Axel Semrau®

LC-GC System Sterols

Product Information

Sterol analytics

Determination of sterols is required for example during quality control of oils. An essential parameter for the purity and quality of oils is the composition of the incorporated sterols. The common analytical procedure for a determination of the total sterol content and the percentage distribution of different sterols in fats and oils is time-consuming and requires numerous manual sample preparation steps.

According to the ISO 12228 procedure, the sample is saponified and then purified using solid phase extraction. After neutralization and concentration, the remaining analytes are further purified by use of a preparative thin layer chromatography. Subsequently, a manual removal of the fraction from the TLC plate is required. The gained fraction is derivatized and finally chromatographically analyzed using GC-FID. Consequently, the expenditure of time and error rate are quite high due to the manual steps.

The method for the determination of sterols in oils described here is based on the LC-GC technology by Axel Semrau®, which was successfully applied already for analytics of MOSH/MOAH in foodstuff and packaging. It is in routine application in numerous laboratories. Saponification and other preparation steps are performed fully automated by use of a RTC PAL autosampler with independent syringes. In the process, one syringe dispenses the reagents required for saponification and another, smaller syringe performs the injection into the HPLC device. Only the concept of the PAL sampler which uses numerous different syringes allows for a full automation. The subsequent purification is run by HPLC.

A 700 µL HPLC fraction which contains the sterols is precisely transferred to the gas chromatographic system and detected via FID. All disturbing components which are usually separated by TLC are elegantly separated by LC here and therefore are kept away from the GC.

Manual interventions as for example concentration are not necessary which avoids potential contamination. The only manual step is the weighing of the sample and placing it into the autosampler. After approximately two hours the final results are available.

Extensive testing in customer's laboratories using various samples and comparing them to those with known sterol composition verify, that the here described method is accurate and provides reproducible results. Analytical parameters of the procedure all are equally precise as the ISO method or even outperform it.

The entire control is performed by the user-friendly software CHRONOS. Therefore, even complex methods are simple to handle. The LC-GC solutions by Axel Semrau® are pre-installed in application laboratories, tested and delivered to the customer ready-to-use. Thus, the quickest possible continuation of the routine measurement is assured.

Benefits of the sterol system

- high sample throughput
- high degree of automation
- no risk of contamination
- excellent reproducibility
- best possible sensitivity

- expandable to other applications, e.g. determination of mineral oils, determination of alkyl esters or stigma studies
- investment safety
- short training period due to installation of the ready-to-use method and introduction to the system
- qualified support

System components

- Agilent 1260 HPLC pump with UV detector and degasser (alternatively Knauer Azura HPLC system or Shimadzu LC 20)
- RTC PAL autosampler for automated saponification and purification
- Agilent 7890B with FID
- CHRONECT LC-GC Interface to couple the HPLC with the GC
- data system including controlling and evaluation software
- accessories and consumables

The following figures present some examples of chromatograms from the application. The LC-GC coupling supplies two chromatograms at the same time:

- signal of the UV detector from the HPLC
- FID signal of the sterols

It is evident, that this separation method allows for a distinction between Δ5 and Δ7 sterols which can hardly be achieved by the manual method. After analysis, the HPLC column is backflushed and reconditioned. This is performed in parallel to the GC run and therefore assures highest sample throughput and stable initial conditions.

This contamination can easily be seen within the sterol distribution. The amounts of brassicasterol and campesterol in the blended oil (red) are significantly higher than in pure olive oil (blue) (cf. Fig. 4).

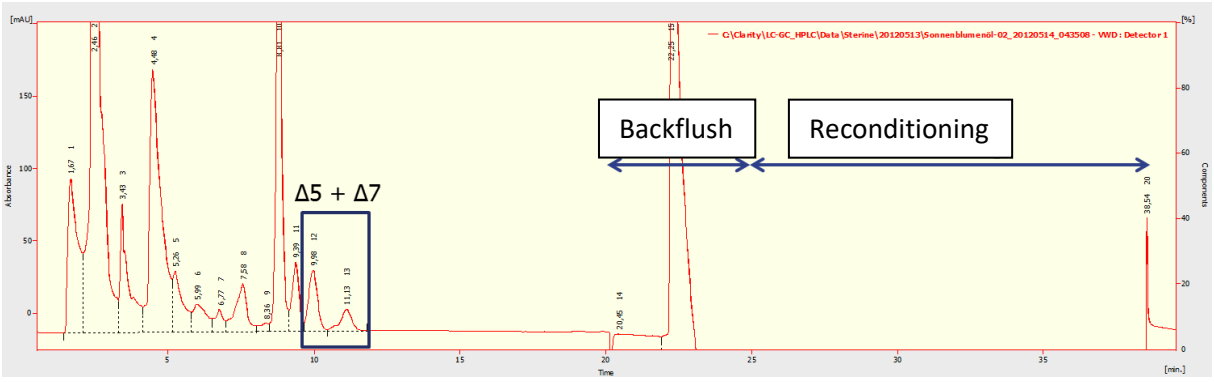

Figure 1: HPLC chromatogram.

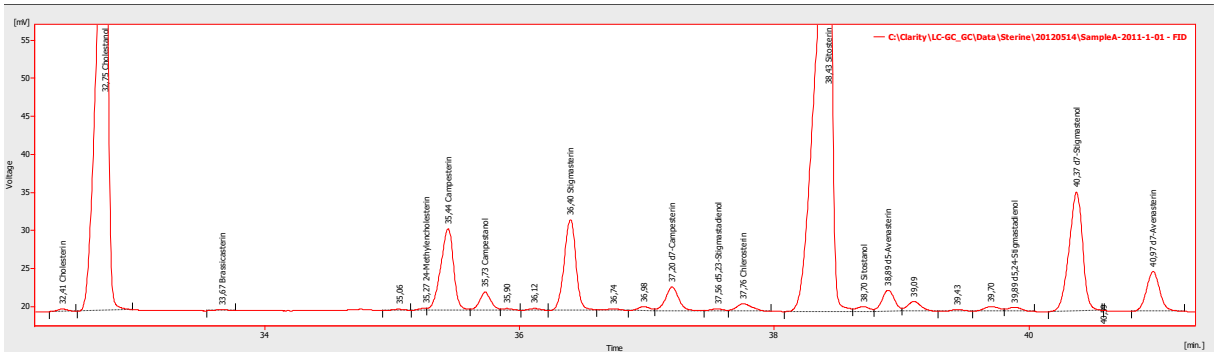

Figure 2: GC chromatogram of a sunflower oil.

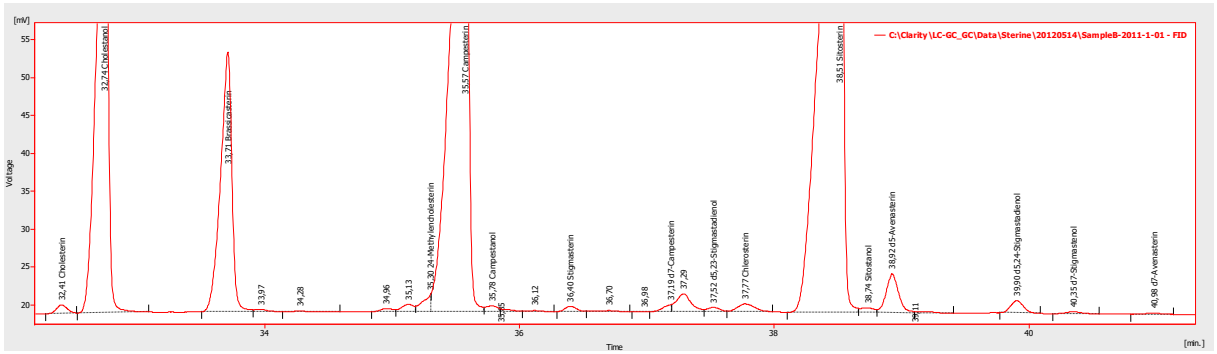

Figure 3: GC chromatogram of a rapeseed oil.

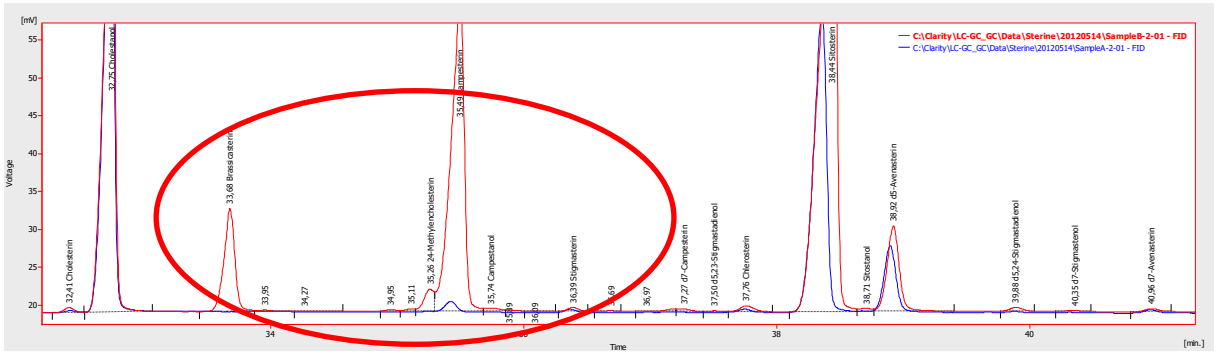

Figure 4: Chromatogram of an olive oil blended with rapeseed oil.

Subject to technical changes

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This LC-GC solution is a development by Axel Semrau ®