Development of an analytical method for the determination of 2- and 3-MCPD fatty acid esters in fish and fish products

1 Scope

3-MCPD (3-Monochloropropane-1,2-diol) is a food processing contaminant formed by heating foods containing a source of salt and fat. The IARC has classified 3-MCPD as a "possible human carcinogen" (group 2B) [1]. 3-MCPD in food is present not only in unbounded form, but also as mono- or di-esters of fatty acids. Further identified forms are 2-MCPD (2-Monochloropropane-1,3-diol) and glycidyl esters.

Limited data on occurrence of these contaminants in fish and fish products have been published [2].

The aim of this project was to develop an analytical method for the specific determination of 2- and 3-MCPD fatty acid esters in fish and fish products.

3 Materials & Methods

(I) Reference substances

• 1,2-bis-Palmitoly-3-Chloropropanediol
• 1,2-bis-Palmitoyl-3-Chloropropanediol-d5
• rac-1,3-bis-Stearoyl-2-Chloropropanediol
• Glycidyl stearate

(II) GC-MS

• GC: Varian CP-3800
• MS: Saturn 2200

Quantification

Single-point calibration

3-MCPD and 2-MCPD are quantified via single point calibration of the respective ions. 3-MCPD is quantified via the ions 149, 150, and 201. 2-MCPD is quantified via the ions 196, 201, and 3-MCPD-d5 via the ions 146, 147, and 196.

The developed method is robust, quickly and easily practicable.

- Key step for specific determination of 2-/3-MCPD ester (Fig. 1): Solution free of chloride → Ammonium sulphate solution (40 %)

- Limit of detection: ~ 30 µg/kg

- Successfully preliminary validation with internal and external reference material (Fig. 2)

- Comparable values by external method comparison (Fig. 3)

- Investigation of various product groups shows variable contents (Fig. 4)

Figure 1: Basis-Method [3] for determination of a sum parameter of 2-/3-MCPD ester and Glycidylester (left side) and developed method for specific determination of 2-/3-MCPD ester (right side). The circles represent the optimized steps of the method development.

Figure 2: Preliminary validation of the developed method with certified (MPHL n milk powder high/low content of 2-/3-MCPD) and internal reference material

Figure 3: Comparison of the developed method with methods of an external commercial laboratory

Figure 4: Investigation of various products from different product groups (min. and max. content, Median, 10th and 90th percentile)

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