USE OF DART-QDA FOR THE RAPID AUTHENTICATION OF FOOD AND DIET SUPPLEMENTS

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INTRODUCTION

Food and dietary supplement fraud is closely monitored. In order to protect consumers, samples need to be rapidly screened to determine the authenticity of the product. Direct Analysis in Real Time (DART) is an ambient ionization technique that eliminates the need for extensive sample preparation and chromatography. For these analyses, DART was coupled to the single quadrupole mass spectrometer (QDa). This provides a compact and easy to use system that can rapidly acquire data for authenticity testing. Authentication in two different matrices was performed; cinnamon samples and polyunsaturated fatty acid (PUFA) oil supplements.

Cinnamon samples were authenticated based on species identification. Cinnamaldehyde, coumarin, and methyl cinnamate ions were used to rapidly distinguish four cinnamon species.

The source of PUFA oil supplements (fish vs. plant) were authenticated by identifying the PUFAs present in each supplement. The composition of PUFAs was compared to the label claims of each oil supplement.

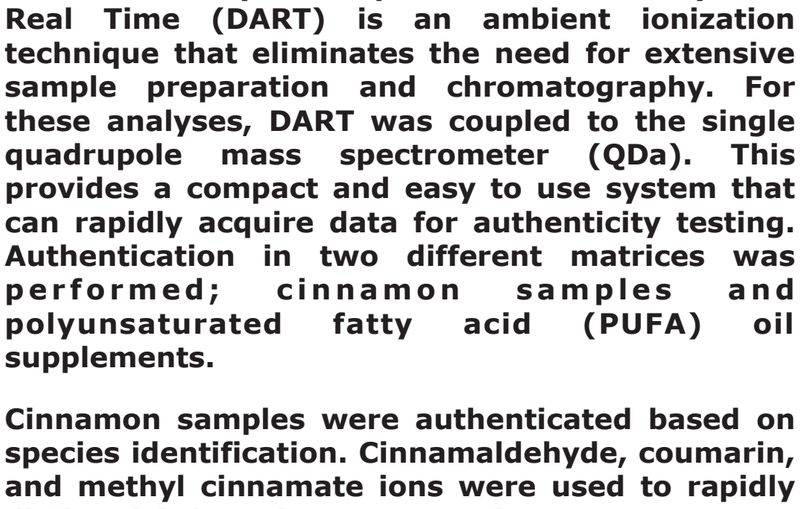

Figure 1. Images depicting DART-QDa sampling module (A) 12 spot QuickStrip card (B) automation of QuickStip card into ionization zone (C) top down look at DART ionization into QDa.

METHODS

Instrumentation and Software:

- Waters QDa (Performance) equipped with IonSense DART-SVP
- Linear X Rail module utilizing 12 spot QuickStrip sampling cards
- MassLynx software
- DART-SVP web based controller, version 5.0.0

DART-QDa Methods:

	Cinnamon	PUFA oils
	DART Method	
Ion Mode	Positive	Negative
Run Temperature (°C)	450	200
Sampling Speed (mm/sec)	1	0.5
Exit Grid Voltage (V)	350	-350

	QDa Method	
Ion Mode	Positive	Negative
Cone Voltage (V)	5	15
Mass Range (amu)	50 - 500	SIR (see table below)
Sampling Frequency (Hz)	2	2

Fatty acid	Abbreviation	SIR m/z
Palmitic Acid	PA	255.2
Alpha Linolenic Acid	ALA	277.2
Linoleic Acid	LA	279.2
Oleic Acid	OA	281.2
Eicosapentaenoic Acid	EPA	301.2
Docosahexaenoic Acid	DHA	327.2

CINNAMON ANALYSIS

Species identification of whole stick and ground cinnamon was performed to distinguish among four species of cinnamon:

- *Cinnamomum Verum*
- *Cinnamomum Cassia*
- *Cinnamomum Burmannii*
- *Cinnamomum Loureiroi*

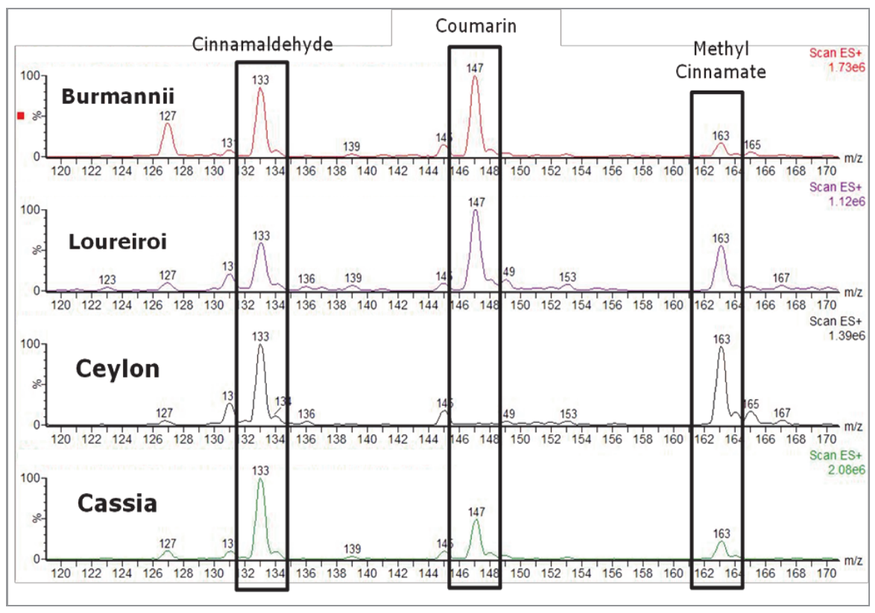

Figure 2. DART-QDa analysis of four known ground cinnamon species.

Based on the signature Cinnamaldehyde/ Coumarin/Methyl Cinnamate ratios, species identifications were made in the remaining unknown samples:

Sample	Form	Species Identification
C1	ground	Burmannii
C2	ground	Burmannii
C3	ground	Cassia
C4*	ground	Ceylon
C5	ground	Ceylon mix
C6*	ground	Loureiroi
C7	ground	Burmannii
C8*	ground	Burmannii
C9	ground	Burmannii
C10*	stick	Ceylon
C11*	stick	Burmannii
C12*	stick	Burmannii
C13	ground	Cassia
C14	ground	Burmannii
C15*	ground	Cassia

*Indicates species of sample identified on label. Used as known sample for analysis

FATTY ACID SUPPLEMENT ANALYSIS

Fatty acid dietary supplements containing known amounts of saturated and omega 3, 6, and 9 fatty acids were tested for percent composition compared against the label claim:

PUFA Supplement	Expected	Experimental
Fish Oil	60% EPA 40% DHA	59% EPA 41% DHA
Safflower Oil	63% LA 22% OA 15% PA	55% LA 33% OA 11% PA
Flax Seed Oil	67% ALA 16% LA 16% OA	57% ALA 21% LA 22% OA

Full fatty acid profile of the three oil supplements (Figure 3) shows a discrepancy in the flax seed oil supplement. DHA is derived from a fish source. Therefore a plant based oil, such as flax seed, should not contain DHA. Contamination during the manufacturing process is likely.

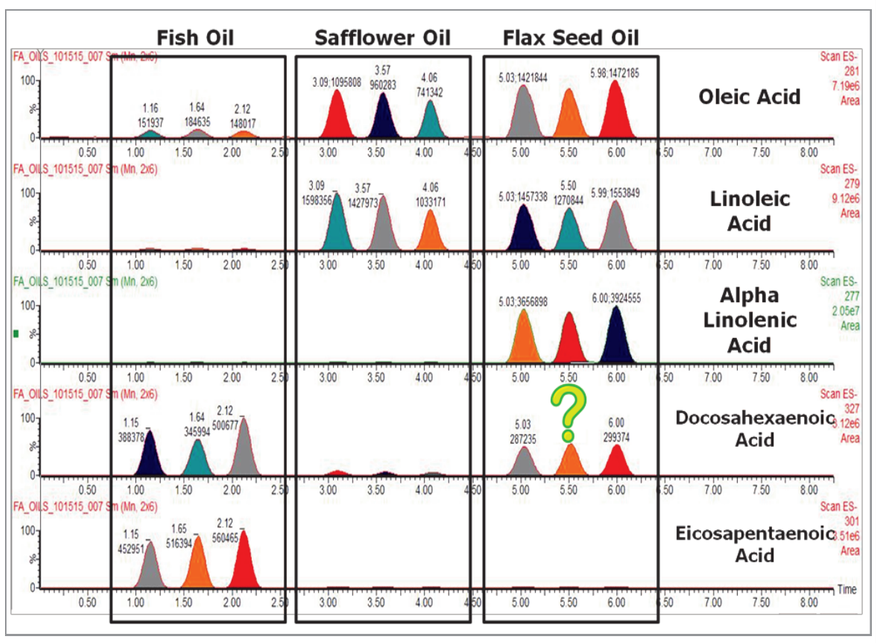

Figure 3. Extracted SIR chromatograms showing the fatty acids present in each of the oil supplements (n=3).

CONCLUSIONS

- **DART-QDa is a rapid technique successfully applied to the analysis of spice and oil supplement samples.**
- **Besides basic dilution in solvent of PUFA oils, no sample preparation was required prior to analysis.**
- **Analysis of each sample is performed in seconds.**
- **The four cinnamon species produced unique mass spectral patterns used to make species identifications of both whole stick and ground cinnamon unknown samples.**
- **The contents of the fatty acid supplements were identified and compared to the label claim of the supplement.**
- **A possible manufacturing contamination was identified in the flax seed oil supplement.**
- **DART in combination with QDa is a user friendly system that could be used on location as an authentication tool for quick screening of samples.**

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