

Sample Preparation for HPLC Analysis of Confectionery

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Like all foodstuff, confectionery is subjected to strict quality controls. Parameters of interest are, for example, nutritional value, moisture or fat content, or the quantification of particular ingredients, such as vitamins or alkaloids. Typically, chromatographic methods like High Performance Liquid Chromatography (HPLC) are used to analyse food samples. Most analytical methods only require a few milligram or gram of sample; the previous size reduction/homogenisation process ensures that the small analysis sample is representative of the entire laboratory sample, thus allowing for reproducible results. Moreover, homogenised samples show a much better extraction behaviour.

Confectionery occurs in very different textures: it can be hard, sticky, greasy, or moist and is frequently inhomogeneous. For the important step of sample preparation, laboratory mills such as offered by Retsch in a variety of designs, are indispensable tools. With a suitable mill and the corresponding accessories all types of samples can be easily and reproducibly homogenised. Generally, the sample preparation process needs to be adapted to the sample characteristics as well as to the requirements of the subsequent analysis to avoid falsified results.

When selecting grinding tools and parameters it should be taken into account that the characteristics of the sample must not be altered by the grinding process. Different analysis methods call for different ways of sample preparation. For HPLC analysis a particle size distribution between 0.5 and 0.75 mm is ideal.

The Cologne-based institute IQ.Köln (Institute for Promotion of Quality in the Confectionery Industry) is specialised in the analysis of confectionery. The institute is accredited according to DIN EN ISO 17025 for food testing using methods such as HPLC with UV/VIS, fluorescence and tandem-MS detection and also gas chromatography with flame ionisation and MS detection.

Homogenisation of Hard Candy and Toffee

The institute uses Retsch's knife mill GRINDOMIX GM 200 to homogenise hard candy and toffee. The cutting effect of the sharp knife blades allows for thorough grinding of hard as well as very sticky sweets. The GM 200 produces representative samples within seconds. Features such as variable speed, a comprehensive selection of grinding containers and lids and easy cleaning of the grinding tools make this mill a versatile tool in the food lab.

A typical homogenisation process involves 100 g of hard candy which is first roughly ground for a few seconds in reverse mode with the blunt side of the knife. The following step involves operation in interval mode for another 15 seconds with 4,000 rpm. Further pulverisation to a size below 0.5 mm is achieved by grinding for 6 to 12 seconds at 6,000 rpm. This step by step procedure prevents the sample – which has a high sugar and starch syrup content - from sticking to the knife as is often the case in household mixers. Just

like the brittle hard candy the tough-elastic toffees are deep-frozen together with the steel grinding container before they are submitted to size reduction. The lab team of IQ.Köln is highly satisfied with the performance of the GM 200 when grinding these difficult samples. All parts of the mill which come into contact with the sample material (container, lid, and knife) are conveniently cleaned in the dishwasher.

Figure 1. Knife Mill GRINDOMIX GM 200



Analysis of Vitamin C

Hard candy is often enriched with vitamin C which can be quantified by HPLC. Ascorbic acid is also frequently used as a preservative. The ground candy is dissolved in water; the vitamin C is stabilised with the help of metaphosphoric acid; after membrane filtration the sample solution is analysed by reversed-phase HPLC and subsequent UV detection. The standard solvent is phosphate buffer with a flow rate of 1 ml/min; under these conditions the vitamin C elutes after 3.7 minutes.

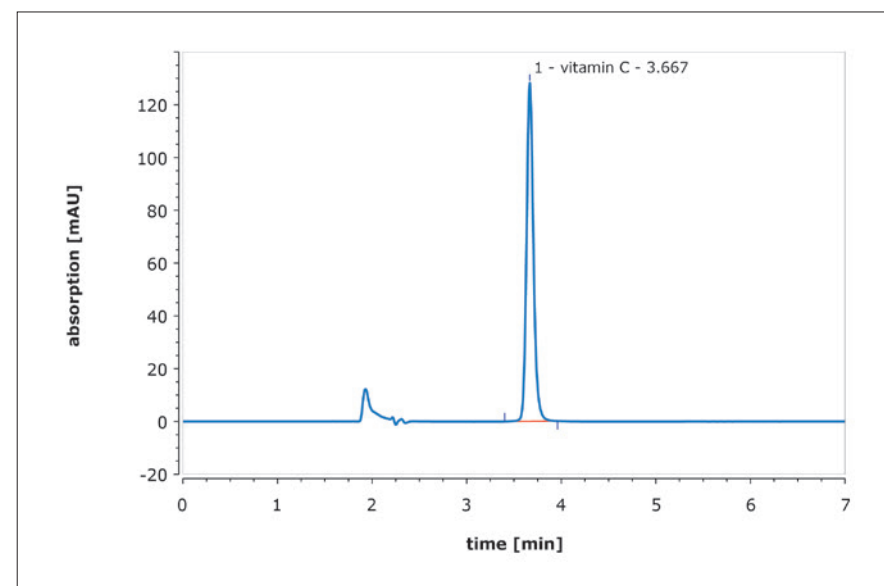


Figure 2. Chromatogram of vitamin C analysis with reversed-phase HPLC

Fatty, Granular Food Samples such as Cocoa Beans

The best combination of size reduction mechanisms for medium-hard, granular food stuff like grain or cocoa beans is impact and shearing. The cocoa bean bursts open through impact, and the smaller parts are then further reduced in size by shearing. The Ultra Centrifugal Mill ZM 200 is ideally suited for this job; this rotor mill grinds the sample between rotor and fixed ring sieve by impact and shearing. The material is fed to a hopper, falls onto the rotor and is thrown outward by centrifugal acceleration. When the sample hits the wedge-shaped rotor teeth running at high speed, it is precrushed and then pulverised between rotor and ring sieve. The sample only remains in the grinding chamber for a very short interval so that the characteristic properties are not altered.

The lab team at IQ.Köln uses the ZM 200 to grind 100 g of cocoa beans with a 1.5 mm distance sieve at 18,000 rpm for 10 seconds and obtains a mean particle size below 0.75 mm. With a distance sieve there is more space between rotor and sieve which helps to reduce frictional heat and prevents the apertures from being blocked by fatty particles. The pulverised sample is then collected in a cassette. This is part of a patented principle where the cassette containing the sample is removed together with the ring sieve, thus ensuring sample recovery without loss and prevention of cross contaminations. For small amounts up to 20 ml Retsch offers a mini cassette with matching rotor and ring sieves.



Figure 3. Cocoa beans before and after grinding in the ZM 200

Alkaloid Analysis

The alkaloids contained in cocoa beans and cocoa nibs, which are mainly theobromine and caffeine, are also quantified by HPLC. Cocoa beans contain up to 0.2% caffeine and 1 to 2.5% theobromine. The invigorating effect of theobromine on the human organism is much weaker than that of caffeine; however, it has a positive effect on the mood.

The ground beans are first cooked in water and then clarified. After centrifugation and membrane filtration the supernatant is analysed by reversed-phase HPLC and subsequent UV detection. A water/ethanol mixture (80%/20%) is used as standard solvent at a flow rate of 1 ml/min. Under these conditions theobromine elutes after 3.5 minutes and caffeine after 9.1 minutes.

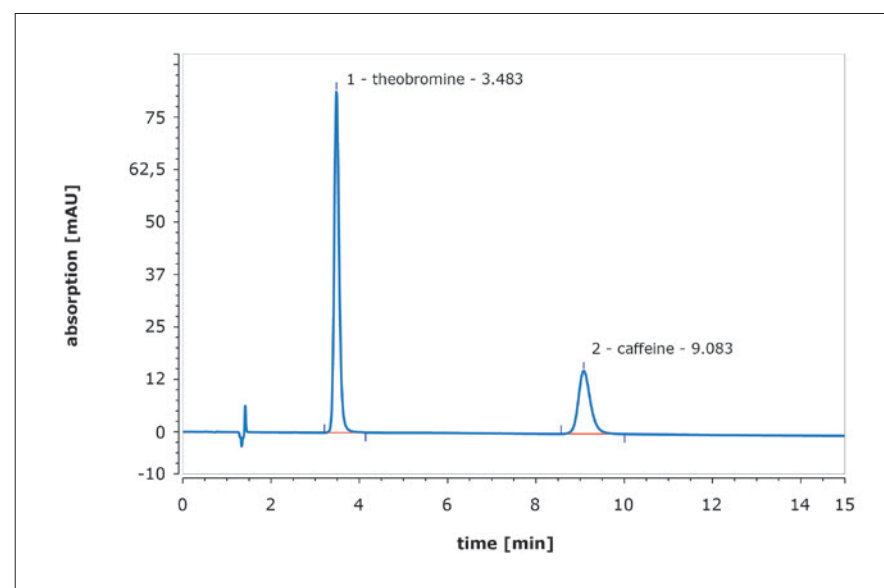


Figure 4. Chromatogram of alkaloid analysis by reversed-phase HPLC

Conclusion

Retsch offers rotor and knife mills which are perfectly suited to grind all types of food quickly, reproducibly and without interfering with subsequent analysis. A wide selection of accessories allows for flexible adjustment to material properties and sample volumes. Tough-elastic samples or those containing volatile components are best processed by cryogenic grinding, which means embrittling the sample material with dry ice or liquid nitrogen. The use of Retsch mills and grinders greatly improves the efficiency of a food laboratory, particularly if a variety of sample types needs to be processed on a daily basis.



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Centrifugal Evaporation Aids Quest for Culinary Perfection

At the renowned Midsummer House Restaurant in Cambridge, UK, the chefs have been experimenting with centrifugal evaporation, using the **Genevac** Rocket evaporator.

Using the centrifugal evaporation technique, Midsummer House chefs are taking large volumes of liquid and concentrating them down. These concentrates can be used to increase the depth of flavour of sauces or stocks or to create unique flavour combinations

At Midsummer House, Head Chef Daniel Clifford's cooking has a modern-focus which is underpinned by classical French technique. It is increasingly common to find scientific equipment in the kitchens of Michelin-starred restaurants: liquid nitrogen, water baths, dehydration and freeze dryers enable modern chefs to experiment with taste and texture in ways that were previously impossible. But while technological theatre can make a meal into a show-stopper, the success of every dish ultimately depends on its flavours.

"We are still learning with the Rocket Evaporator and testing lots of products on it," commented Chef Clifford. "It really is so much fun for our chefs to taste the clarity of ingredients and see how pure we can make the flavours."

At Midsummer House the centrifugal evaporation technique is already used with a wide range of components, including a quail consommé (for ravioli), celery juice (incorporated into sorbet) and crab stock (to be made into crab butter). Centrifugal evaporation does not need high temperatures to work so in contrast to other methods, for example reducing a sauce down on the stove, the flavours are not affected. This is especially important with the subtle flavours like cucumber or beetroot.

But just as the Midsummer House is at the cutting edge of gastronomy, the Genevac Rocket has redefined centrifugal evaporation. Most evaporators are designed for use with small amounts of liquid, often just a few centilitres. Making enough concentrate for a single serving of sauce might take days. The Rocket, however, is able to rapidly transform litres of liquid into millilitres of concentrate. By using an innovative low-temperature steam system this can be achieved in just a few hours.

Chef Daniel Clifford concluded: "We are always striving to better our techniques to offer the best food in the industry. There is no other evaporator that can achieve these goals. The Genevac Rocket is unique."

For further information on the Rocket Evaporator please visit www.genevac.com/movie/rockets/

For More Info, email: 32676pr@reply-direct.com



Food & Beverage Analysis

Food Tracker for Cook and Chill Processes



Food Tracker systems from **Datapaq** ensure highly efficient process monitoring and HACCP documentation at the click of a key. In addition to complete temperature profiles and extensive alarm and reporting functions, they offer multiple simultaneous food lethality calculations (F0/Pu value, D-value, chill rate Critical Control Point) and an optimisation function that predicts the effect of parameter changes on the process without repeated trial runs. A state-of-the-art 6 or 8-channel data logger from the MultiPac21 series guarantees high accuracy, high resolution, and large memory without data loss. Designed for efficient operation, it manages eight consecutive measurement runs before data download to a computer. A start/stop button and an alternative time or temperature trigger provide true ease of use.

The logger features a serial and a USB interface. For even more direct access and speedier process set-up even from the control level, users can choose additional data transmission via radio as well as real-time data export via OPC ("Open Platform Communications"). The recommended thermocouples are type K and type T which are specially designed to meet the stringent requirements of food processors. The logger has an IP67 waterproof rating and an operating temperature range of -40 to 85°C. For even harsher environments, the Food Tracker program includes a range of thermal barriers for all kinds of applications from freezer to fryer.

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Fast, Accurate Diagnostic Tests for Safe Food and Water Now Available from UK Distributor



From detecting antibiotic residues, allergens or pathogens in food, to uncovering milk and cheese fraud, Zeulab, are leaders in the production of tests that can do all this and more. Their leading product range is now distributed in the UK by **Cambio**.

The detection of antibiotics in milk products is compulsory across the EU and levels must comply with the Maximum Residue Limits (MRL) set by the EU. Zeulab's microbial growth inhibition test, Eclipse, is a simple, ready-to-use test that detects antibiotic residues in milk with a sensitivity that complies with EU-MRLs. It's a one-step test that takes just 2½ hours and works for cow, sheep, goat or buffalo milk.

Similarly, if cow's milk is being used to replace more expensive buffalo milk in mozzarella cheese, Zeulab's IC-BOVINO offers a rapid dip-stick test to detect the presence of cow's milk that would rule out a mozzarella cheese's valuable Protected Designation of Origin (PDO). A range of similar tests are available to detect, for example, goat's milk in sheep's cheese or cow's milk in goat's cheese.

E. coli O157 is a widely feared pathogen sometimes found in raw or undercooked beef, unpasteurised milk or sewage-contaminated water. Zeulab's Microfast e.coli O157 is a rapid, easy-to-use test that, after a normal pre-enrichment period, provides an answer in 5-10 minutes, letting you make decisions quicker and reduce assay costs.

Allergens can be equally dangerous in food, and Zeulab offers a range of kits for detecting milk, egg, or gluten. The kits are presented in a range of formats: PROTEON ELISA tests for quantification of allergens, and PROTEON EXPRESS which are fast strip tests that give results within 10 minutes of food sample extraction. Alongside these, the PROTEON EXPRESS SWAB has been specifically designed for the detection of allergen surface contamination without the need for any equipment beyond that contained in the kit.

In addition, Zeulab offers tests for toxins associated with shellfish poisoning, including their OkaTest, which detects okadaic acid; DomoTest, which detects domoic acid; and SaxiTest, which detects saxitoxins. Similar tests pick up toxins in drinking or recreational water, including MICROCYSTEST, a rapid enzymatic test for detecting microcystins in water.

For More Info, email: 32824pr@reply-direct.com

Convenience and ISO Formulation are key to µPrep™ Half Fraser Broth ISO (+FAC) Success



Lab M is seeing demand grow quickly for the company's recently launched µPrep™ Half Fraser Broth ISO (+FAC), a complete primary enrichment broth designed for use in the isolation of *Listeria* spp. from foodstuffs. Media in Lab M's µPrep™ range are supplied ready-to-reconstitute in 20 litre bags. They are designed primarily for high throughput laboratories where speed, convenience, reliability and cost-effectiveness are high priorities.

"Since we launched µPrep™ Half Fraser Broth ISO (+FAC) in July 2014, customers have been very clear that simple reconstitution with no mess and no autoclaving is a significant factor in their choice of media format. However, the fact that the Lab M medium is based on the formulation of ISO 11290, and meets the performance requirements of ISO/TS 11133, is highly influential in their final choice of product," said Wendy Martindale, Director of Sales and Marketing for Lab M.

Reconstitution simply requires attaching a standard laboratory deionised or reverse osmosis water supply, via the Lab M filter accessory, to the bag. The medium reconstitutes rapidly, requires no additional sterilisation or supplementation and is ready to dispense and use in minutes.

All Lab M media are manufactured in powdered form, ensuring rapid cold dissolution and avoiding any of the reconstitution issues that can arise with granulated media, making them ideally suited for use in the µPREP™ format. Robust double-skinned bag construction and a dual port system ensure that leakage is not an issue.

For More Info, email: 33064pr@reply-direct.com

Simple Test for Enterobacteriaceae Gives Results in 1 - 7 Hours



The MicroSnap product range from **Hygiene International** is simple rapid test for bacteria giving results in the same shift, and the range is extended to include Enterobacteriaceae. The measurement of Enterobacteriaceae is a useful indicator of surface hygiene and product quality in the manufacture of heat processed food. Simple, rapid tests such as the specific ATP test system (MicroSnap EB) provides results in a working shift of <8 hours. The test has many applications and a wide dynamic range thus saving time and money and providing an effective tool for routine testing in production facilities.

The test can be applied to solid surfaces for environmental monitoring as well as raw material or finished product testing. It has an excellent agreement with cultural plate count methods and has a huge dynamic range of 1 to 10,000 bacteria in a single test device such that it can be used for presence / absence test or enumeration purposes. This means that less sample preparation or dilution is required thus saving time, labour and material costs as well as minimising the requirements for laboratory facilities.

MicroSnap EB (Enterobacteriaceae) is a simple two-step test procedure with a total time to result of 7 hours 15 seconds. The sample can be either a surface swab or 1ml liquid sample or food suspension that is mixed with a proprietary enrichment broth in an all-in-one device and incubated for 7 hours at 37°C after which a 0.1ml aliquot is transferred (using the device itself) to a specific end detection device for measurement in the EnSURE luminometer giving results in 15 seconds. The specificity of MicroSnap EB was determined by measuring its ability to detect 39 different bacteria in pure culture (22 Entero's and 17 Non-Entero bacteria). Both the inclusivity and exclusivity were measured at 95%.

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