Analysis of Vomitoxin and T-2 Toxin by LC-MS

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Introduction

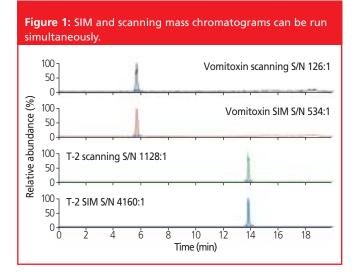
Grain and grain products may be susceptible to a variety of fungal infections. Fusarium species fungi are known to produce a number of mycotoxins including Vomitoxin (DON) and T-2 Toxin. These mycotoxins may evolve pre-harvest in the field or post-harvest during processing, storage and transportation. The presence of these mycotoxins can lead to human and animal toxicology and in the severest cases, death. The US FDA (United States Food and Drug Administration) has issued 'advisory' limits for Vomitoxin in grain and grain products as follows:

- 1 ppm: Finished wheat products for human consumption.
- 5 ppm: Grain and grain by-products destined for swine and other non-ruminant species.
- 10 ppm: Grain and grain by-products for ruminating beef and feedlot cattle older than four months.

Additional restrictions on percentage make up of the diet are also advised. Fusarium infections of grain products affect not only the vigor of the crop itself leading to lower yields, but also the quality of the crop as a feedstuff. The severity of effect of the mycotoxin contamination is to a large extent dependent on the age and species of the particular livestock. In particular, pigs are susceptible to even low levels of Vomitoxin in their feed.

Goal

This report describes a simple LC–MS method for the determination of fungal mycotoxins. It will allow analysts to develop and optimize sample preparation, extraction and clean-up techniques for these important contaminants. The flexibility of simultaneous SIM and scanning acquisition will facilitate the development of quantitative and qualitative methodologies.



Chromatographic Conditions

LC Column: 150 × 2.1 mm Packing: 5 μm C18 Flow-rate: 200 μL/min

Mobile phase: water/methanol

Gradient: 80/20 to 20/80 in 10 min, held 5 min

Injection volume: 10 µL Column temperature: 40 °C

Mass Spectrometer Conditions

Instrument: Finnigan Surveyor MSQ Ionization: electrospray; Polarity: positive

Needle voltage: 3 keV Probe temperature: 350 °C Cone voltage: 90 eV

Scan range: 100-650 Da; Scan rate: 0.8 s/scan

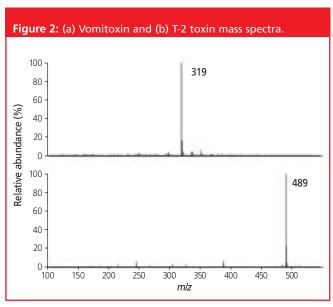
Data acquisition: Centroid

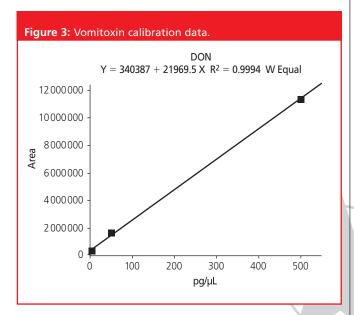
Selected ions: m/z 319 (M+23) and 489 (M+23)

Dwell time: 100 ms

Results and Discussion

Figure 1 shows the unique capability of the Finnigan Surveyor MSQ to collect selected ion monitoring (SIM) and scanning data from the same run. The improvement in signal to noise from the scanning (upper) to the selected ion (lower) acquisitions is shown on the pseudomolecular ion traces for both Vomitoxin (DON) and T-2 Toxin, respectively. This allows low-level quantitative data and qualitative survey data to be obtained from the same injection.





The full-scan mass spectral data obtained from this run is shown in Figure 2. Pseudomolecular ion species, presumably sodium ion adducts, were observed at [M+23] for both the Vomitoxin (upper) and the T-2 Toxin (lower). The calibration data from 5–500 pg/µL for Vomitoxin is shown in Figure 3. The selected ion monitoring chromatogram for the 5 pg/µL standard shows a signal to noise ratio of 28:1 and the correlation co-efficient (R²) was 0.9994. For T-2 Toxin a similar calibration line was observed (not shown) giving a signal to noise of 7:1 for the 5 pg/µL standard level and a correlation coefficient (R²) of 0.9982.

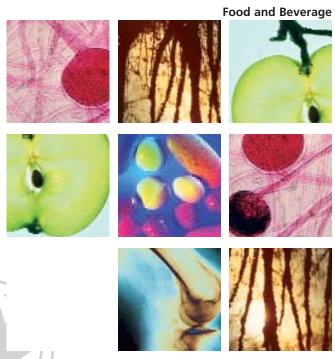
Conclusions

A simple LC–MS method for the separation and analysis of these important fungal metabolites is shown. The data show excellent linearity and sensitivity for these compounds that would readily allow determination on contamination to below ppm levels in grain samples when combined with a suitable extraction technique. The unique capability of the Finnigan Surveyor MSQ to acquire both SIM and scanning data from the same sample injection is demonstrated.



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Analysis of carboxy-THC in urine matrix



Available from Thermo Electron is an application note describing the analysis of carboxy-THC in a urine matrix. The application uses the company's Finnigan TRACE DSQ and demonstrates its ability, in the single ion monitoring mode, to locate and confirm a drug in a given urine sample. The SIM scan function of the TRACE DSQ is reportedly suitable for any method that requires the detection and quantification of the lowest levels of a compound.

Thermo Electron Corp., Austin, Texas, USA.

Pesticide analysis



Thermo Electron offers an application note that demonstrates the improvement of pesticide analysis with the application of variable damping gas, VDG, for GC–MS ion trap systems.

Combining MS–MS with variable damping gas is said to improve the detection limits by a factor of 4–5 from the default damping gas flow. Complete method parameters and examples are provided.

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