Analysis of acrylamide from water according to DIN EN ISO 38413-6

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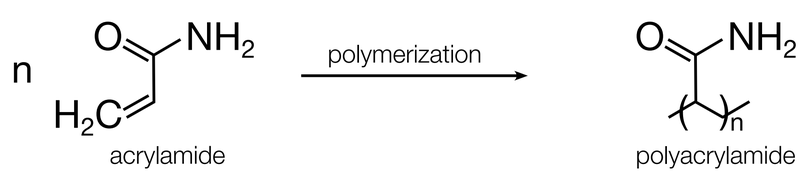
Introduction:

Polyacrylamides are often used in the water industry as coagulant for water clarification. Residual amounts of acrylamide monomers in drinking water, included in the purification treatments, are observed. In animal tests carcinogenic and mutagenic properties have been observed. Subsequently amounts of acrylamide in drinking water are limited by the german law [1]. Hence the interest in highly sensitive analysis for acrylamide has increased. The most important german guidelines for analysis of acrylamide are described in DIN EN ISO 38413-6 method [2]. This determination method provides a solid-phase extraction and high perormance liquid chromatographic method by mass spectrometric detection for acrylamide.

The first part of this work deals with a methodology for sample preparation of water analysis including a solid-phase extraction (SPE) method. SPE is carried out successfully on an activated carbon with methanolic elution. The activated carbon phase is highly porous and suited to the DIN specification of a specific surface higher than 1000 m^2/g. The recovery is compared with the requirements of DIN EN ISO 38413-6. The second part of this work points out the optimal high performance liquid chromatographic conditions on NUCLEODUR® C_{18} Gravity for acrylamide. The influence of the amount of methanol in the sample solution is analyzed. The identification of acrylamide in this work was carried out by ESI mass spectrometry.

Compound of interest



Sample pretreatment:

- the sample was treated with 100 mg/L sodium thiosulfate pentahydrate to reduce oxidizing species
- 40 mg/L sodium azide was added to avoid microbiological degradation
- an aliquot of 500 mL of the sample was taken and 50 ng of acrylamide were added

Solid phase extraction

Column type:	CHROMABOND® Carbon A, 6 mL, 1000 mg
Column conditioning:	1 x 8 mL methanol
	1 x 8 mL water
Sample application:	Sample was aspirated at a flow of 20 mL/min
Washing:	1 mL water
Drying:	15 min of nitrogen or air flow
Elution:	5 x 2 mL methanol

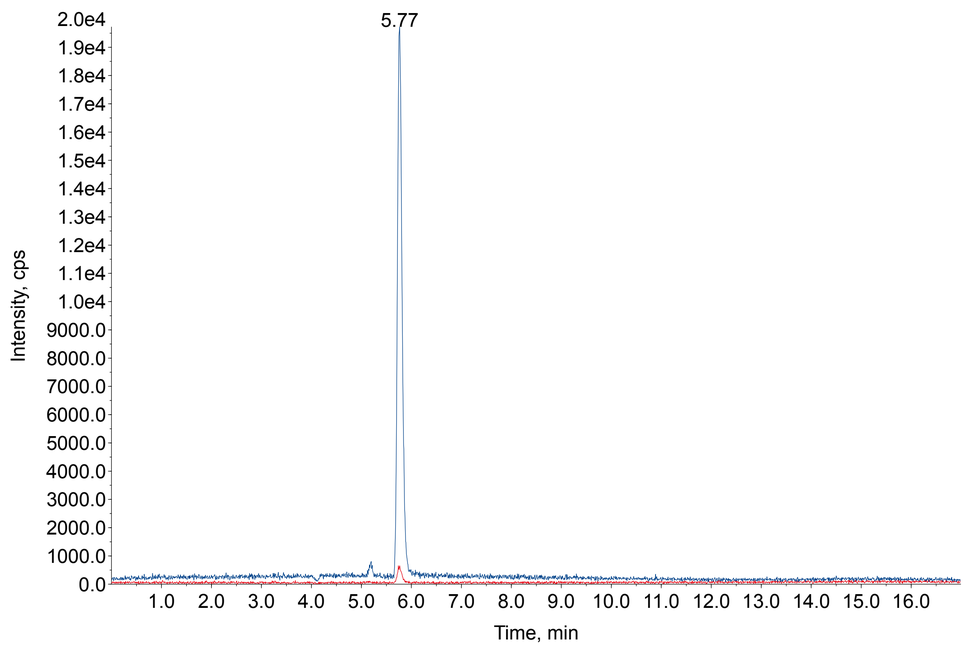
Concentration

Concentration was performed manually. Eluates from the SPE are combined and concentrated to 1mL (methanol content in sample must be 20 % or less).

Further analysis:

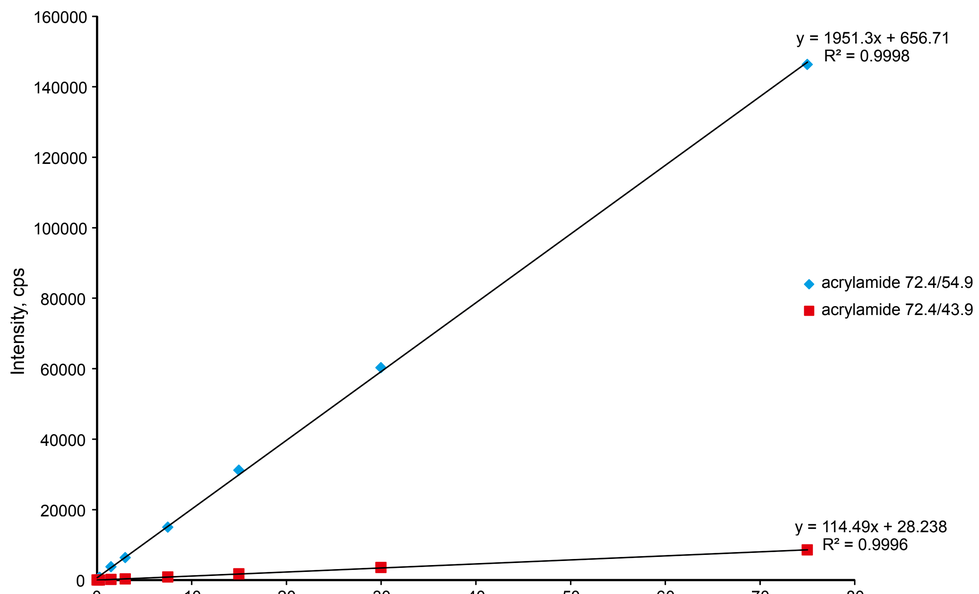
Column:	EC 150/3 NUCLEODUR® C_{18} Gravity, 3 µm
Eluent A:	0.001 % formic acid in water
Eluent B:	0.001 % formic acid in methanol
Gradient:	10 % B in 10 min to 100 % B, back to 10 % B in 2 min, hold for 5 min
Flow rate:	0.25 mL/min
Temperature:	60 °C
Injection:	10 µL
Detection:	MS/MS, AB Sciex API 3200
Ion source:	Turbo Spray (ESI)
Scan type:	MRM
Polarity:	positive
Curtain gas:	15 psig
Ion spray voltage:	5000 V
Temperature:	650 °C
Gas 1 (nebulizer):	60 psig
Gas 2 (turbo gas):	50 psig
CAD gas:	6 psig

Chromatogram of an acrylamide standard solution



Retention Time:	5.77 min
MRM of acrylamide	72.4/54.9, 72.4/43.9

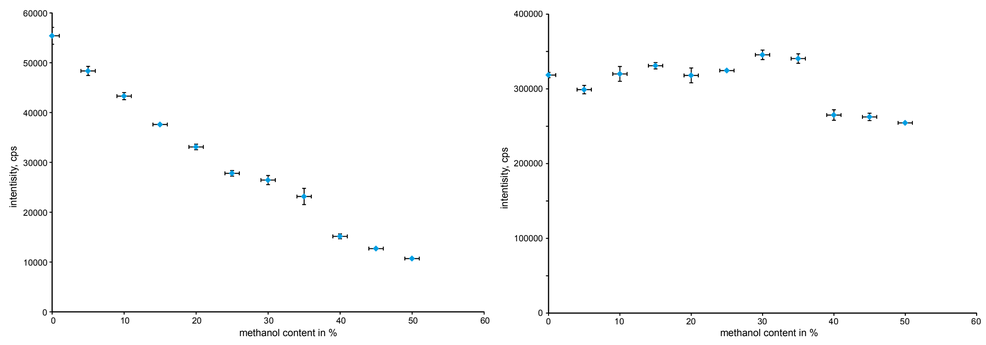
Calibration curves of acrylamide



Comparison of recovery rate

Recovery rate:	81 % (SD: 5 %; n=6)
Recovery rate required by DIN EN ISO 38413-6:	≥ 75 %

Influence of methanolic content in sample solution on peak height and peak area



Summary

The results of this work show that the solid phase extraction of acrylamide with CHROMABOND® Carbon A is very well suited. This application proposal shows that the enrichment of acrylamide with CHROMABOND® Carbon A fulfills all requirements of DIN EN ISO 38413-6. Using highly porous, spherical particles for SPE of acrylamide leads to excellent recovery rates.

Base material:	activated carbon	✓
Specific surface:	> 1000 m^2/g	✓
Recovery rate	≥ 75 %	✓

Methanolic content in the sample solution should be lower than 20 % after sample concentration.

References:

[1] Verordnung über die Qualität von Wasser für den menschlichen Gebrauch, (Trinkwasserverordnung - TrinkwV 2001)

[2] Determination of acrylamide – Method using high performance liquid chromatography with mass spectrometric detection (HPLC-MS/MS)

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