

## G-5 DETERMINATION OF AMINO ACIDS IN TEA BY HYDROPHILIC INTERACTION LIQUID CHROMATOGRAPHY COUPLED TO HIGH RESOLUTION MASS SPECTROMETRY

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Methods used for amino acid analysis are usually based on a chromatographic separation of the amino acids present in the sample. Current chromatographic techniques require postcolumn derivatization, unless the sample is analyzed using precolumn derivatization. Derivatization of free amino acids is necessary either to increase their detection sensitivity on ultraviolet-visible and fluorescence detectors, or to improve their chromatographic separations on conventional reversed phase columns. Amino acids are polar molecules that can be separated better using hydrophilic interaction liquid chromatography (HILIC) without precolumn derivatization. Underivatized amino acids can be sensitively detected by means of mass spectrometry. Analysis of free amino acids in foods by using HILIC coupled to mass spectrometry offers great advantages over the existing chromatographic methods in terms of analysis speed, accuracy, and cost. In this study, a liquid chromatography system (Thermo Scientific Accela) coupled to an orbitrap high-resolution mass spectrometry (Thermo Scientific Exactive) operated in positive electrospray ionization mode was used for the analysis of amino acids. Chromatographic separations were performed on an Atlantis HILIC column using a gradient mixture of acetonitrile and 100 mM aqueous formic acid solution. High-resolution mass spectrometry was used to scan ions between  $m/z$  50 and 300 with ultra-high resolving power. Ground tea (1 g) was extracted three times with 10 ml of hot water. The aqueous extract was diluted with acetonitrile (1:1) and injected onto HILIC column. The analytical conditions successfully resolved the peaks of amino acids in a total runtime of 6 min. Retention and molecular mass data were used to confirm the presence of amino acids in samples. Orbitrap mass spectrometry analysis verified the molecular masses of identified amino acids in tea samples with very high mass accuracy (<2 ppm). These amino acids detected in tea include theanine, glutamic acid, glutamine, serine, alanine, aspartic acid, asparagine, arginine, tyrosine, valine, phenylalanine, leucine, isoleucine, tryptophan, proline, lysine, histidine, and glycine. In principle, the method described here is applicable to any food matrices for high throughput analysis of amino acids.

**Keywords:** Amino acids, HILIC, high-resolution mass spectrometry, tea

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## G-6 CHEMICAL CHARACTERIZATION OF A TRADITIONAL FISH PREPARATION (MISSOLTINO) OBTAINED FROM SALTED AND DRIED TWAITE SHAD

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Missoltino is a traditional Italian fish product obtained from salted and dried twaite shad (*Alosa fallax lacustris*), an endemic fish of northern Italian lakes. A large amount of these fish is caught near its reproductive period, during the end of spring. This reason had led local professional fishermen to find a way to preserve the fish for all the year using an old processing techniques. Briefly, the freshly-caught twaite shad, weighing about 80 g, are eviscerated and then salted using fine sea salt. The exceeding salt is removed by water washing and the fish are naturally dried in a room for 3-5 days. Afterwards fish are arranged in layers in metallic containers and pressed at ambient temperature for a long period, nearly 3 months. These containers are closed with a wooden lid and the pressure is progressively increased with a crank handle. The aim of this work was to chemically characterize this fish product, prepared using two different levels of salt and at different time of ripening, in order to identify the optimal processing technique that resulted in best quality properties. Thirty six samples of twaite shad caught in Como lake were collected and used to make missoltini with 2 different salt concentrations, 80 g kg<sup>-1</sup> (A) and 40 g kg<sup>-1</sup> (B). Four samples were sampled: after catching, during salting, after 30 days, after 60 days and after 90 days of pressing. Chemical composition, TBARS value, histamine, fatty acid composition and flavor volatile compounds were determined in all samples. Fresh twaite shad showed a lipid percentage of 6.7±2.41. This percentage increased progressively in missoltini while the moisture content decreased during drying and pressing. The salt percentage had a direct influence on the lipid content. In missoltini made using 80 g kg<sup>-1</sup> of salt lipids were lower than in missoltini made using 40g kg<sup>-1</sup> of salt. This difference was statistically significant

**Keywords:** Twaite shad, processing conditions, volatile compounds, fatty acids, salt