

Background: Mineral oil hydrocarbons (MOH) were defined as the collective mixture of n -C₁₀– n -C₅₀ paraffins, naphthenic and aromatics hydrocarbons derived from petroleum and synthetic oils. Basically, this definition is determined by current analytical technologies, *i.e.* LC-GC and a series of sample pretreatment methods. Over the past decade, with growing concern over MOH contamination in food, the levels in various food products and food contact materials had been significantly reduced. A notable example is the evolution of detection standards for mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) in vegetable oils, which had updated from EN 16995:2017 to ISO 20122:2024, featuring a lower limit of quantification. Since MOH analysis is highly matrix-dependent, pretreatment methods varied among laboratories worldwide, even when equipped with LC-GC instruments. For instance, defatting was performed using either silica gel column adsorption or saponification; alumina purification may be manual or automated; and the most complex epoxidation methods included more than five variants, such as *m*-CPBA and PFA methods, which differed in reaction solvent systems. Additionally, the MOSH & MOAH chromatographic profile could be distorted by any interfering compounds within the n -C₁₀– n -C₅₀ boiling range, compromising the accurate delineation of the chromatographic contour. Therefore, ensuring consistency in analytical results among laboratories is of critical importance. This poster shared several measures commonly adopted in our laboratory to guarantee result consistency.

Aim: Although the current methods for detecting MOSH and MOAH in various foods are not yet optimal, ensuring comparability and consistency of analytical results among different laboratories is of great importance. This facilitates accurate decision-making by regulatory authorities, enables business clients to smoothly conduct trade and trace contamination sources, and supports continuous product quality improvement.

Methods

Ensuring the LC-GC instrument's performance and baseline stability: The LC column must effectively separate MOSH and MOAH, and the retention gap and GC columns must function optimally. Maintain the purity of the carrier gas and ensure the FID detector operates effectively. Given the high concentration factor required for the solvent, it's essential to use pure solvents and reagents to prevent any interferences from solvent impurities, thus maintaining a stable baseline. Any abnormal baseline uplift or fluctuation must be avoided as it can directly affect the method's quantitative detection limit.

Ensuring the accuracy of MOSH/MOAH and RT mix standard materials and QC samples: Accurately maintain the concentrations of MOSH/MOAH standard solutions (comprising 9 or 10 analytes) and RT mix used for quality monitoring. The MOSH and MOAH concentrations in QC samples, such as Gravex 913 and SN500, are lot-dependent; therefore, periodic re-certification against primary standards is recommended to prevent drift.

Ensuring the necessity and accuracy of the purification methods: The saponification process must prevent emulsification, and it's essential to remove polar substances from *n*-hexane phase when required. For MOH in water-alcohol solutions, a second extraction with *n*-hexane is usually necessary. Monitor the recoveries of the 9/10 standard substances in the MOSH/MOAH standard solution, particularly the quantitative internal standard, as its accuracy is crucial for result consistency. The Alox purification method requires careful consideration to balance the instrument's sensitivity to natural hydrocarbons in the sample matrix without underestimating MOSH results. In the epoxidation method, nearly all samples require epoxidation. To ensure that residual polar substances from epoxidation do not compromise instrument performance, all such substances must be removed before analysis. For certain flavoring samples, like garlic oil, which had extraordinarily high olefin contents, as it was necessary to improve the epoxidation method since conventional epoxidation methods were unable to remove the olefins in the sample. In order to achieve sufficient epoxidation, the sampling amount could have to be reduced, but resulting in the higher LOQ value. Increasing the amounts of *m*-CPBA or PFA used while avoiding the risk of explosion that may be caused by vigorous reactions (Fig 1). In addition, there were significant differences in the MOAH contents when different epoxidation methods are used (Fig 2).

Preliminary experiments are crucial: since the interference from unconventional sample matrices is unknown, pre-experiments are essential to establish the appropriate purification methods. Additionally, for samples with unusually high or low concentrations of target substances, pre-experiments are necessary to determine the optimal amount of internal standard to add and the suitable injection volume for the LC-GC instrument.

For special sample matrices, it's essential to use "classical" offline methods to individually prepare MOSH and MOAH before analyzing them on the instrument.

Regular participation in proficiency testing and inter-laboratory comparisons is essential to ensure that the analysis results are comparable with those of peers. Fig 3 presented an olive oil sample that is representative of MOSH & MOAH contamination. This sample was utilized for an inter-laboratory comparison of analysis results in 2024, involving 10 laboratories in mainland China those all had the capability to detect MOH in food and FCM.

Conclusion: Due to the different matrix interferences that interfere with MOSH & MOAH analysis in foods, detecting low levels of MOH poses a great challenge to the laboratory. Proficient in mastering the evolution of the sample pretreatment methods, and exploring the advantages and disadvantages of different methods, reaching consensus through result comparison to ensure the consistency of results from different laboratories.

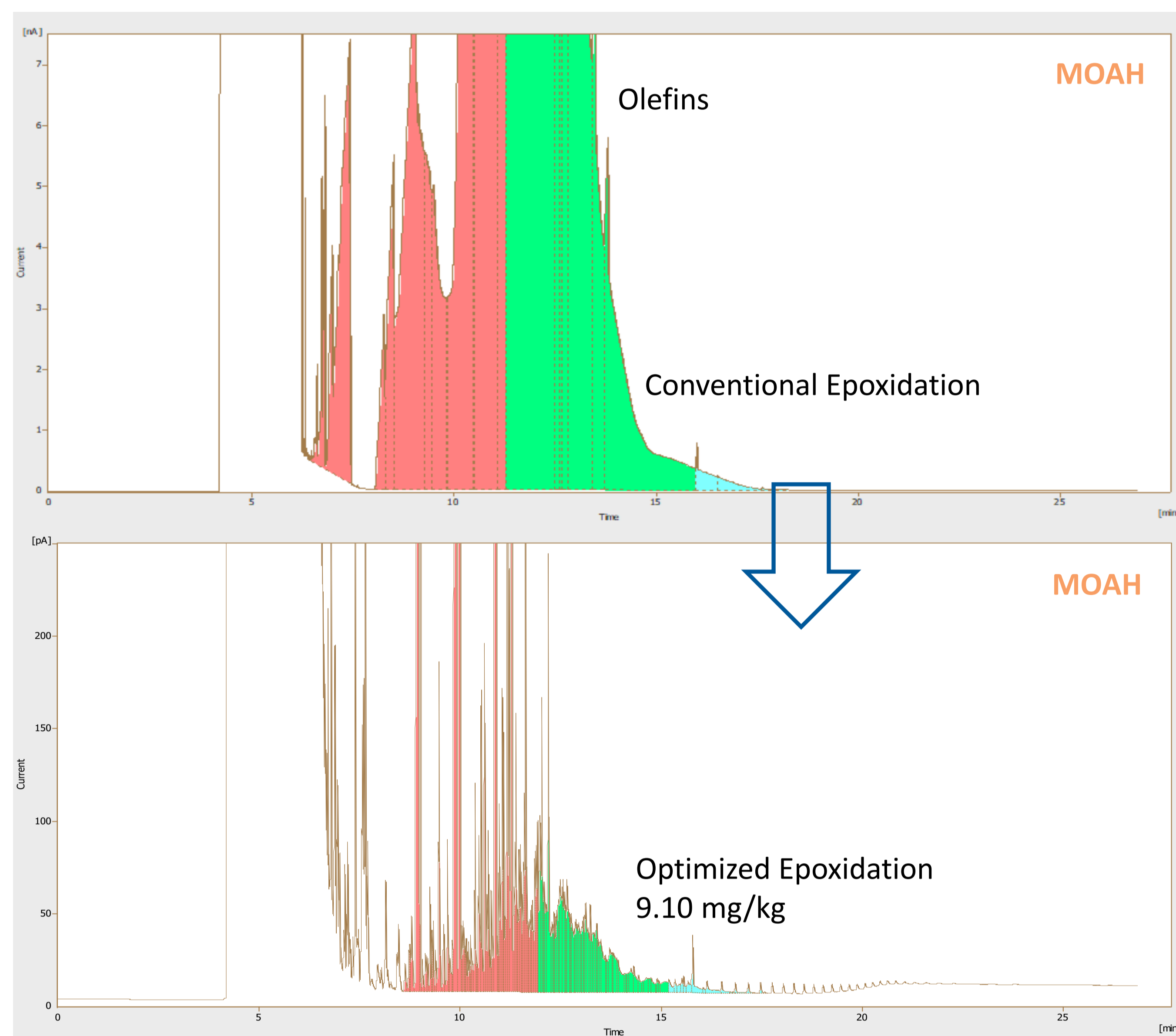


Fig 1 The MOAH chromatograms of a garlic oil before and after epoxidation optimization

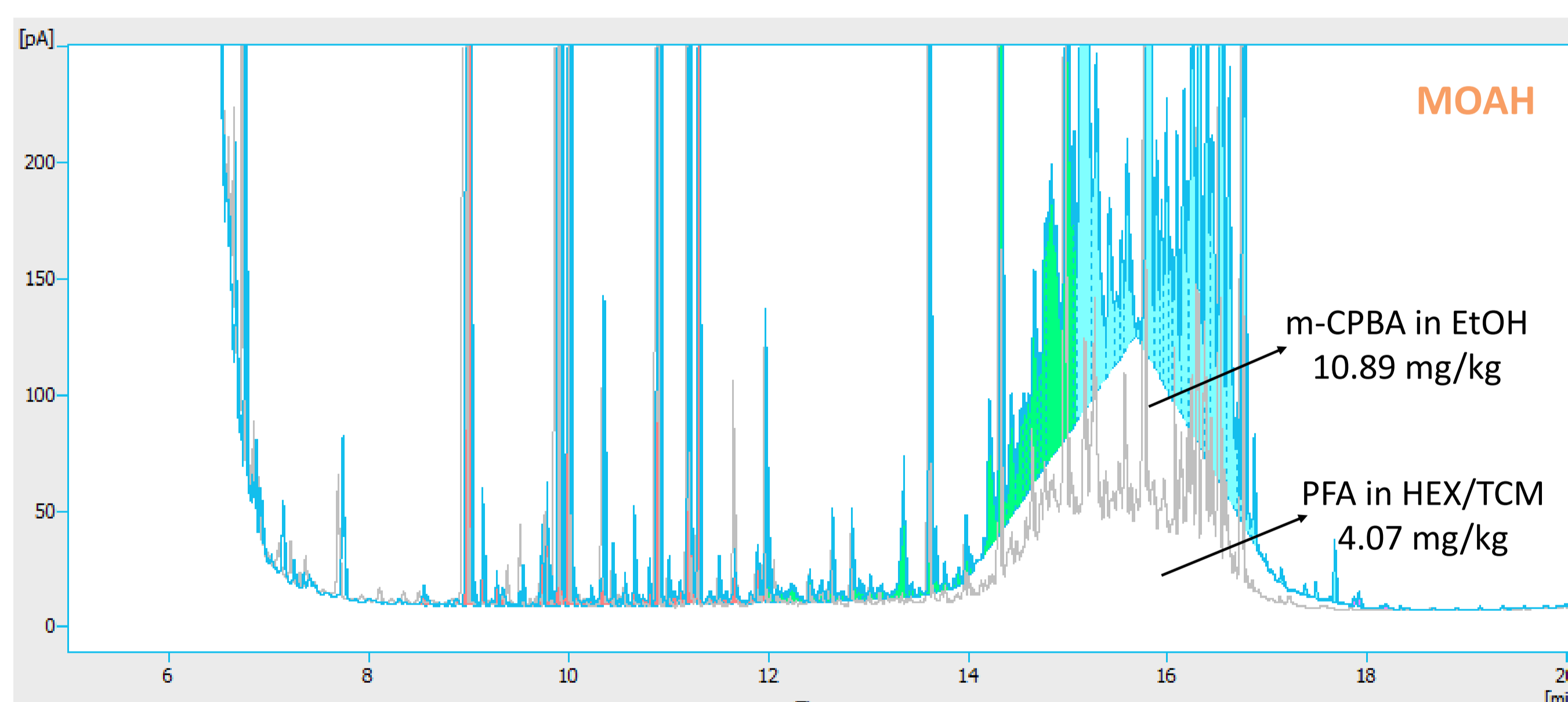


Fig 2 The chromatograms of MOAH in a GMO sample with different epoxidation methods

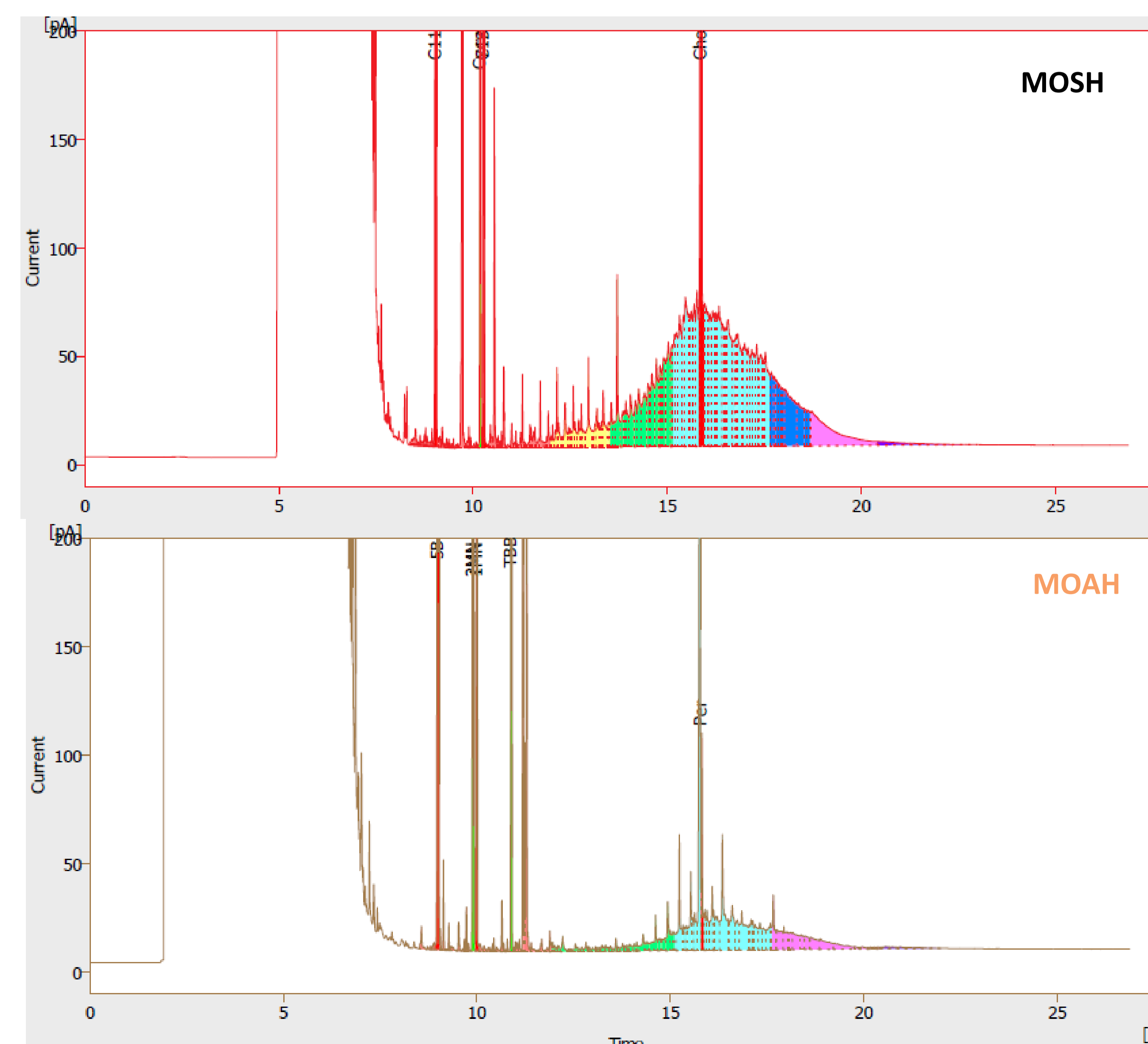


Fig 3 The chromatograms of MOSH & MOAH in the olive oil for interlab comparison