CORVINUS
UNIVERSITY of
BUDAPEST

Faculty of Food Science

# ORIGIN DETERMINATION OF DIFFERENT TYPES OF SAMPLES USING ICP-MS

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PhD thesis summary and statement

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**PhD program**

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| **Name:** | Doctoral School of Food Science |
| **Discipline:** | Food Science |
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**The approving signature of the Head of the Doctoral School and the Supervisor:**

The candidate has met all the requirement determined in the Doctoral Code Book of the Corvinus University of Budapest. He took the observations and suggestions arising during preliminary examination when reworking this dissertation, thus the dissertation can be put to public defense.

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**The local Doctoral Council for Life science of the Corvinus University of Budapest has been assigned in the resolution 04/06/2013 the following Thesis Commitee for the public defence.**

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# 1 Introduction

Nowadays origin determination is a widespread and frequently used expression. We are talking about origin determination in case of a single word or in case of ethnical group, but fingerprint or DNA samples which were collected during a judicial proceeding for identifying the possible perpetrator also belong to this subject. Origin determination methods are usually used for identifying implements, materials and objects which were found during archaeological excavation. One of the main advantages of HACCP (Hazard Analysis and Critical Control Point) used in the food industry among others that it is possible to trace the producer, the manufacturer and the processor of all single food products back.

Through the above mentioned examples you may admit that the expression “origin determination” cover a huge area. It is used in forensics science as well as in geochemistry, archaeology, and food industry or even in history and crime prevention and detection. From this list in many cases the origin determination raises analytical problems among others for proving the origin. In my dissertation I intended to introduce a wide range of these analytical applications by looking for a solution for difficulties in origin determination in case of areas or disciplines which are far from each other.

Food industry is one of the area was selected by myself. If we are thinking about the series of food scandals in the recent years like Brazilian paprika containing high concentration of aflatoxin, or could we imagine the harm which could be caused for a vintner if the competitor wine on the shelves of the shops have the same quality and origin as his own product (only) on the basis on the notation which product is cheaper but in fact its origin and quality is doubtful, they are not meet with the notation. It is evident that the origin determination plays a big impact on food industry.

To determine the origin is important not only in case of foodstuffs. There is a discipline which is far from food science but the origin determination and other examinations based on the same analytical approach may be also very justified. This discipline is the nuclear forensics. In recent years the smuggling of nuclear and other radioactive materials has been come to the front. Primarily after the collapse and disintegration of the former Soviet Union some of these materials have gone out of control, which may be a possible danger. The phenomenon of nuclear smuggling and illicit trafficking has led to development of a new branch of science: nuclear forensics. This science is about to answer specific questions after seizure, like the age, the dangerous level, the intended use and of course the origin of the material.

In my thesis I attempt to develop an analytical method based on elemental composition and isotope ratio determination which is able to determine the origin of wine and paprika in the group of foodstuff and uranium-oxide fuel pellets from nuclear field.

The role of trace element analysis is very important in origin determination because the distribution of element in the material may give some information about the origin in geographical and technological sense. The determination of trace element fingerprint may be suitable for determining the origin of agricultural and nuclear materials. By examination of trace elements the elemental fingerprinting of food-, environment and nuclear samples may be possible. In case of food science the main purpose is to identify the elements (principally heavy metals and rare earth elements) which concentrations change significantly in the function of geographic region but preferably does not change during food processing. The examination of combined effect of typical contaminations originated from environment and technological steps in case of nuclear samples, primarily uranium-oxide pellets, may be relevant in origin determination.

By the examination of naturally occurring relative stable isotope ratios is possible to follow for example the water cycle, the migration of insects and several other natural processes. It is possible to judge by measurement of stable isotope ratios based on the natural variation to the origin in case of foodstuff and judge by measurement of isotope ratios artificially changed ($^{235}$U/$^{238}$U) to the intended use or to the date of production ($^{234}$U/$^{230}$Th).

For determining the geographical origin especially for agricultural samples the determination of heavy isotope ratios are prevalently used. One of these heavy elements is strontium (Sr) which $^{87}$Sr/$^{86}$Sr isotope ratio is different depending on geographical region. Unfortunately these results are not always perfect in case of samples which are originated geographically too close from each other or have quite similar properties. Nevertheless if the measurements of 87Sr/86Sr isotope ratio are complemented with independent method, for example with a trace element determination within given region, the result of origin determination may be more reliable.

For determining the elemental composition of the samples appropriate analytical techniques are available. One of these techniques is the mass spectrometry which is fit for purpose, widely used and the most effective method for precise and accurate determination of concentrations and isotope ratios. Out of these methods Inductively Coupled Plasma Mass

Spectrometry (ICP-MS) is developed in a most dynamic way, which main feature are the outstanding high sensitivity, precision and accuracy.

Using ICP-MS for origin determination it is necessary to apply a proper sample preparation procedure. Although in some instances (e.g. Laser Ablation ICP-MS, which is quasi non-destructive) the sample preparation step could be omitted, the methods applied in this dissertation were destructive ones. This is due to the lack of (or limited access of) solid etalons for concentration determination for certain examination (e.g.: food analysis) and in many cases the sample is not suitable for this kind of sample introduction. Therefore I used combined sample preparation steps known from literature during the sample preparation procedure. After these procedures the prepared, digested samples and their component which are important for our purpose are introduced to the ICP-MS in liquid state.

## 2 Objectives

The aim of my work was to develop sample preparation methods, which is one of the most important parts of inorganic mass spectrometry, for determining relevant isotopes precisely and less disturb by spectral interferences for trace element and origin determination. Validation of the developed method is a critical point of the successful application in the future, therefore I put emphasis on that and I applied certified reference materials and interlaboratory comparison using independent method during the validation process. For testing the reliability of the developed and validated method real sample with well-known origin were determined.

The objectives of my dissertation were grouped according the type of the samples. Accordingly:

Objectives in case of methods are applied for origin determination of food:

- Method development
    - Development of sample preparation method need for trace element determination which is based on critic of different published methods, during improvement and method development may use as basic approach.

    - Development of sample preparation method and associated analytical procedure which is suitable for determining relevant isotope composition appropriate for origin determination and able to minimize the possible interferences.

- Validation

    - After the determination of performance characteristic the developed method was validated using certified reference materials and independent analytical methods.

- Analysis of real samples

    - The developed methods were tested for origin determination of different foodstuffs (paprika, wine) from different processing stage which origin were well-known.

Objectives in case of methods are applied for origin determination of food:

- Method development

    - Development of sample preparation method for rare earth element determination

    - Development of sample preparation method for trace element determination

- Validation

    - After the determination of performance characteristic the developed method was validated using certified reference materials and independent analytical methods.

- Analysis of real samples

    - The developed methods were applied for origin determination of nuclear forensics samples

# 3 Materials and Methods

The experiments were carried out in three different laboratories. The experiments for selecting the method for sample preparation of wine were carried out in the Department of Applied Chemistry of the Faculty of Food Science of the Corvinus University of Budapest using a quadrupole ICP-MS and in the ICP-MS Laboratory of the Institute of Isotopes of the Hungarian Academy of Sciences using a high resolution ICP-MS. In the latter one, additional experiments were carried out for analysis of trace elemental fingerprint of wine samples, sample preparation and elemental fingerprint analysis of paprika samples and the sample preparation and analysis of nuclear forensics samples. The preparation of wine samples and the Sr isotope ratio determination of wine and paprika sample were carried out in the VIRIS Laboratory of Analytical Chemistry Group of Department of Chemistry of the University of Natural Resources and Life Sciences, Vienna (Universität für Bodenkultur Wien, Department für Chemie, Abteilung für Analytische Chemie, VIRIS Labor) using a multicollector MC-ICP-MS instrument.

During my experiments I always used high purity chemicals or reagents, but since these experiments were carried out different laboratories the type, the brand and the purity of these reagents was or might have been different.

For operating the ICP-MS high purity (99.996%) argon gas was used. The concentrated (65%) nitric acid was Suprapur® purity. Milli-Q high purity water (18.2 MΩ resistivity) was used. The $H_2O_2$ (30%) was used for sample preparation and its purity was analytical grade. The working standard solutions were prepared using Merck IV multi elemental ICP standard solution in 1000 mg $l^{-1}$ concentration, Claritas PPT I, II and IV multi elemental ICP standard solution in 10 mg $l^{-1}$ concentration and As, Rb, U, Zr and Mo mono elemental ICP standard solution in 1000 mg $l^{-1}$ concentration. The rhodium internal standard was prepared using Rh mono elemental ICP standard solution in 1000 mg $l^{-1}$ concentration.

For validation of sample preparation of food samples two different certified reference material, BCR 679 (white cabbage) and NIST 1573a (tomato leaves) were applied.

TRU® and UTEVA® resin was used for separating uranium. For validation of the method Morille and BCR-2 (basalt) certified reference material were used.

For strontium separating Sr specific resin was used with its 100-150 µm particle size and for method validation a NIST SRM 987 reference material was used.

# 4 Results

## 4.1 Food Analysis (examination of wine and paprika)

I have compared several sample preparation method applied for preparing wine samples, which – by applying minor changes – may also be used to determine paprika samples. Based on those examinations the microwave digestion method using only nitric acid proved the most suitable one.

Both in the case of paprika and wine I have succeeded in developing a procedure which is suitable for determining the elemental fingerprint of the samples and assessing the results statistically. For the examination I used 98 wine samples originated form Austrian wine regions and 30 pieces paprika samples originated from different, but known area of several country. I have measured the $^{87}Sr/^{86}Sr$ isotopic ratio in both sample types, which may be a complementary method in determination of origin. For determining the Sr isotope ratio a multicollector MC-ICP-MS was applied.

Such origin determination proved to have relatively low efficiency on samples which are geographically too close to each other but in many cases the combined method led the evaluation to the right results. However, results turned out to be reliable in the case of samples which are distant from each other.

The methods were validated using certified reference materials and in an interlaboratory comparison study and were tested using real samples. According to the results and the performance characteristics it may be declared that the method is suitable for distinguishing samples that are geographically distant from each other and for determining the origin using an appropriate database.

## 4.2 Nuclear Forensics

I have developed two combined methods to determine the elemental fingerprint (including rare earth elements) of high uranium content samples (seized uranium-oxide and uranium concentrate) using extraction chromatographic separation, UTEVA and TRU extraction chromatographic resin. The uranium separation was necessary for minimizing the reduction of ionization efficiency and the contamination of the instrument. The methods were validated analysing certified reference materials and in an interlaboratory comparison study and the adaptability were tested using real samples (uranium ore, uranium concentrates and uranium-oxide).

The developed methods have sufficiently low detection limit and are capable to determine the elemental fingerprint of the above-mentioned samples. Additionally, they prevent the contamination of the device from uranium. The calculated decontamination factor of these methods is $10^6$ for uranium, which indicate the successful separation of uranium from the measurands. Other performance characteristics like detection limit, recovery, repeatability were also satisfactory. The recovery of the developed method was between 92-105%.

According to the results and the performance characteristics it may be declared that the methods are appropriate for separation, determination of elemental fingerprint and origin determination of nuclear (primarily uranium-based) samples using an appropriate database.

## 5 Thesis statements

1. A comparison of sample preparation procedures, developed for determining trace element content and geographical origin of foodstuffs from high processing stage (wines) applied mass spectrometry, reviewed in scientific journals was performed and the simplest procedure was selected which is able to be used for examination of foodstuffs from different procession stage including highly processed wine and lowly processed paprika sample. The method was tested by analysing five different types of wines using two independent measurement techniques (ICP-SFMS and ICP-CC-QMS). I declared that the method was suitable for determining 22 elements (Al, As, B, Ba, Bi, Cd, Co, Cr, Cu, Fe, Ga, Li, Mn, Mo, Ni, Pb, Rb, Sr, Tl, U, Zn, Zr),whose limits of detection are between 0.0004 ng $g^{-1}$ and 0.49 ng $g^{-1}$ depending on the particular element using ICP-SFMS technique.

   This mass spectrometric method based on trace element determination was improved with combination this method with $^{87}Sr/^{86}Sr$ isotope ratio determination. By examination of foodstuffs from different processing stages I have determined the follows:

   a. In the example of paprika sample I proved that the accuracy of the analytical method is not influenced by technological process (the original elemental fingerprint is not changed) and the combination of methods based on trace element fingerprint and $^{87}Sr/^{86}Sr$ isotope ratio determination is suitable to separate samples from lower processing stage, originating from different countries, from each other.

   b. Based on the analysis of wine samples I proved that the combinations of strontium isotope ratio trace element determination methods are suitable to separation of wines which geographical origin quite close together provided that the genetics of the individual loam are different properly. Examining the geochemical conditions of the above mentioned loam and area further clarification is expected.

2. I developed two new analytical methods for nuclear forensics purposes in which the uranium matrix was separated from the analyte used extraction chromatography on one hand for reducing the signal suppression caused by uranium matrix and on the other hand the possibility of contamination of the instrument and the cross-contamination of samples are also reduced.
   a. The first method in which the uranium is separated using TRU extraction chromatography resin is suitable for determining 14 rare earth elements (Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sm, Tb, Tm, Yb) which limit of detection is between 0.0024 ng g$^{-1}$ and 0.2 ng g$^{-1}$ depending on the particular element.
   b. The other method in which the uranium is separated using UTEVA extraction chromatography resin is suitable for determining 26 element (Al, As, Ba, Bi, Ca, Cd, Ce, Co, Cr, Cu, Fe, Ga, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sr, Th, Ti, Tl, Zn, Zr) which limit of detection is between 0.0031 ng g$^{-1}$ and 12.3905 ng g$^{-1}$ depending on the particular element.

The developed methods were validated using certified reference materials and independent analytical methods, and it was showed that these methods are able to clearly make distinction among nuclear forensics samples (uranium fuel pellets) with different origin or from different seizures.

## 6 Publications

*In journals with impact factor*

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*Hungarian full-text*

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