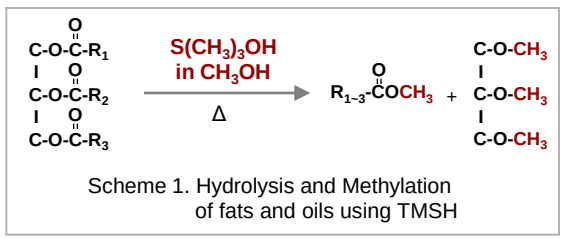
# Notes on Reactive Pyrolysis of Fatty acids using Trimethylsulfonium hydroxide

**[Background]** The official method for the analysis of fatty acids in fats and oils instructs analysts to use boron trifluoride ($BF_3$); however, because it is a cumbersome procedure and the high toxicity of $BF_3$, simpler and safer analytical procedures are of interest. Reactive pyrolysis using a Py-GC/MS system is one possibility to consider. Often, tetramethyl-ammonium hydroxide (TMAH) is used as the organic alkali; however, TMAH is known to cause isomerization of the polyunsaturated fatty acids. Recently, Ishida et al. have reported that trimethylsulfonium hydroxide (TMSH) will reduce the degree of isomerization during the hydrolysis and methylation of the fatty acids[1)] Isomerization is known to be directly related to the amount of TMSH used. This report illustrates how the level of isomerization is influenced by the amount of TMSH added to the sample.

**[Experimental]** Soybean oil (10 µg) was placed in a sample cup (Eco-cup L), 2~4 µL of a methanolic solution of TMSH (0.01~0.2 M) was added to the cup. The reaction temperature was 350ºC. 80% recovery was typically achieved.

**[Results]** The major constituent in oils (fats) such as soybean oil are triglycerides. As shown in Scheme 1, reactive pyrolysis using TMSH , results in the formation, via an ester-exchange reaction, of methyl derivatives of fatty acids (FAMEs). Pyrograms of soybean oil obtained using varied amounts of TMSH are shown in Fig. 1. Methyl esters of fatty acids are observed. The peak area ratio of C18:2 : C18:3 is larger in pyrogram A. Isomers presumably formed though isomerization of C18:2 are observed in pyrogram B.These results show that increasing the amount of TMSH increases the isomerization of the polyunsaturated fatty acids. To determine the optimal amount of TMSH to use, the dependence of the FAME peak areas on the amount of TMSH was determined – Fig. 2. The peak area of C16:0 is independent of the TMSH concentration. This is attributed to the fact that the reaction efficiency for C16:0 is high. On the other hand, the isomerization of C18:2 and C18:3 depends on the conditions. A TMSH amount of 0.2 x $10^{-6}$ mol, which is 10 times the chemical equivalent of the sample, results in the maximum area counts. Increasing the amount of TMSH beyond 10 times reduces the peak area counts, This indicates that larger amounts of isomerized products have formed. Based on these results, the optimal amount of TMSH is 10 times the chemical equivalents of the fatty acids in the sample. Compositional analysis of the FAMEs in soybean oil using reactive pyrolysis Py-GC/MS are comparable to those obtained using $BF_3$ derivatization.




Scheme 1. Hydrolysis and Methylation of fats and oils using TMSH

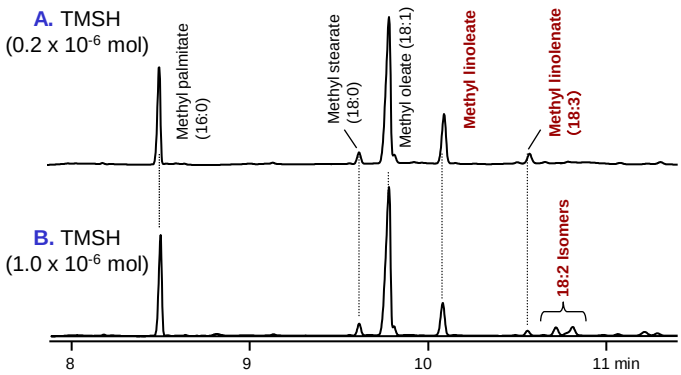



Fig. 1: Pyrograms of soybean oil with varied amounts of TMSH

Py temp.: 350ºC, Oven temp.: 40 – 240ºC(20ºC/min), Column: UA-CW (polyethylene glycol) (L=30 M, i.d.=0.25 mm, df=0.25 µm), Carrier gas He: 1 mL/min, split ratio: 1/50

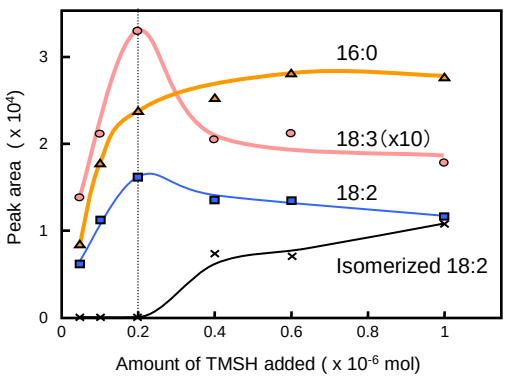



Fig. 2: Amount of TMSH vs. peak areas of fatty acids

1) Y. Ishida et al. J. Anal. Appl. Pyrolysis 49 (1999) 267-276

**Keywords :** Fats and Oils, Fatty acids, Reactive Pyrolysis, TMSH, $BF_3$

**Products used :** Multi-functional pyrolyzer, Vent-free GC/MS adapter, UA-CW

**Applications :** Foods and related industries

**Related technical notes :** PYA2-022E, PYA2-018E

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