

Carbon isotopic ratios and enantiomeric distributions of selected volatiles as parameters for the quality assessment of *Citrus* liqueurs determined by HS-SPME coupled to GC-C-IRMS, and ES-GC.

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Abstract

Liqueurs derived from *Citrus* fruits, generally obtained from maceration of the peel or the whole fruits of lemon, mandarin and bergamot in ethanol, water and sugar, are a category of spirit drinks in which the addition of nature-identical flavouring substances and preparations is forbidden. The traditional production methods and the protection of geographical indications of spirit drinks are governed by the Regulation (EC) No 110/2008 of the European Parliament and of the Council. Authenticity assessment of home-made and commercial *Citrus* liqueurs was performed using Headspace-Solid Phase Microextraction (HS-SPME) coupled to Gas Chromatography-Combustion-Isotope Ratio Mass Spectrometry (GC-C-IRMS). Additional analyses were performed on all the samples, by means of enantioselective Gas Chromatography (Es-GC), measuring the enantiomeric distribution of the chiral volatile components, extracted by the same HS-SPME technique. Moreover, Gas Chromatography-Mass Spectrometry (GC-MS) analyses were also conducted by using HS-SPME, in order to obtain information on the qualitative aspects of the samples. Additional analyses also revealed the lack of the monoterpene fraction in some commercial samples. The results obtained by GC-C-IRMS on the liqueurs were compared with the carbon isotopic ratio ranges determined on authentic cold-pressed lemon, mandarin and bergamot essential oils. In particular, it emerged that the carbon isotope ratio of the volatile compounds of all the home-made drinks fell into the correspondent authenticity range of the cold-pressed essential oil, while some commercial products did not match the correspondent ranges of authenticity. GC-C-IRMS, ES-GC and GC-MS coupled with HS-SPME have shown to be a complete and rapid tool for the quality control investigation of *Citrus* liqueurs. The results obtained by these techniques were in good agreement, revealing the non-natural *Citrus* aromas in some commercial liqueurs, as well as assessing the genuineness of the home-made ones.

Passata di pomodoro authenticity checks using $\delta^{18}\text{O}$ analysis

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Abstract

Tomato puree (passata di pomodoro) represents a classic and emblematic Italian product around the world and is of considerable importance for the Italian economy. It has recently been estimated that 15% of industrial tomato comes from China in paste form (http://affaritaliani.libero.it/green/coldiretti_pomodoro_cina280311.html). For these reasons Italian law (Ministerial Decree of 23 September 2005) provides a very strict definition of 'passata di pomodoro' and establishes that the use of tomato concentrates over 12° degrees Brix and subsequent dilution to obtain 'reconstituted passata' is not permitted. Furthermore it indicates $\delta^{18}\text{O}$ measurement in vegetal water (UNI ENV 12141 method) as the official method for detecting fraudulent dilution, but does not report any reference values for authentic passata. Trifirò et al. (2007) verified that passata effectively shows $\delta^{18}\text{O}$ values significantly higher than products obtained by diluting tomato paste and proposed a minimum threshold value of -1.6‰ for authentic passata with Brix values between 7.5 and 10°. In this work we analysed authentic samples of passata, paste and diluted concentrate in order to check the validity of this threshold value and to establish a $\delta^{18}\text{O}$ limit value for passata produced by diluting products with a Brix value lower than 12°. A large number of samples of Italian 'passata di pomodoro' and paste from Italy, Greece, Turkey and China have been officially collected by the Italian Ministry of Agricultural, Food and Forestry Policy since 2009. Analysis of the $\delta^{18}\text{O}$ of vegetal water was performed on these samples using an Isotope Ratio Mass Spectrometer (IRMS), interfaced with a CO_2 equilibration system, according to the ENV 12141 method. The results essentially confirmed that -1.6‰ can be considered as the limit $\delta^{18}\text{O}$ value for authentic passata with a Brix of over 7.5°. Furthermore a $\delta^{18}\text{O}$ of -2.5‰ can be considered as a limit value for passata coming from the dilution of products with a Brix level of less than 12°. All the diluted pastes showed $\delta^{18}\text{O}$ values far below these limits. This research was funded by the Italian Ministry of Agricultural, Food and Forestry Policy.