ANALYSIS OF ACRYLAMIDE IN FOOD

Dedicated to Professor Dr. Werner Baltes

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Abstract: Since the first discovery of the presence of acrylamide in a variety of food products in April 2002, numerous methods have been developed to determine the acrylamide monomer in heat-treated carbohydrate-rich food. These detection methods are mainly MS-based, coupled with a chromatographic step using LC or GC. The Food Chemistry Institute (LCI) of the Association of the German Confectionery Industry (BDSI) therefore established a detection method by means of aqueous extraction plus a cleaning step and LC-MS/MS detection, making great efforts to ensure internal and external validation. Citing potato crisps as an example, we will in the following show how the German manufacturing companies have gone to great pains to reduce acrylamide levels in their products.

Key words: acrylamide, LC-MS/MS, potato crisps, minimization

1. INTRODUCTION

Publication of the report on acrylamide intake from food, authored by the M. Törnquist and co-workers (2002), confronted food scientists and the food industry with a completely new dimension of toxicologically relevant food components, a so-called foodborne toxicant. Since then, there have been many publications outlining analytical methods for determining acrylamide in diverse matrices (e.g. Rosen and Hellenäs, 2002; Tareke et al., 2002; Biedermann et al., 2002a). The LCI has established the use of an LC-MS/MS method, extensively validating by testing quality assurance sample on a daily basis.

Since the occurrence of acrylamide in food first became known, the German potato chips industry has acted in the interests of preventive...
consumer protection and has correspondingly quickly introduced and successfully implemented extensive concentration-reducing measures (Matissek, 2002). To this end, the Food Chemistry Institute (LCI) of the Association of the German Confectionery Industry (BDSI) has conducted numerous systematic analyses on behalf of the member companies of the BDSI.

2. EXPERIMENTAL DETAILS

2.1 Materials

Acrylamide (99+%) was supplied by Sigma (Taufkirchen, Germany) and deuterium-labeled acrylamide-d₃ (> 99%) by LCG Promochem (Wesel, Germany). HPLC-grade acetonitrile came from Aldrich (Seeze, Germany) and the syringe filter used was a Rotilabo® Nylon 0.45 µm from Roth (Karlsruhe, Germany). All other reagents used for the analysis of acrylamide were of analytical grade.

CAUTION: acrylamide and acrylamide-d₃ are hazardous and must be handled carefully.

2.2 Sample Extraction

The samples were homogenized, fat-rich samples were defatted with n-hexane by way of extraction and 20 ml of water and 400 µl of internal standard acrylamide-d₃ (5 µg/mL) were added to 2 g of the homogenized sample. The samples were extracted by ultrasonic treatment (15 min, 60°C) and 20 ml of acetonitrile was added. Clean-up of the extracts was performed using 500 µl of Carrez I and II respectively and the samples were then centrifuged (4500 rpm for 10 min, 4°C). Before injection into the LC-MS/MS system, the supernatant was passed through a syringe filter (0.45 µm, Roth Rotilabo® Nylon).

2.3 LC-MS/MS Analysis

Mass spectrometry measurements were performed using a HPLC-system Series 200 (Perkin Elmer, Rodgau, Germany) coupled with a API 2000 mass spectrometer (Applied Biosystems, Darmstadt, Germany). Analytical separation was achieved using a Lichrospher 100 CN 5 µm (250 x 4 mm) with a guard column 5 µm (Merck, Darmstadt, Germany). The elution mode was isocratic, using a mixture of acetonitrile and water (0.5:99.5, v/v)