Compositional analysis of polyunsaturated fatty acid oil by one-step thermally assisted hydrolysis / methylation in the presence of trimethylsulfonium hydroxide (TMSH)

[Background] Thermally assisted hydrolysis and methylation (THM) in the presence of an organic alkali such as tetramethylammonium hydroxide (TMAH) has been a powerful method in the determination of chemical compositions of polymers and ester compounds including lipids. However, it has been pointed out that strong alkalinity of TMAH caused the isomerization and/or degradation of polyunsaturated fatty acid (PUFA) components during the THM reaction. Here, one-step THM-GC in the presence of TMSH for the determination of fatty acid components especially PUFA in lipid samples is described.

[Experimental] Triarachidonin, soybean oil, and sardine oil were used as supplied. Solutions of various concentrations of TMSH and tetramethylammonium hydroxide (TMAH) were prepared. A Py-GC equipped with a capillary column and with FID and MS as detector was used. The pyrolysis was done at 350˚C, at which fatty acid methyl esters were obtained at highest yields.

[Results]. Figure 1 shows the typical chromatograms of the soybean oil sample obtained by one-step THM-GC in the presence of (a) TMSH and (b) TMAH, respectively. As anticipated, many additional isomer peaks of C18:2 and C18:3 derivatives resulted from the thermal isomerization in the presence of TMAH are observed, while these are hardly seen in the case of (b) TMSH. Table 1 summarizes the chemical compositions of fatty acid components in the soybean oil obtained both by the one-step THM-GC in the presence of TMSH and TMAH together with those by the offline transmethylation along with the recovery data of components. The fatty acid compositions obtained using 0.2M of TMSH were in fairly good agreement with those obtained by the offline GC method showing fairly high and constant recoveries about 80% for every fatty acid component. When sardine oil was used as sample, the fatty acid composition obtained by THM-GC were in a good agreement with those by offline GC method with a good reproducibility.

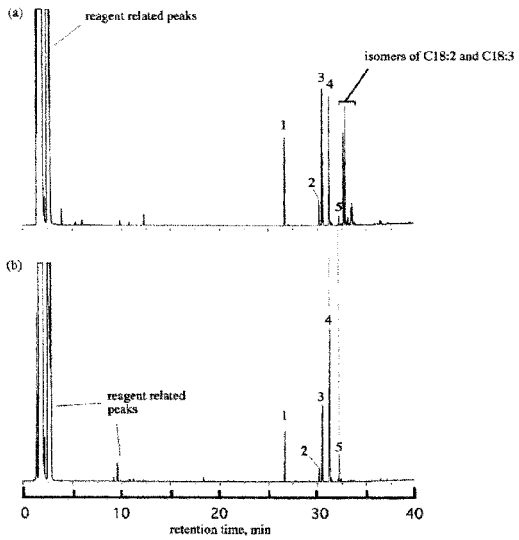

Figure 1. Chromatogram of soybean oil obtained by one-step THM-GC at 350˚C (a) in the presence of 0.15M of TMAH (b) in the presence of 0.2M TMSH. Peaks of fatty acid methyl esters: 1, C16:0; 2, C18:0; 3, C18:1; 4, C18:2; 5, C18:3

Table 1. Chemical composition and recovery of fatty acid components in soybean oil obtained by one-step TM-GC

		Fatty acid						
		16.0	18.0	18:1	18:2	18:3	Isomers of 18:2&18:3	Total
		Compositions (mol%)						
Offline method*		13.2	4.0	22.6	54.6	5.7	nd	100
One step THM-GC								
THSH	0.2M	13.4(74.6)	3.7(76.5)	22.8(81.5)	54.8(80.6)	5.4(75.5)	nd	100
TMAH	0.05M	13.4(18.2)	3.6(17.3)	22.1(21.0)	55.2(19.2)	5.7(18.2)	nd	100
	0.1M	13.2(43.6)	3.7(42.2)	22.4(51.5)	48.2(37.7)	5.1(36.5)	7.4	100
	0.15M	13.3(83.5)	3.9(82.5)	23.2(99.6)	22.5(40.5)	1.1(22.1)	34.8	100

*Offline transmethylation by TMSH followed by GC measurement

Data in parenthesis: recovery obtained from the observed molar peak intensity normalized by sample weight.

*Contents excerpted from Y. Ishida, S. Wakamatsu, H. Yokoi, H. Ohtani, S. Tsuge, *J. Anal. Appl. Pyrolysis,* 49 (1999) 267-276

Keyword : Thermally assisted hydrolysis and methylation, Trimethylsulfonium hydroxide, Fatty acid oil, Pyrolysis

Applications : Lipid analysis

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