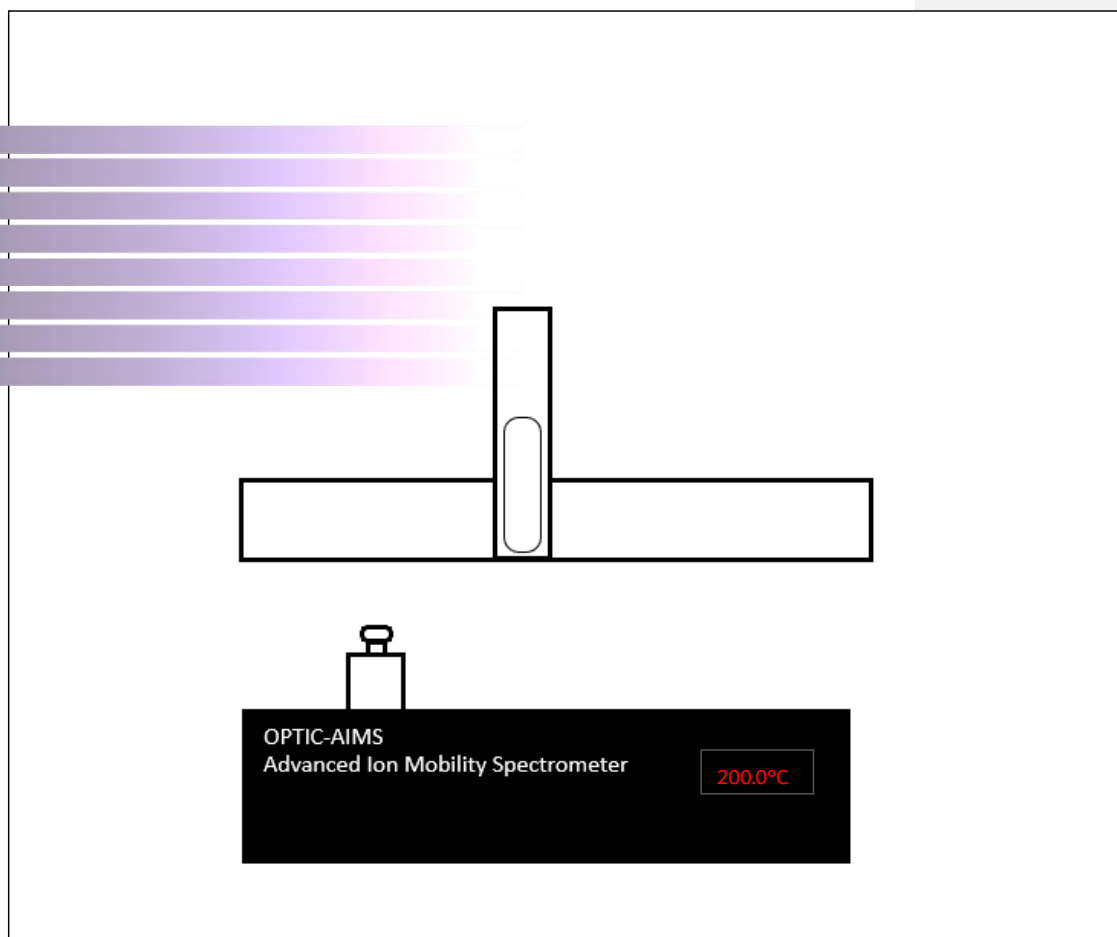


Detection of Ochratoxine A in fresh Paprika using OPTIC-AIMS



Keywords: OPTIC, AIMS, Ochratoxine A

Introduction:

Because of the toxic nature of Ochratoxine A, its maximum level in the food needs to be controlled in accordance with the guidelines established in the EU and other countries. In the EU Regulation No 1881/2006, the maximum permitted level depends on the type of product. The lowest permitted amount is 0.5 µg/kg.

It is not possible to measure Ochratoxine A directly from the raw sample without some form of sample cleanup. There are 3 basic sample cleanup methods: QuEChERS, SPE met SAX and an Immunoaffinity column. In our experiments, the full sample preparation with paprika was done without cleaning. This is to verify if the matrix does not influence the analysis outcome.

Analytical conditions:

Analyzer: OPTIC-4-AIMS, GL Sciences B.V. and MaSaTech s.r.o.
 Transfer Line: Fused Deactivated Silica, 0.25mm ID x 0.1 m long
 Transfer Line Temperature: 200°C
 Injection mode: Large Volume Injection
 Sample: 10 µL sample with 2 µg/kg Ochratoxine A with Matrix of Red Paprika
 Inlet Temperature Program: 45°C -> 30.0 °C/sec -> 500°C
 Column Flow: 20.0 mL/min conditioned Air
 Venting: 75 ml/min conditioned Air, 6 sec
 Drift tube temperature: 150°C
 Drift Flow: 900 ml/min conditioned Air
 Analysis Time: 120 seconds (including cool down time. Ready for next injection)

Peak Discovery:

Step 1: Validate if the solvent peaks do not overlap with the peak(s) of Ochratoxine A.

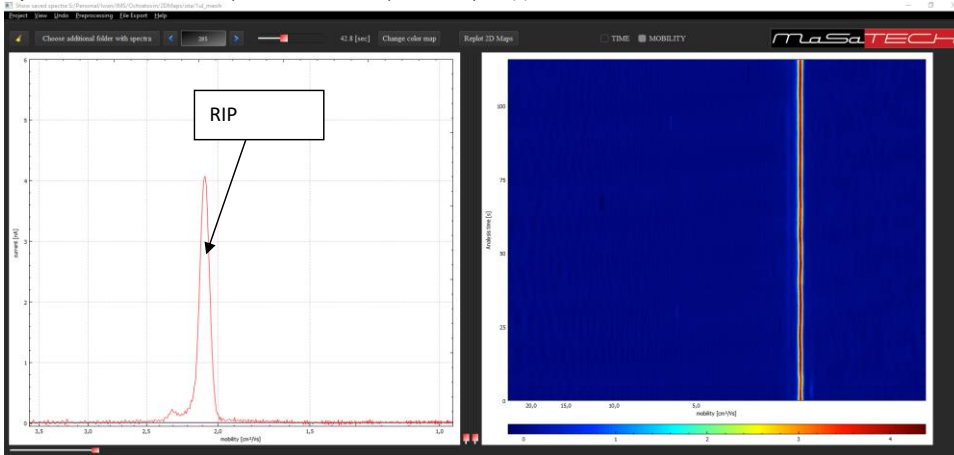
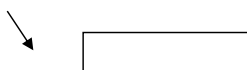


Figure 1. Plasmagram of 1 µL injection of pure Methanol.



Step 2: 1 μL injection of Ochratoxine A, 10 $\text{ng}/\mu\text{L}$ (Ochratoxin Analytical Standard -Sigma Aldrich, 34037-2ML-R)

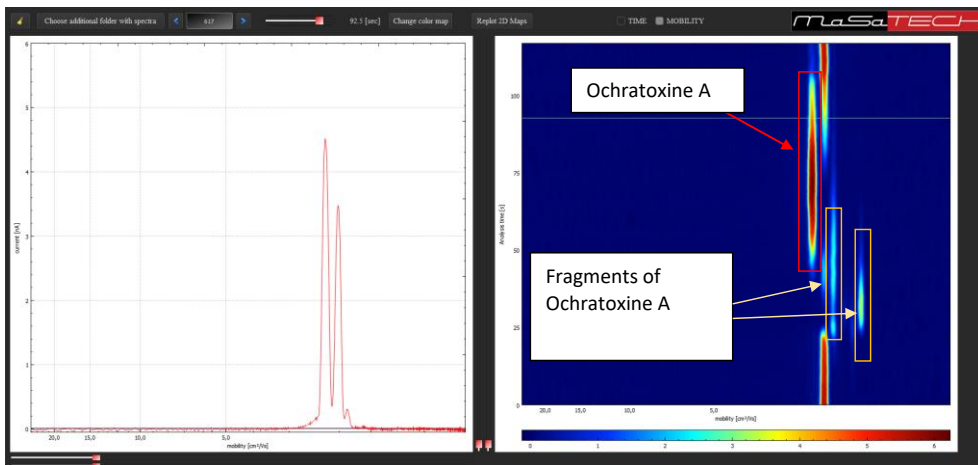


Figure 2. Plasmagram of 1 μL injection of 10 $\text{ng}/\mu\text{L}$ Ochratoxine A in Acetonitrile

Step 3: Multiple dilutions of Ochratoxine A for 10 μL injections with the following concentrations: 0.1, 0.01, 0.001, 0.0001, 0.00001 $\text{ng}/\mu\text{L}$

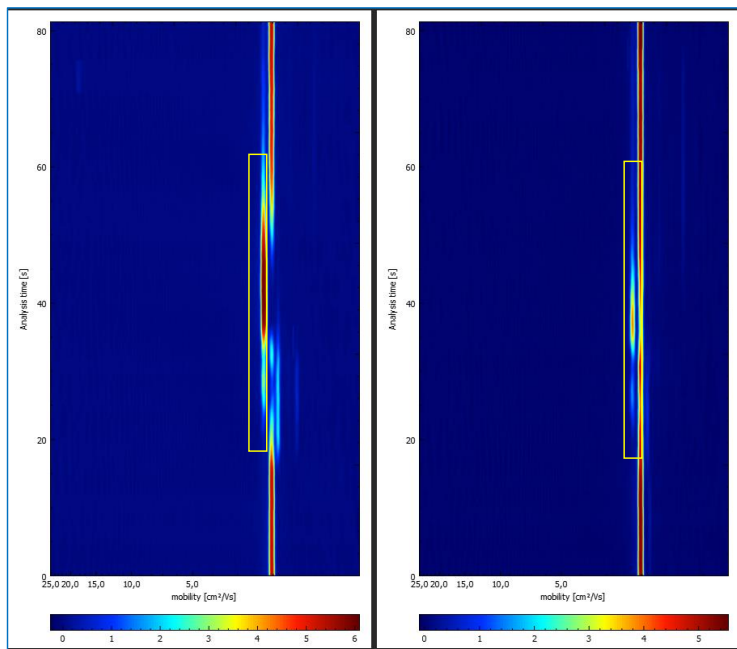


Figure 3. 0.1 and 0.01 $\text{ng}/\mu\text{L}$ Ochratoxine A in Methanol (10 μL injection)

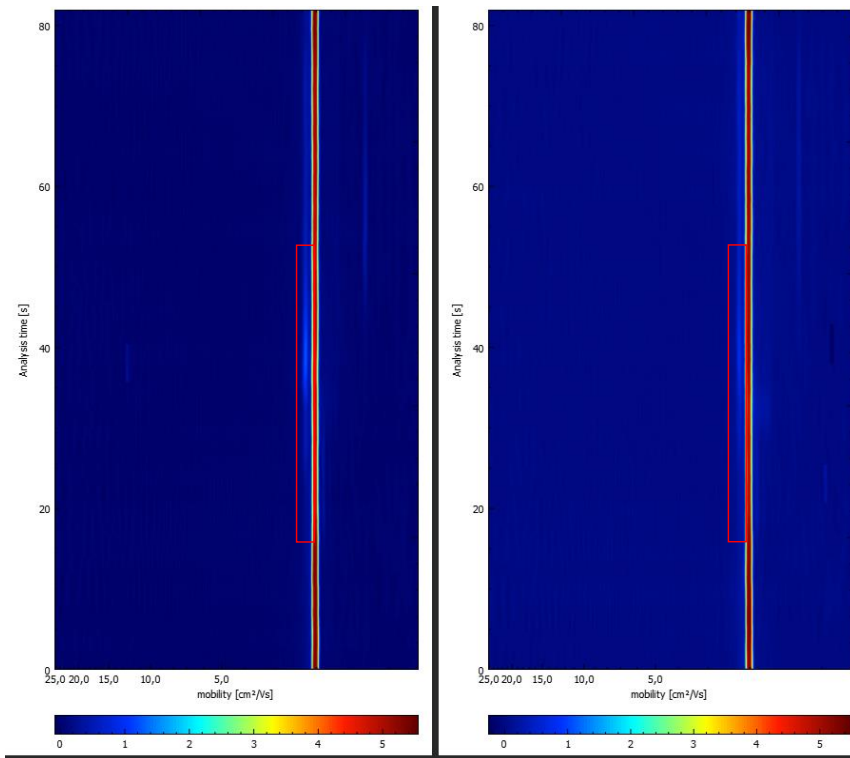


Figure 4: 0.001 and 0.0001 ng/μL Ochratoxine A in Methanol (10 μL injection)

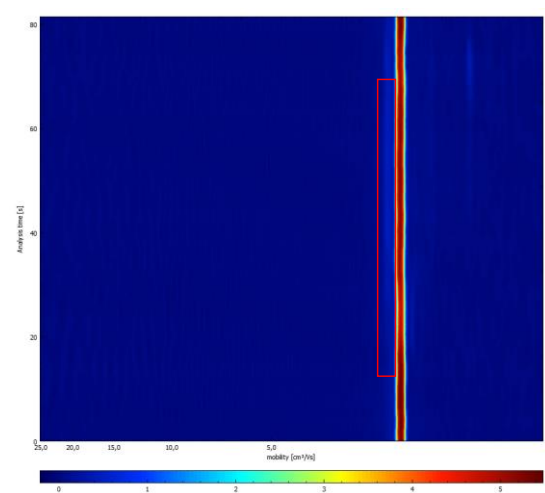


Figure 5: 0.00001 ng/μL Ochratoxine A in Methanol (10 μL Injection)

Analysis:

Below are the results of analysis of the Paprika matrix with spiked Ochratoxine A(Ochratoxine A went through a cleanup step).

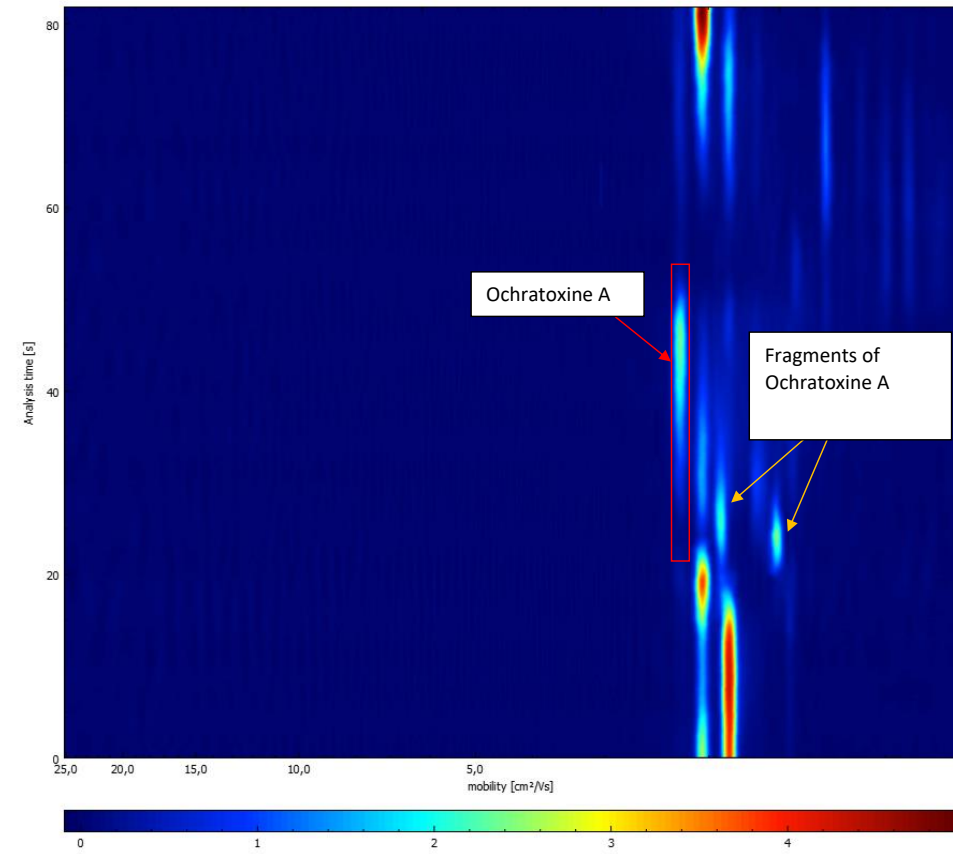


Figure 6: 10 µL injection with 2 µg/kg Ochratoxine A, cleanup step was done using InertSEP-SAX (5010-61644, GL Sciences Inc.).

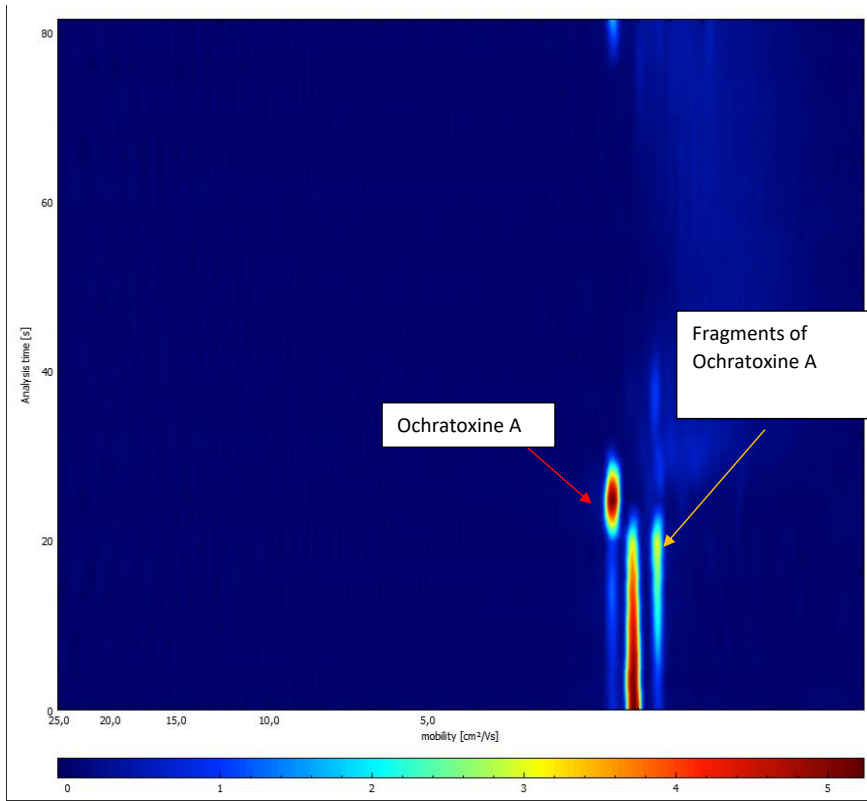


Figure 7: 10 μ L Injection of 2 μ g/kg Ochratoxine A using Quechers cleanup step (8010-1001, GL Sciences Inc.).

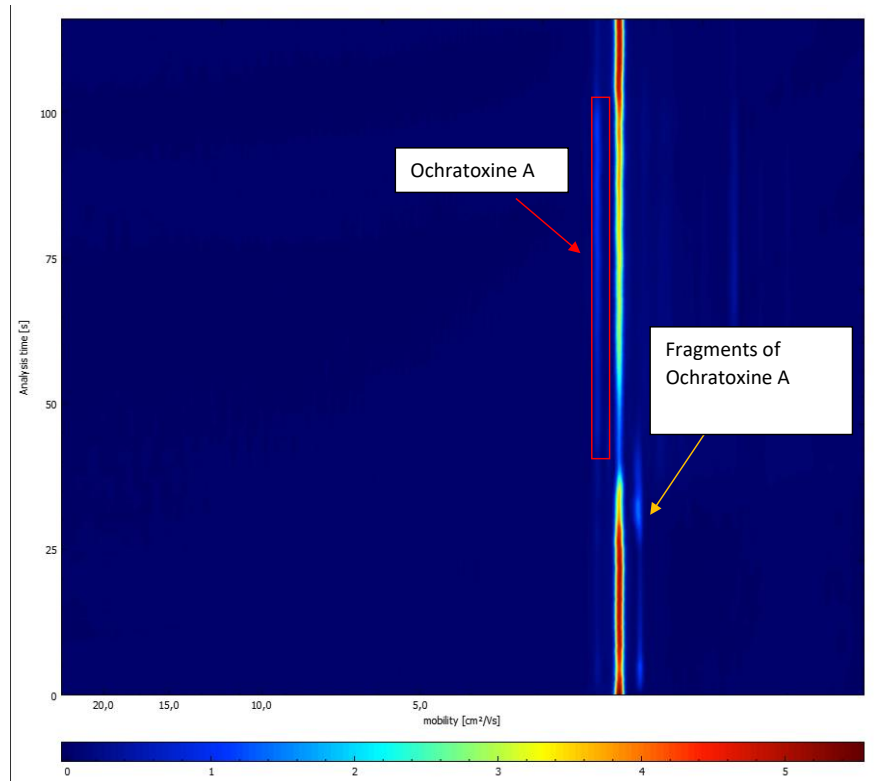
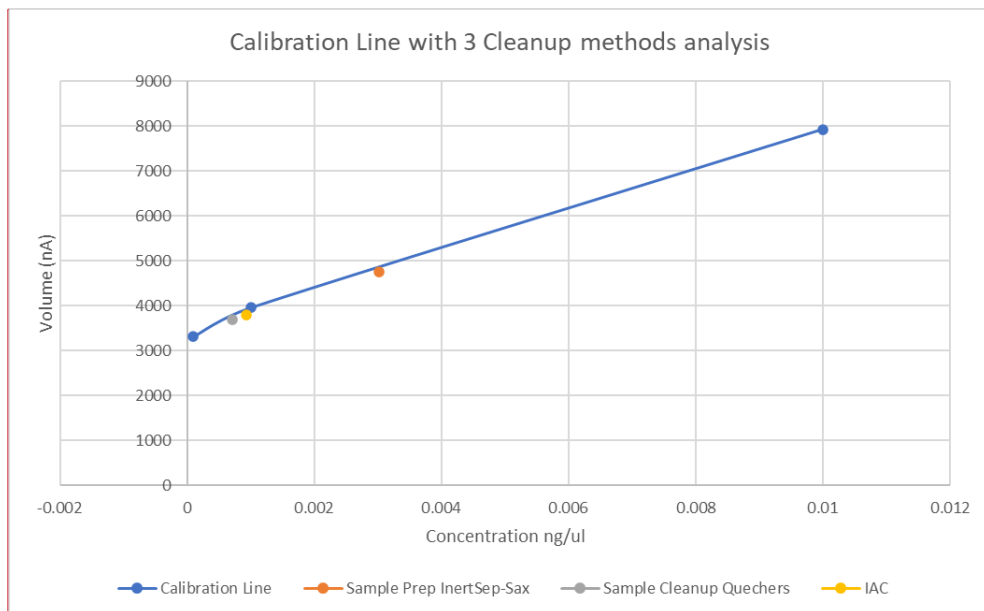


Figure 8: 10 μ L of 2 μ g/kg Ochratoxine A cleanup step was done using Immunoaffinity Column (Ochraprep, R-Biopharm Rhône Ltd.).



Met opmerkingen [NG1]: Use straight line approximation!

Figure 9: Calibration curve for Ochratoxine A.

Conclusions:

As it is shown in the plasmagrams, the OPTIC-4-AIMS analyzer is very well capable of detection of Ochratoxine A.

After the paprika sample clean-up, the analysis can be done in 120 seconds. This also includes the cool-down time. to the ready state for the next injection.

The AIMS system runs on conditioned air and needs a very little amount of solvent for the sample preparation. At the same time, an LC system consumes about 500 liter of solvent per year, and this is only for the mobile phase with flow rate of 1 mL/min.

The OPTIC-4-AIMS needs only 10 µL per injection The sample preparation is not added to the calculations.

The system is very green in this sense. It can be used even in a mobile laboratory.

References:

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- Determination of ochratoxin A at the ppt level in human blood, serum, milk and some foodstuffs by high-performance liquid chromatography with enhanced fluorescence detection and immunoaffinity column cleanup" methodology and Swiss data by Bernhard Zimmerli*, Rudolf Dick.
- Determination of ochratoxin A in licorice root using inverse ion mobility spectrometry by Mohammadreza Khaledi, Mahmoud Sheikh-Zeinoddin, Mahmoud Tabrizchi.
- Determination of Ochratoxin A in cereals and feed by SAX-SPE clean up and LC Fluorimetric Detection by A. Biancardi, A. Riberzani.
- Efficiency of Various Solvents in the Extraction of Ochratoxin A from Naturally Contaminated Wheat and Corn by Ryan J. Malone, Bruce R. Malone, Carrie K. Maune.

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