

4th European Chemistry Congress

May 11-13, 2017 Barcelona, Spain

Assessment of bound 3-monochloropropanediol (3-MCPD) and glycidol content in fats and oils by gas chromatography-ion trap tandem mass spectrometry

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An analytical method for determining bound 3-monochloropropanediol (3-MCPD) and glycidol in fats and oils based on gas chromatography-ion trap mass spectrometry (GC-MS/MS) technique has been developed and applied to the study of concentration levels of these compounds in margarine and olive, palm and sunflower oils. 3-MCPD and glycidol are food contaminants that have been classified as carcinogenic by the *International Agency for Research on Cancer* (IARC). They have been detected in wide range of food such as bread, coffee, pastries, etc., which are produced at high temperatures, as well as in refined animal and vegetable fats and oils, and in foods containing them. In oils and fats, 3-MCPD originates when triglycerides are hydrolyzed to mono- and diglycerides and they then undergo a substitution reaction with chlorine atoms, with temperatures above 140 °C being required. Glycidol was first detected in palm oil due to its high content of diglycerides and the high temperatures used in its refining. In 2014, the European Union (EU) issued a Recommendation to Member States about the need to assess the concentration levels of these contaminants in certain foods, as well as to develop new analytical methods for their determination in order to protect the health of European consumers. The developed method consists of two parallel tests, A and B, which are carried out using basic catalysis. 3-MCPD-d5 is used as internal standard in order to obtain reproducible results. In assay A, both bound 3-MCPD and glycidol are determined. A transesterification of 3-MCPD and glycidol esters is carried out with sodium methoxide in methanol, whereby both compounds are released. The time of this reaction is strictly controlled in order to avoid the conversion of part of 3-MCPD in glycidol. The reaction is stopped by adding an acidic solution of NaCl, which causes the conversion of free glycidol in 3-MCPD. After a clean-up step with isohexane, free 3-MCPD and the internal standard are extracted with ethyl ether:ethyl acetate (6:4, v/v), derivatized with phenylboronic acid (PBA) and determined by GC-MS/MS. The test B is carried out analogously to test A, but in this case the transesterification reaction is stopped by the addition of an acidic solution of a non-chlorinated salt, NaBr. Under these conditions free glycidol reacts to give a product other than 3-MCPD, which does not interfere in its determination. Therefore, in test B only 3-MCPD is determined. The glycidol ester content is determined from the difference between the 3-MCPD ester content calculated in tests A and B. The quantification was performed in MS/MS mode what allowed a significant reduction in the background noise. Ions m/z 91 (147 → 91) and m/z 93 (150 → 93) were selected for the measurement of 3-MCPD phenylboronate and 3-MCPD-d5 phenylboronate, respectively.

Biography

M L Fernández de Córdova is a full professor in Analytical Chemistry at the University of Jaén. She has published more than 75 papers in reputed journals as well as numerous book chapters. Her main researches involve the development of automatic methods of analysis and the determination of contaminants in food.

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