Investigation of Critical Factors of 3-MCPD Esters Determination

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3-Chloropropane-1,2-diol (3-MCPD) and 3-MCPD fatty acid esters are food-borne contaminants formed during processing of various raw materials and the production of various foodstuffs (e.g. bread and cereal-based products). Recently, relatively high levels of 3-MCPD esters have been found in refined vegetable oils and several oil-based products. Despite the recognition of the occurrence of 3-MCPD esters in foods, the comparison of the levels reported is extremely difficult as there is no official validated method for the determination 3-MCPD esters. With an ongoing effort to optimise and validate a reliable method, a need to understand and evaluate the individual steps in the 3-MCPD analysis emerges.

This work is focused on the comparison of the most common analytical methods used for determination of 3-MCPD fatty acid esters. Direct analysis of 3-MCPD esters at trace levels is complicated. Faster routine methods are based on the release of free 3-MCPD from 3-MCPD fatty acid esters by transesterification (either in acidic or alkalic environment) and its determination after derivatisation by GC-MS.

The comparison of the analytical procedures using different type of transesterification – in methanolic solution of either sulphuric acid or sodium methoxide – showed significant differences in the method recovery; that suggests the possibility of decomposition (or even an additional formation) of the analyte during the analytical procedure. As the degradation of 3-MCPD in alkalic media was observed, several critical factors of the method based on alkalic catalysed transesterification were studied in detail, particularly the effect of the transesterification time, the impact of different pH value of the sample after transesterification, the influence of various salts used during the sample preparation and the type of the internal standard (3-MCPD-d_5 v. 3-MCPD-d_5 dipalmitate).