

CHRONECT Workstation Cholesterol



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Introduction

The determination of cholesterol is routinely performed in egg-containing foods. The aim is to determine the amount of the ingredient whole egg or egg yolk, as these are valuable components. This determination according to the Paragraph 64 method involves time-consuming, manual steps. It requires many solvents and sometimes derivatization reagents. The CHRONECT Workstation Cholesterol facilitates the analysis by a fully automated analysis approach, starting from the ground sample. The manual effort is dramatically reduced; the solvent consumption is also significantly lower. Turnaround time and analysis quality are improved in this way and the consumption of resources is also reduced.

System setup

Basis of the CHRONECT Workstation Cholesterol is the LC-GC technology of Axel Semrau in combination with an automated sample preparation. The LC-GC technology connects an HPLC via a special interface with a GC equipped with a FID. The coupling is done via an interface consisting of a control unit and a heatable valve unit.

The temperature of the valve unit can be varied from room temperature to 150 °C. The temperature control effectively prevents condensation of the HPLC solvent in the valve unit and improves the stability of the system significantly. An integrated flushing of the valve unit with carrier gas prevents tailing of the solvent and possible carryover.

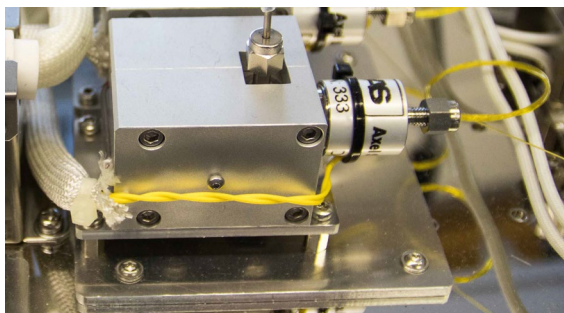


Figure 1: Heatable valve unit.



Figure 2: Control unit.

The HPLC system consists of a binary pump and a UV detector, which is used to check the HPLC chromatogram and to verify the correct fractionation. HPLC is used to separate cholesterol from matrix components. A fraction of the HPLC containing the cholesterol is then transferred from the LC directly to the GC. The measurement of cholesterol is performed as in the official method with a FID.

The control of the whole system is done user-friendly by the software CHRONOS by Axel Semrau. The XYZ robot CHRONECT Robotic integrated in the system takes over the sample preparation steps, which are based on the manual procedure of the Paragraph 64 method. The sample is saponified automatically. After neutralization an automated liquid-liquid extraction is performed. This is followed by automated drying using Na₂SO₄. Afterwards the again diluted extract is injected into the LC-GC-FID system. After the analysis, the HPLC is backwashed. This removes any remaining matrix and significantly improves the operational reliability and lifetime of the chromatographic columns.

If samples containing starch are analyzed, the described automatic procedure is extended by an enzymatic starch hydrolysis; this is also part of the Paragraph 64 method.

The use of LC-GC technology allows the elimination of a derivatization step. The obtained peak form of the cholesterol is very good even without derivatization.

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Comparison with the reference method

The determination of the egg content with the CHRONECT Workstation Cholesterol is largely based on the specifications of the official method. The samples are saponified, extracted and starch-containing samples are subjected to enzymatic starch hydrolysis.

Nevertheless, processes had to be changed and adapted for an automated approach. In an extensive comparison of the automated method with the official approach, good matches of the results were found for different matrices.

Compared to the reference method, the consumption of solvents is approximately halved. With the CHRONECT Workstation Cholesterol up to 18 samples can be measured within 24 hours. The time required to prepare these samples is about one hour.

Table 1: Comparison with reference methods.

[mg/100 g]	Procedure acc. to § 64 LFGB	Fully automated LC-GC method	ACTUAL/SET [%]
Sponge cake	171	162	95
Egg pasta	49	45	92
Swabian tagliatelle	107	100	93
Pyramid cake (Crumb)	238	236	99
Butter cookie	46	49	107
Spätzle	81	83	102
Salad mayonnaise	38	40	105
Mayonnaise	59	63	107
Egg liqueur	247	255	103

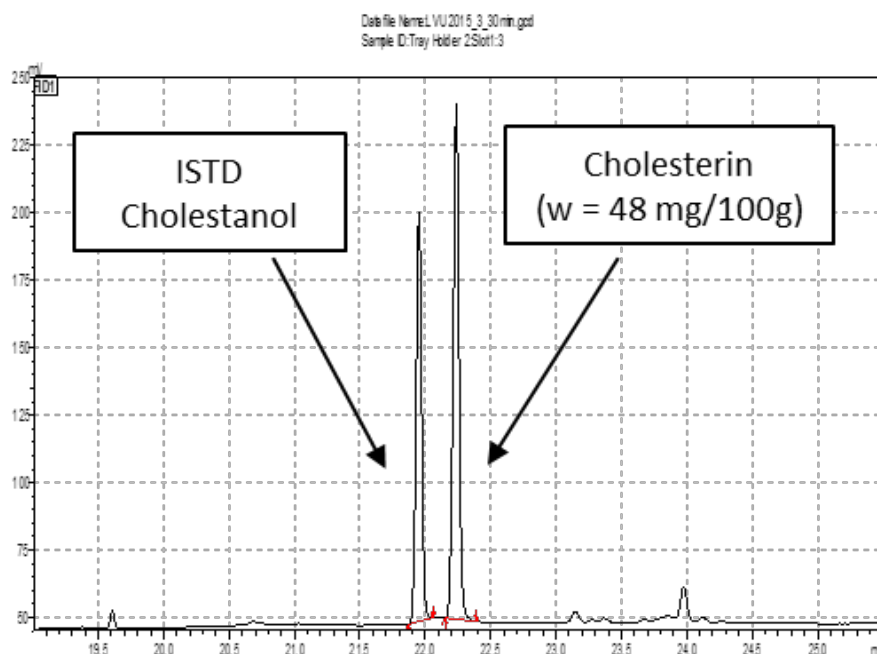


Figure 3: Chromatogram of a butter cookie sample. Cholesterin = Cholesterol.

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Commissioning

CHRONECT Workstations are commissioned in advance to ensure that the systems function properly. In the course of a comprehensive Factory Acceptance Test not only the correct technical function but also the analytical performance is checked. This test run is repeated after the installation in a Site Acceptance Test during installation in the customer's laboratory. In this way the analytical accuracy is verified. The system is ready for use immediately after installation.

Summary

The CHRONECT Workstation Cholesterol allows a fully automatic determination of the egg content in foodstuff and provides comparable results to the reference method. Up to 18 samples can be measured within 24 hours, thus reducing the turnaround time and significantly increasing the sample throughput. Due to the FAT/SAT concept, the CHRONECT Workstation Cholesterol is immediately ready for use and therefore an ideal system for laboratories that determine egg content in food.

The CHRONECT Workstation Cholesterol is a common development by Landeslabor Berlin-Brandenburg and Axel Semrau.

Subject to technical changes

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