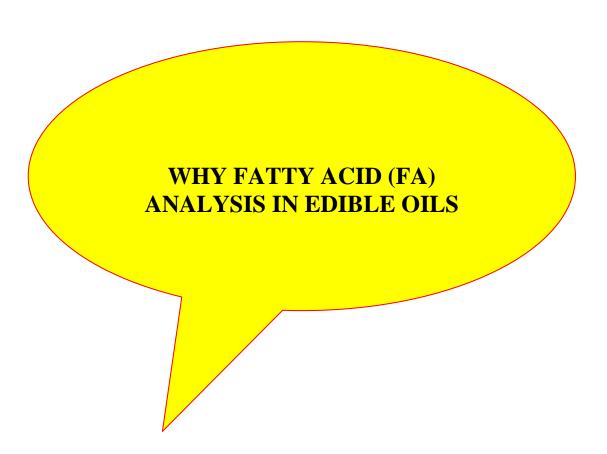
# DETERMINATION OF FATTY ACIDS IN EDIBLE OILS BY CAPILARY GC

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The content of fatty acids as well as the ratio between unsaturated and saturated fatty acids is important parameter for determination of nutritional value of certain oil (1). Therefore the newest trend in food processing industry is notifying the composition of edible oils and other food commodities on the content of each individual fatty acid.

# **METHODS FOR FATTY ACIDS ANALYSIS**

The most of the proposed methods [2-5] for fatty acid analysis by capillary GC involve their derivatization with BF<sub>3</sub> in methanol or methanolic H<sub>2</sub>SO<sub>4</sub>, because these highly polar compounds tend to form hydrogen bonds and are often absorbed on the column.



In this issue simple, fast, precise and reliable method for the determination of long chain fatty acids (FA): C<sub>16:0</sub>; C<sub>18:0</sub>; C<sub>18:1</sub>; C<sub>18:2</sub> and C<sub>18:3</sub> in edible oils is described. Procedures of isolation of the FA from edible oils [1], and capillary gas chromatographic analysis [2,3] were developed and optimised. After saponification with KOH in methanol (0.5 mol/L), FA were liberated by addition of 25% (V/V) HCl (pH=3) and extracted with petroleum ether (40-70). The underivatized FA are separated and quantified directly by capillary gas chromatography (GLC) on HP-FFAP column.



## INSTRUMENTAL

HP model 5890 series II (plus) gas chromatograph with an HP automatic liquid sampler and a flame-ionisation detector (FID) was used with a capillary HP-FFAP column (25 m x 0.32 mm id., 0.52  $\mu$ m film thickness).

The carrier gas (nitrogen) flow rate was 82cm'sec<sup>-1</sup> and the split ratio was 1:10. The injection port was maintained at 230 °C and the FID at 260 °C. Oven temperature was set at 180 °C increasing for 2 °C'min<sup>-1</sup>. The final oven temperature was maintained at 230 °C (4 minutes).

#### **MATERIALS**

60 samples of sunflower, peanut and olive oil were taken for fatty acid analysis from the market.

## REAGENTS AND STANDARDS

All solvents and standards were of analytical grade and were purchased from Merck (Germany). The fatty acid standard oil was obtained from Supelco. Stock solutions of fatty acids were prepared in acetone.

# **PROCEDURE**

0.025 g of oil were saponificated in 5 ml methanolic KOH (0.5 mol·L<sup>-1</sup> solution) by refluxing for 35 minutes at 100 °C in tightly sealed Pyrex tubes. After cooling the pH value of the solution was adjusted to 3 with 25% HCl (V/V) and fatty acids were extracted into 10 mLof petroleum ether (40-70). 5 ml of the extract containing fatty acids were diluted to 50 ml with petroleum ether (40-70). Aliquots of the extracts were injected into the column. Identification of the individual fatty acids was achieved by comparison with authentic reference standards.

# **RESULTS AND DISCUSSION**

Most of the analytical methods for fatty acid analysis in oils by capillary GC [2-5] include their esterification to fatty acid methyl esters (FAMES) with various esterification agents. In order to avoid this esterification step, an analytical method for direct analysis of some long chain fatty acids (C<sub>16:0</sub>; C<sub>18:0</sub>; C<sub>18:1</sub>; C<sub>18:2</sub> and C<sub>18:3</sub>) in edible oils was developed and optimised. FA were isolated from oils by saponification performed with 0.5 mol·L<sup>-1</sup> KOH in methanol, liberated by adding HCl (25% v/v) to pH=3 and extracted with petroleum ether (40-70). The underivatized FA were separated and quantified directly by capillary GC on HP-FFAP column, which enable accurate and rapid determination within 30 minutes (figure 1).

Standard mixed oil was analysed using this direct method and esterification method with BF<sub>3</sub> in methanol and the results obtained are given in Table I.

Because of excellent recovery (R), which was found between 99.0-100.0 %, the use of internal standard in order to compensate for the losses is not necessary.

With this method described all major FA ( $C_{16:0}$  -  $C_{18:3}$ ) in edible oils can be quantified with good repeatability (RSD between 2.0-2.4 %). The method is also applicable to the analysis of minor FA ( $C_{16:1}$ ;  $C_{20:0}$  -  $C_{24:0}$ ) in edible oils and also in other products.

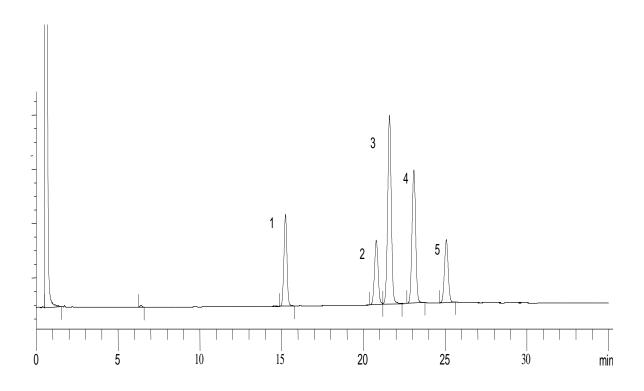


Figure 1. Chromatogram of fatty acids in standard oil on HP-FFAP 1.  $C_{16:0}$ ; 2.  $C_{18:0}$ ; 3.  $C_{18:1}$ ; 4.  $C_{18:2}$ ; 5.  $C_{18:3}$ 

TABLE I

Comparison of direct method and esterification method for FA analysis by GLC

Fatty acid	Concentration in standard oil (%, m/m)	Direct determination		Esterification method	
		R (%)	RSD (%)	R (%)	<b>RSD</b> (%)
$C_{16:0}$	10.0	99.2	2.1	89.3	4.2
$C_{18:0}$	10.0	100.0	2.3	89.8	4.5
$C_{18:1}$	50.0	99.5	2.0	90.2	4.1
$C_{18:2}$	10.0	99.2	2.4	90.3	3.9
$C_{18:3}$	20.0	99.0	2.3	89.9	4.6

n=40



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