



Sybille Merkle^{1,2}, Horst Karl¹, Jan Fritsche^{2*} Hochschule für Angewandte Wissenschaften Hamburg Hamburg University of Applied Sciences

Max Rubner-Institut, Institut f
ür Sicherheit und Qualit
ät bei Milch und Fisch, Hamburg, Germany
 HAW Hamburg – Ulmenliet 20 – 21033 Hamburg – Tel.: +49-(0)40-42875-6163 – Jan.Fritsche@haw-hamburg.de

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

• MS: Saturn 2200

(II) GC-MS

Quantifying ions:

- GC: Varian CP-3800 3-MCPD: 149, 150, 201
 - 2-MCPD: 196, 201
 3-MCPD-d₅: 146,147,196
- Quantification

Single-point calibration



Fig. 2: Preliminary validation of the developed method with certified (MPH/L = milk powder high/low content of 2-/3-MCPD) and internal reference material



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



Taking off n-Hexan, evaporation →Dissolution in isooctane →GC-MS analysis

Fig. 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.

4 Conclusion

- · 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)



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

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(1) IARC (International Agency for Research on Cancer) (2012). IARC Monographs 101, S.349-374. (2) EFSA (European Food Safety Authority) (2013). EFSA Journal 2013;11(9):3381. (3) DGF (Deutsche Gesellschaft für Fettwissenschaft) (2010). Einheitsmethode C-VI 17 (10). Abteilung C1. WVG Stuttgart, Germany. (4) OKOTEST (2012). Stäbchenprobe. Heft Nr. 10. S. 82-86.