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Application News

GC-MS GCMS-QP2020 NX

Fast Analysis of Fatty Acids in Brans by GC/MS

No. M306 A

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User Benefits

- Increased productivity with 10 mins run time for FAME 37
- EI/CI mode in the same batch to never misidentify a target FAME peak
- Constant linear velocity mode to easily convert the method to GC-FID

■ Introduction

Fatty acids, particularly those with a high degree of unsaturation and a carbon backbone of a middle chain length, are known as functional nutrients.

Shimadzu Corporation Japan has entered the second year of the three year collaborative research with National Agriculture and Food Research Organization (NARO) on the amount of functional nutrients (e.g. fatty acids) in foods such as brans, tea leaves and rice.

As a part of the collaboration, fatty acids (Fig. 2) were quantitated in 48 bran samples. Details of the results are off limits to the public at the moment of this writing.

However, the otherwise time-consuming run of the 48 samples was cut to 10 mins by optimizing a method without using a high heat ramp rate, which warranted this application news.

Fig. 1 GCMS-QP2020 NX

■ Methods - Extraction

Extraction was performed according to Fatty Acid Methylation Kit (Nakarai USA Inc., P/N MAK224-1KT) with the sample weight of 0.1 ±0.005 g.

■ Methods – Analysis

Table 1 Instrument Configurations

GC-MS	: GCMS-QP2020 NX
Auto Injector	: AOC™-20i Plus
Auto Sampler	: AOC-20s Plus
Analytical Column	: DB-FastFAME (20 m × 0.18 mm I.D., df=0.20 µm) P/N: G3903-63010
Glass Insert	: Split liner with wool

Table 2 Analytical Conditions

GC	
Inlet temp.	: 250 ˚C
Injection Mode	: Split
Spit ratio	: 10
Carrier gas	: Helium
Control Mode	: Constant linear velocity (53.4 cm/s)
Column oven temp.	: 60 ˚C (1 min) → (40 ˚C /min) → 200 ˚C (3 mins) → (25 ˚C /min) → 250 ˚C (1 min) Total 10.50 mins
Purge flow rate	: 5 mL/min
Sample Inj. volume	: 1 µL
MS	
Ion Source Temp.	: 230 ˚C
Interface temp.	: 250 ˚C
CI reagent gas	: Isobutane
Measurement Mode	: Simultaneous Scan/SIM (FAAST)
Scan mass range (*m/z*)	: 35-600 at scan speed of 20000 µ/s

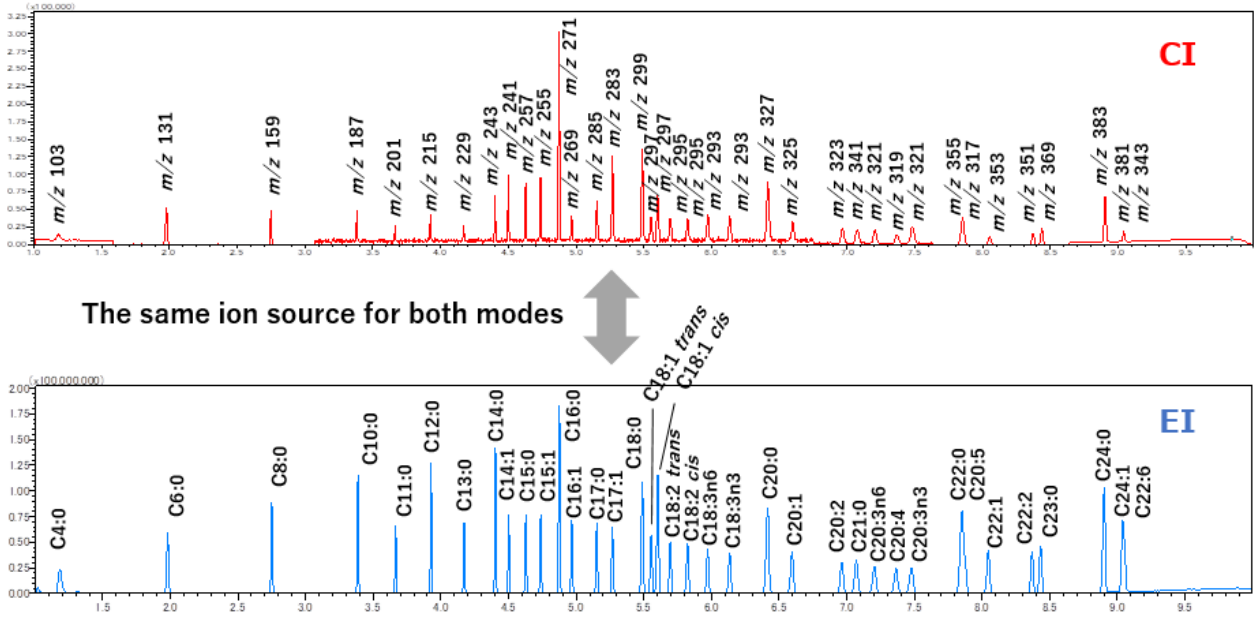


Fig. 2 CI and EI Chromatogram at 20 µg/mL with Smart Ion Source

■ Smart EI/CI Ion Source

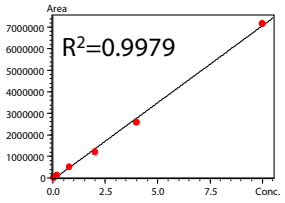
It is of paramount importance to have CI spectra for FAME analysis. CI spectra helps determine elution positions of FAMEs that might vary due to column lot difference and column conditions on the day of analysis.

Smart EI/CI ion source allows a seamless switching of EI and CI ionization modes within the same batch (Fig. 2). When the identity of a peak is questioned, the CI data from the same batch, as opposed to comments such as "retention time won't change" or "vendor catalog said this peak is this", will satisfy customers and auditors alike.

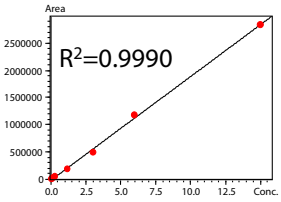
The *m/z* in Fig. 2 are quasi-molar masses for illustration and not necessarily those obtained.

■ Calibration Curve

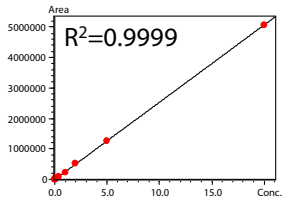
The calibration curves (2 µL injection) were obtained with EI ion source.



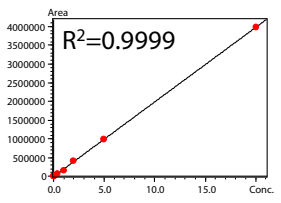
C4:0 (20 ng/mL to 10 µg/mL)



C16:0 (30 ng/mL to 60 µg/mL)

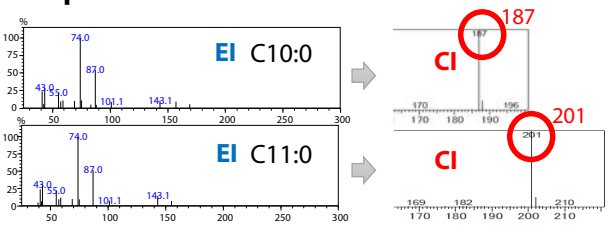


C18:3 (10 ng/mL to 20 µg/mL)



C20:4 (10 ng/mL to 20 µg/mL)

■ Importance of CI Mode



■ Summary

Fast analysis of fatty acids in brans were performed with Smart EI/CI ion source. CI data was acquired in the same batch without changing the ion source. It is of utmost importance to have CI data ready at hand to ensure the confidence in peak identity on the day of analysis, given the variability due to the column lot and column conditions.

Calibration curves for Fame 37 were all satisfactory with R^2 of greater than 0.990. 48 bran samples, the results of which are in non-disclosure agreement, were analyzed with the calibration curves.

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