CHRONECT Workstation Sterols

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Sterol analytics

The determination of sterols is part of the quality control of oils. An essential parameter for the purity and quality of oils is the composition of the contained sterols. The common analytical method for determining the total sterol content as well as the percentage distribution of different sterols in fats and oils is time-consuming and requires numerous manual sample preparation steps. According to the procedure described in ISO 12228, the sample is first saponified and then purified by solid phase extraction. After neutralization and concentration, the analytes are further purified by preparative thin-layer chromatography. Afterwards, the fraction must be removed manually from the DC plate. The manually obtained fraction is then analyzed chromatographically after derivatization by GC-FID. The many manual steps involved in this method make it very time-consuming and error-prone

The method described here for the determination of sterols in oils is based on the introduced LC-GC technology by Axel Semrau, which is already in routine use for the analysis of MOSH/MOAH in food and packaging in many laboratories. Sample saponification as well as other preparations are performed fully automatically with a CHRONECT Robotic Autosampler. A syringe is used for dosing the reagents necessary for saponification. Another smaller syringe carries out the injection into the HPLC. Only the concept of the CHRONECT Robotic with several different syringes allows a complete automation. The subsequent purification is performed by HPLC. The 690 µL HPLC fraction containing the sterols is transferred directly into the gas chromatographic system and detected by FID. All interfering components, which are otherwise separated by thin layer chromatography, can be elegantly removed by the LC and kept away from the GC.

Manual intervention, such as concentration, is not necessary, thus eliminating potential points of contamination. The only manual step is to weigh the samples and transfer them to the autosampler. After about two hours, the result is available.

By comparing numerous samples with known sterol content and distribution, it was verified in customer laboratories that the presented method is precise and produces reproducible results. The analytical characteristics of the method are equal or superior to those of the ISO method in all points.

The entire control of the system is done in an extremely user-friendly way by the CHRONOS software. This makes even complex procedures easy to use. The LC-GC-solutions of Axel Semrau are pre-installed in the application laboratory, tested and installed directly ready for use at the user. These Factory and Site Acceptance Tests ensure the fastest possible start of the routine measuring operation.

Benefits of the CHRONECT Workstation Sterols

- high sample throughput
- high degree of automation
- low risk of contamination
- excellent reproducibility
- best possible sensitivity
- expandable to further applications e.g. determination of mineral oils, determination of alkyl esters or stigmastadiene
- Investment security
- short familiarization time by installation of the finished method and training
- qualified support

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System components

- Agilent 1260 Infinity II HPLC pump with UV detector and degasser (alternatively Shimadzu LC-40)
- CHRONECT Robotic RTC for automatic saponification and purification
- Agilent 8890 with FID (alternatively Shimadzu GC-2030)
- CHRONECT LC-GC-Interface for coupling HPLC with GC
- Data system with control and evaluation software
- Accessories and consumables

The following figures show sample chromatograms. The LC-GC coupling delivers two chromatograms simultaneously:

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

It is to be recognized that with this procedure a separation of Δ5- and Δ7-sterols takes place, which is hardly possible with the manual method. After the analysis the HPLC column is backflushed and reconditioned, this is done parallel to the GC run and ensures highest sample throughput and stable initial conditions.

This contamination can be easily detected by the sterol distribution. It can be seen very clearly that the proportions of brassicasterol and campesterol in the blended oil (red) are considerably higher than in pure olive oil (blue) (see Fig. 4).

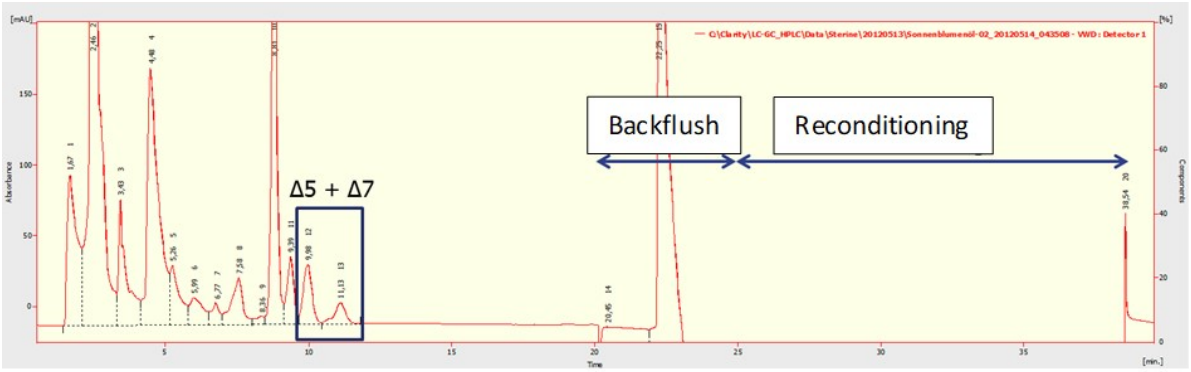

Figure 1: HPLC chromatogram.

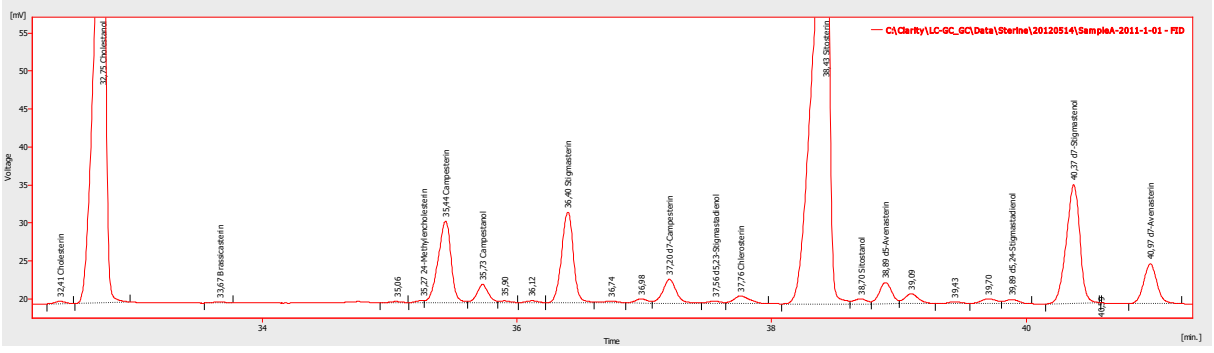

Figure 2: GC chromatogram of a sunflower oil.

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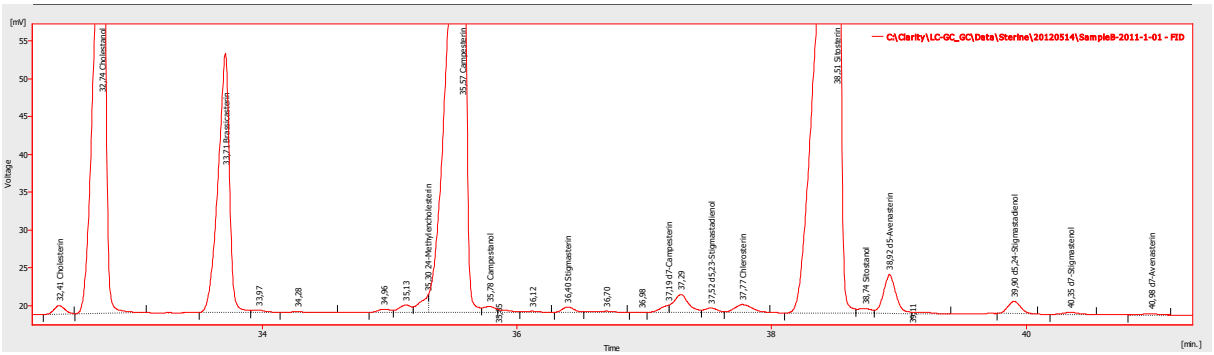

Figure 3: GC chromatogram of a rape seed oil.

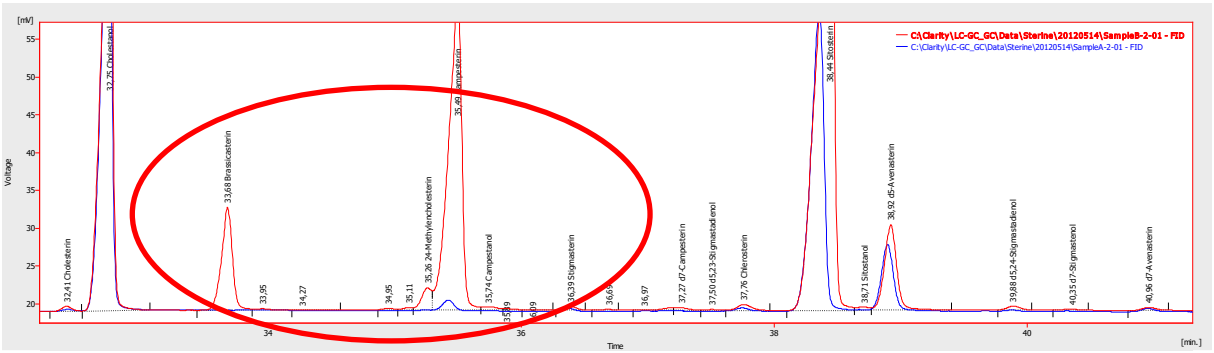

Figure 4: Chromatogram of an olive oil blended with rape seed oil.

The CHRONECT Workstation Sterols is a development by Axel Semrau.

Subject to technical changes

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