical system:

- It is possible to collect SEC fractions without detriment to the separation capacity
- Frequently used SEC organic solvents (for example, tetrahydrofuran(THF)) can be utilized in combination with a spotting device and MALDI analysis
- MS analyses are achieved with adequate peak sensitivity from a practical viewpoint
- Semi-automatic analysis may be performed
- Individual precursor ions may be selected for MS/MS analysis, providing valuable structural information •

Shimadzu's Prominence Ultrafast LC and Shim-pack XR-ODS Columns Offer Practical

**Approach to Conserving Acetonitrile** 



Faced with a worldwide shortage of acetonitrile (ACN), labs are seeking ways to reduce consumption of this common HPLC solvent. To this end, **Shimadzu's Prominence UFLC/UFLCXR** systems and **Shim-pack XR-ODS** ultra-fast liquid chromatography columns minimize ACN consumption through high-speed analysis, yet retain the flexibility of conventional analysis.

Engineered for low carryover, the valves on the Prominence autosamplers require

little or no additional rinsing after injection. The injection cycle time of the autosampler is 10 seconds, further reducing mobile-phase consumption. Additionally, for situations requiring operating under pressures up to 66 MPa, **UFLCXR** permits the substitution of methanol for ACN. The development of new methods using methanol will further assist in the reduction of acetonitrile usage.

Shim-pack XR-ODS columns enable users to shorten analysis time and drastically reduce ACN consumption without high pressures (< 30 MPa) by using a 2.2  $\mu$ m particle size. Used with the Prominence UFLC system, a Shim-pack column uses only 2.3 mL of ACN for analysis of alkylphenones compared to 10.5 mL with a 5  $\mu$ m column. In addition to reduced acetonitrile usage, analysis is five times faster with the use of the Shim-pack XR-ODS and specialized high-pressure hardware is not required for operation.





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