

SoxROC Extraction Unit Operation Manual Version E



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2. Introduction

The OPSIS LiquidLINE SoxROC Extraction Unit is an automated instrument for general hot solvent extractions and for extraction of fat according to official methods. The instrument is based on Randall and Twisselmann techniques, using hot solvent and a closed system to reduce extraction time versus manual Soxhlet. However, to determine fat, several other steps such as weighing samples and drying are also required. Final analytical result will not only depend on the solvent extraction but also these steps. It is therefore important, not only to read this manual, but to follow the OPSIS LiquidLINE application notes.

As most analyses carried out on this instrument involves the use of hot solvents, it is important that the operator reads or is informed about what is written in chapter 3 “User Safety”.

To get a brief understanding of how the SoxROC Extraction Unit works, please refer to chapter 4 “Function”.

In chapter 5 “Operation” the different available functions will be described.

Chapter 6 “How to run an Extraction” describes the typical steps for a complete extraction using the SoxROC Extraction Unit. One or several of these steps might be excluded depending on application and purpose of the extraction.

To get the best from your instrument, it is important to maintain and service it correctly. This is described in chapter 7 “Maintenance and Service” and chapter 8 “Installation”.

3. Safety

The SoxROC Extraction Unit is protecting the operator from any hazardous actions, e.g. it is not possible to run a program with open safety door and it is not possible to start a program if the hotplate temperature is higher than desired by the operator. Care has also been taken to minimize explosion risks if hot solvents come in contact with the hotplate or if there are hot solvents inside the instrument.

However, as the methods described involve the use of hot solvents, every user should read the Safety Instructions or be instructed by the laboratory manager. Below you can find the instruction.

3.1. USER SAFETY

The instrument may only be used by laboratory personnel and other persons who have knowledge and/or experience of doing chemical analysis and dealing with solvents.

Applications not mentioned in this document are improper. In particular, it is forbidden to use the instrument in the following instances:

- Use of the instrument in rooms which require ex-protected instruments.
- Use of samples which can explode or inflame due to shock, heat, friction or spark formation.
- Use in conjunction with solvents containing peroxides.
- Use with other glassware than the original.
- Use with samples that react with solvent.

It shall be noted that:

- Modifications and upgrades to the instrument shall only be carried out by authorized service personnel.
- Debug menus in the Instrument is only to be used by authorized service personnel.

3.2. SAFETY SYMBOLS



General Hazard



Flammable Hazard



Crushing hazard



Electrical shock hazard



Hot Surface

Explanations used in this manual



Important, Please Note



Please Note, Gloves should be used

3.3. PRODUCT SAFETY SYSTEMS

The instrument is designed and built in accordance with state-of-the-art technology. Nevertheless, risks to users, property, and environment can arise when the instrument is used carelessly or improperly. If the equipment is not used in a manner specified by this document, the protection provided by the equipment may be impaired.

3.3.1. Instrument Related Hazards

Pay attention to the following safety notices on the instrument:



There are potentially hot surfaces during operation, especially at the hotplate (up to 300°C). Always be aware of the risk of being burned.



Do not remove covers and other parts protecting from electricity. Always keep the areas of electric parts, such as power supply plug, mains switch, etc. dry.



The supplied air tube should always be connected to the instrument and the inlet of the tube should be connected to clean air, with no solvents or flammable gases nearby.

There is a potential risk if explosive solvent vapours accumulate within the instrument housing. Please always take care to:

- Always use the instrument in a well-ventilated area.
- Beware of damaged or cracked glass parts, damaged seals or fire hazard.
- Always be aware of the explosion risk when working with hazardous substances or with substances of unknown composition.
- Always provide a good ventilation within or in the vicinity of the system.

3.3.2. Maintenance and Service

The Operator is responsible for ensuring that recommended daily and monthly user maintenance is performed on the Instrument. Failure to do so might impair the functionality and/or shorten the lifespan of the instrument.

The operator is responsible to schedule regular Maintenance with authorized service personnel only. Only OPSIS LiquidLINE Spare parts should be used in the instrument.

3.3.3. Safety Sensors

The instrument is equipped with several safety systems.

- A sensor will identify if the safety door is closed. No program is allowed to start with an open door. The safety door protects operators from broken glass in case of an accident or explosion.
- The hotplate is equipped with two separate over-temperature protection systems. The SoxROC will always alert the operator and close down the system if the temperature exceeds 30°C compared to desired program temperature.
- A sensor monitors the air pressure inside the electronics cabinet. No program is allowed to run without a safety pressure inside the SoxROC.
- All solvent valves are ATEX classified.
- Accidental leak of solvents onto the hotplate should always be cleaned as soon as possible. If necessary, remove the front plate and clean the area below the hotplate. See chapter 7.4.2, Remove Solvent inside of SoxROC Instrument.



Please note that the air tube on the back of the instrument must be used and placed in a good location to ensure safe operation. Air is used to maintain air pressure inside the electronics cabinet and must be connected to clean air (no risk for flammable solvents).



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4. Function

4.1. GENERAL

This section describes the general function of your SoxROC Extraction Unit. The instrument is a fully automatic system that automatically will boil, rinse and dry samples.



A hotplate (1) controlled by software will maintain an operator controlled temperature for the boiling and rinsing steps. This hotplate will automatically move up at the beginning of the boil and move down when reaching the drying step. The hotplate is monitored by two separate temperature systems to ensure safety.

The samples should be placed inside cups (2) and thereafter placed in the sample tray. The sample tray is placed into the instrument. The tray carrier is inserted and the safety door is closed with the handle (3). Solvent can be added directly in the cups or afterwards, via the refill inlet at the top of the SoxROC (4).

A condenser (5) is placed above each cup and will ensure that all vapours are cooled. The condenser requires cooling water to function properly. The condensed solvent will be collected into an adapter (6). Solvent will flow back to the cup when the solvent inside the adapter reaches above 11 ml. The cup, seal and condenser are a closed system.

Solvent can be removed from the system via the adapter and PTFE valves. The valves can be controlled with opening interval, opening time and amount of opening times.

All solvent will be collected in the recovery flask (7). Use the release handle (8) to lock and unlock the flask to the SoxROC. The handle secures the placement of the flask but solvent will always pass, regardless of position of the handle.

All operations can be controlled via the display (9). Power on and off is done with the power button (10).

The front cover (11) can be removed in case there is a need to clean the interior of the instrument.

4.2. STEP BY STEP OPERATION

The SoxROC is built to do hot solvent extractions and follow a three step process with boiling, rinsing and drying. The parameters for the complete extraction process can be programmed by the operator and followed on the display.

4.2.1. Boiling with extraction



Six cups with samples are placed into the SoxROC instrument. One of six programs can be chosen and set by the operator. All programs follows the same procedure, also known as Randall, Goldfisch or Twisselman extraction.

- The operator places inserts the sample tray with cups, closes the safety door, selects a program and press the START/STOP button.
- The SoxROC will start the heating.
- The hotplate will move upwards and dock with the cups when it reaches the desired temperature. High pressure between the hotplate and the cups will ensure efficient heat transfer and good sealing of the system.
- Solvent is heated by the hotplate and evaporates into the condenser. The vapour will condense and be collected into the adapter above the cup. Additional solvent will flow down from the adapter to the cup when the adapter is full. The sample inside the cup is extracted.
- The boiling, condensing and extraction will repeat several times until the boiling time has been reached.
- Once the boiling step is completed the adapter valve will open on all positions and the solvent inside is released. Solvent will be collected in the recovery flask at the front of the SoxROC. Solvent removal can be controlled in the extraction

program. The thimble should be above the boiling solvent after the removal of solvent.

- The program switches to the next step when the removal of solvent has been completed.

4.2.2. Rinsing with Extraction



The purpose of the rinsing step is to remove the remaining extractable matter from the thimble and ensure that all is collected inside the cups.

- Once the removal of solvent is completed in the boiling step the rinsing will begin. The solvent evaporates, vapour in the condenser is condensed and flows down to the adapter and then finally into the cup. The condensed solvent will wash final traces of soluble matter from the sample when reaching the cup. The sample inside the cup is extracted.
- The boiling, condensing and extraction will repeat several times until the rinsing time has been reached.
- Once the rinsing step is completed, the adapter valve will open on all positions and the solvent inside is released. Solvent will be collected in the recovery flask at the front of the SoxROC. The amount of solvent to remove can be controlled. All remaining solvent should be removed at this step in most applications.
- The program switches to the next step when the removal of solvent has been completed.



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4.2.3. Drying



The purpose of the drying step is to remove all solvents from the SoxROC system as well as to initiate the drying of the extractable matter. Please note that a final drying step in an oven is also necessary.

- Once the rinsing step is completed, the hotplate will undock with the cups and move downwards. This is to ensure that there is no overheating of the soluble matter at the bottom of the cups. The hotplate will also stop heating.
- Adapter valves will open on all positions and the solvent inside the adapter is released. The valves will be open for the duration of the drying step. All solvent in the SoxROC will be collected in the recovery flask at the front of the SoxROC.
- The drying step, with open valves, will continue until the drying time has been reached.

Enabling or disabling as well as the time of drying can be controlled in the program.

5. Operation

An extraction is done in several steps and might be different for different applications. However, the most common way to do an extraction is described below.

- The operator will
 - Homogenize and pre-dry the sample.
 - Weigh the sample.



- Insert the sample in a thimble and if necessary add cotton to ensure reliable results.



- Weigh an empty cup with boiling stones.

- The operator will use the SoxROC Extraction Unit to
 - Boil and extract matter from sample (boiling).
 - Rinse and continue to extract sample (rinsing).
 - Dry sample (drying).

- The operator will
 - Dry the cup (with extracted matter) in an oven.
 - Keep the cup (with extracted matter) under dry conditions, using a desiccator.

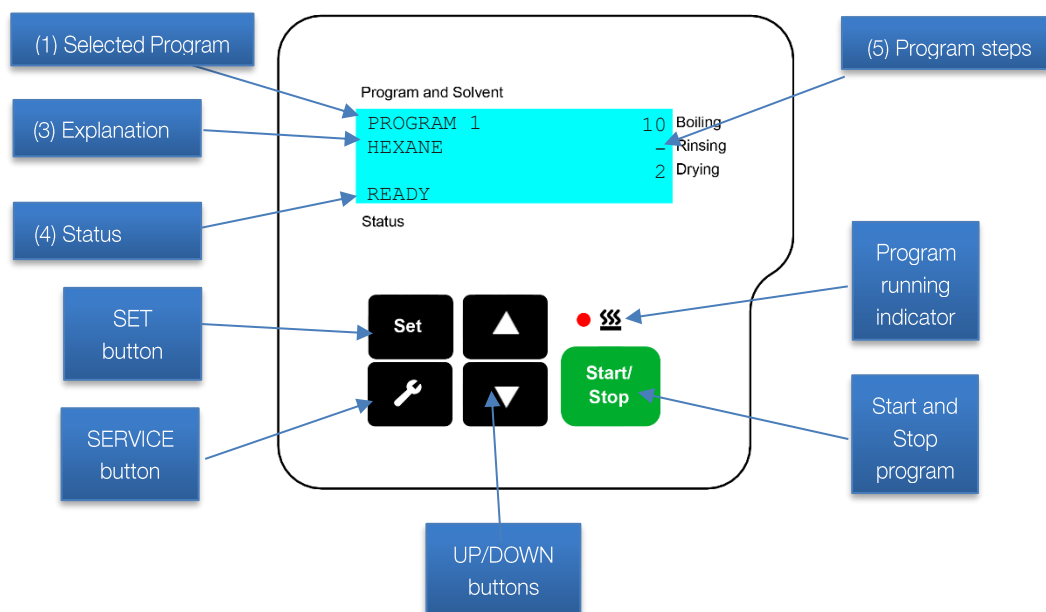


- Weigh the cup with the extracted matter.
- Calculate the final extracted matter vs sample.

This section will describe the menus to operate the SoxROC Extraction Unit. For more details on the complete process, please consult the OPSIS LiquidLINE application notes.

5.1. GENERAL GUIDELINES

The SoxROC can be controlled with the four button panel and the display.



Buttons on the Front Panel



Press the SET button to adjust the program settings. SET button is also used to confirm choices inside the SET menu and to continue with next menu choice inside the SET menu.



Press the SERVICE button to adjust the instrument settings. SERVICE button is also used to confirm choices inside the SERVICE menu and to continue with next menu choice inside the menu.



Press UP and DOWN to toggle between choices and enter values into the instrument.



Press START/STOP to start a program or cancel a current operation. The warning light will always be light whenever an operation is running.

Display Messages

- The Selected program or the selected menu item can be seen in the upper left corner of the display. For each program there is also an option to add one pre-defined comment to the program name (i.e. Pet eth 40/60, hexane, other, etc.).
- The status of the SoxROC can be seen in the lower left corner. When running a program the status will be boiling, rinsing or drying. A ready message will be displayed when the unit is ready to start. “Needs cooling”, “Purging wait” or “Close door” will be displayed in case the SoxROC is not yet ready.
- The remaining time for each step can be seen in the upper right corner.

5.2. START UP

The SoxROC Extraction Unit is started with the power on button at the side of the instrument. During initiating the instrument will pump air into the electronics cabinet and the message “purging – wait” will be displayed. The message will remain until there is pressure inside the electronics cabinet. It is possible to use SET and SERVICE menus but not starting a program while purging.

The system will also perform an automatic control of all systems during start-up and if any error is found, a warning message will be displayed.

5.3. HOME

The instrument will reach the Home menu after start-up, from there it is possible to control the SoxROC Extraction process. The message “Ready” will be written as soon as pressure has been reached inside the electronics cabinet.

The following choices can be done in the Home menu:

- SET will access the programming menu where it is possible to adjust the program parameters for the currently selected program.
- SERVICE will access the instrument settings menu where it is possible to adjust instrument settings.
- UP/DOWN will allow selection between the six different programs in the SoxROC.
- START/STOP will start the currently selected program.

5.3.1. Home Menu Status Messages

It is not possible to start a program if any of the below messages are displayed in the Home menu. The SoxROC will give a sound if the START/STOP button is pressed while these are displayed:

- “Purging – Wait”: This message indicates that the pressure inside the electronics cabinet has not been reached. Normally this should be achieved within 1 minute from start-up of the SoxROC. Please wait.
- “Needs Cooling”: This message indicates that the selected program is set with a lower temperature than is currently on the hotplate. The system is cooling down but will not allow start until temperature is within 10°C from set program. Please adjust boiling temperature of the program, select a different program or wait until temperature has been reached.



- It is possible to check the current temperature of the hotplate by pressing and holding down the SERVICE button from the Home menu. The display will automatically return to the Home menu after releasing the button.
- “Close door”: It is not possible to start a program with open safety door. Please close door to continue.

5.4. SET MENU

Pressing SET from the Home menu will allow modification of the selected program. The name of the program will be displayed at the top of the display and the specific menu choice will be displayed below. This menu consists of a step by step programming where each press on SET will confirm choice and continue to next step.

It is not possible to leave the Set menu until the programming is finished. In case of an error when entering a setting, then please continue with all steps until the Home menu is reached. Re-start the programming from there.

The following programming steps are available:

- SET SOLVENT. It is possible to give each program a unique name by selecting different solvent names. Set name with UP/DOWN buttons. Press SET when done.



Please note, this setting only affects the displayed name and does not affect the program in any other way. Selecting solvent name does not affect over-temperature protection.

- SET BOIL TEMP (30-300°C). Set boiling temperature with UP/DOWN buttons. Press SET when done. Please note, it will not be possible to start the program in case the hotplate is 10°C above the set temperature.



The over-temperature will automatically be set at the boiling temperature plus 30°C. The SoxROC heating will shut-down in case the hotplate becomes warmer than this during boiling.

- SET BOIL TIME (0-999 min). Set time of the boiling step with UP/DOWN buttons. Press SET when done.

- SET REDUCE (0-20): Set the amount of times the adapter shall open to reduce the solvent levels prior to the rinsing step. The adapter contains 11 ml of solvent and each time the valve opens this volume will be removed. Set amount of openings with UP/DOWN buttons. Press SET when done.



Please take care to program this setting according to OPSIS LiquidLINE application notes. It is important to reduce the solvent level so that the thimble inside the cup is above the boiling solvent to achieve correct results.

- SET RINSE TIME (0-999 min): Set time of the rinsing step with UP/DOWN buttons. Press SET when done.

- SET RECOVER (0-20): Set the amount of times the adapter shall open to remove the final solvent prior to the drying step. The adapter contains 11 ml of solvent and each time the valve opens this volume will be removed. The boiling will continue during the recovery step. Unless there is an application to keep a certain level of solvent at the end of the process then all solvent should be removed at this step. Set amount of openings with UP/DOWN buttons. Press SET when done.

- SET DRYING (0-999 min): At the drying step the pivoting hotplate will be lowered and the heating of the cups will stop. The valves will open and empty the final remains from the adapters during the drying time. If time is set to “---“ then the valves will remain closed during this step and the program will finish. Set time with the UP/DOWN buttons. Press SET when done.

The programming is completed after the drying step and will return to the Home menu.

5.5. SERVICE MENU

Pressing SERVICE from the Home menu will access the Service menu. The Service menu will allow modification of general instrument settings. Adjustment in this menu affects all six programs in the SoxROC.

The menu consists of a step by step programming where each press on SERVICE will confirm the choice and continue to the next step. It is not possible to leave the menu until all choices has been displayed.

The following steps are available:

- TEMPERATURE SET SCALE. This option allows selection between Celsius or Fahrenheit temperature scales. Set with UP/DOWN buttons and press SERVICE when done.
- VALVE CYCLE SET OPEN TIME (0-999,0 s). The valve opening time allows detailed control of the valve opening time. There should always be enough time to ensure that a full adapter can be emptied. Set integer values using the UP/DOWN buttons. Set decimal values by pressing SET and thereafter using the UP/DOWN buttons. Press SERVICE when done.



Please do not adjust this parameter unless given in an OPSIS LiquidLINE application note.

- VALVE CYCLE SET INTER TIME (0-999,0 s): The valve cycle interval time allows detailed control of the time delay between valve openings. There should always be enough time to ensure that all adapters are filled with solvent between each emptying step. Set integer values using the UP/DOWN buttons. Set decimal values by pressing SET and thereafter using the UP/DOWN buttons. Press SERVICE when done.



Please do not adjust this parameter unless given in an OPSIS LiquidLINE application note.

- AIR PUMP POWER SET PWM-HI: The Air pump High Power Set controls the power of the air pump when there is no pressure inside the electronics cabinet. The air pump will operate at this higher effect until pressure is achieved. The pressure is controlled by a pressure sensor inside the SoxROC. Set with UP/DOWN buttons and press SERVICE when done.



Please do not adjust the default unless instructed by a service engineer.

- AIR PUMP POWER SET PWM-LOW: The Air Pump Low Power Set controls the power of the air pump when there is pressure inside the electronics cabinet. The air pump will operate at this lower effect so that pressure can be maintained. Set with UP/DOWN buttons and press SERVICE when done.



Please do not adjust the default unless instructed by a service engineer.

The instrument settings is done after the Power set and will return to the Home menu.

5.6. RUNNING A PROGRAM

It is possible to start an automatic program from the Home menu. Pressing START/STOP will initiate the automatic extraction process.



Please check that the recovery flask is inserted into the instrument and empty prior to starting the extraction process. Please check that a sample tray with cups is inserted and that the cooling water tap is open.

- Initial safety control with safety door, air pressure and temperature is always controlled before a program will start.
 - If door is open then a message will be displayed to inform the operator to close the door.
 - If air pressure has not been achieved inside the electronics cabinet, the message “Purging wait” will be displayed.
 - If current temperature of the hotplate is above the set over-temperature then “Needs cooling” will be displayed in the menu.
- Heating of the hotplate will start immediately after the initial safety controls. The water valve will open and cooling of the condensers will start.
- The hotplate will raise when the desired temperature has been reached and the boiling step will commence. Boiling will continue until the desired boiling time has been reached. During the extraction addition of solvent is possible. The solvent is added through the refill inlet. The remaining boiling time will be shown in the display.
- Solvent will be removed at the end of the boiling step. A counter to indicate the openings of the valves will be shown in the display.

- The Rinsing step will continue with boiling and then removal of solvents when the desired time has been reached. The remaining rinsing time will be shown in the display.
- Solvent will be removed at the end of the rinsing step. A counter to indicate the openings of the valves will be shown in the display.
- The hotplate will automatically go down at the drying step. Depending on the program, the valves might open and the drying will continue. The remaining drying time will be shown in the display.
- The SoxROC will inform that the extraction is finished. Press START/STOP to reach the Home menu.

It is possible to stop the extraction at any time by pressing the START/STOP button.

5.6.1. Status Messages during a Program

The display will show the remaining time of the different steps in the upper right corner of the display. Additional status messages will also be shown during the extraction.

- Heating 50°C/150°C indicates that the hotplate is heating but has not yet reached the desired temperature. 50°C is the current temperature and 150°C is the desired temperature in this example.
- Boiling, Rinsing and Drying indicates the current step in the program.
- Aborted: Indicates that the extraction was aborted due to an internal sensor giving an alarm. This can be due to low pressure in electronics cabinet, open safety door or power cut. Consult a Service Technician if this happens repeatedly. Press START/STOP to return to the Home menu.
- Interrupted: Indicates that the program was interrupted and stopped. Typically because the STOP button has been pressed. Press START/STOP to return to the Home menu.

6. How to Run an Extraction

6.1. PREPARING FOR AN EXTRACTION

1. Prepare the samples. Insert sample into thimble and add cotton as described in the application note. Insert boiling stones and thimble into cup.
2. Depending on preference, add solvent directly inside the cups or add solvent later when the cups are placed inside the SoxROC. Closed solvent addition can be done via the refill inlet at the top of the instrument. The solvent is added through the refill inlet using a pipette, rinsing bottles, a dispenser or other solvent delivery device. Use a 6 mm wide tube.



3. Place the cups with samples in the sample tray and insert that into the SoxROC Extraction Unit. Close the safety door.



The sample tray should always be used to ensure good sealing of the cups. It is recommended to always insert six cups even if less samples are extracted.

4. Power on the SoxROC Extraction unit. Wait until the air pressure has been reached inside the electronics cabinet, the message “Purging wait” will disappear.
5. Open the cooling water to the SoxROC (tap water/chiller).
6. Ensure that there is enough empty space inside the recovery flask to allow for solvent during the process.

6.2. RUN A PROGRAM

7. Select a stored program with UP/DOWN or modify a current program by pressing SET.
8. Start the program with START/STOP.



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9. At the end of the program. Remove the sample tray with the cups.



Please take care that the cups and sample tray will be hot. Use gloves when removing the sample tray.

7. Maintenance and Service

The SoxROC is built with durable material but in order to keep your instrument working, it is important to do regular maintenance.

You should always call for service technician if any unexpected warnings are displayed on the instrument. Some parts are recommended to be replaced on a regular basis to assure proper operation. Original parts from OPSIS LiquidLINE should always be used.

7.1. DAILY MAINTENANCE AND CLEANING

Though the SoxROC Extraction unit is designed to resist normal solvents, a clean instrument will always last longer. Therefore it is recommended to follow below recommendations.

- Before start, use a wet cloth to wipe off any spillages from the instrument. It is important that the cloth is damp to avoid build-up of static electricity.
- It is recommended to empty the instrument from any solvents at the end of the day by running a program with drying on.
- In case of glass cups. Check these for cracks, especially on the surface which faces the seal.
- The hot plate should be cleaned regularly after it has been cooled down.



It is important to ensure that there is nothing, such as stones or glass pieces, on top of the hotplate. Cups might break, due to the intense pressure, if there are residues left.

7.2. MONTHLY MAINTENANCE

The SoxROC Instrument should be controlled more thoroughly at a monthly interval to ensure a working instrument.

- Check the seals between the cups and the condenser. Run a program with the following settings.

P -1-	Vol ml	t °C	Drain -s-	Int -s-	Boil -m-	Red I	Rins - m-	Red II	Dry - m-
PE40/60	90	90/150	5	120	5	3	5	5	Yes

- 90 °C for Al-cups or 150 °C for glass cups. Measure the volume of the solvent in the recovery flask and calculate the recovery.

Observe any damages or leakages.

- Check the tubes, hoses and hose connections for defects (cracks, brittle areas) and replace damaged hoses.
- Visually inspect all glass parts for defects (cracks, stars, and splintering) regularly. The glass parts should be cleaned using common commercial cleaners (for example, mild soap solutions). Dirt that is stuck inside the condenser spirals will normally not affect the performance of the instrument and is cleaned during the regular maintenance by a Service engineer.
- The hotplate should be cleaned regularly after they have cooled down. Remove dirt adhering to the plate with e.g. ethanol. Then clean the plate with a moist cloth and dry it.
- Sometimes the tray carrier will require additional grease. This is added during the yearly maintenance but may also be added when needed.

7.3. YEARLY MAINTENANCE

A yearly maintenance of your instrument should be performed by an OPSIS LiquidLINE certified service engineer. Please consult your OPSIS LiquidLINE contact to get advice on yearly maintenance.

7.4. CLEANING AND EXCHANGE OF COMPONENTS

7.4.1. Cleaning of Cups

The aluminium cups should be cleaned by hand to avoid that the protective layer is removed from the surface. Cleaning of glass cups can be done in a dish washer.

7.4.2. Remove Solvent inside of SoxROC Instrument

The instrument should be switched off and cleaned in case solvents or other liquids enter into the instrument. Follow these instructions:

- Let the instrument cool down to avoid burns.
- Remove any visible solvents on the outside of the instrument. Use a wet cloth to wipe off any spillages from the instrument. It is important that the cloth is damp to avoid build-up of static electricity.
- Open the front cover. Take a firm grip at the holes of the front plate and drag the plate towards you. No tool is needed.
- Clean the area below the hotplate.
- Re-attach the front cover to the SoxROC.

7.4.3. Exchange of Seals

It is easy to exchange the seals on your SoxROC. Please follow these steps:

- Power off your instrument and remove any sample tray rack or cups.
- Open the safety door and use your hand to remove the seals from the adapters. The seals are soft and they will easily follow.



Care should be taken with solvents. Please use gloves when removing the seals.

- Insert new seals by hand. Make sure that the complete seal is fitted in the track that is prepared on the adapter. The flat side of the seal should face upwards.

7.5. EXTRACTION ADJUSTMENTS

7.5.1. Adjusting Settings for Solvent Removal

Please always follow the instructions in the OPSIS LiquidLINE application note to adjust your instrument towards your application. Typically each program is adjusted with the SET REDUCE (0-20) and SET RECOVER (0-20) options. In case there is need for further adjustment of the complete instrument, then settings in the SERVICE menu might be required.

- Please adjust the VALVE CYCLE SET OPEN TIME in case there are problems to empty the complete adapter during each reduce or recover.
- Please adjust the VALVE CYCLE SET INTER TIME in case there is not enough time to fill all adapters between each reduce or recover.



These settings are programmed in factory and should not be changed unless instructed in an application note or by a service technician.

7.5.2. Specific Applications and Specific Seals

Please check the seals at regular intervals to prevent leakage and low recovery of solvents. Change seals 1-2 times per year.

Please note that standard seals are in Viton which might not be able to withstand all solvents. PTFE/Butyl seals are recommended in case of solvent usage of Acetone, Acetonitrile, Diethyl ether or Ethyl Acetate.



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8. Installation

We recommend that the installation should be done by an authorized OPSIS LiquidLINE service technician.

8.1. INSTALLATION REQUIREMENTS

- Laboratory bench, with 70 cm wide and 60 cm deep free space. The use of a fume hood/fume cupboard is recommended.
- Electrical supply 230V \pm 10%.
- Cold water tap and drain within 1.5 m from the instrument.
- Clean air to feed the electronics cabinet. It is important that the air inlet is placed far from solvents.

8.2. UNPACKING AND ASSEMBLY

Carefully remove the packing material and make sure there are no transport damages. If so you should directly contact your OPSIS LiquidLINE representative.

8.3. CONTENTS OF THE PACKAGE

The following components are included together with your SoxROC.



1 SoxROC Extraction Unit with attached power cord, PVC tube to connect to air inlet, water tube to connect to water inlet and PVC tube to connect to water drain, user manual and testing protocol.



1 Recovery Flask.



Selection of cups, aluminium or glass.



Selection of thimble holders, thimbles and seals.
(seals are mounted inside instrument)



1 Cup stand for sample tray.



1 sample tray, should be used when inserting cups inside the SoxROC.

8.4. CONNECTING AND STARTING THE SOXROC

1. Place the SoxROC at the designated laboratory bench.



The SoxROC instrument is heavy and care should therefore be taken when lifting. Please avoid to hold the instrument in the safety door handle.

2. Connect all tubes to the SoxROC Extraction Unit.
 - a. Ensure that cooling water inlet on the SoxROC is connected to a cold water tap. Use the supplied water tube. Check that the water tap is open.
 - b. Ensure that a cooling water outlet is going to the drain. Use the supplied PVC tube.
3. Connect the Air tube to the Air inlet of the SoxROC. Make sure that the open end is in fresh air and outside any fume cupboard. A longer tube might be needed.



The supplied air tube should always be connected to the instrument and the inlet of the tube should be connected to clean air, with no solvents or flammable gases nearby.

4. Connect the Power to the SoxROC and switch on the instrument

9. Technical Data

Operating Temperature	5°C - 40°C
Relative Humidity	max 80 %
Power Supply	190-240 VAC, 50-60 Hz, 10A
Power Consumption	max 1300W (SX-360-A) max 600W (SX-320-A)
Dimensions (WxHxD)	630 x 520 x 540 mm (SX-360-A) 440 x 520 x 540 mm (SX-320-A)
Weight	63 kg (SX-360-A) 57 kg (SX-320-A)

10. Declarations and Requirements

10.1. DECLARATION OF CONFORMITY



Declaration of Conformity

Identification of apparatus: SoxROC Extraction Unit SX-320
SoxROC Extraction Unit SX-360

Model/type: SoxROC Extraction Unit

Manufacturer: OPSIS AB
Box 244, SE-244 02 Furulund, Sweden
Phone: +46 46 72 25 00

The undersigned hereby declares that the above-referenced product, to which this declaration relates, is in conformity with the provisions of:

- Council Directive 2014/30/EU (February 26, 2014) on Electromagnetic compatibility (EMC),
- Council Directive 2014/35/EU (February 26, 2014) on Electrical Safety: low-voltage electrical equipment,
- Council Directive 2006/42/EC (June 9, 2006) on Safety of Machinery,
- Council Directive 2011/65/EU (June 8, 2011) on Restriction of the use of hazardous substances (RoHS 2).

The below harmonised standard specifications have been applied:

Safety:

ANSI/ISA-61010-1 (November 5, 2012) Safety Requirements for Electrical Equipment for Measurement, Control, and Laboratory Use - Part 1: General Requirements

Electromagnetic Compatibility:

Emission: EN 61000-6-3 (2007)

Immunity: EN 61000-6-2 (2005)

October 8, 2018

Svante Wallin
President OPSIS AB



Representantes / Distribuidores Autorizados

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10.2. FCC REQUIREMENTS (FOR USA AND CANADA)

English:

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to both Part 15 of the FCC Rules and the radio interference regulations of the Canadian Department of Communications. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications.

Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Français:

Cet appareil a été testé et s'est avéré conforme aux limites prévues pour les appareils numériques de classe A et à la partie 15 des réglementations FCC ainsi qu'à la réglementation des interférences radio du Canadian Département of Communications. Ces limites sont destinées à fournir une protection adéquate contre les interférences néfastes lorsque l'appareil est utilisé dans un environnement commercial.

Cet appareil génère, utilise et peut irradier une énergie à fréquence radioélectrique, il est en outre susceptible d'engendrer des interférences avec les communications radio, s'il n'est pas installé et utilisé conformément aux instructions du mode d'emploi. L'utilisation de cet appareil dans les zones résidentielles peut causer des interférences néfastes, auquel cas l'exploitant sera amené à prendre les dispositions utiles pour pallier aux interférences à ses propres frais.

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.

Sample h	Small cups			Large cups		
	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

List of Application Notes

Updated December 2020, Please contact your OPSIS LiquidLINE representative in case a more updated list is required.

Kjeldahl method and/or KjelROC instrument

LA1000 Application Guide (included in manual)

LA1001 The Importance of Catalyst in Kjeldahl

LA1009 Determination of total nitrogen in milk

LA1010 Determination of nitrogen in wheat

LA1011 Determination of nitrogen in pet food

LA1012 Determination of nitrogen in fish meal

LA1013 Determination of nitrogen in corn (maize) starch

LA1018 Determination of nitrogen in rice

LA1019 Determination of nitrogen in water

LA1022 Determination of protein in soy sauce

LA1023 Determination of nitrogen in soil

LA1024 Determination of TVB-N in fish

LA1026 Determination of protein in nuts

LA1028 Determination of protein in pasta

LA1031 Determination of SO₂

LA1031 Determination of SO₂ according to Chinese National Standards (CNS)

LA1033 Determination of alcohol in wine

LA1036 Determination of protein in Wine

Hot Solvent methods and/or SoxROC Instrument

- LA1002 Application Guide (included in manual)
- LA1003 Extraction of palm oil
- LA1004 Extraction of fat in potato chips
- LA1005 Extraction of total fat in pet food
- LA1006 Extraction of fat in biscuits
- LA1007 Extraction of fat in chocolate
- LA1008 Extraction of total fat in chocolate
- LA1014 Determining the gel content of Ethylene Vinyl Acetate
- LA1015 Extraction of total fat in hard Cheese
- LA1016 Extraction of fat in coconut milk
- LA1017 Extraction of fat in fish Meal
- LA1020 Extraction of dioxins and SVOC in food
- LA1021 Extraction of SVOC and PAH in soil
- LA1025 Extraction of fat in nuts
- LA1029 Extraction of crude and total fat in poultry feed
- LA1034 Determination of fat in Waste water
- LA1035 Extraction of Total fat in Milk powder

FiberROC instrument

- LA1027 Determination of crude fiber in cattle feed



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