Compositional analysis of fatty acids in food samples by THM-GC/MS

Part 1: Optimal reaction time for the use of *m*-TFPTAH reagent at room temperature

[Background] In the compositional analysis of fatty acids in food samples, methyl esterification of fatty acids using a boron trifluoride (BF_3)-methanol reagent has traditionally been used in combination with GC analysis. This method allows methyl esterification of polyunsaturated fatty acids (PUFAs) without cis-trans isomerization, but the sample preparation procedures are cumbersome. Although thermally assisted hydrolysis and methylation (THM)-GC/MS is known as a rapid analytical method, cis-trans isomerization would be caused when tetramethyl ammonium hydroxide (TMAH) is used as a reagent. On the other hand, it has been known that the compositional analysis of PUFAs in lipid samples can be effectively conducted with suppressing the isomerization reactions in the presence of 3-(trifluoromethyl)phenyl trimethylammonium hydroxide (*m*-TFPTAH; Fig. 1) as a reagent[1]. In this study, the optimal reaction time at room temperature between a food sample (algae oil) and m-$TFPTAH_2$ for the analysis by THM-GC/MS was examined.

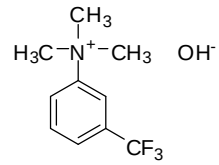

Fig. 1 Structural formula of *m*-TFPTAH

[Experimental] 3 µL of a hexane solution of algal oil (2 µg/µL) was put in a sample cup, followed by addition of 5 µL of a methanolic solution of *m*-TFPTAH (5 wt%). The sample cup was left at room temperature for 0 - 60 min, followed by the GC/MS.

[Results] As shown in the upper part of Fig. 2, methyl docosahexaenoate (DHA; C22:6; with six double bonds) and its isomers were detected in the chromatogram obtained for a reaction time of 0 min. For 30 min and 60 min reaction times, the peaks of isomerized compounds mostly disappeared as shown in the lower part of Fig. 2. It was found that PUFAs were fully esterified while suppressing isomerization when the reaction time was 30 min or longer. Thus, the optimal reaction time for the esterification with *m*-$TFPTAH_2$ was determined to be 30 min. In the next note (PYA1-171E), compositions of fatty acids in food samples obtained by the preset *m*-TFPTAH method are compared with those obtained by the conventional BF_3-methanol method.

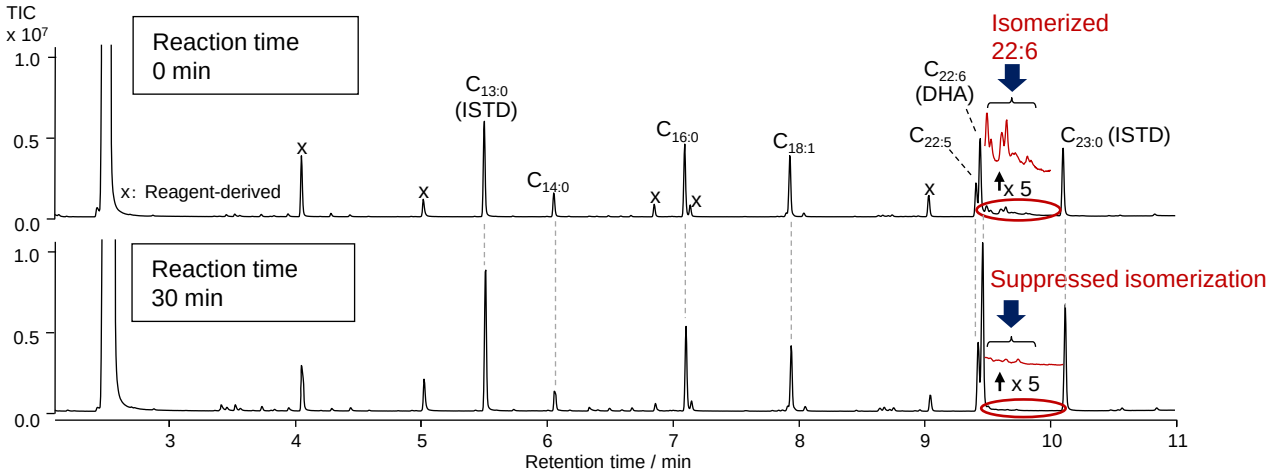

Fig. 2 Chromatograms of algae oil obtained for reaction times of 0 min (upper) and 30 min (lower).
($C_{m:n}$: where m is the number of carbons, n is the number of unsaturated bonds)

Furnace temp.: 400 ºC, Furnace-GC interface temp.: 280 ºC, GC inj. temp.: 250 ºC, Separation column: UA+-5 (*L*=15 m, i.d.=0.25 mm, df=0.25 µm), Column flow: 1.0 mL/min (He), Sample amount: 6 µg, Reagent: *m*-TFPTAH 5 wt% methanol solution 5 µL.

1) Freeman, R. R. *et al., Lipid Tech*., 23, 254-256 (2011).

Keywords : Oil and fat, Edible oil, Compositional analysis, Fatty acid, Thermally assisted hydrolysis and methylation

Products used : Multi-functional pyrolyzer, Vent-free GC/MS adapter, UA+-5, Eco-Cup LF

Applications : Food analysis, Lipid analysis, Quality control (QC)

Related technical notes : PYA1-171E (Part 2), PYA2-023E, PYA2-030E

Please forward your inquiries via our web page or send us a fax message.

R&D and manufactured by :
Frontier Laboratories Ltd.
Phone: (81)24-935-5100 Fax: (81)24-935-5102
www.frontier-lab.com