

BPX70

FATTY ACID DISTRIBUTION IN EDIBLE OILS

Quantitative and Qualitative Analysis of Fatty Acid Methyl Esters (FAME) Using a 0.53mm ID Capillary Column

INTRODUCTION

- Rapeseed Oil
- Sunflower Oil
- Canola Oil

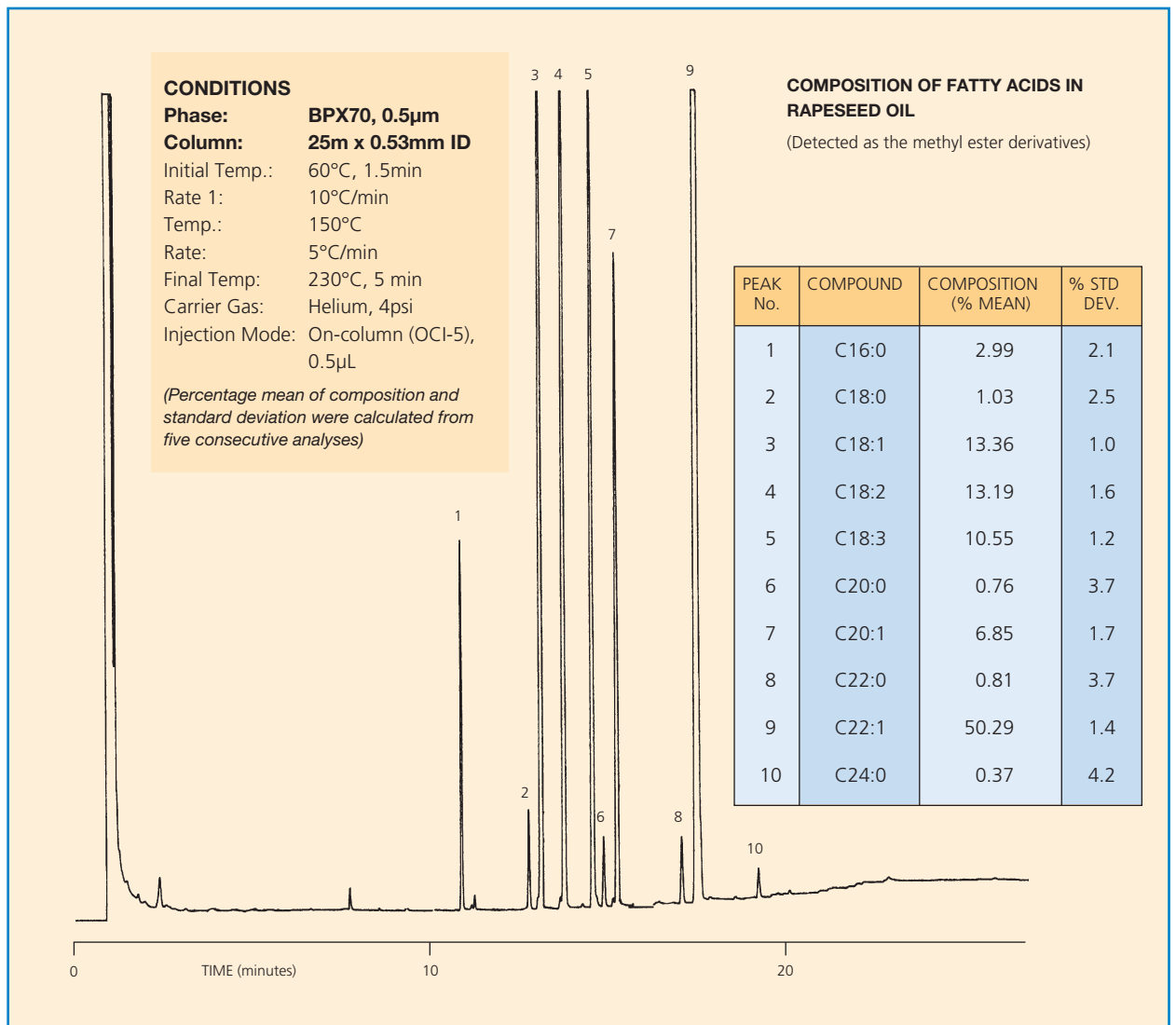
It is of great importance, both for economic and medical reasons, that the fatty acid distribution in edible oils be identified and characterized.

Research has shown that certain compounds in edible oils, particularly the saturated fatty acids, are detrimental to our health. High levels of these components have been shown to increase the occurrence of cardiovascular diseases.

Also associated with the health consideration, is the quality control of edible oils. It is of great importance that the quality of an edible oil be as stated by a supplier or manufacturer, because unidentified components or uncharacterised fatty acid distribution can affect its value.

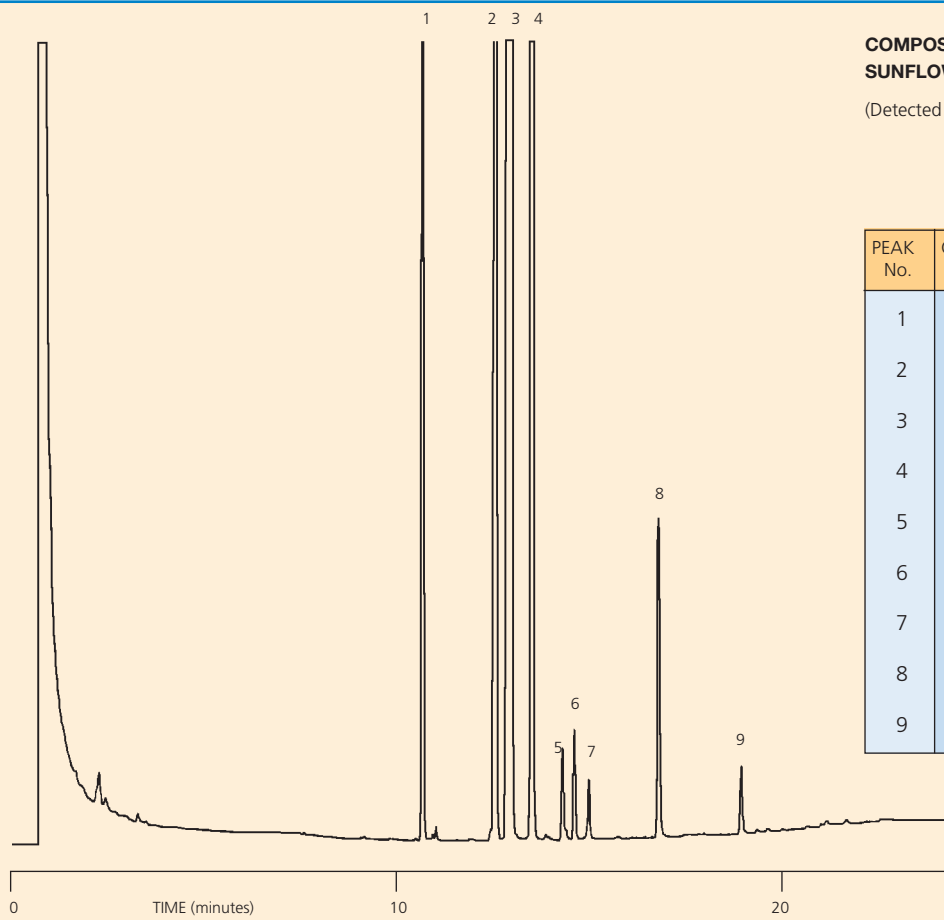
It is, therefore, essential that a capillary column be available to provide both quantitative and qualitative information on the composition of the numerous edible oils consumed throughout the world.

APPLICATION NOTE



COMPOSITION OF FATTY ACIDS IN SUNFLOWER OIL

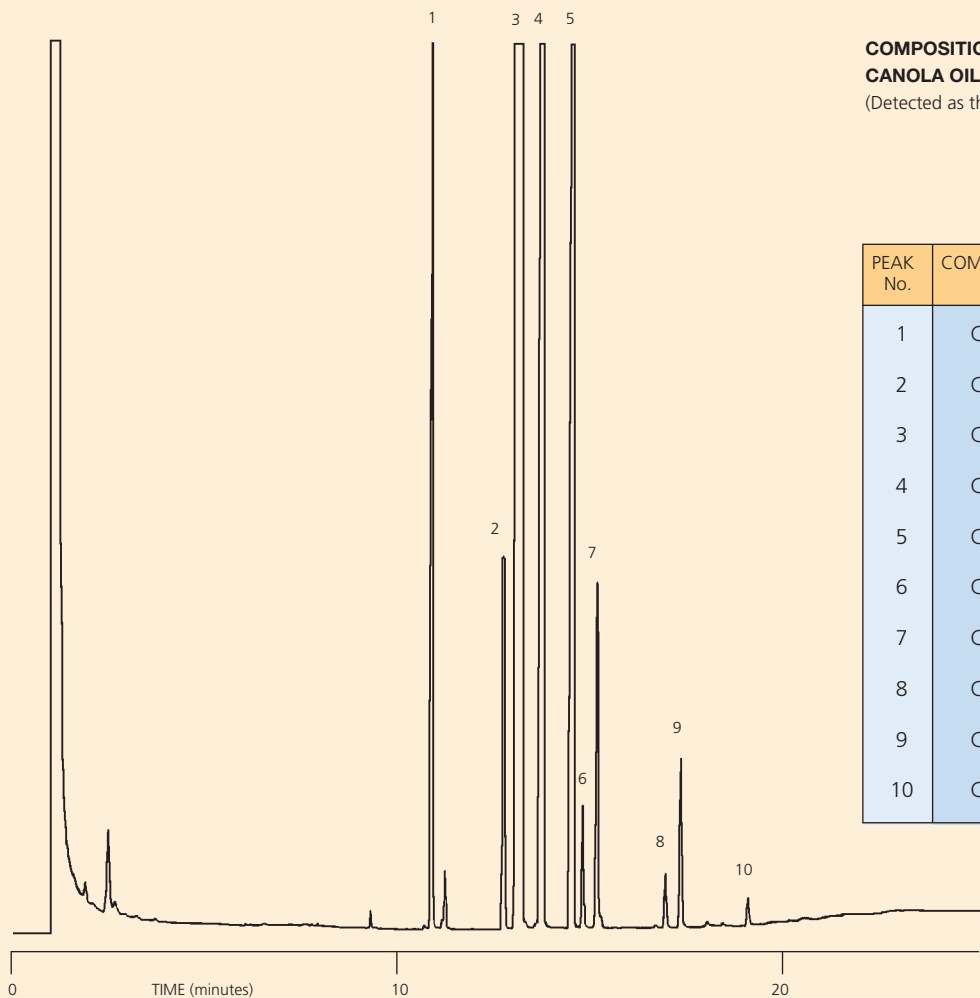
(Detected as the methyl ester derivatives)



PEAK No.	COMPOUND	COMPOSITION (% MEAN)	% STD DEV.
1	C16:0	3.82	1.8
2	C18:0	6.25	1.0
3	C18:1	69.12	2.9
4	C18:2	17.04	1.0
5	C18:3	0.40	4.1
6	C20:0	0.50	1.4
7	C20:1	0.29	1.4
8	C22:0	1.48	1.3
9	C24:0	0.35	5.1

COMPOSITION OF FATTY ACIDS IN CANOLA OIL

(Detected as the methyl ester derivatives)



PEAK No.	COMPOUND	COMPOSITION (% MEAN)	% STD DEV.
1	C16:0	4.21	2.6
2	C18:0	1.94	2.5
3	C18:1	58.04	2.6
4	C18:2	21.51	2.7
5	C18:3	10.24	2.7
6	C20:0	0.61	1.6
7	C20:1	1.71	1.7
8	C22:0	0.29	3.1
9	C22:1	0.88	1.5
10	C24:0	0.15	3.3

BPX70 is a thermally modified siloxane phase containing a high concentration of cyanopropyl groups. It is ideal for the separation and characterization of saturated and partially unsaturated fatty acids (as the methyl ester derivative) found in edible oils. Baseline separation of consecutive unsaturated fatty acids, (i.e. C18:1, C18:2, C18:3, etc.) even when present in greatly varying concentrations, can be achieved on large inside diameter capillary columns.

BPX70 coated inside 0.53mm ID capillary tubing is a versatile approach to edible

oil analysis as it allows short analysis times, high loading capacity, and being a bonded phase, excellent column life.

However, to be a truly versatile column, reproducibility of an analysis is a necessity. With a standard deviation averaging less than 2.5% from five consecutive analyses of rapeseed, sunflower and canola oil, the BPX70 column could only be regarded as an excellent choice for this analysis.

PERFORMANCE PROFILE

What conditions do I require to maintain

adequate separation of a FAME mixture?

Once the column ID, length and carrier gas has been chosen, the two remaining parameters which can effect the separation are:

- Carrier Gas Velocity
- Sample Loading

CARRIER GAS VELOCITY

Often we are told to operate our capillary column at the optimum gas velocity, as varying from this value will result in a loss of resolution. Although correct, the decrease in efficiency (separating power) is not as significant as would be first thought. Resolution and efficiency do not have a linear relationship, therefore, to affect the resolving power of a column, a substantial change in gas velocity is generally required.

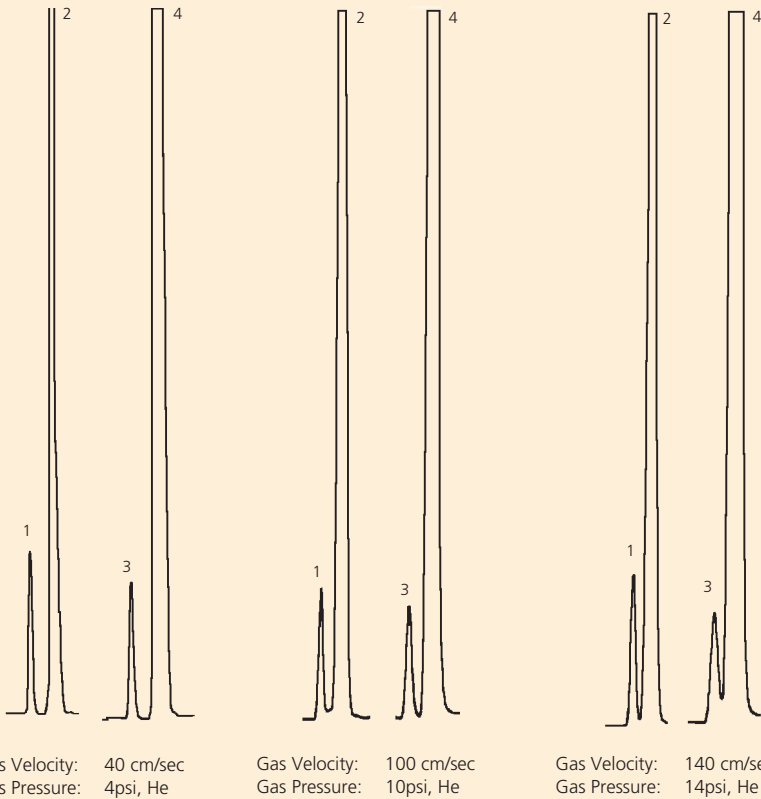
The analysis of two pairs of closely eluting fatty acid methyl ester isomers from rapeseed oil, (C18:0/C18:1 and C22:0/C22:1) demonstrates the effect on resolution with increasing carrier gas velocity (Fig. 1). A gas velocity of greater than 100cm/sec for helium is needed before baseline resolution of the isomers is lost on a 0.53mm ID BPX70 column. Therefore, a working gas velocity range from optimum (25cm/sec) to 100cm/sec can be used without sacrificing the resolution required to ensure qualitative and quantitative results with a ratio of 100:1.

Figure 1.

CARRIER GAS VELOCITY EFFECTS

Phase: BPX70, 1.0µm
Column: 25m x 0.53mm ID
 Initial Temp.: 60°C
 Rate: 5°C/min
 Final Temp.: 230°C
 Injector: OCI-5

Components
 1. C18:0
 2. C18:1
 3. C22:0
 4. C22:1



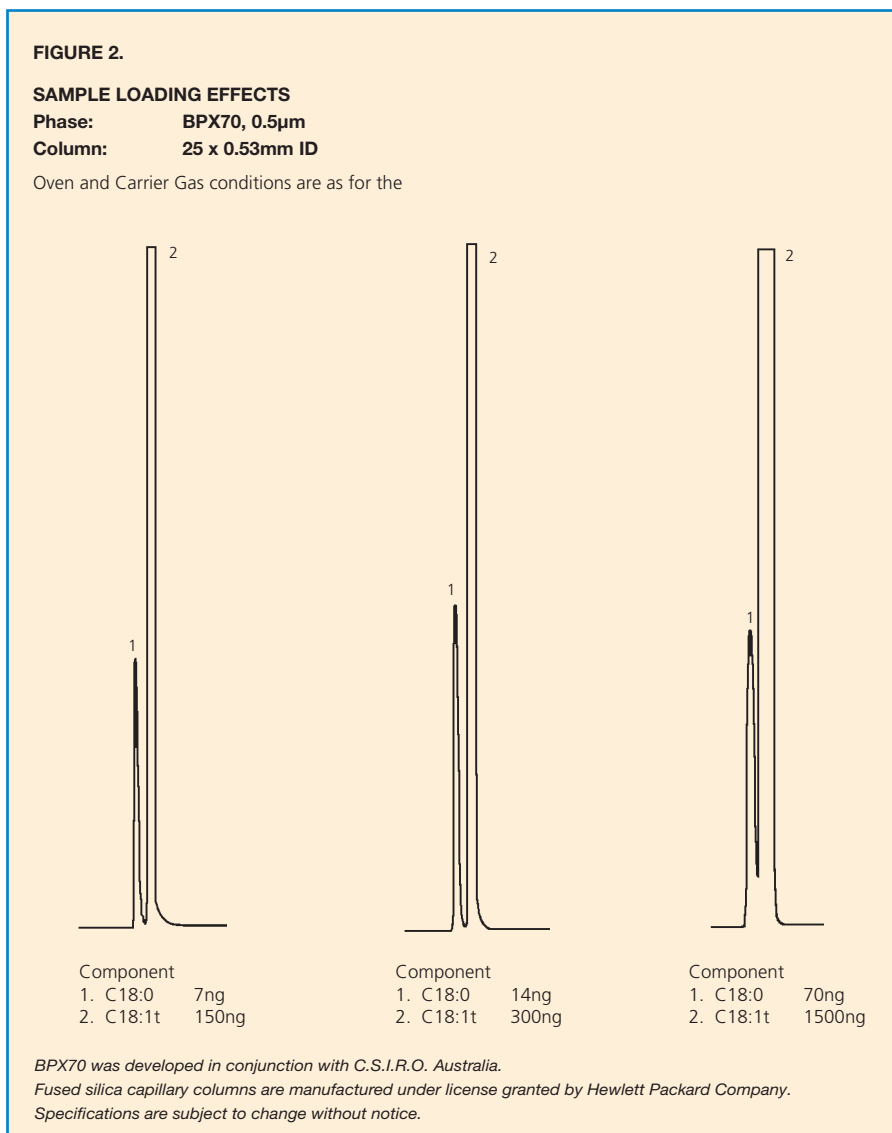
RECOMMENDED OPERATING CONDITIONS

Carrier Gas Velocity (Helium): 30-100cm/sec, 3.5-10 psi
 Sample Concentration (as FAME)
 On-column: 0.2-0.8 mg/ml, (0.02-0.08% solution)
 Split Injection: (20:1 split) 4-16 mg/ml, (0.4-1.6% solution)

SAMPLE LOADING

Sample overloading is a parameter often overlooked, particularly when the chromatographer is familiar with only packed column technology or has only recently begun to use capillary columns. Sample loading can effect the quality of an analysis as much as either column diameter or length. The analysis of edible oils presents an even greater problem. Unfortunately, though comprising of only a relatively small range of fatty acids, their distribution in edible oils is never equal. In fact, it is not uncommon to find two consecutive isomers present with a ratio of 100:1.

Figure 2 provides an indication of the level of sample loading that a 0.53mm ID BPX70 column can accommodate before resolution is affected. The C18:1 trans isomer (elaidic acid) has been used (eluting closer to the C18:0 than the cis isomer) to demonstrate the large sample range that can be analyzed using this capillary column. An increase in sample loading from 300ng to 1500ng only results in a small loss of resolution between the two esters. To maintain baseline separation of the two isomers, a sample loading of up to 700ng for the largest component could be analysed before baseline resolution is lost.



* Denotes 10 meter column

ID (mm)	FILM (µm)	12 meter Part No.	15 meter Part No.	25 meter Part No.	30 meter Part No.	50 meter Part No.	60 meter Part No.	120 meter Part No.
0.1	0.20	054600*	—	—	—	—	—	—
0.22	0.25	054601	—	054602	054612	054603	054613	—
0.25	0.25	-	054621	—	054622	—	054623	054624
0.32	0.25	054605	—	054606	054616	054607	054617	—
0.53	0.5	—	054619	054610	054620	—	—	—

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