

Determination of the fatty acid composition in oils and fats in high throughput

with the CHRONECT Workstation FAMEs High Throughput



Application note 1906

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Introduction

Rapid determination of fatty acid compositions (FAC) in natural fats and oils for production control is an important application for many refineries and contract laboratories. Natural fats and oils are composed of a variety of organic compounds, including fatty acids and mono/di/triacylglycerols. Fatty acids form a diverse group of molecules consisting of repeating methylene groups that influence the hydrophobic character of the molecule. Due to their biological, structural and functional role, fatty acids are important components of the human body. For example, they support the transport of proteins across the cell membrane. Their composition can vary to fulfill these functions. Dietary intake of fatty acids therefore has implications for human health and needs to be monitored for all oil- and fat-containing products. The general approach for determining FAC consists of optional fat extraction followed by transesterification. The resulting free fatty acids are then separated by gas chromatography and the

percent composition is calculated. Based on the FAC, the individual concentration of the monitored fatty acids is determined for the final product. The analytical method has been used in the laboratory for decades but has a low throughput due to a GC run time of up to 50 minutes per sample. Often, multiple gas chromatographs (GC) are used in a single laboratory for FAC determination only, in order to be able to measure enough samples in one day.

The CHRONECT Workstation FAMEs High Throughput established a novel approach by placing an autosampler over two GCs, each with two injectors that prepares and injects up to four samples in individual batches. The sample preparation follows the official AOCS Cd2-66 method and simultaneously prepares and injects four samples within 48 minutes. The GCs run a temperature ramp for optimal separation of the sought-after fatty acids. Intelligent overlap and parallel sample preparation result in the ability to measure over 100 samples per day; an important criterion for routine laboratories.

Device setup

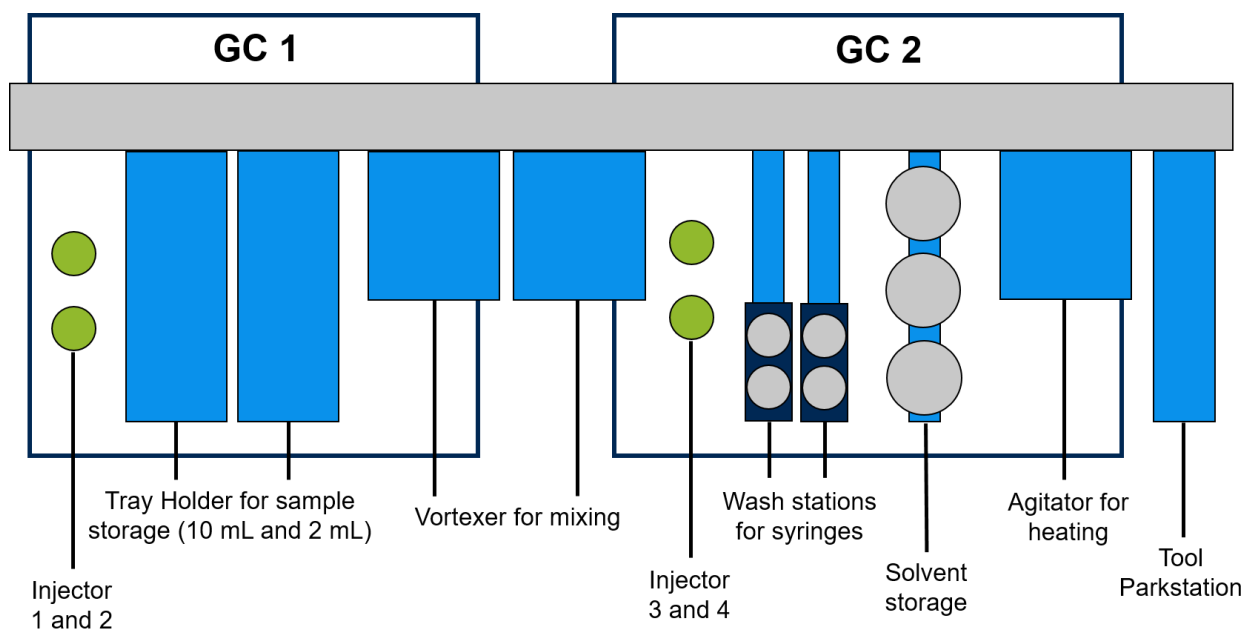


Figure 1: Schematic setup of the CHRONECT Robotic RTC/RTC Autosampler with modules for FAME analysis according to AOCS Cd2-66.

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The CHRONECT Workstation FAMEs High Throughput consists of a CHRONECT Robotic Dual Head Autosampler and several modules for sample preparation, such as solvent stations, two wash stations as well as an agitator and two vortex mixers (Figure 1). Samples are preprepared in 10 mL vials (approximately 100 mg of fat or oil) and then prepared and injected fully automatically.

The autosampler is installed on two GCs, each equipped with two split/splitless injectors (injector 1,2,3 and 4) and two FID detectors. Each GC thus has 2 channels and a CHRONECT Workstation FAMEs High Throughput up to 4 channels (Figure 1).

Measurement parameters and results

All four injectors of the two GCs are operated at isothermal temperatures of 250 °C. The GCs run an oven program with a ramp to 225 °C within 41 minutes. All other measurement parameters are listed in Table 1.

On two CHRONECT Workstation FAMEs High Throughput systems, an oil sample is analyzed on all 8 channels to demonstrate the reproducibility and stability of the system. The results are shown in Table 2. From each sample, 100 mg of oil was weighed into a 10 mL vial and placed in a rack on the autosampler.

After creating the sample list, each of the four samples are prepared on the system and injected into the GCs.

Table 1: Measurement parameters of the GCs for FAMEs analysis.

Injector					SSL, 1 µL injection volume, splitless (split 1:20 after 1 minute), carrier gas H ₂				
Temperature [°C]		Heating rate [°C/min]		Hold time [min]		Total [min]			
250		-		41		41			
Pressure regulation		Constant pressure, 150 kPa							
Separation column		60 m BPX 70, 250 µm ID, 0.25 µm film thickness							
Oven program									
Temperature [°C]		Heating rate [°C/min]		Hold time [min]		Total [min]			
145		-		6		6			
175		5		13		25			
225		5		6		41			
Detector		Temperature		Combustible gas		Air		Make-up gas	
FID		250 °C		H ₂ bei 30 mL/min		Synth. air at 300 mL/min		N ₂ at 25 mL/min	

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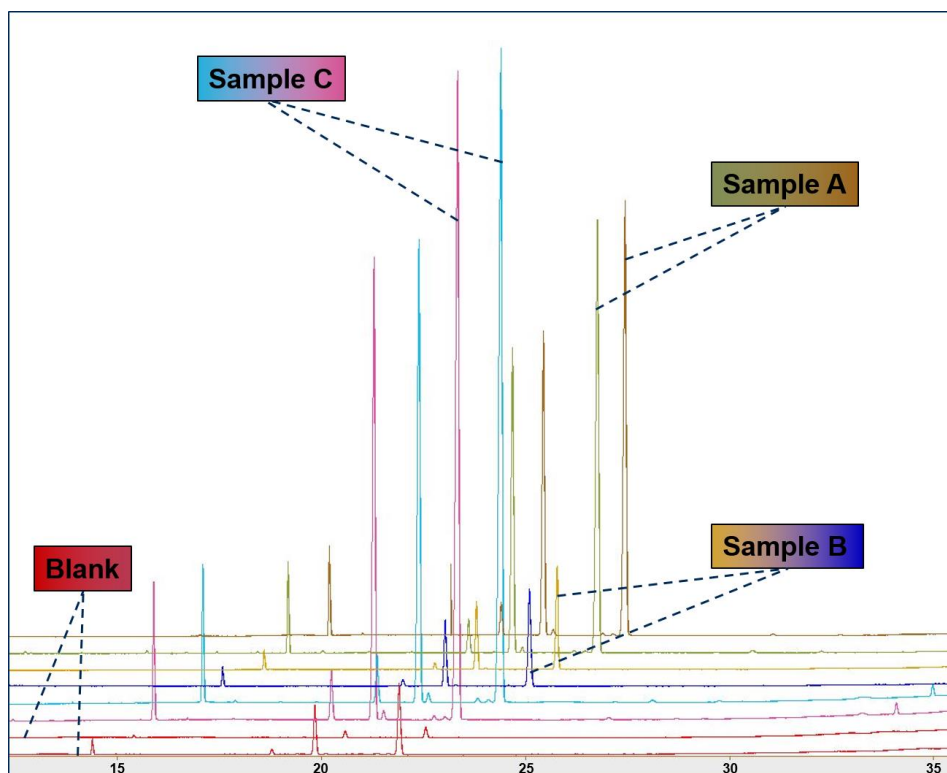


Figure 2: Exemplary chromatograms of a batchwise preparation and analysis of three samples and one blank in duplicate within 48 minutes.

Table 2: Results of a vegetable oil measured on two CHRONECT Workstation FAMEs High Throughput systems. A reference vegetable oil was analyzed on two systems with 4 channels each within 96 min each (including 48 min GC run time).

Reference		System 1				System 2				Statistics		
Vegetable oil		Channel 1	Channel 2	Channel 3	Channel 4	Channel 5	Channel 6	Channel 7	Channel 8	Average [%]	RSD [%]	Average WF [%]
Fatty acid		Area [%]	Area [%]	Area [%]	Area [%]	Area [%]	Area [%]	Area [%]	Area [%]	Area [%]	Area [%]	Area [%]
C12:0	6	6	6	6	6	6	6	6	6	6	2	101
C14:0	3	3	3	3	3	3	3	3	3	3	2	99
C16:0	34	34	34	35	34	34	34	34	34	34	1	100
C18:1	56	56	57	56	57	57	57	57	57	57	0	100
Area sum [%]	100	100	100	100	100	100	100	100	100			

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Evaluation of the results

An oil sample was prepared and analyzed on two systems a total of eight times, achieving a relative standard deviation for the four fatty acids considered (C12, C14, C16 and C18) of < 2.5 %. Using nominal values, additional recovery for the composition of the four fatty acids was found to be between 98 and 101%. Further analysis of reference samples showed similar results. With the CHRONECT workstation FAMES High Throughput it is possible to obtain robust values for the fatty acid composition in a routine laboratory within a short time. Using the CHRONOS software, it is also possible to choose between batch preparations of two, three or four samples, depending on sample requirements. A routine laboratory can thus manage a total of more than 200 samples per day using two CHRONECT workstations. The workstation is not tied to any GC manufacturer and can be adapted to a variety of different GCs.

The CHRONECT Workstation
FAMES High Throughput is a
development by Axel Semrau.

Subject to technical changes

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