Acrylamide
Analysis of acrylamide in chocolate, chips, waffles, coffee and cacao powder

Application Note

Food Testing & Agriculture

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Introduction
Analysis of acrylamide in chocolate, chips, waffles, coffee and cacao powder using GC/MS with Agilent VF-5ms after liquid-liquid extraction with Agilent Chem Elut.
Conditions

Technique: GC-capillary

Column: Agilent FactorFour VF-5ms, 0.25 mm x 30 m fused silica (df = 0.25 μm) (Part no. CP8944)

Temperature: 50 °C (0.8 min) → 70 °C, 20 °C/min → 130 °C, 6 °C/min → 300 °C, 120 °C/min (5 min)

Injection: PTV, Splitless, T = 230 °C

Detection: MS; Positive chemical ionization (CH₄)
Single ion monitoring (m/z: 72, 75)

Carrier Gas: He, 0.5 mL/min

Injection Vol.: 2 μL

Courtesy: Institut Kirchoff Berlin GmbH, Berlin, Germany

Peak identification

1. acrylamide m/z = 72, 1,512 µg/mL
2. D3-acrylamide m/z = 75, 0.524 µg/mL
Sample Preparation

Sample preparation with Chem Elut cartridges to improve standard liquid/liquid extraction.

Chem Elut cartridges contain a high-purity, inert diatomaceous earth. The aqueous sample is applied to the dry Chem Elut cartridge. The analytes are then simply extracted with a water-immiscible solvent. No shaking is necessary and no emulsions are formed.

Part no. of Chem Elut products used in this application:

12198007 for 10 mL aqueous extract
12198008 for 20 mL aqueous extract

Acrylamide in low-fat matrices

Acrylamide is extracted from the matrix using an acetonitrile/water (85:15) mixture. For internal standard, methylacrylamide is added. The extract is filtered and centrifuged. Part of the extract is used for liquid-liquid extraction using ethyl acetate and Chem Elut. The eluent is dried, dissolved in ethyl acetate and the internal standard is added.

Acrylamide in high fat matrices

Acrylamide is extracted from the matrix using an acetonitrile/water (85:15) mixture and the fat is removed using hexane. For internal standard, methylacrylamide is added. After phase separation the extract is filtered and centrifuged.

Part of the extract is used for liquid-liquid extraction using ethyl acetate and Chem Elut. The eluent is dried, solved in ethylacetate and the internal standard is added.

Acrylamide in coffee and coffee-type samples

Acrylamide is extracted with water from the homogenized sample. For internal standard, methylacrylamide is added. The extract is centrifuged. The centrifuged sample is “cleared” with Carrez solution I and II followed by filtration. Part of the extract is used for liquid-liquid extraction using ethyl acetate and Chem Elut. The eluent is dried, dissolved in ethyl acetate and the internal standard is added.
Acrylamide in chocolate, cone. 1814 µg/kg, recovery 79%
Acrylamide in chips, cone. 3000 µg/kg, recovery 90%

Acrylamide in cacao powder
Acrylamide in coffee and chocolate

![Graph showing the abundance of acrylamide at specific times and mass-to-charge ratios.](graph.png)