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Title: Determination of short chain carboxylic acids in vegetable oils and fats using ion exclusion chromatography electrospray ionization mass spectrometry

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1 Determination of short chain carboxylic acids in vegetable oils and fats using ion  
2 exclusion chromatography electrospray ionization mass spectrometry

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6 Abstract

7 A new method for quantification of short chain C<sub>1</sub>-C<sub>6</sub> carboxylic acids in vegetable oils and fats by  
8 employing Liquid Chromatography Mass Spectrometry (LC-MS) has been developed. The method  
9 requires minor sample preparation and applies non-conventional Electrospray Ionization (ESI) liquid  
10 phase chemistry. Samples are first dissolved in chloroform and then extracted using water that has been  
11 spiked with stable isotope labeled internal standards that are used for signal normalization and absolute  
12 quantification of selected acids. The analytes are separated using Ion Exclusion Chromatography (IEC)  
13 and detected with Electrospray Ionization Mass Spectrometry (ESI-MS) as deprotonated molecules. Prior  
14 to ionization the eluent that contains hydrochloric acid is modified post-column to ensure good  
15 ionization efficiency of the analytes. The averaged within run precision and between run precision were  
16 generally lower than 8 %. The accuracy was between 85 and 115 % for most of the analytes. The Lower  
17 Limit of Quantification (LLOQ) ranged from 0.006 to 7 mg/kg. It is shown that this method offers good  
18 selectivity in cases where UV detection fails to produce reliable results.

19

20 Keywords: Vegetable oil; Short-chain carboxylic acid; Ion exclusion chromatography; LC-MS;  
21 Electrospray

## 22 1. Introduction

23

24 A variety of oils and fats are typical raw materials for the production of renewable diesel fuels. The  
25 current trend is towards increasing the share of wastes and residues such as animal fats, Used Cooking  
26 Oil (UCO) and different acidic palm oil residues like Palm Fatty Acid Distillate (PFAD) as much as possible.  
27 Such materials are of lower quality than refined vegetable oils and contain higher amounts of impurities.  
28 Acidic components such as short chain carboxylic acids form one important impurity class of renewable  
29 feedstock. The amount of acidic components needs to be determined prior to production for process  
30 optimization because of their corrosive nature. In addition, short chain fatty acids such as butyric and  
31 valeric acids and their isomers are volatile and have strong odor that can cause occupational safety  
32 issues in the manufacturing process. Currently only a few analytical methods exist in the literature for  
33 these analytes and sample matrices [1].

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