AN number: LA1002
Date: 2015-01-08
Revision: 1.0

Application Guide SoxROC Solvent Extraction

As the extraction might involve the use of hazardous and hot solvent it is strongly recommended to use protective glasses and gloves. The SoxROC Operation Manual chapter 3 Safety should be read before starting any work.

INTRODUCTION

Soxhlet extraction is one of the most commonly used methods for determination of total fat. This is mainly because it is fairly simple to use and is the officially recognized method for a wide range of fat content determinations. Nowadays other extraction techniques tend to be more accurate and more generally applicable and can therefore often replace the old standard method for official analysis of many food materials.

The SoxROC Extraction Unit is based on different techniques, using hot solvent and a closed system for optimal analytical conditions, still giving the same results as the classical and well accepted Soxhlet. OPSIS LiquidLINE has developed the SoxROC Extraction unit to increase the effectiveness of this process.

This Guide is describing the method in a general way and will help to use the SoxROC Extractor. To benefit from all features it is recommended to also read the SoxROC Operation Manual.

During the first period, BOILING, the thimble with sample is immersed in the boiling solvent. To prevent sample floating out from the thimble it is recommended to put a plug of cotton above the sample. Samples like impregnated paper or pieces of plastic can be extracted without using a thimble, just place the sample in the metal coil. For a smooth boiling it is recommended to use boiling chips. Small porous stones or glass balls can be used.

After the BOILING step a controlled volume solvent is collected in the recovery flask, thus lowering the solvent surface in the extraction cups below the thimble. Extraction continues, with the RINSING phase. Hot solvent vapours surround the sample and simultaneously condensed solvent washes out any remaining extractable matter (fat).

At the end of the extraction the solvent is removed in a step by step process, leaving the cups with only extractable matter (fat/ lipids). The cups are dried and weighed, the percentage extractable matter (fat) is calculated.

For some applications the extraction is used as a sample preparation step. Then it is often preferable if the solvent is not completely removed as the extractable matter might change character if dried/heated.

Equipment & Accessories

- SoxROC Extraction Unit
- Extraction Cups, glass or aluminium
- Analytical balance with a capacity of 200g. If the balance has a blue tooth connection the Excel macro for fat analysis from Opsis LiquidLine eliminates all manual recording of weights.
- Oven (100 °C) to dry samples and extraction cups prior to and after the extraction. If the extracted matter is very heat sensitive a vacuum oven should be considered
- Desiccator for cooling cups after drying before weighing
- PTFE holder or Filter holders
- Thimbles (standard size 25x80 mm and 33x80 mm). If hydrolysis is done the special filters for the HydROC should be used also for the extraction step
- Cotton wool to prevent sample from floating out from the thimble/hydrolysis filter
- Celite or sand. Some sample (e.g. meat) should be mixed with Celite or sand to allow full contact with the solvent
- Measuring cylinder (100ml) or a Dispenser to add solvent

Establishing a method

For many samples dedicated Application Guides are available. If not the method and the instrument settings must be established and verified. In general the sample preparation is very important for the final result. The finer it is ground or cut the extraction will be more sufficient. Below are some hints.

Sample volume

A general rule is that higher sample volume gives a more representative result. However, to save solvent and time there are limitations. Also the size of the extraction thimble will set limits. Some samples might cause foaming during the extraction, to reduce this problem often a smaller sample size is a solution. The amount to be extracted has to be on a reasonable level to minimize the weighing errors. Therefore to select the optimal sample size often is a compromise. When the desired sample volume is selected, the average sample height in the thimble should be recorded as this will indicate the proper solvent volume, as listed in the table below.

Solvent volume

During the first step, BOILING, the sample should be completely immersed in the solvent. Therefore the solvent volume is correlated to the sample volume/height.

1. Put the selected sample volume in the thimble to be used, never exceed half of the height. Note the sample height in millimetres.
2. Read the recommended solvent volume in the table below.

	Small cups			Large cups		
Samle h	Volume (ml)	Reduce I	Reduce II	Volume (ml)	Reduce I	Reduce II
10	65	3	4	80	4	5
15	75	4	4	95	5	5
20	85	4	4	105	5	5
25	95	5	4	115	6	5
30	100	6	4	130	7	5
35	115	6	4	140	8	5
40	120	7	4	150	9	5

Note: If the solvent volume used exceeds 115ml/cup the optional large volume recovery kit is recommended. Alternatively the standard recovery flask should be emptied after the first reduction period.

INSTRUMENT SETTINGS

Dependent on sample and solvent different temperatures and boiling/rinsing times are used. Also the type of thimble and extraction cup needs different settings. The SoxROC can be set to almost any extraction program required.

Temperature

In the dedicated application guides the needed temperature is indicated. It depends on the boiling point as well as the type of extraction cup used. Al-cups transfer heat more efficient than glass therefore a lower temperature can be set. If no application guide is available for the specific need the temperature is set as follows.

1. Fill the cups with 90 ml solvent (70 ml if the smaller cups are used).
2. Select a program with the actual solvent. If not listed use OTHER.
3. Set BOILING temperature 20 degrees above the boiling point of the solvent (80 if glass cups are used).
4. Set BOILING time to 15 minutes. Remaining alternatives are set to 0.
5. Insert the cups and start extraction. Make sure the cooling water is on.
6. In a couple of minutes the boiling should start. Sufficient temperature is if the condensation speed is 2-3 drops/sec. If there is no condensation in the coolers after 6-10 minutes the temperature has to be raised. Press START/STOP to interrupt and raise the temperature another 10 degrees according 3-6.

Time and number of reduces

In the BOILING and RINSING steps both time and number of reduces can be set. The number of reduces depends on the initial solvent volume and are listed in the table above.

The time needed for a complete extraction depends on the sample as well as the solvent. In general the SoxROC is four to five times faster than classical Soxhlet. The only way to find an optimal extraction time is to make a series of extractions. The BOILING step can often be set to a third of the total time. Start by making a programme with 20 min BOILING and 40 min RINSING and run a couple of analyses. If the results are satisfactory, the expected level and a low relative error, the time is sufficient. If not, the times have to be longer. Increase total time step by step by 15 minutes until stable results.

If the results were satisfactory already after the first attempt, probably the time can be reduced.

If the aim is to have the extract (fat) completely separated from the solvent the DRYING time should be set to five minutes or longer.

A TYPICAL EXTRACTION ANALYSIS STEP BY STEP

1. Weigh the empty and dried extraction cup with boiling chips. W1.
2. Fix the thimble onto the PTFE holder or into the Filter holder
3. Weigh the prepared sample. W2 and put it into the thimble.
4. Dependent on sample type dry the thimble with sample.
5. Put the holder with the thimble into the extraction cup
6. Fill the required volume solvent into the cup. Note the solvent can be filled through the top of the condensers after the cups have been inserted in the instrument by using a dispenser equipped with a Teflon tube
7. Place the Sample Tray with all cups 2/6 dependent on model into the SoxROC and lower the handle.
8. Select the program to be used.
9. Press START/STOP. When correct temperature is reached the hot plate docks with the cups and the extraction starts.
10. After pre-set extraction is finished the hot plate goes to the lower position.
11. After DRYING, when the display indicates “COMPLETED”, remove the cups
12. Dry the cups with boiling chips and weigh W3 (Note for some applications solvent is left in the cup and no weighing is of interest)

$$\% \text{ extractable matter (fat)} = 100*(W3 - W1) / W2$$

Sometimes the weight difference of the thimble with sample (or just a piece of sample) before and after the extraction is used for the calculation. Notable is that the thimble with remaining solvent has to be carefully dried before the final weighing.

SAMPLE PREPARATION

One of the most common solvent extractions is fat determination of food and feed samples. Sampling and homogenization of such samples prior to extraction is important for the result. Dry samples should be ground, the finer the better. Semi solid samples, like meat and cheese, can be minced using a kitchen mixer or a proper homogenizer.

Dependent on local regulations many sample types have to be hydrolysed prior to the solvent extraction. The procedure is described in the general Application Guide for the HydROC.

Liquids can also be extracted but requires specific preparation dependent on sample type. Detailed information can be found in the dedicated Application Guides.

SOLVENT RECOVERY

The design of the SoxROC allows almost all solvent to be recovered. Dependent on sample and if cotton is used in the thimble some solvent will always be lost here. Solvent collected in the recovery flask can normally be re-used without any further treatments. However, dependent how efficient the drying of the sample prior to the extraction is done some moisture might be adsorbed in the used solvent. If so the results can suffer. Solvent mixed with water is not the same as pure solvent.

Note: If diethyl ether is used it always has to be tested for peroxides before re-use.

REFERENCES

OG1012 SoxROC Operation Guide