Supplementary data

HS-SPME gas chromatography approach for underivatized acrylamide determination in biscuits

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Table of Contents

Table S1. List of ingredients used in the biscuit recipes.

	Modifications		
Original	**Recipe 1**	**Recipe 2**	**Recipe 3**
Cereals (68%): wheat flour, rice flour, whole wheat flour, rye flour	✓	Substitution of 50% of the wheat flour content rice flour	✓
Sunflower oil	✓	✓	✓
Sugar	✓	✓	✓
Glucose-frutose syrup	✓	✓	✓
Salt	✓	✓	✓
Raising agents (Sodium, Calcium and Ammonium carbonates)	✓	✓	✓
Vitamins	✓	✓	✓
Skimmed milk	✓	✓	✓
-	+ 3.5% asparaginase*	-	+ 2% pectate*

* w/w, in relation to the total flour content.

Table S2. Types of commercially available fibre coatings used in this work [36].

Stationary phase	Coating (µm)	Polarity	Extraction mechanism
PDMS	100	Nonpolar	Absorption
PA	85	Polar	Absorption
CW/DVB*	65	Polar	Adsorption
CAR/PDMS	75	Bipolar	Adsorption
PDMS/DVB	65	Bipolar	Adsorption
DVB/CAR/PDMS**	50/30	Bipolar	Adsorption

PDMS- polydimetylsiloxane; PA- polyacrylate; CW- Carbowax; CAR – carboxen; DVB – divinylbenzene. * This fibre has been taken out from the market. ** This stationary phase compromises a 50/30 DVB/CAR on PDMS fibre.

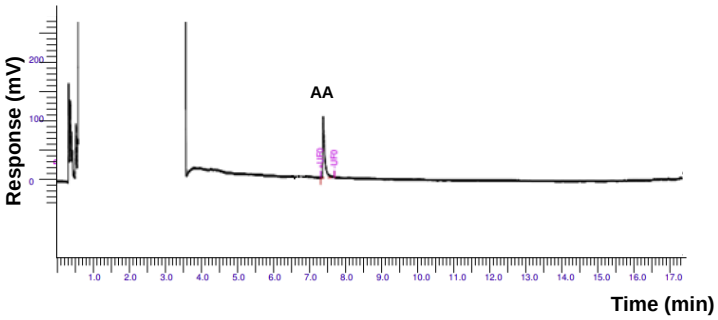

Figure S1. Example of a GC-FID chromatogram obtained for AA with a concentration of 5 mg/vial using a PA fibre.

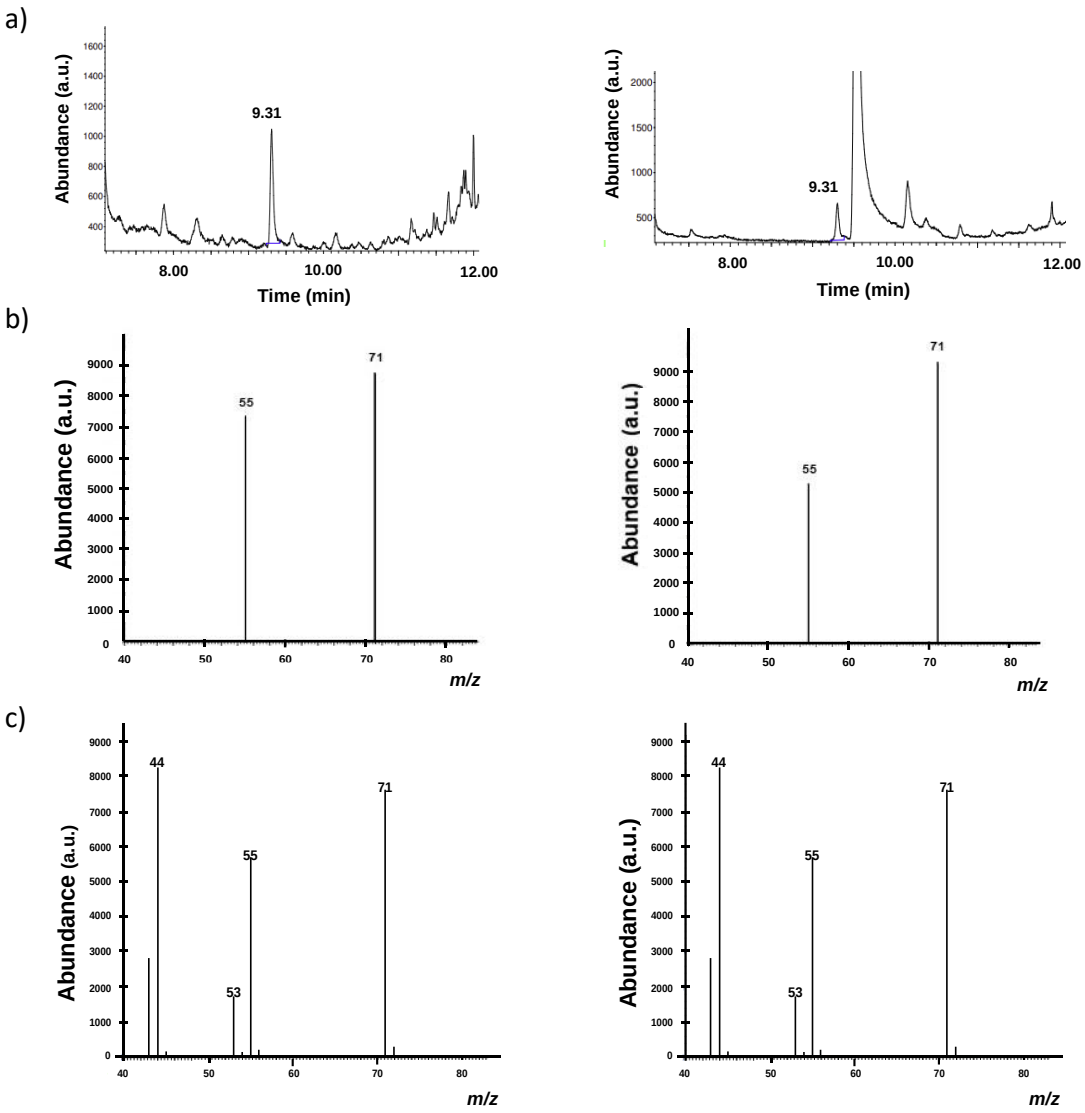

Figure S2. GC-MS chromatograms obtained for (a): (**left**) standard AA solution (8 µg/vial) and (**right**) biscuits sample with AA detection (retention time: 9.31 min); ion extraction spectrum of AA using *m/z* 71 and 55 (b), and literature full scan spectrum data for AA (c).