

Development of a method for identification of FAMES in fat from pork by GC-MS

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Abstract

A GC-MS method was developed for qualitative and quantitative analysis of FAMES. Mass spectrometry was used both in scan mode and SIR. The method was optimized to separate C6:0 to C24:1*n*-9 methyl esters. Mass spectrometry in scan mode was used for qualitative and quantitative analyses of FAMES, while SIR was used for quantitative analyses of FAMES when *m/z* fragments and retention times were known. There were two different SIR methods, each using different ions. The ions in SIR method #1 were four fragments with relatively high intensity in the upper part of the mass spectra. SIR method #2 included the three ions with the highest intensity in the mass spectra.

Fatty acid methyl esters were quantified using RRF and IS. Two IS were used, C11:0 ethyl ester (EE) and C19:0 ME. Ethyl ester C11:0 was used to quantify C6:0 to C14:0 MEs, and C19:0 ME was used to quantify C14:1*n*-5 to C24:1*n*-9 MEs. The RRF for the FAMES varied, depending on which IS and method that were used.

The LOD and LOQ of FAMES varied both within different compounds, and between scan and SIR mode. The LOD of FAMES using scan mode varied from 0,021 to 0,150 µg/mL, for SIR method #1 from 0,001 to 0,096 µg/mL, and for SIR method #2 from 0,002 to 0,114 µg/mL.

The difference between measured and theoretical concentration of FAMES, together with the precision were determined. In scan mode the recovery was between 81,7-123,7% with RSD from 0,1 to 9,8%, for SIR method #1 the recovery was between 89,6-120,4% with RSD from 0,03 to 6,8%, and for SIR method #2 the recovery was between 95,1-152,1% with RSD from 0,1 to 20,4%.

The stability of FAMES compared with that of C19:0 ME was examined. Standards of FAMES were stored at room temperature and -20 °C. For saturated and unsaturated FAMES stored at -20 °C in 12 weeks the recovery was 86-121%. For standards of FAMES stored at room temperature there was some loss due to evaporation. The recovery for most of the FAMES was 80-120% after 2 weeks storage.

The method was used for qualitative and quantitative analysis of fatty acids in ham (Noroc). Phospholipids and triglycerides were extracted from ham with solid phase extraction. The fatty acids bonded to lipids were transesterified to ME, and analysed with GC-MS in scan mode and SIR method #1. For most of the quantified FAMES the difference between scan mode and SIR method #1 was less than 20%, but the difference between the methods increased when the concentration was approaching LOQ.

GC-MS is a suitable method for analysis of FAMES, and scan mode was used for qualitative and quantitative analysis, while SIR was used for quantitative analysis of FAMES with known *m/z* fragments and retention times. The advantage with SIR compared to scan mode, is a gain in sensitivity in SIR mode.