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Information Bulletin
21 2002

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with Classic Soxhlet Extraction

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Introduction

Extraction procedures for sample preparation and sample clean-up have been established in many areas of analytical chemistry. The separation and reclamation of analytes from solid matrices represents a broad application base for solid-liquid extractions. The type and amount of solvent has a major influence on the extraction results. Currently, the time factor is of ever increasing importance. Keeping solvent consumption and extraction time to a minimum is a goal of most laboratories.

For official food monitoring, the extraction conditions are precisely laid down in Section 35 Methods LMBG (e.g. L01.00-20). For example, fat extraction must be performed according to Soxhlet Standard extraction method (extraction by boiling on reflux). This procedure requires a great deal of time and uses a large amount of solvent.

The new Büchi Extraction System B-811 is tested as to whether a minimization of solvent use and a savings in time can be realized without a loss in reliability of analytical results.

Theoretical Basis of Extraction

The basis of all extraction procedures is described in Nernst's distribution law. A substance distributes itself between two immiscible solvents so that the ratio of this concentrations in the two solvents is constant. The system specific constant is described as the Nernst distribution coefficient $K_{D.}$

$$K_D = c_u / c_l$$

In this equation, c_u and c_l correspond to the substance concentrations in the upper and lower phases. More meaningful, however, is the extraction efficiency w of the substance derived from the equation.

$$w = 1 - [1/(1+K_D V]^n$$

The extraction efficiency w is determined by the Nernst distribution coefficient K_D, the phase ratio V [V = V (upper phase)/V(lower phase)] and the number of extraction cycles n which are carried out. The step which determines the speed of the solid-liquid extraction is the suspension of the equilibrium state of the analyte between the solid and the liquid phase. The amount and surface of the sample are very important, as well as the temperature and polarity of the solvent. As a rule of thumb in reaction kinetics: the reaction speed doubles with an increase in reaction temperature of 10°C. A reduction in extraction time can be achieved through the use of hot extraction with high boiling solvents.

BÜCHI Extraction System B-811

Advantages of the BÜCHI Extraction System B-811

The Büchi Extraction System B-811 completely automates four different extraction modes.

- Soxhlet Standard
- Soxhlet Warm
- Hot Extraction
- Continuous Flow Extraction

In addition to the Soxhlet Standard and Continuous Flow Extraction, one achieves significant reduction of extraction times with the warm and hot extraction modes. Because of the high heating capacity and optimal heat transfer, solvents with boiling points up to 150°C can be used. The inert design of the extraction system allows contamination-free operation with all common solvents. The method-integrated evaporation and drying of the extract has been especially advantageous by eliminating the use of a rotary evaporator. All four extraction

modes can be performed without changing the glassware components. The volume of solvent used for extraction can be adjusted to the sample volume with an easily adjustable light sensor which triggers a glass valve.

Materials and Methods

The materials from which the fat will be extracted will include: ready-to-serve meals, ground beef, cheese and rissoles. Hydrolysis of the samples is performed prior to extraction; the fat is determined gravimetrically after extraction. Results of Classic Soxhlet extraction after 4 h extraction time with 250 ml petroleum ether is in each case compared with the results from a petroleum ether extraction with the Extraction System B-811 after various extraction times (2 h, 2.5 h, and 3 h).

Results and Discussion

Evaluation and comparison of all analytical values generates the following results: a solvent volume of 120 ml is sufficient for an extraction, but the time factor has to be considered carefully.

Various extraction times (2 h, 2.5 h, and 3 h) were tested. In some samples a two hour extraction time is sufficient (food with low fat content), other samples require three hours for full extraction. As a general advice, an extraction time of three hours is recommended.

In the following table the values of the standard method Section 35 LMBG are compared with the values from extraction with the Büchi Extraction System B-811. The solvent used was petroleum ether 40/60; the solvent volume could be reduced from 250 ml (standard method Section 35 LMBG) to 120 ml (extraction with Büchi Extraction System B-811). Table 1 shows the fat content in percent (%). The extraction time required in each case with the Büchi Extraction System B-811 is listed in parenthesis after the values. The standard method (Section 35 LMBG) regulates four hours of extraction.

Another main focus of this examination was the handling and use of the Büchi Extraction System B-811. The menu-driven operation is clearly laid out, and the programming is easy to do. The different programs can be stored and quickly recalled. In order to get uniform and reliable results, the same conditions must be used throughout. This is guaranteed by the program-controlled process. The state of the extraction is visible at any time on the display.

Table 1: Fat content of food in %

Sample	Fat determination according to § 35 LMBG	Fat determination with Büchi Extraction System B-811	
Rissoles	14.6	14.5	(2 h)
Ready-to-serve food (a)	3.3	3.3	(2.5 h)
Ready-to-serve food (b)	2.3	2.3	(3 h)
Ground beef (a)	13.8	13.9	(3 h)
Ground beef (b)	12.7	12.7	(3 h)
Cheese (a)	30.2	29.9	(3 h)
Cheese (b)	43.1	43.2	(3 h)

In addition, there are integrated safety features which immediately display errors during the extraction. Should errors occur at a certain position during extraction, this position goes into stand-by. The other unaffected positions continue the process to completion. The entire extraction process is monitored from start to finish.
Extraction experiments concerned exclusively with the optimization of the extraction cycles have not yet been carried out.

Summary and Conclusion

The results of this test indicate that the Büchi Extraction System B-811 represents a good extraction alternative to the Section 35 LMBG procedure in daily routine analysis. Reliable results are achieved together with savings in time and reduced solvent use. In addition, solvent can be recovered separately for further applications. A change of solvents is also possible. The integrated monitoring functions of the Büchi Extraction System B-811 allow the operator to use the equipment safely and reliable.

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