MACHEREY-NAGEL
Chromatography application note

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

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Abstract

This application note describes the determination of residues of sweeteners from fresh water in lower µg/L range using SPE for sample clean up und analyt concentration. After eluent exchange, the eluates from SPE are finally analyzed by HPLC-MS/MS.

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]. 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.

Compounds of interest

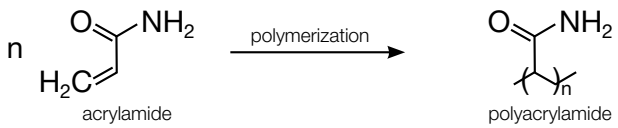

Figure 1: Polymerization of acrylamide.

Sample preparation

Sample pretreatment – alkaline hydrolysis

- 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, (REF 730167)

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 1 mL (methanol content in sample must be 20 % or less).

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Analysis of acrylamide from water

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

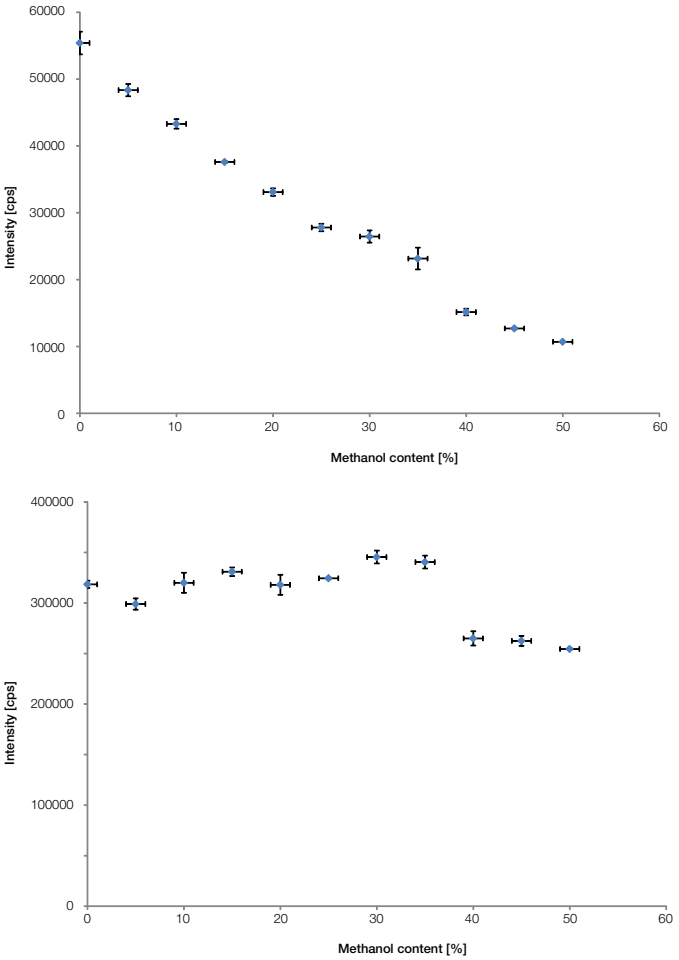

Figure 2: Influence of methanolic content in sample solution on peak height and peak area for the analysis of acrylamide.

Subsequent analysis: HPLC-MS / MS

Chromatographic conditions:

Column:

EC 150/3 NUCLEODUR® C_{18} Gravity, 3 µm, (REF 760083.30)

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

MS conditions:

API 3200; ion source ESI, positive ionization mode

Curtain gas 15 psig, ion spray voltage 5000 V, temperature 650 °C, nebulizer gas 60 psig, turbo gas 50 psig, CAD 6.0 psig

MRM transitions

Analyte	$[M+H]^+$	Q_1 (Quantifier)	Q_2 (Qualifier)
acrylamide	72.4	54.9	43.9

Table 1: MRM transitions for the analysis acrylamide.

Chromatogram of an acrylamide standard solution

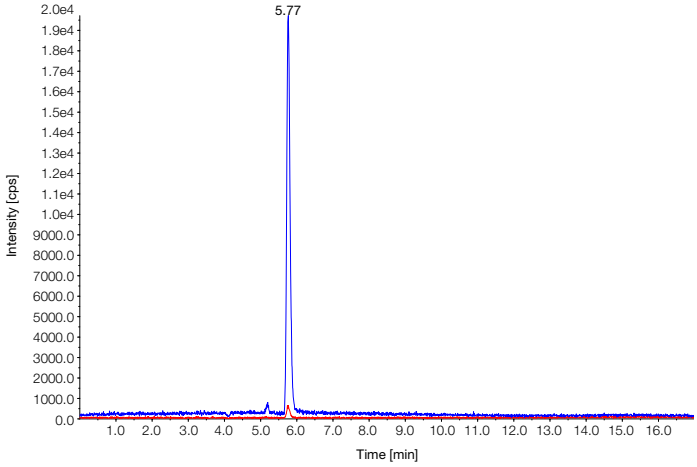

Figure 3: Chromatograms of HPLC-MS/MS analysis.

Calibration curves of acrylamide

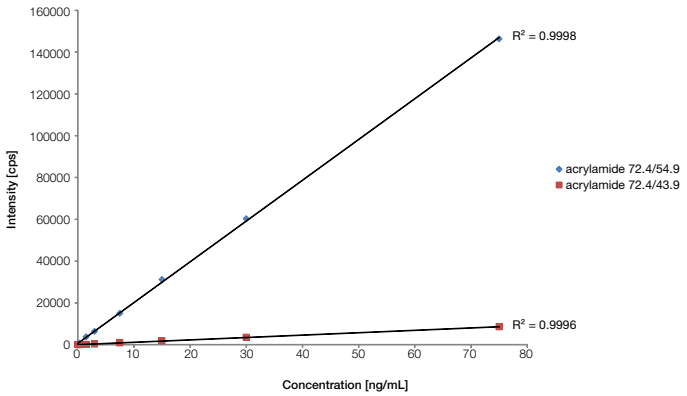

Figure 4: Calibration curves of acrylamide.

Comparison of recovery rate

Recovery rate:

81 % (SD: 5 %; n=6)

Recovery rate required by DIN EN ISO 38413-6:

≥ 75%

Analysis of acrylamide from water

Summary

The results of this work show that CHROMABOND® Carbon A is very well suited for the solid phase extraction of acrylamide. 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 – Methode using high performance liquid chromatography with mass spectrometric detection (HPLC-MS/MS).

Additional information

The following applications regarding "Analysis of acrylamide from water according to DIN EN ISO 38413-6" and further applications can be found on our online application database at *www.mn-net.com/apps*:

SPE: MN Appl. No. 306140

HPLC: MN Appl. No. 127530

Product information

The following MACHEREY-NAGEL products have been used in this application note:

REF 730167, CHROMABOND® Carbon A, 6 mL, 1000 mg
REF 760083.30, EC 150/3 NUCLEODUR® C_{18} Gravity, 3 µm
REF 702293 Screw neck vials N 9, 1.5 mL
REF 702107 N 9 PP Screw cap, yellow, center hole,
silicone white / PTFE red