

Zeitschrift: Mitteilungen aus Lebensmitteluntersuchungen und Hygiene = Travaux de chimie alimentaire et d'hygiène
Herausgeber: Bundesamt für Gesundheit
Band: 93 (2002)
Heft: 6

Artikel: Two GC-MS methods for the analysis of acrylamide in foods
Autor: Biedermann, Maurus / Biedermann-Brem, Sandra / Noti, Anja
DOI: <https://doi.org/10.5169/seals-981744>

Nutzungsbedingungen

Die ETH-Bibliothek ist die Anbieterin der digitalisierten Zeitschriften. Sie besitzt keine Urheberrechte an den Zeitschriften und ist nicht verantwortlich für deren Inhalte. Die Rechte liegen in der Regel bei den Herausgebern beziehungsweise den externen Rechteinhabern. [Siehe Rechtliche Hinweise.](#)

Conditions d'utilisation

L'ETH Library est le fournisseur des revues numérisées. Elle ne détient aucun droit d'auteur sur les revues et n'est pas responsable de leur contenu. En règle générale, les droits sont détenus par les éditeurs ou les détenteurs de droits externes. [Voir Informations légales.](#)

Terms of use

The ETH Library is the provider of the digitised journals. It does not own any copyrights to the journals and is not responsible for their content. The rights usually lie with the publishers or the external rights holders. [See Legal notice.](#)

Download PDF: 11.05.2025

ETH-Bibliothek Zürich, E-Periodica, <https://www.e-periodica.ch>

Two GC-MS Methods for the Analysis of Acrylamide in Foods

Maurus Biedermann¹, Sandra Biedermann-Brem¹, Anja Noti¹, Koni Grob¹, Peter Egli² and Hugo Mändli²

¹ Official Food Control Authority of the Canton of Zurich, Zurich

² Labor Veritas, Zurich

Received 4 November 2002, accepted 19 November 2002

Introduction

On 24 April 2002 the Swedish National Food Administration published data and comments on disturbingly high concentrations of acrylamide in some foods (1). Acrylamide causes tumors in laboratory animals. The discovery was considered particularly alarming as the acrylamide-containing foods include products regularly consumed in rather large quantities, such as potato chips, French fries, roast potatoes, breakfast cereals, and crisp bread. The World Health Organization (WHO, (2)) and the Scientific Committee for Food (SCF) of the European Union (3) confirmed the issue as a "serious concern".

There are primarily two approaches for the analysis of acrylamide in food, based either on GC-MS or on HPLC-MS-MS. The GC-MS method described by *Castle et al.* (4) included extraction of the food sample with water, bromination of the double bond of acrylamide, extraction into ethyl acetate, and purification by gel permeation chromatography (GPC). It was recently revisited by the same group (5). *Tareke et al.* (6) used a similar method. *Rosen and Hellenäs* (7) described a HPLC-MS-MS method involving extraction into water, clean up by SPE, and HPLC on a Hypercarb column.

As the methods used by the Swedish laboratories were disclosed only recently, new methods were developed in many laboratories. This paper describes two GC-MS methods which were developed by two different groups. Despite regular contacts, they evolved differently, and since both have their merits, we decided to describe them separately. They do without bromination and clean up and are, therefore, faster than the GC-based methods described so far.

Method 1

Outline of the method

The steps of method 1, from the Official Food Control Authority of the Canton of Zurich, are listed in figure 1. A minimum of water is added for the swelling of solid parts. Extraction is performed with 1-propanol and the propanol/water evaporated in order to avoid the difficult extraction of acrylamide from an aqueous phase. Acetonitrile enables a simple clean up by restricted solubility of the salts and the defatting with hexane.

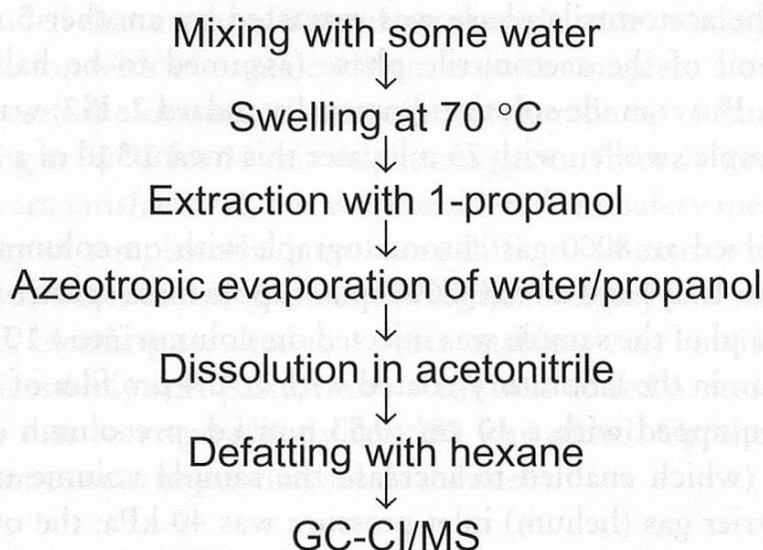


Figure 1 The steps of method 1

Procedure

Acrylamide, methacrylamide and butyramide were from Fluka, Buchs, Switzerland, D₃-acrylamide from Cambridge Isotope Laboratories (Andover, USA). 1-Propanol, reagent grade, was from Scharlau (Barcelona, Spain).

Samples were allowed to swell adding water in an amount normally corresponding to three times the weight of the sample (more for exceptionally dry samples). Taking into consideration homogeneity and availability of the sample, often 25 g of sample and 75 ml of water were combined in a 150 ml beaker glass. 500 µg/kg of the two internal standards methacrylamide and D₃-acrylamide (internal standard 1, IS1) was added, i.e. 1 µl per 1 g of sample of a 500 mg/l acetonitrile solution. After mixing (Polytron, Kinematica, Luzern, Switzerland), the homogenate was allowed to swell during 30 min at 70 °C in a water bath. The beaker glass was covered by aluminium foil to prevent evaporation of water.

10 g of the homogenate was weighed into a 100 ml centrifuge glass with a screw cap and thoroughly mixed with 40 ml of 1-propanol. When the solids form lumps,

mixing was supported by a blender (Polytron). 10 ml (8.4 g) of the supernatant (possibly after centrifugation of some 12 ml of turbid supernatant) was transferred to a 25 ml pointed flask. Fifteen droplets (about 200 mg) of a vegetable oil were added and the water/propanol removed in a rotary evaporator at about 50 Torr and 60–70°C in the water bath. Evaporation was stopped as soon as no liquid was left. The vacuum needs to be adjusted if bumping is observed.

The residue from the evaporation, consisting of fat/added oil and often much salt, was extracted with acetonitrile and defatted with hexane. 3 ml acetonitrile and 20 ml hexane were added and mixed with the sample with the help of an ultrasonic bath. The acetonitrile (lower) phase was transferred into a 10 ml reagent glass with screw cap by means of a Pasteur pipette, losing acetonitrile rather than carrying along hexane. The acetonitrile phase was extracted by another 5 ml hexane, now transferring 1.5 ml of the acetonitrile phase (assumed to be half) into a 1.5 ml autosampler vial. Butyramide solution (internal standard 2, IS2) was added. For the common 25 g sample swollen with 75 ml water this meant 5 µl of a 25 mg/l solution in acetonitrile.

GC-MS involved an 8000 gas chromatograph with on-column injector (ThermoQuest, Milano, Italy) and an SSQ7000 quadrupole mass spectrometer (Finnigan, San Jose, USA). 1 µl of the sample was injected on-column into a 10 m × 0.25 mm i.d. separation column in the laboratory coated with an 0.4 µm film of Carbowax 20M. The latter was equipped with a 40 cm × 0.53 mm i.d. precolumn deactivated with Carbowax 20M (which enabled to increase the sample volume up to 5 µl when needed). The carrier gas (helium) inlet pressure was 40 kPa; the oven temperature was programmed at 15°/min from 70°C (1 min) to 220°C (2 min).

Mass spectrometry involved positive ion chemical ionization (CI) with methane as reagent gas. The ion source was at 100°C. Usually selected ion monitoring (SIM) was used at *m/z* 72 (acrylamide), 75 (D₃-acrylamide), 86 (methacrylamide) and 88 (butyramide).

For normal samples, acrylamide concentrations were determined using D₃-acrylamide of IS1 as internal standard and applying a calibrated response factor. They were recalculated on the basis of methacrylate as a control. The recovery of the analytical procedure was checked by the ratio of the peak areas of butyramide/D₃-acrylamide. For other analyses, such as the determination of the potential of acrylamide formation and elimination of acrylamide (8, 9), D₃-acrylamide was used to monitor the behavior of acrylamide and methacrylamide was used as internal standard. The recovery was checked by the area ratio of butyramide/methacrylamide.

Discussion of the method

Swelling of solids

Extraction of acrylamide from the food is a critical step since acrylamide may be firmly enclosed, e.g., in dark solids of roasted potato; concentrations are likely to

be particularly high in these particles. Method development has to overcome the problems that spiking experiments are not suitable to check extraction of enclosed material and that it is difficult to predict the material which is most difficult to extract.

To check for completeness of the extraction, numerous potentially difficult samples were extracted a second time under more severe conditions, using accelerated solvent extraction (ASE). The residues from the first extraction were again swollen with water and extracted with 1-propanol at 60°C and 1500 psi pressure (at 80°C, some acrylamide was formed as soon as the water is removed). Extraction was considered complete when the second extract contained less than 10% of the acrylamide concentration of the first extract.

It was concluded that extractability of acrylamide varies widely. For some samples, direct extraction with an organic solvent is quite complete. For others, swelling with water increased the amount of extracted acrylamide up to hundred times. The proposed swelling at 70°C for 30 min might exaggerate (mostly extractions without heat treatment were satisfactory), but was considered as a safety measure accounting for the possibility of samples which are more difficult to extract than those tested.

The amount of water added (3 times the sample weight) was adjusted such that, on the one hand, blending resulted in a paste enabling easy mixing. When mixing resulted in an excessively stiff paste, more water was added or less sample weighed in, which meant that correspondingly less IS2 was added. On the other hand, the sample should not turn as liquid that solids sediment.

Extraction

Compared to water as extraction solvent, 1-propanol has several advantages: (i) it yields a clean solution mostly without centrifugation; (ii) it also extracts lipids which might hinder extraction with water; (iii) it can be evaporated rather easily; and (iv) it azeotropically removes some 28% of water (somewhat less in presence of high concentrations of salt).

The volume of 1-propanol added was selected to reliably remove the water (in the worst case, a wet sample introduces nearly 10 g of water into the evaporation step). As propanol was left up to the end of evaporation, the salts were precipitated before acrylamide was deposited, i.e. enclosure of acrylamide into solids could be avoided.

Solvent evaporation

Since extraction of acrylamide from aqueous solutions was considered rather difficult, it was preferred to eliminate the water required for swelling by (azeotropic) evaporation. Evaporation is also critical, however. Acrylamide is largely lost if 1-propanol/water is completely evaporated without a residue to retain it on the wall of the round flask. Most samples introduce fat and other retaining

materials, but not all of them to a sufficient extent. As the procedure anyway includes defatting, edible oil was added to all samples.

Evaporation should be kept under control. On the one hand, vastly excessive time on the rotary evaporator may still result in losses of acrylamide. On the other, incomplete removal of 1-propanol may disturb defatting. The process was timed (about 4.5 min) and the last step kept under surveillance.

As an alternative, water was removed with acetonitrile (azeotropic mixture containing 16 % water). Acetonitrile, the solvent of the defatting step, has the advantage that evaporation does not need to be complete. However, since even substantial losses have no adverse effect on the results (see below), the use of relatively substantial amounts of acetonitrile for this step did not seem to be justified. Furthermore, precipitation of polar by-products did not result in the same efficiency of the clean up as restricted re-dissolution.

Clean up

The propanol/water extract may contain substantial amounts of by-products which need to be removed before GC analysis (on-column injection!). Most salts are removed by their limited solubility in the acetonitrile used to extract the residue obtained from evaporation. The fat/oil is removed with hexane, which is not miscible with acetonitrile. Acrylamide is sufficiently polar to remain in the acetonitrile phase: the combined two hexane phases contained between 4 and 8 % of the acrylamide and up to 12 % of the methacrylamide and butyramide used as internal standards. In the chromatograms, no interferences were noted and the life time of the separation system was sufficiently long to discourage additional clean up.

GC-MS analysis

GC analysis was designed to minimize adsorption effects. It involved a short column coated with Carbowax of intermediate film thickness (also keeping column bleed low). The precolumn was deactivated by rinsing the raw fused silica with a 0.1 % solution of Carbowax in dichloromethane, heating for 30 min to 280°C under carrier gas and removal of the non-lacquered and non-bonded material by dichloromethane (10). This procedure resulted in good inertness. Retention power is rather high (11), but of no concern for the relatively short precolumn used. Also to minimize adsorptivity, temperature programming was fast, resulting in high elution temperatures (around 150°C).

Injection occurred by the on-column technique since it was found to be more reliable than splitless injection. The price to pay for good quantitative performance is a relatively rapid contamination of the precolumn: after some 30–50 analyses, the precolumn and the first 15 cm of the column inlet were removed (two thirds of the samples being potato products). For the about 1500 analyses performed so far, five columns were spent.

MS involved positive ion chemical ionization (CI). Chromatography of the polar acrylamide on a polar stationary phase causes that potentially coeluted, mostly less polar materials are likely to have a substantially higher molecular mass. Using electron impact ionization (EI), many substances are fragmented to the ions 71 and 55 amu, i.e. those resulting from EI of acrylamide. To avoid such interferences, this fragmentation was largely suppressed by CI. In fact, the chromatograms recording the protonated compounds of interest were cleaner than those obtained with EI. methane was used as reagent gas since ammonia resulted in a somewhat variable mixture of the adducts with a proton and ammonia ($M^+ + 1$ and $M^+ + 18$).

Figure 2 shows the relevant sections of the GC-MS chromatograms from the 100 µg/l calibration mixture and from roast potatoes containing 20 µg/kg of acrylamide (near the detection limit). They show no relevant interfering peaks. Sensitivity could easily be increased by injecting a larger sample volume. The left chromatograms were obtained using an almost new column. The right chromatograms show substantial peak broadening resulting from 200 analyses previously performed on this column.

Detection limit and linearity

The detection limit of the method was largely determined by interfering material and was usually below 20 µg/kg; for potato products it was below 10 µg/kg (see also fig. 2). If the signal to noise ratio is too low, it can easily be improved by increasing

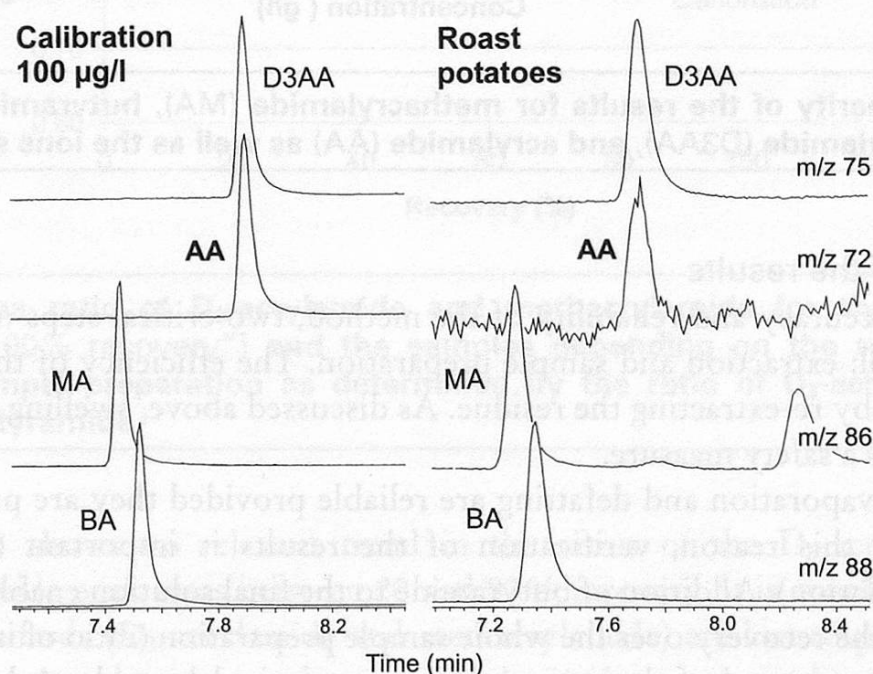


Figure 2 Relevant sections of chromatograms from a calibration and the extract from roast potatoes

the sample volume injected (up to 5 μ l if the precolumn is of the size indicated). Attention must be paid to sometimes strongly varying acrylamide concentrations in the samples analyzed (up to a factor of 1000) and the resulting risk of carryover.

Linearity of the results was regularly checked, primarily to keep possible adsorption in the GC column under control. As sample preparation results in dilution by a factor of about 6, 600 μ g/kg acrylamide in the sample corresponded to about 100 μ g/l in the solution analyzed (recovery assumed to be 100%). The range of concentrations shown in figure 3, 10–500 μ g/l, corresponded to about 60–3000 μ g/kg in the sample. A minor deviation was found only when the column was seriously contaminated (strongly broadened and tailing peaks).

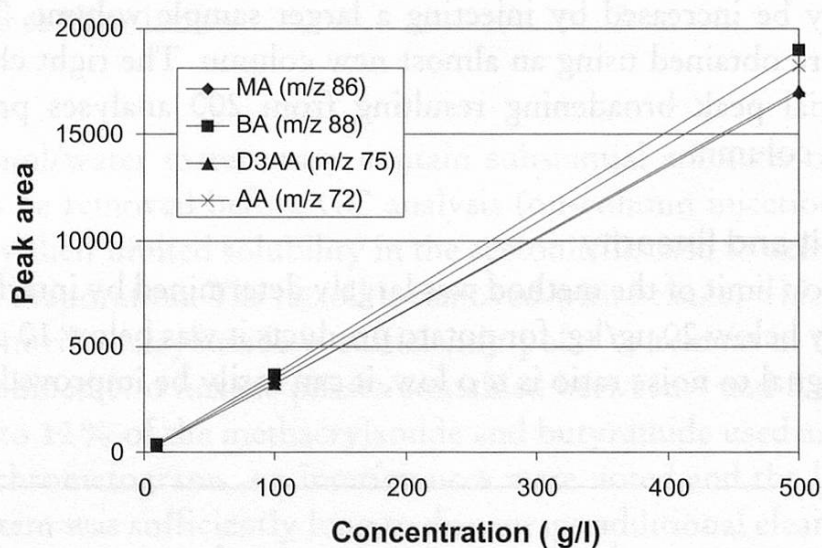


Figure 3 **Linearity of the results for methacrylamide (MA), butyramide (BA), D₃-acrylamide (D3AA), and acrylamide (AA) as well as the ions specified**

Accuracy of the results

For the accuracy and reliability of the method, two critical steps must be kept under control: extraction and sample preparation. The efficiency of the extraction was checked by re-extracting the residue. As discussed above, swelling at 70°C was introduced as a safety measure.

Solvent evaporation and defatting are reliable provided they are properly performed. For this reason, verification of the results is important (and largely replaces validation). Addition of butyramide to the final solution enables the determination of the recovery over the whole sample preparation (ratio of internal standards) and the control of the procedure for every single sample. A low recovery results in a reduced sensitivity of the method (no relevant problem), but might affect the accuracy of the results if losses of the internal standards and acrylamide were different.

The effect of a loss during sample preparation on the quantitative result was tested through the ratio of methacrylamide and D₃-acrylamide, assuming that D₃-acrylamide behaves similarly as acrylamide and that the difference in losses between methacrylamide and D₃-acrylamide would be larger than between D₃-acrylamide and acrylamide. Figure 4 shows the ratio of the areas of D₃-acrylamide and methacrylamide as internal standards in samples or as components of the calibration mixtures. Above the point of 100 % recovery, the ratios from the 10 µg/l and 100 µg/l calibration solutions are reported (no losses by sample preparation). These values were obtained from 34 measurements performed over about two weeks. The mean ratio was 0.95, with a relative standard deviation of 6.1 %. For the 49 samples analyzed during the same period, the mean ratio was 0.98, with a relative standard deviation of 5.9 %. This suggests that the ratios for the calibrations and the samples are equal and that the standard deviation is largely determined by the GC-MS analysis.

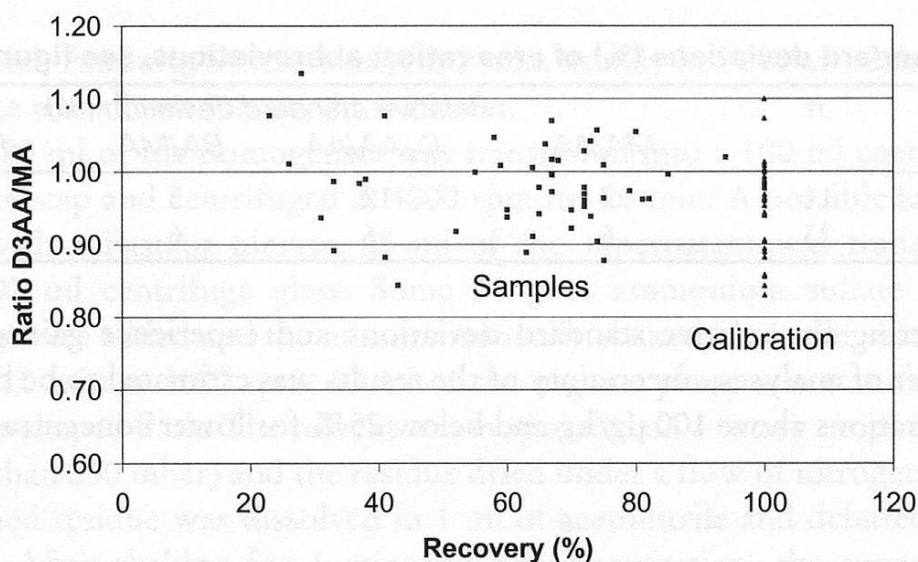


Figure 4 Area ratio of D₃-acrylamide and methacrylamide for the calibration ("100% recovery") and the samples depending on the recovery over sample preparation as determined by the ratio of D₃-acrylamide and butyramide

Figure 4 shows no relevant trend in the ratios of the D₃-acrylamide and methacrylamide peak areas between 20 and 90 % recovery. It is concluded that the internal standards (D₃-acrylamide and methacrylamide) are lost to practically the same extent as acrylamide, i.e. that reduced recoveries over sample preparation do not affect the accuracy of the results. No minimum could be determined for an acceptable recovery, but, as a rule, evaporation and defatting was repeated if the recovery dropped below 40 %.

Precision of the results

To estimate the precision of the results, it is assumed that the extraction from the food is complete, i.e. that extraction does not contribute to uncertainty. As shown above, the main contribution to the standard deviation of the results was from the GC-MS analysis. As the stability of the area ratios depends on the instrument and its performance, this contribution to uncertainty is more characteristic for a given instrument than for the method.

The contribution of the GC-MS analysis to uncertainty was derived from the area ratios (response factors) of the calibration solutions (the 10 µg/l solution injected 11 times and the 100 µg/l solution injected 23 times over about 2 weeks). As shown in table 1, relative standard deviations were between 6 and 14 %. They include contributions from chromatography (injection, separation process), MS analysis, and integration. As the ratio of the chromatographically almost equal D₃-acrylamide and acrylamide was not more stable than the others, probably MS analysis and integration were the primary sources of variation.

Table 1

Relative standard deviations (%) of area ratios; abbreviations, see figure 2

Solution	n	Relative standard deviation (%)			
		MA/AA	D ₃ AA/AA	BA/MA	BA/D ₃ AA
10 µg/l	11	7	12	5	14
100 µg/l	23	6	6	6	9

Considering the relative standard deviations and experience gained during a large number of analyses, uncertainty of the results was estimated to be below 15 % for concentrations above 100 µg/kg and below 25 % for lower concentrations.

Method 2

Outline of the method

The steps of method 2, from Labor Veritas, Zurich, are listed in figure 5. As for method 1, water is added to the sample for the swelling of solid parts. Extraction from the water phase is performed with 2-butanone. After evaporation of the solvent, the residue is dissolved in acetonitrile and defatted with hexane.

Procedure

Acrylamide and 2-butanone (puriss. p.a.) were from Fluka, Buchs, Switzerland, propionamide from VWR International, Dietikon, Switzerland.

Samples were allowed to swell adding water in an amount corresponding to 10 times the weight of the sample; normally 200 ml of warm water (60°C) was added to 20 g of sample, considering the homogeneity and availability of the sample. 10 µg/g of internal standard was added (propionamide, 1 g/l in acetonitrile). After

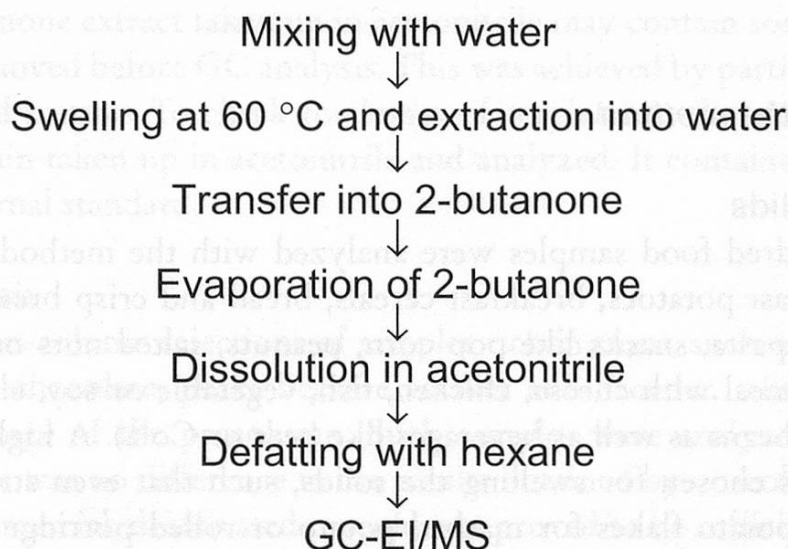


Figure 5 **The steps of method 2**

mixing (Rotel, Aarburg, Switzerland) for 2 min, usually in a 600 ml beaker glass, the homogenate was allowed to swell for 20 min.

About 80 ml of the homogenate was transferred into a 100 ml centrifuge glass with a screw cap and centrifuged at 4000 rpm for 20 min. A possible fat layer was removed with a Pasteur pipette. 25 ml of the supernatant was transferred into another 100 ml centrifuge glass. Some 20 g of ammonium sulfate and 25 ml 2-butanone were added and the mixture manually shaken for 1 min. After centrifugation for 10 min at 4000 rpm, 20 ml of the organic phase was transferred into a 100 ml pear-shaped flask. The solvent was evaporated on a rotary evaporator (50°C, not lower than 250 mbar) and the residue dried under a flow of nitrogen.

The dried residue was dissolved in 1 ml of acetonitrile and defatted with 3 ml of hexane. After shaking for 1 min and phase separation, the supernatant was discarded, the acetonitrile transferred into an autosampler vial and this solution analyzed by GC-MS.

GC-MS involved a Mega 5300 gas chromatograph equipped with an on-column and a split/splitless injector (Fisons, Milano, Italy) and an ITD 400 mass spectrometer (Finnigan, San Jose, USA). 1–2 µl of the sample was injected on-column or splitless into a 100 cm×0.32 mm i.d. precolumn deactivated with diphenyl tetramethyl disilazane (BGB Analytik AG, Anwil, Switzerland), connected to a 30 m×0.32 mm i.d. separation column coated with a 0.25 µm film of FFAP (BGB Analytik). The carrier gas (helium) inlet pressure was 75 kPa; the oven temperature was programmed at 10°/min from 110°C to 230°C and at 25°/min to 250°C (1 min). For splitless injection, the injector was set to 250°C, the splitless period to 5 s.

Mass spectrometry involved positive ion electron impact ionization (EI), usually with full scan monitoring (m/z 42–80). Acrylamide concentrations were calculated

from the ratio of the peak areas of acrylamide and propionamide. The ion source was at 200°C.

Discussion of the method

Swelling of solids

Several hundred food samples were analyzed with the method (potato chips, French fries, roast potatoes, breakfast cereals, bread and crisp bread, biscuits and crackers, flour, pasta, snacks like pop corn, peanuts, salted nuts or tortilla chips, ready-to-serve meal with cheese, chicken, fish, vegetable or soy, chocolate, cocoa powder, coffee beans as well as beverages like beer or Cola). A high water/sample ratio (10:1) was chosen for swelling the solids, such that even strongly swelling foods, such as potato flakes for mashed potato or rolled porridge oats, could be homogenized.

Extraction

A two-step procedure was chosen: extraction of the matrix with water followed by extraction of the aqueous extract with 2-butanone. Whilst solids tended to coagulate when extracted with a water/solvent mixture, they remained loose in 100 % water and thus facilitated the process. To obtain a clear extract, the sample needed to be centrifuged.

The difficult extraction of acrylamide from the aqueous phase into an organic solvent is achieved with 2-butanone and the help of salting out with ammonium sulfate. Ammonium sulfate also has the advantage of minimizing the water content in the organic phase. After centrifugation, three phases were usually obtained: a rather solid layer of matrix material separating the supernatant clear 2-butanone solution from the aqueous phase.

Extraction was almost complete. Potato chips containing 600 µg/kg acrylamide were extracted twice. After removing the first extract by centrifugation, the second extraction under stronger conditions (30 min, 70°C in an ultrasonic bath) gave less than 8 % (<50 µg/kg) of the result of the first extraction.

Solvent evaporation

The evaporation of the solvent is a critical step also for this method. 2-Butanone can, however, be removed under considerably milder conditions than a mixture of water and propanol, i.e. the addition of a retaining material is unnecessary. To rule out a loss of acrylamide in the rotary evaporator, a last portion of solvent was removed in a stream of nitrogen.

An additional time on the rotary evaporator of 15 min led to a similar (about 25 %) loss of acrylamide and internal standard. 570 µg/kg acrylamide resulted from acrylamide-free bread crumb to which 500 µg/kg acrylamide had been added.

Defatting

The 2-butanone extract taken up in acetonitrile may contain some fat/oil which needs to be removed before GC analysis. This was achieved by partitioning between acetonitrile and hexane. To check for losses of acrylamide, the evaporated hexane extract was again taken up in acetonitrile and analyzed. It contained neither acrylamide nor internal standard.

GC-MS analysis

After few on-column injections of samples rich in sugar, such as breakfast cereals, biscuits or chocolate, peaks became smaller and broader, which required the frequent exchange of the precolumn. Such samples were analyzed with splitless injection. There was no difference in the results obtained by on-column or splitless injection. Commercial, deactivated precolumns proved to be sufficiently inert.

Electron impact ionization of acrylamide primarily yielded the ions m/z 71 and 55, which may also originate from co-eluting substances. In fact, using columns shorter than that used, maltol (3-hydroxy-2-methyl-4-pyrone) was eluted with the same retention time and a similar intensity ratio of the ions m/z 71 and 55 as acrylamide. Heptanoic acid is another compound which may interfere with acrylamide. For this reason, quantitation was carried out separately with the total ion current and the masses m/z 70–72 and 55, and the results were averaged. When the three results significantly differed, the sample was reanalyzed by scanning through a wider mass range in order to check for overlapping components. The internal standard was measured as sum of m/z 73 and 74. Chemical ionization was not available.

Detection limit and linearity

The detection limit was usually 50 $\mu\text{g/kg}$. It was 100 $\mu\text{g/kg}$ for food containing interfering substances, such as chocolate and coffee.

The linearity of the results was checked by spiking bread crumb with acrylamide. The resulting three calibration graphs are shown in figure 6.

Recoveries and precision of the results

Recoveries were determined by the addition of 200 or 770 $\mu\text{g/kg}$ of acrylamide to bread crumb followed by 6-fold complete analysis. As shown in table 2, at the lower concentration, the recoveries averaged 101.7%, at the higher 93.9% of the acrylamide added.

The relative standard deviation was calculated as 5.9% for the series of the 200 $\mu\text{g/kg}$ addition and of 8.0% in the series with 770 $\mu\text{g/kg}$ of acrylamide being added. As there are additional factors contributing to the uncertainty of the results, we estimate the latter as $\pm 20\%$ for concentrations below 100 $\mu\text{g/kg}$ acrylamide in the sample and $\pm 10\%$ at higher contents.

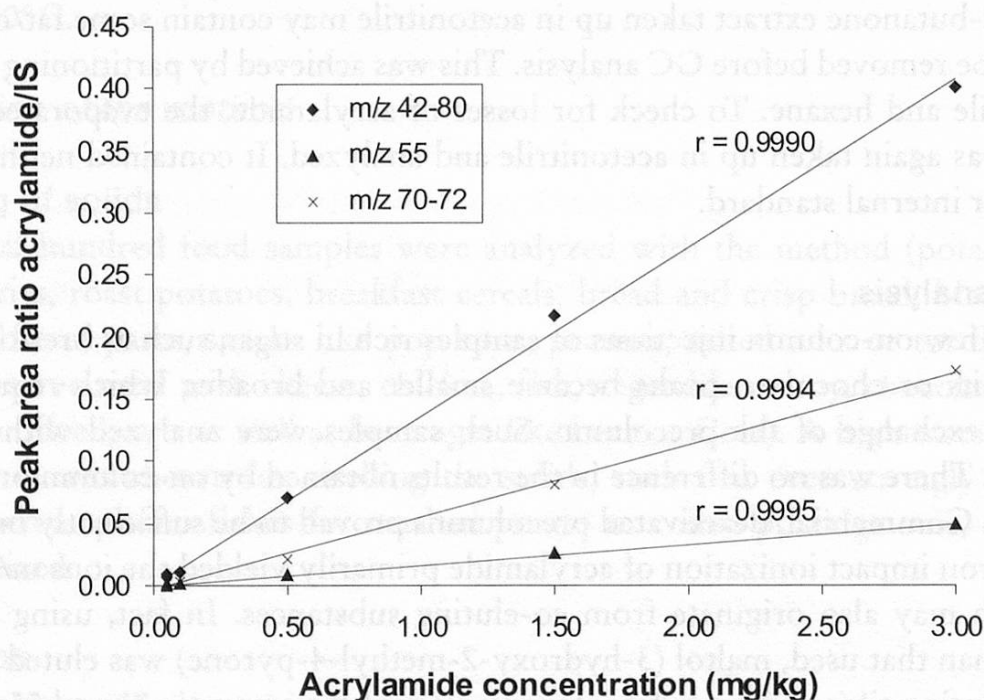


Figure 6 Linearity of the results for acrylamide

Table 2

Recoveries and standard deviation of method 2 for bread crumb

Addition: 200 µg/kg of acrylamide		Addition: 770 µg/kg of acrylamide	
Determination	Recovery (%)	Determination	Recovery (%)
1	97.0	1	94.8
2	98.0	2	79.2
3	103.0	3	98.7
4	112.0	4	100.0
5	104.0	5	94.8
6	96.0	6	96.1
Mean recovery	101.7	Mean recovery	93.9
Standard deviation	6.0	Standard deviation	7.5
Relative standard deviation (%)	5.9	Relative standard deviation (%)	8.0

Conclusion

Both methods were in routine use for about half a year and applied to almost all types of food known to contain acrylamide. Results obtained were compared between the two methods, but also with other laboratories, always with highly satisfying agreement. The results of official ring tests have not been evaluated so far.

Summary

Two GC-MS methods for the determination of acrylamide in food are presented. Both start by swelling of possibly solid material with water. Method 1 extracts acrylamide from this preconditioned material with 1-propanol, method 2 with water. According to method 1, the water is evaporated azeotropically with 1-propanol, according to method 2 acrylamide is extracted from the aqueous extract with 2-butanone. Salts and other polar materials are eliminated by restricted solubility in acetonitrile, fat by partitioning between acetonitrile and hexane. Chromatography is performed on Carbowax columns. Detection occurs by MS with CI or EI.

Zusammenfassung

Zwei GC-MS Methoden für die Bestimmung von Acrylamid in Lebensmitteln werden beschrieben. Beide beginnen mit der Quellung möglicherweise fester Bestandteile mit Wasser. Methode 1 extrahiert Acrylamid aus der vorbehandelten Probe mit 1-Propanol, Methode 2 mit Wasser. Gemäss Methode 1 wird das Wasser durch azeotrope Destillation mit 1-Propanol entfernt; Methode 2 extrahiert Acrylamid mit 2-Butanon aus dem wässrigen Extrakt. Salze und andere polare Materialien werden durch beschränkte Löslichkeit in Acetonitril entfernt, Fett durch Verteilung zwischen Hexan und Acetonitril. Die gaschromatographische Trennung erfolgt auf Carbowax-Kapillaren, die Detektion massenspektrometrisch entweder mit Elektronenstoss- oder chemischer Ionisation.

Résumé

Deux méthodes chromatographiques GC-MS permettant de déterminer l'acrylamide dans les produits alimentaires sont décrites. Elles débutent par le gonflement des composants solides avec de l'eau. L'acrylamide est extrait avec du propanol-1 dans la première méthode et avec de l'eau dans la deuxième. Dans la méthode 1, l'eau est éliminée par distillation azéotrope du propanol-1 tandis que dans la méthode 2 l'acrylamide est repris de l'extrait aqueux dans du butanone-2. Les sels et les autres matériaux polaires sont éliminés du fait de leur solubilité limitée dans l'acétonitrile; les matières grasses sont réparties entre l'hexane et l'acétonitrile. La séparation par chromatographie gazeuse est réalisée sur une phase stationnaire Carbowax et la détection est effectuée par spectrométrie de masse en mode ionisation chimique ou électronique.

Key words

Acrylamide, Food, Gas chromatography-mass spectrometry

References

- 1 Swedish National Food Administration: Information about acrylamide in food. www.slv.se/Download/Document/approvedDocs/enginformationakryl.htm, 24. April 2002.
- 2 World Health Organization, Press Release WHO/51 27 June 2002.
- 3 Opinion of the Scientific Committee on Food on new findings regarding the presence of acrylamide in food (expressed on 3 July 2002).
- 4 Castle, L., Campos, M.-J. and Gilbert, J.: Determination of acrylamide monomer in hydroponically grown tomato fruits by capillary GC-MS. *J. Sci. Food Agric.* **54**, 549–555 (1991).
- 5 Castle, L., Clark, D.B., Lloyd, A.S., Philo, M.R. and Speck, D.: GC-MS and LC-MS/MS methods used to verify the report of acrylamide in food (text to be published).
- 6 Tareke, E., Rydberg, P., Karlsson, P., Eriksson, S. and Törnqvist, M.: Acrylamide: a cooking carcinogen? *Chem. Res. Toxicol.* **13**, 517–522 (2000).
- 7 Rosen, J. and Hellenäs, K.-E.: Analysis of acrylamide in cooked foods by LC tandem MS. *The Analyst* **127**, 871–879 (2002).
- 8 Biedermann, M., Biedermann-Brem, S., Noti, A. and Grob, K.: Methods for determining the potential of acrylamide formation and its elimination in raw materials for food preparation, such as potatoes. *Mitt. Lebensm. Hyg.* **93**, 653–667 (2002).
- 9 Biedermann, M., Noti, A., Biedermann-Brem, S., Mozzetti, V. and Grob, K.: Experiments on acrylamide formation and possibilities to decrease the potential of acrylamide formation in potatoes. *Mitt. Lebensm. Hyg.* **93**, 668–687 (2002).
- 10 Grob, K., Grob, K. and Grob, G.: Preparation of apolar glass capillary columns by the barium carbonate procedure. *J. High Resol. Chromatogr. Chromatogr. Commun.* **1**, 149–155 (1978).
- 11 Grob, K. and Neukom, H.P.: Method for testing retention gaps and data on the GC retention power of differently deactivated capillary tubes. *J. Chromatogr.* **323**, 237–246 (1985).

Corresponding author: Koni Grob, Official Food Control Authority of the Canton of Zurich, P.O. Box, CH-8030 Zurich