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Analysis of Acrylamide in Different Foodstuffs
Using Liquid Chromatography Tandem Mass Spectrometry and Gas Chromatography Tandem Mass Spectrometry

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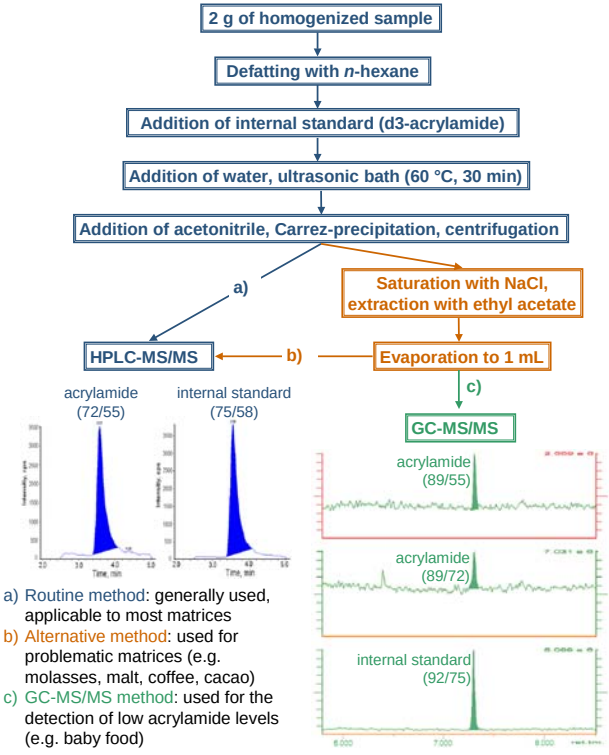
Introduction

In April 2002 the Swedish National Food Administration reported the finding of alarmingly high levels of acrylamide in heat-treated potato products and other baked goods [1]. Many researchers have confirmed the presence of acrylamide in different processed foods and it was shown that its concentration might reach levels as high as several mg kg^{-1} depending on the composition and processing of the food. As acrylamide is a potential carcinogen, a world-wide monitoring of this substance in various food products has started. The analyses of acrylamide was first performed by GC-MS following bromination to 2,3-dibromopropionamide [2]. Although this method is very sensitive, the derivatisation is laborious and time consuming. Recently, some methods were developed which omit the time consuming derivatisation step and measure acrylamide directly after extraction and clean-up using either GC-MS [3] or HPLC-MS-MS [4, 5]. The aim of this study was to develop a simple and fast method applicable to a wide range of food category groups and suitable for routine analysis.

Method



a) Routine method: generally used, applicable to most matrices
b) Alternative method: used for problematic matrices (e.g. molasses, malt, coffee, cacao)
c) GC-MS/MS method: used for the detection of low acrylamide levels (e.g. baby food)

Results and Discussion

Acrylamide analysis in different foods

Acrylamide levels over a wide range of different food products were analysed using both using HPLC-MS/MS and GC-MS/MS. Altogether more than 10,000 samples were screened (Fig. 1). Two different sample preparation methods for HPLC-MS/MS analysis were developed and optimised with respect to a high sample throughput on the one hand and a robust and reliable analysis of difficult matrices on the other hand. The first method (routine method) was applicable to most foods like potato chips, French fries, cereals, bread, and roasted coffee, allowing the analysis of up to 60 samples per technician per day. The second preparation method (alternative method) is not as simple and fast but enables the analysis of difficult matrices like cacao, soluble coffee, molasses, or malt. In addition, this method produces extracts which are also well suited for GC-MS/MS analysis (GC-MS/MS method). This method has proven to be a sensitive and selective method offering two transitions for acrylamide even at low levels down to 1 µg/kg.

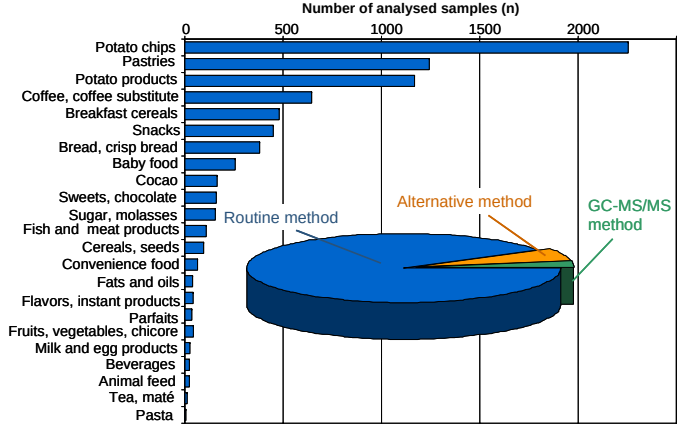


Fig. 1: Number of analyzed samples and distribution of the methods used for analysis

Table 1 summarises the coefficients of variation (CV) (determined by 10-fold analysis) and the limit of quantification (LOQ) for different foods determined using the respective extraction method and HPLC-MS-MS or GC-MS/MS analysis.

Tab. 1: Mean, coefficient of variation (CV), and limit of quantification (LOQ) for different food matrices, **a) Routine method, b) Alternative method, c) GC-MS/MS method**

Matrix	Mean (µg/kg)	CV (%)	LOQ (µg/kg)
Potato chips[a]	620	9.6	30
Crisp bread[a]	439	3.6	30
Butter biscuit[a]	546	2.7	30
Cacao[b]	190	9.1	30
Coffee, roasted[a]	282	9.2	30
Coffee, soluble[b]	816	4.1	30
Baby food[c]			5

Causes for variability of acrylamide levels

Recurrent analysis of acrylamide in the same food product (same recipe, same manufacturing process) revealed a wide variability of acrylamide levels which were mostly higher than the 10 % attributable to analytical variations (CV). Moreover, acrylamide levels vary widely in different product samples within one batch. It was shown for potato chips that acrylamide concentrations analysed in different packages of one batch may vary as much as +/- 50 % from the mean. However, it can be assumed that the inhomogeneity of the sample matrix (compare Fig.2) or the stability of acrylamide in the food (compare Fig. 3) may contribute to the variability of acrylamide levels, as well.

Homogeneity

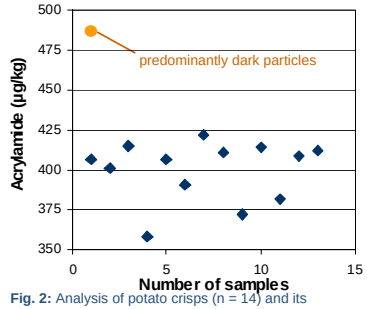


Fig. 2: Analysis of potato crisps (n = 14) and its predominantly dark particles (n = 1)

Stability

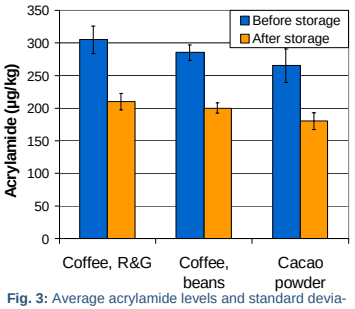


Fig. 3: Average acrylamide levels and standard deviations (n = 10) of roasted and ground (R&G) coffee, coffee beans and cacao powder analyzed before and after 3-month and 6 month storage at 10 – 12 °C, respectively

Conclusion

The occurrence of acrylamide in various food products makes it very difficult to develop one method which is suitable for all purposes and matrices. Therefore, two different methods were used, one which is very simple, fast and applicable to most samples and a second one which gives reliable results even for difficult matrices like soluble coffee or cacao. For the analysis of acrylamide at low levels GC-MS-MS turned out to be a suitable method since levels up to 5µg/kg can be quantified by two characteristic mass transitions without derivatisation. However, measurement uncertainties are not only caused by analytical variations but rather by inhomogeneity of the sample matrix or by the instability of acrylamide in the food during storage. The variability of acrylamide levels in food depends on the nature, the manufacturing process, and partly on the storage conditions of the respective food sample. Altogether, this can causes variations of up to 50 % which exceed the analytical variability by nearly a factor of 5.

Literature

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