MILESTONE
HELPING CHEMISTS

Fragrances

Microwave Green Extraction of Natural Products

Operator Manual

MA217

MILESTONE
HELPING CHEMISTS

Thank you for selecting our microwave system.

We are sure that you will be completely satisfied with the performance of this new unit entering your laboratory. We invite you to read carefully this user manual and to keep it in reach for convenient and fast consulting. The person who will be using this unit should have received an appropriate training from a Milestone trained technician. For any possible clarification or any request for assistance please contact either our Representative in your country:

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Tutorial Videos

How to work with ETHOS X for terpene extraction

We are glad to share Tutorial Videos that both shows you the process and also walks you through the procedure steps.

Our Tutorial Videos show easy to follow and clear instructions about:

- Equipment set up and the needed guidelines to assemble the reactor and the distillation module.
- How to properly prepare the raw material prior the extraction
- How to use the software and set the extraction method
- How to check the extraction progress
- How to perform the extraction work-up
- How to keep the system at best working conditions

SEE THE TUTORIAL VIDEOS HERE

http://www.milestonesrl.com/tutorial-video-terpene

SCAN ME

1 SAFETY RULES

General Information

Fragrances configuration

- Please read carefully this operator manual before starting the system and follow its prescriptions with the utmost care.
- In case the system has been delivered with additional accessories (i.e. the terminal is an additional accessory), also read carefully the instruction manuals of such accessories.
- This Operator Manual is part of the delivery, hence must be always kept together with the instrument on its working place.
- It is imperative that every person operating this system has read and fully understood this manual.
- The non-observance of the instructions contained herein or improper use may involve damages/injuries that are not covered by product liability!
- The national ruling and international safety norms in working environment shall in any case be observed under user's responsibility.

ETHICS	The instruments commercialized by Milestone are intended for the use in strict compliance with all applicable laws and in a manner that promotes public safety and/or linked to approved scientific or medical research purposes.Milestone does not endorse, encourage or promote the use of its instruments for any illegal activities.No liability for any consequences, direct or indirect, is accepted if the Fragrances configuration is used in any way in which the instrument is not intended to be used.

- It is also user's responsibility to keep constantly updated all safety norms ruling in the country or operation.

Information = indicates important information/recommendations for the user.

Attention = means that the non-observance of the warning may cause damages to unit or properties.

Warning means that the non-observance of the warning may cause injuries, even severe, to people.

Safety rules for Start-up

- The first start-up of the system must exclusively be carried out by authorized and trained service engineers appointed by Milestone.

Safety in operations

- The unit must exclusively be operated for its intended use, otherwise the protection can be compromised.
- The unit must only be operated within the power limits set forth by the technical specifications.
- The unit must be operated exclusively by specially trained people.
- All electrical parts must always be protected from wet and humidity.
- The unit must be kept clean.
- Basic rules of chemistry will always apply. The basic rules of chemistry are valid also for handling chemicals in this unit. Working with chemicals, always take all those safety measures that are usually required (i.e. lab coat, protective gloves, protective glasses mask, exhausts hood, etc.)

Safety in service operations

- In case of failure or damage of the unit, the intervention of an authorized and trained service operator must be requested. The unit shall not be used until successful repair is completed. The unit includes components in which lethal high voltage of 4kV can be reached. Improper repairs by unauthorized people may cause microwave leakage and consequent injuries to personnel.
- Do not operate the unit, if a minimum doubt exists about its trouble-free operation and good functioning.
- Service can only be carried out by authorized and trained service engineers of the company Milestone. Works on the electrical and electronic components of the system must be carried out exclusively by specialized electrical/electronic engineers.
- Before every repair – service – installation, always make sure that the unit is disconnected from the power supply line and cannot involuntarily be switched on.

No manipulation of the unit allowed

- Constructive changes/modifications of the unit and its accessories are forbidden, in particular any openings in the sample chamber must be carried out, because microwaves can be released there.

Parts replacement

- In order to guarantee the correct functioning of the unit and user's safety, all replacement parts must be exclusively original supplied by Milestone. Original spare parts are guaranteed by high quality control on the material and during the production.

Customer support

- The Warranty and the Service Report are included in the documentation delivered together with the unit.
- After the installation, you are kindly requested to send us the filled in warranty card via fax or e-mail (form on the website).
- In case of required unit reparation, you should send us the filled in Service Report including precise description of the errors.
- For any request or order, please contact your local area representative or directly Milestone.

Other hints

- The microwave unit can be provided with several sensors and built-in devices, which are in part described in this user manual. This does not mean that all the options are installed in your unit, but the supply depends on your order.
- The unit must be connected to a socket with protective grounding.
- Be careful that voltage and frequency of the power supply match with the values indicated on the shield on the unit back.
- The system must be operated with the original power supply cable supplied together with the unit.
- The power supply cable must be in perfect conditions and must not show any damage.
- The unit must be operated only in upright position.
- Ventilation ports in the housing - for cooling the inside components – must not be covered or obstructed by any object.
- The unit must not be installed or stored in an explosive, corrosive or conductive atmosphere (dust, vapor, gases).
- During program runs, the unit must be carefully attended, to enable timely recognition of operator's improper use or error. The unit must under no circumstances (i.e. at night) run unattended.
- Metal parts in sample chamber

  No metal parts must be introduced in the sample chamber of the microwave unit (except for components incorporated in the original construction). The resulting arc effect can cause damages.
- Flanges in the sample chamber

  Unused flanges must be sealed with the provided screw or plug caps. In any case fingers or objects in operations should not be held during operations. Flanges should not be modified.
- The microwave unit is exclusively intended for heating materials destined to chemical and analytical laboratory applications.
- Magnetic stirrer/People with peacemakers

  If the unit is provided with the magnetic stirrer, then there is a strong magnetic field inside the sample chamber. Peacemaker wearers are at risk when in the immediate vicinity of the device.
- PC control device

  If the microwave unit is controlled by a terminal or a PC, no changes can be operated on the control unit. The installation of electronic cards or of additional software can compromise the function of microwave unit control software.

Instructions for safety operations

- All samples must be processed only in the glass reactors tested and released by MILESTONE.
- The operator must carefully follow the appendixes 5.1 and 5.2 of this manual to set up the glassware correctly before starting the extraction.
- MILESTONE does not take up the responsibility for an accidental breakage of the glassware.
- The system is thought to perform solvent-free microwave extractions. The use of any organic solvent is therefore strictly forbidden.
- The system is thought to perform extractions at atmospheric pressure. Working under pressure is strictly forbidden.
- The boiling point of water imposes the maximum temperature reached during an extraction process (100°-110°C).

- When following Milestone application notes, all parameters, settings and equipment shall be noted and followed. The proposed application notes by Milestone are only to be considered as general guidelines and must be adapted to each single sample or used material.
- In anycase, the pursuing of different applications must be discussed with Milestone Application Specialist prior to be applied.
- Application requirements must be met.
- If damages occur to the microwave unit, in particular to the door, door frame, welded joints and ventilation openings, the microwave unit may no longer correctly work. The microwave unit must be shipped back to Milestone for a safety check.

Care of glassware and accessories

Glassware and accessories such as glass reactors, distillation module, holders etc. should be regularly checked and cleaned to avoid damages. Never use organic solvents for plastic parts cleaning (except for PTFE) (i.e. acetone attacks the PSU). Water residues must be thoroughly dried, because they heat in the microwave with the risk of damages. For any requests, contact Milestone or the authorized distributor in your country.

Maintenance and care

- Please, be sure that the sample chamber is always clean. Contamination in the door area can obstruct the correct door sealing, letting out microwave irradiation.
- Before each run, check and remove eventual contamination in the sample chamber, in particular the area near the door and its wall.
- Sediments in sample chamber such as contamination and solvent residues etc. can cause burns and damages.
- Normally a gentle cleaning with a soft cloth or paper towel, dried or moistened with water, should be carried out.
- To remove strong contamination, you can use mild detergents.
- Do not use any abrasive cleaner on the sample chamber walls and on the door, because they could damage the PTFE coating.
- If the microwave unit is equipped with a QP sensor for digestion, do not use cleaning agents containing alcohol or solvents, because such substances are responsible for the sensor output signal for several days, and the sensor cannot be used.
- Contamination on the external side of the housing can be removed with a damp cloth and mild detergents.
- The unit life, in particular of the electrical and electronic parts, is mainly depending on the environmental conditions. The unit should not be installed under a hood, but in a well-ventilated room, free from acid vapors and corrosive atmosphere. Although the housing is stainless steel made and the critical parts are coated, components like plugs, electronic boards, etc. can be damaged by corrosion. These parts cannot be manufactured in more resistant materials. Thus the most common problems are due to corrosion and chemical attack to these components.
- All components in direct contact with samples, sample vessels, etc. must be handled with the utmost care, i.e. the parts must be free from chemical residues/traces and dried before use. This principle is of course well unknown in the trace analysis, but not always properly taken into account.
- Dust in the laboratory atmosphere is very frequently responsible for deposits in the ventilation area (air inlet and exhaust, etc.) and must be removed, to prevent overheating and/or damages.
- Check that the exhaust tube is regularly introduced into its opening. If the pipe is disconnected, reintroduce it until it is firmly fixed.

2 SENSORS

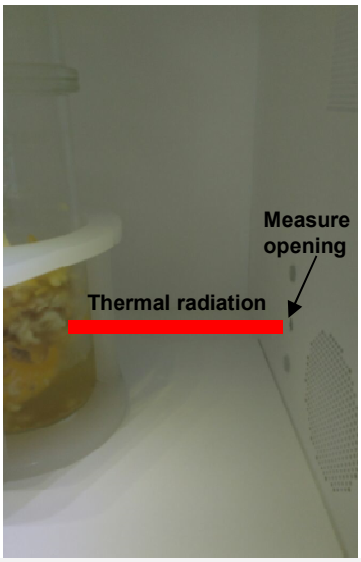
Temperature sensor

2.1.1 Contactless temperature control (T2)

The contact-less temperature control measures the infrared heating radiation from samples located before the measuring port.

The measuring of the infrared warm radiation from a body depends on the following factors:

- Emission factor of the material
- Surface nature
- Body distance and dimensions
- Body geometry



IR-TC-Sensor

Even if the boiling point of water imposes the maximum reachable temperature, the control of the temperature (T2) is of great benefit to monitor constantly the extraction.

2.1.2 Care Instructions

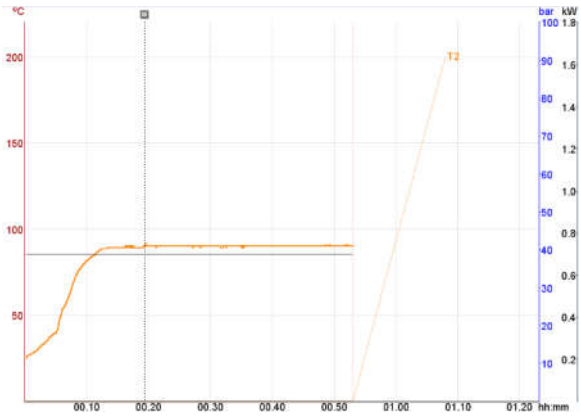
The IR sensor is calibrated by the manufacturer using the supplied accessory, while the recalibration can be performed by a service contract.

To ensure a correct measuring of the IR sensor, the sensor must be weekly cleaned with a cotton bud (i.e. the measuring port and the sensor window located there). In case of unusual/abnormal program runs, the IR sensor must be cleaned after every run.

Power

The system can reach a maximum power of 1800W, accomplished by the presence of two magnetrons of 900 W each. The power must be set at the beginning and remains constant during the extraction process. It is extremely important to set the right power before starting the extraction process, since it is a function of the amount of the sample loaded. To understand how to set the power properly, please refer to the Application Book.

On the right, you can find a typical temperature curve of a performed extraction. As shown, the power remains constant during the whole run, while temperature reaches approximately the boiling point of water.



3 BASICS OF MICROWAVE EXTRACTION

Introduction

Fragrances can be extracted from several botanical sources using the ETHOS X Fragrance Package. For the extraction of fragrances there are not any particular solvent requires. There are differing conditions based on the material being extracted. The categories of botanical are Fresh, Dried, and Frozen. The main factor in utilizing the ETHOS X for extraction is water content. The material needs to have enough moisture for the hydrodistillation to work properly without degrading the plant material.

The suitable method to perform extraction of fragrances is included in the control software. With these tested programs, a large variety of samples can be successfully extracted. Specific method are described in the Application book as well as the general guideline to optimize and prepare new methods.

Essential oil extracted from the fragrances kit shown in same case strong solvent properties. Store the essential oil in glass vials and use the proper PPE.

Preparation

The material being extracted should be pre-treated prior to extraction in the ETHOS X. The material should be weighed and placed into a large mixing container. For some samples, for example orange peel, a grinding could improve the extraction efficiency. Next water will need to be measured by weight and added at the appropriate proportion based of the category of material being used as described below. The contents of the mixing container are then mixed thoroughly. Using gloved hands for this works extremely well as the material can be kneaded like dough which causes water to incorporate within the plant material. After mixing, the soaked contents will need to sit for 15-20 minutes to be certain the water is completely throughout the plant material prior to introducing it to the vessel of the Fragrance package for the ETHOS X. The amount of water will be determined based off the material being utilized of the three categories above, the water amount is critical in successful fragrances extraction.

Most of the fragrances will be extracted and isolated from the plant material when the botanical is processed through the ETHOS X Fragrance Package. Below are some general guidelines to follow for the differing materials being used as the starting material based on the above categories.

3.1.1 Fresh Botanical

Fresh material is material that has been harvested from the plant without any further processing. The material is still moist with the natural water content of the plant. Freshly harvested material should have about 75% water content naturally. Fresh will be considered material that has been harvested not long ago and has not had much time to dry out; thereby still incorporating the moisture naturally from the growing season. Anything else will fall into another one of the categories to follow.

Fresh material should be pre-treated with 1 gram of water to 1 gram of material (for example: 500g of Fresh material should have 500g of water should be added to it).

The ratio is 1 to 1 Fresh to water, respectively.

3.1.2 Dried Botanical

Dried material is plant material that has had its' moisture removed either on purpose or naturally by time after harvest. This material has had the water removed, one way or another, and will need substantial water to be added with it to induce the extraction of the fragrances using microwave hydrostatic distillation. Please read below carefully:

Dried material needs to be pre-treated with 3 grams of water to every 1 gram of plant material (for example: 500g of Dried material needs to have 1500 g of water added to it). Soaking time might be increased to 30 minutes with larger batch sizes to help offset the lack of water within the material.

The ratio is 1 to 3, Dried to water, respectively

Dry material will require more soak time and excess water for good extraction. Be certain to mix this material extremely well, using the fingertips to break up any larger clumps until a fine, spongy, completely soaked material is obtained in the mixing container. Before the appropriate soak, be certain to tamp the soaking material down so that it is level along the top. It will be correct when at the beginning of the soak, pressing a thumb into the material will leave an indentation that has a small pool of water at the lowest point. At this point re-flatten the top to remove the indentation created and let soak for the appropriate time. Each batch might be different, so soak at least 30 minutes to be certain that moisture is incorporated throughout all plant material. Unfortunately, as each batch of this material will be slightly different, an exact time nor amount of water can not be pre-deduced. This material will require a little experimentation, however, 1 to 3 is a good starting ratio of Dry material to Water, respectively, with a 30 minutes soak. Slight adjustments might be necessary.

3.1.3 Frozen botanical

Material that has been harvested and immediately frozen. If it has been processed in any way prior to freezing (ground or dried, and then frozen), treat it as Dried material. Frozen material normally will have ice crystals collected on the outside of the buds during the freezing process. These crystals add water into the mixture, so a lesser amount of moisture needs to be reincorporated. Allow this material to warm to room temperature prior to soaking to allow efficiency when utilizing the microwave energy. This material should soak just like the other forms prior to extraction.

Frozen material should be allowed to warm to room temperature, then pre-treated with 1 grams of water to every 1 gram of plant material (for example: 500g of Frozen material should have 500g water added).

The ratio will be 1 to 1, Frozen to water, respectively.

NOTE: Again; this is for immediately frozen material after harvesting. TREAT ALL OTHER FROZEN MATERIAL AS DRIED MATERIAL.

3.1.4 Fragrances Extraction

The method to run a successful extraction of fragrances will depend on run characteristic factors. For power input, those factors include size of vessel, weight of plant material used, and the total interior volume of the vessel filled. The length of the run will depend on fragrances extraction and quality of extract required. One of the key aspects of proper method execution is initial heating of the vessel and its' contents. This, again, will take a little experimentation to dial in the time and energy required utilizing the vessel(s) and material being used. The temperature reader (IR for the ETHOS X; T2 in the method and display) is not used to control the program. This field should be untouched when creating a method, and

when using a “Fragrance Method” that comes pre-installed the ETHOS X software to build a method should also remain unchanged from the factory settings. Below are some general starting guidelines:

3.1.4.1Loading

Loading the vessel will describe how the energy profile will need to be to get good extraction of fragrances. The best, most efficient distillations will happen when the vessel is almost filled to the lid. This allows the best efficiency in getting to reflux, holding the temperature, and extracting the fragrances. This also allows the shortest, hottest path to the reflux condenser with the best efficiency. A large void between the material and the lid will result in less efficient distillation, and possible non-distillation in all desired fragrances as they might reflux back into the vessel and not onto the condenser and through the downward condenser to be collected. The closer the vessel is to being completely full to the lid the better the efficiency will work.

3.1.4.2Heating

Depending on the volume of material being used (flower vs. ground for example) the initial energy input (Watts) and holding power will vary. Initially, 5-10 minutes at 1500-1800W can be used to heat up the vessel and contents. When using a larger vessel, a longer heat time will be necessary. Depending on the scale, the energy (Watts) might need to be reduced once the reflux gets to an acceptable level. A good indicator of progress with this is following the IR temperature reading. This reads the interior temperature of the plant material when it is within the vessel. As the vessel and contents heat up, they should approach 94-98 degrees °C; at which time the water should begin to reflux. 94-95 degrees °C temperature range will keep the reflux going and is the temperature that the most desired fragrances will distill out. After reflux is obtained, the power can be reduced to a ratio of around 1 Watt per gram of material.

General power ratio: 1 Watt per gram of material.

This can be used to keep the reflux going and result in good distillation of the desired terpenes. The temperature of the IR reading should be around 94-96 °C. The length of the distillation will vary depending on the amount of material and the efficiency of the distillation. A good stopping point indicator for these distillations is when the power does not change, but the temperature slightly increases to 98-99 °C. At this point the higher-boiling, less-desirable fragrances and aromatics are coming out, so unless absolute yield is an issue instead of quality, this is a good place to stop.

- The 2L vessel can begin with 1200-1500W for several minutes (~3-7 minutes) until reflux is obtained then power reduced to match the amount of plant material in the vessel (~300g material uses ~300-400W to keep reflux). Typical runtime in total: 30-50 minutes

- The 5L vessel follows the same methodology as the 2L but with 5-15 minutes heating time to reflux. The power is then reduced to be equivalent to the plant material weight (~500g; ~500-600W). Typical runtime in total: 40-60 minutes.

- The 12L vessel will require 1800W when using 1.8-2kg of material, so the heating and reflux power will be the same throughout the run; view the temperature to follow progress. Typical runtime in total: 60-90 minutes.

All times above are approximate. The heating parameters/method will have to be adjusted based on the starting material and/or volume. Initial heating time will change based on the type of material being used. The vessel size and volume within the vessel should also be considered; the closer to the lid the pre-wetted material is the more efficient the extraction.

3.1.4.3Distillation progress and fragrances recovery

If the ETHOS X Fragrance Kit has the glass-only distillation apparatus (there is no stainless-steel components on the apparatus; glass only), then one can view the beginning distillation progress by observing the straight glass tube that passes through the roof of the ETHOS. One can see the tube begins to look cloudy and turbid on the inside with the beginning formation of liquid droplets. This normally begins right about 70-80 °C. As the temperature begins to near 90 °C, the tube begins to clear up and the droplets form more towards the condenser. At 92-96 °C a decent reflux is obtained which is used to remove the fragrances.

The above is not used for monitoring when the ETHOS X includes the stainless-steel distillation apparatus; as the output from the top of the microwave and first condenser is steel. The best place to monitor the distillation with the stainless apparatus is in the graduated collection glassware. Once the fragrances begin to collect and the reflux is going well, the Wattage can be reduced as recommended above to keep the reflux going.

Progress can be monitored by viewing the fragrances accumulate on top of the water in the collection glassware. Once the collection begins to slow, the interior temperature will increase to 98-99 °C and the most desirable fragrances have been distilled at this point. This slight temperature change is a good place to stop the distillation as the desired fragrances are removed at this point. Further distillation will remove less-desirable fragrances and aromatics.

The fragrances are then removed by draining from the collection area of the glassware using the attached stopcock. Residual water can be removed by typical laboratory practices (separatory funnel, drying agent and filter, etc.).

Yields are commonly 0.5-2.0% depending on quality of starting material.

The collected fragrances can be exposed to the atmosphere for a couple of hours to help remove highly volatile components. This can be exasperated by using a pipette to agitate the solution occasionally during this time, helping to aerate the contents thereby helping to remove any trapped highly volatile components that azeotroped with the oil.

4 ADDITIONAL DEVICES

Chiller

Depending on the amount of material treated per batch, two different water chillers are suggested: H150-1000 and H150-2100 (both developed and manufactured by LabTech), or equivalent. They differentiate by the maximum power they can supply, since the amount of water to be cooled down strictly depends on the amount and the water content of the plant material in input. In each application note it is clarified how to choose the right chiller for material treated. Please find below the technical specifications of the two chillers.

Tech-Specs	**H150-1000**	**H150-2100**
Temperature range	8°C ~ 35°C	8°C ~ 35°C
Temperature stability	±0.1°C	±0.1°C
Temperature control mode	PID	PID
Cooling capacity	1500 W	2100 W
Pump flux	6L/min@60psi	13L/min@60psi
Pump power	120 W	250 W
Pump type	Magnetic	Magnetic
Heat exchanger model	BOARD	BOARD
Dimension mm(LxWxH)	670x385x625	735x460x703
Weight	78Kg	92Kg

Before to connect the chiller to the glass modules, be sure that the output pressure is max 3 bar

5 FRAGRANCES KIT SET UP

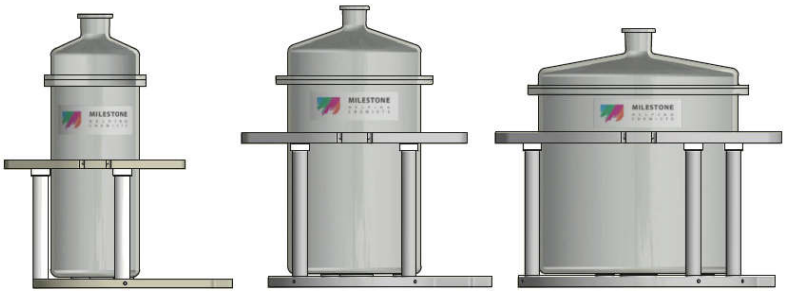
Fragrances kits

Chapter 3 explained how to properly prepare the botanical sample prior the extraction and how to set the right method according to the material type and amount.

Two fragrances kit are available for ETHOS X, a stainless steel and a glass versions. In this chapter it is explained how to set up the fragrances kit.

5.1.1 Fragrances extraction vessels

ETHOS X is a versatile system that offers extraction vessels with different capability and extraction volume from 2 to 12 liters. These can be used interchangeably.

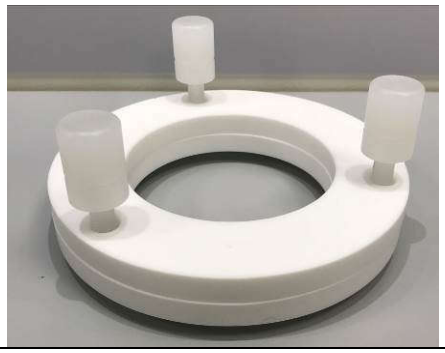


2, 5 and 12-liter glass reactors for fragrances

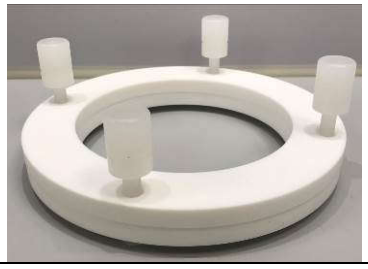
5.1.1.1 List of components

Glass reactor S, complete with glass cover and dedicated holder (PN 70930A) includes the following parts :

P/N	Picture	Description
70931		Glass reactor S (max 2L)
70932		Glass cover for S reactor

70933		Holder for S reactor
90023		PTFE sleeve for joint 29/32
70925		Sealing rings kit, S
70926		Silicon sealing ring

Glass reactor M, complete with glass cover and dedicated holder (PN 70940A) includes the following parts :

P/N	Picture	Description
70941		Glass reactor M (max 5L)
70942		Glass cover for M reactor
70943		Holder for M reactor
90023		PTFE sleeve for joint 29/32
70975		Sealing rings kit, M

70974		Silicon sealing ring

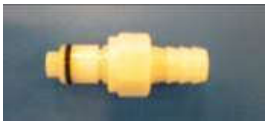
Glass reactor L, complete with glass cover and dedicated holder (PN 70950A) includes the following parts :

P/N	Picture	Description
70951		Glass reactor L (max 12L)
70952		Glass cover for L reactor
70953		Holder for L reactor
90023		PTFE sleeve for joint 29/32

70959		Sealing rings kit, L
70954		Silicon sealing ring

5.1.2 Stainless steel fragrances kit (PN 70990-S)

5.1.2.1 List of components

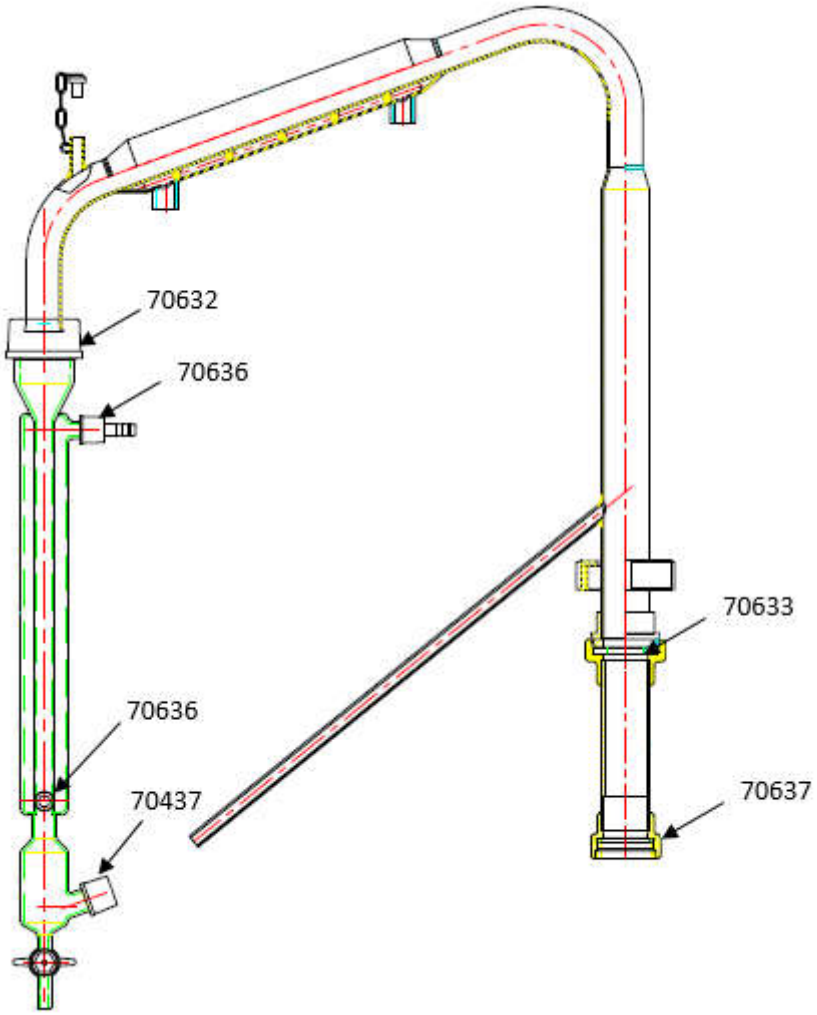
P/N	Picture	Description
70997		Complete stainless steel kit
SL0050		PU tube for cooling water (blue), 1m – 6m required
102697		Connector ¼
91210		Quick connect female water connector
91215		Quick connect male water connector

70965		Glass joint 29/3
70960		Positioning plate
70963		Silicon grease
70629		Adapter reactor stainless steel joint
OR0096IT		Silicon o-ring Øint 17.13mm 2.62mm
OR3100IT		Red silicon o-ring Ø 25.07x2.62mm
85267-IT		Tool for flange condenser joint
70997 includes		
70631		Stainless steel distillation module

70634		Graduated glass connection module (burette)
70635		Stainless steel joint
70435		Silicon/glass joint

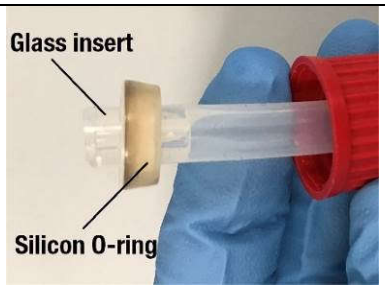
5.1.2.1.1 List of spare o-rings:

In the below picture a list of spare o-rings are reported:



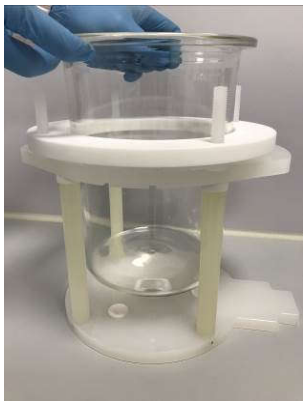
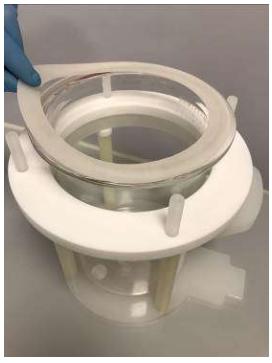
5.1.2.2 Pre-assembling stainless steel condenser

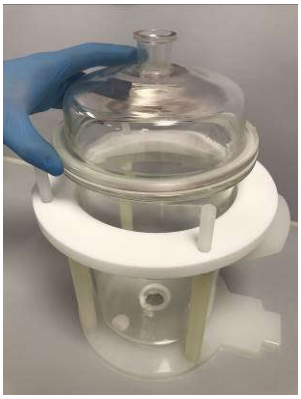
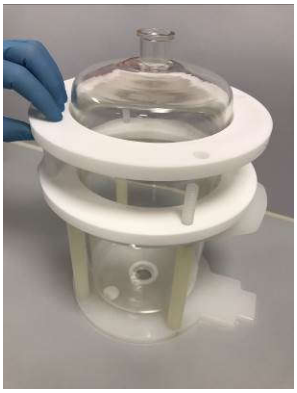
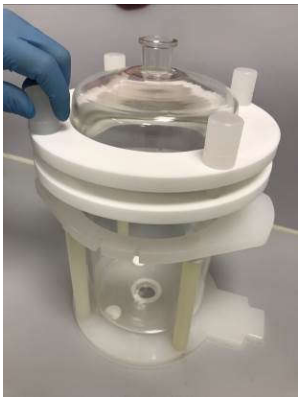
Step	PICTURE	DESCRIPTION
1		Components
2		Connect the cooling tubes (with quick male connector) to the condenser.
3		Insert the screw cap (smaller) and the silicon O-ring onto the silicon tube of the stainless steel condenser.

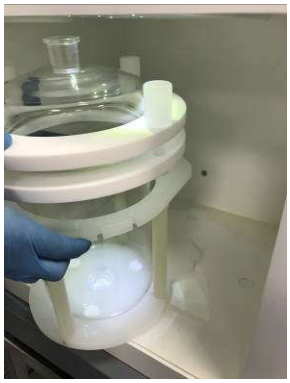
		
4		Insert the screw cap (bigger) and the silicon O-ring onto the stainless steel condenser.
5		Condenser properly pre-assembled.

5.1.2.3 Assembling stainless steel fragrances kit

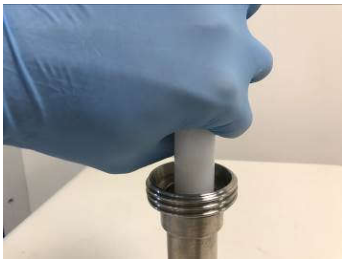
With the botanical inside the reactor properly prepared for the extraction follow the below instruction for mounting the stainless steel fragrances kit

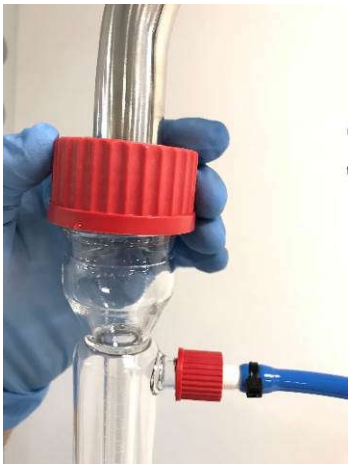
Step	PICTURE	DESCRIPTION
1		Place Bottom Ring of the Sealing Clamp onto the Vessel Holder
2		Place loaded, Vessel into the Vessel Holder
3		Dry carefully the upper flange of the reactor with paper
4		Place Silicon Gasket onto the ground-glass lip of the Vessel

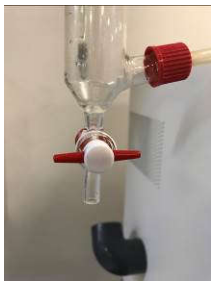
5		Place Vessel Lid centred onto the top of the Silicon Gasket
6		Place Top Sealing Ring onto the Vessel Lid
7		Use the four Poly Tightening Nuts to Secure the Top and Bottom Ring together which seals the Lid to the Vessel • Do not deform ring during this step • Hand tighten to slight tension; tighten across, not in a circle
8		Install the Cavity Positioning Plate into the back to the Ethos X cavity

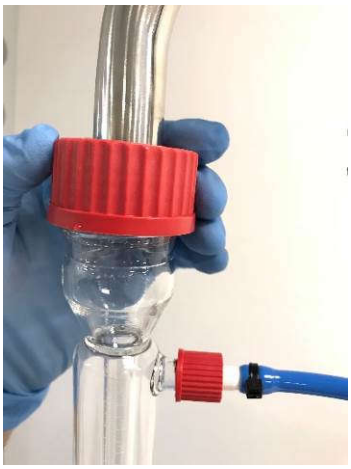
9		Place assembled Vessel into the cavity of the Ethos X
10		Centre the holder into the positioning plate.
11		Insert the PP adapter into the glass connection tube
12		Grease the larger o-ring
13		Install PTFE sleeve onto ground-glass joint; Seat well (as alternative Spread the silicon grease on the glass-ground side of the glass connection tube)

14		Place Pipe through the hole in the top of the Ethos X and into the ground-glass joint of the Vessel Lid
15		Place the stainless steel joint on the glass connection tube on the upper flange.
16		Carefully screw the stainless steel joint on the upper flange holding the glass tube on the top of the glass reactor cover (Hand Tighten snug)

17		Push the PP adapter with the proper tool
18		Place the silicon O-ring on the top of the stainless steel joint.
19	 	Install the pre-assembled condenser onto the stainless steel joint.

20		Be certain Pressure Relief Plug is inserted into the Steel Still Head
21		Connect the graduated burette to the condenser through the red screw cap.

22		Connect the upper cooling tube of the burette (female) to the left cooling tube of the condenser (male).
23		Connect the lower side of the graduated burette to the condenser through the smaller red screw cap.
24		Connect the lower cooling tube of the burette (male) to the “SUPPLY” tube of the water chiller system (female).
25		Connect the right cooling tube of the condenser (male) to the “RETURN” tube of the water chiller system (female).
26		With Petcock closed, unscrew top connection and pre-fill Collection Glass with water until the return drains Overfill again by 20-30mL and let return drain again - verify operational

27		Reconnect top to Steel Still Head
28		Set the Chiller at 8°C and turn it on
29		Select the right method and press start

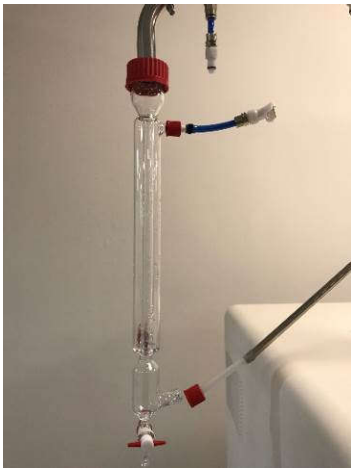
5.1.2.4 Collecting the extracts

After microwave hydro distillation properly performed according to the guideline reported in the chapter 3 follow the below step to collect the product.

STEP	PICTURE	DESCRIPTION
1		Unload the extracts from the graduated burette using the PTFE stopcock.
2		Recover the extracts. A separatory funnel can be used to better separate oil extract from water.

5.1.2.5 Disassembling the fragrances kit

For general cleaning operations, the stainless steel condenser apparatus must be completely disassembled. The following procedure describes how to dismount the main components from the condenser.

STEP	PICTURE	DESCRIPTION
1		Disconnect the “RETURN” cooling tube on the right side of the condenser.
2		Disconnect the “SUPPLY” cooling tube from the lower side of the burette.
3		Disconnect the upper cooling tube of the burette from the left cooling tube of the condenser.
4		Disconnect the lower side of the graduated burette from the condenser.

5		Unscrew the red screw cap on the top of the graduated burette.
6		Remove the graduated burette.
7		Unscrew and remove the stainless steel condenser from the joint on the top of the unit.

8		Unscrew the stainless steel joint from the upper flange on the top of the unit.
9	 	Carefully remove the stainless steel joint from the upper flange. The glass connection tube should come out along with the joint.
10		Remove the glass tube from the stainless steel joint.

11		In case the glass connection tube is remaining inside the flange: remove the stainless steel joint from the upper flange
12		Disconnect the glass connection tube and remove it through the upper flange, holding firmly the glass reactor cover.
13		Remove the glass reactor from the microwave cavity.

5.1.2.6 General cleaning and maintenance

All the glass and stainless steel components can be cleaned with water, ethanol or acetone.

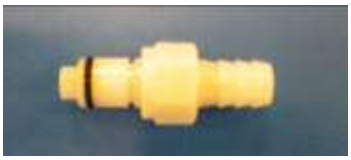
When o-rings and plastics components are contaminated, replace with new one. Below a list of possible components to be replaced:

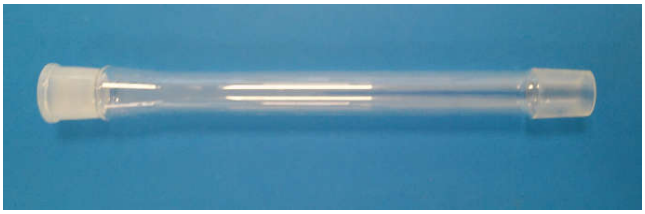
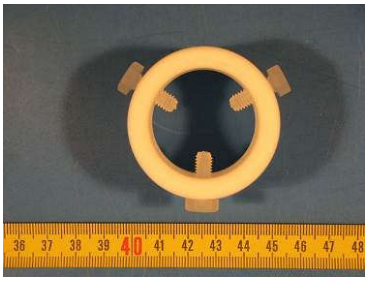
- 90023 PTFE sleeve for joint 29/32
- 70962 Silicon sealing ring
- 70974 Silicon sealing ring
- 70954 Silicon sealing ring
- *70629 Adapter reactor stainless steel joint*
- *OR0096IT Silicon o-ring Øint 17.13mm 2.62mm*
- *OR3100IT Red silicon o-ring Ø 25.07x2.62mm*
- *70629 Adapter reactor stainless steel joint*
- *70435 Silicon/glass joint*
- *70636 Silicon o-ring*
- *70633 Silicon o-ring*
- *70632 Silicon o-ring*
- *70437 Silicon o-ring*

5.1.3 Glass fragrances kit (PN 70990)

5.1.3.1 List of components

P/N	Picture	Description
91326		Glass condenser 280mm (fragrances)
90076		Glass cap
91308		Glass extension tube for distillation module 210mm
70991A		Distillation module

70196		Silicon tube 6 x 10 (length: 2m)
91211		Male connectors (x4)
91210		Female connectors (x4)
SL0050		PU tube for cooling water (blue), 1m – 6m required

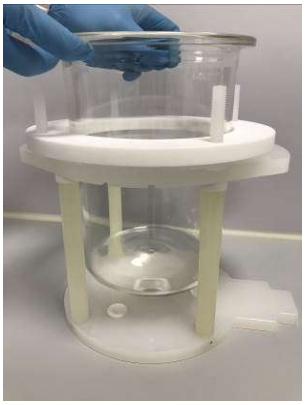
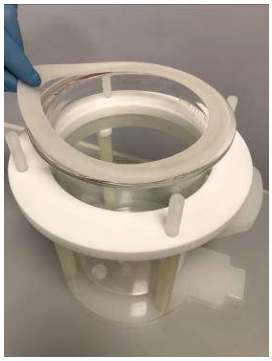
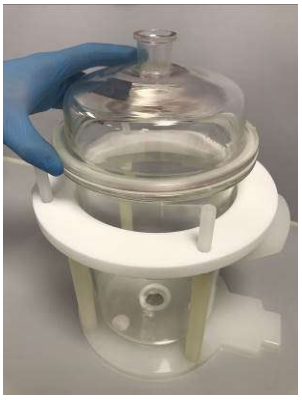
70992		Glass connector for distillation module
70963		Silicon grease
70964		Glass joint F-M 320 mm
SGL0021		Centering adapter for glass tube

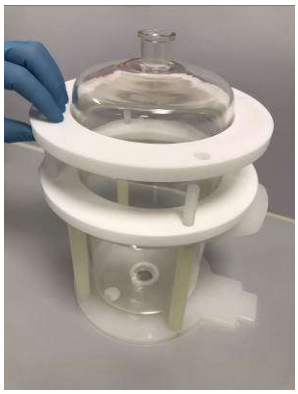
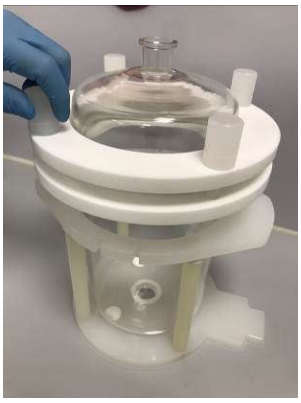
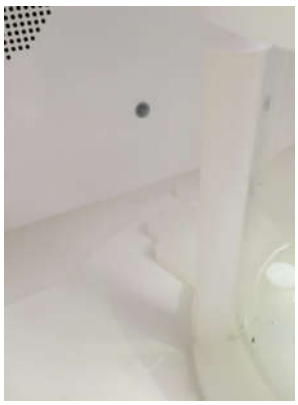
Other components :

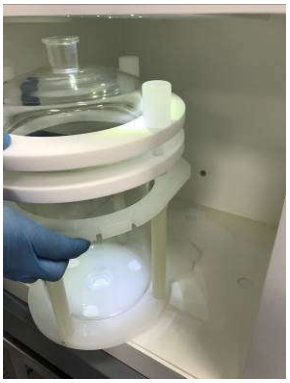
P/N	Picture	Description
70960		Positioning Plate
70961		External glassware support
91206		Pliers
91207		Clamps

5.1.3.2Assembling glass fragrances kit

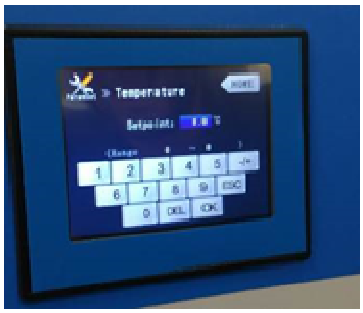
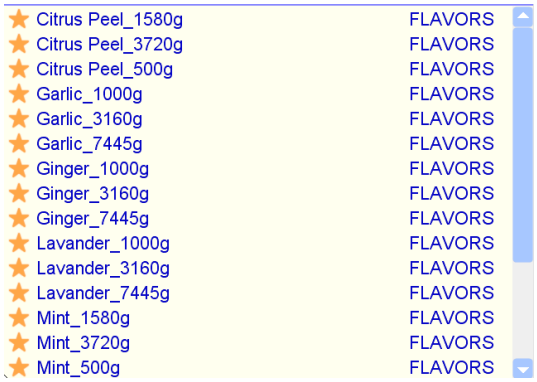
Step	DESCRIPTION	PICTURE
1		Place Bottom Ring of the Sealing Clamp onto the Vessel Holder

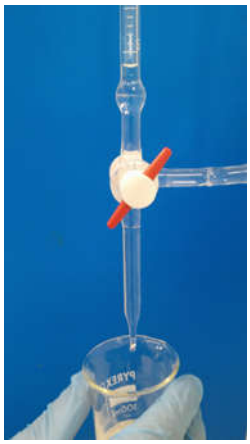
2		Place loaded, Vessel into the Vessel Holder
		Dry carefully the upper flange of the reactor with paper
3		Place Silicon Gasket onto the ground-glass lip of the Vessel
4		Place Vessel Lid centred onto the top of the Silicon Gasket

5		Place Top Sealing Ring onto the Vessel Lid
6		Use the four Poly Tightening Nuts to Secure the Top and Bottom Ring together which seals the Lid to the Vessel • Do not deform ring during this step Hand tighten to slight tension; tighten across, not in a circle
7		Install the Cavity Positioning Plate into the back to the Ethos X cavity
8		Place assembled Vessel into the cavity of the Ethos X
9		Centre the holder into the positioning plate.

10		Place assembled Vessel into the cavity of the Ethos X
		Screw the PTFE holder on the ETHOS X
11		Connect the glass joint to the glass reactor through the hole, placed on the top of the microwave cavity
12		Screw to hold the glass joint

13		Connect the distillation module together with the glass extension tube to the glass joint and fasten it properly to the external glassware support
14		Cover the condenser with the glass stopper
15		Connect the condenser to the connector tube
16		Place the condenser on the top of the distillation module and connect the two condensers using the connector tube

17		Connect the inlet and outlet chiller tubes
18		Set the Chiller at 8°C
19		Fill up the distillation module with water and insert the lass connector for distillation module
20		Set one of the following method the following method and press start

21		At the end of the extraction, collect the essential oil from the distillation module according to the picture

5.1.4 Fragrances for Ethos UP, Ethos Easy and flexiWAVE

Fragrances configuration is compatible with Ethos UP, Ethos Easy and flexiWAVE both in 660 and 480 versions.
When the fragrances configuration is installed by the local supplier, the software will be updated with all the setting to run the fragrances configuration.

Ethos UP, Ethos Easy and flexiWAVE are generally equipped with turntable adapter. Before to install the fragrances kit remove the turntable adapter as reported in the pictures below.

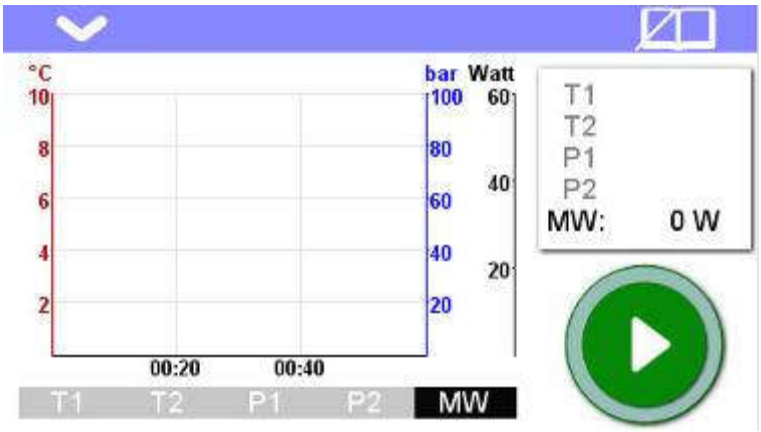
6 EASYCONTROL (T480)

Program start

Switching on the unit, also the terminal is turned on and the program is initialized.

There are different levels of access authorization that are described in the following program description.

Display during start procedure



Start screen

The green button in the lower right part of the screen is to start the method. By clicking the right thumbnails, they will be enlarged and displayed on the centre of the screen.

At time of the unit shipment, the password represented here is assigned.

The password can be changed in the Control Panel.

Administrator:	123456

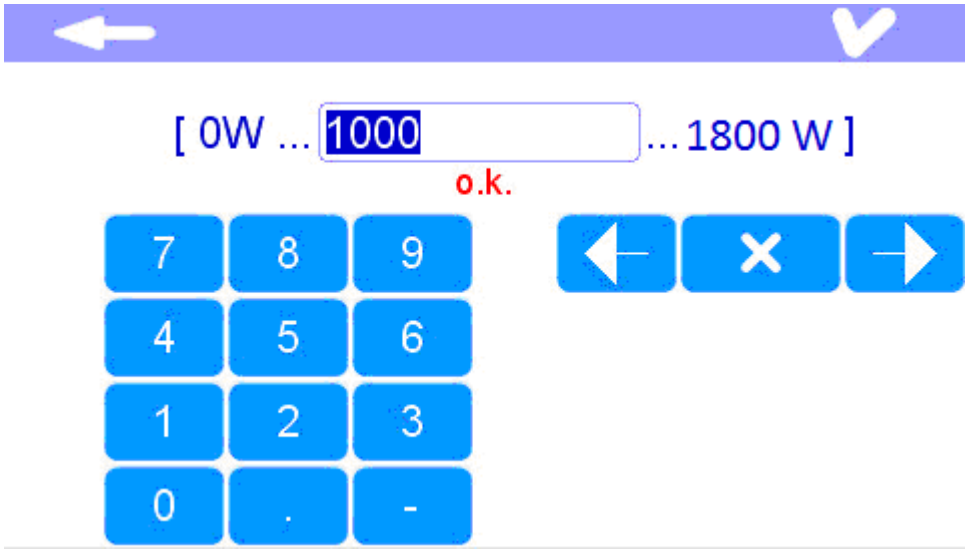
General operations tip

The software operations are carried out by a touch-screen. By touching the screen, the icons or the input fields are activated.

Activated fields are ready for input via a keyboard. By touching again the activated field, a numeric or standard keyboard appears on the screen.

6.1.1 Numeric input via touch-screen

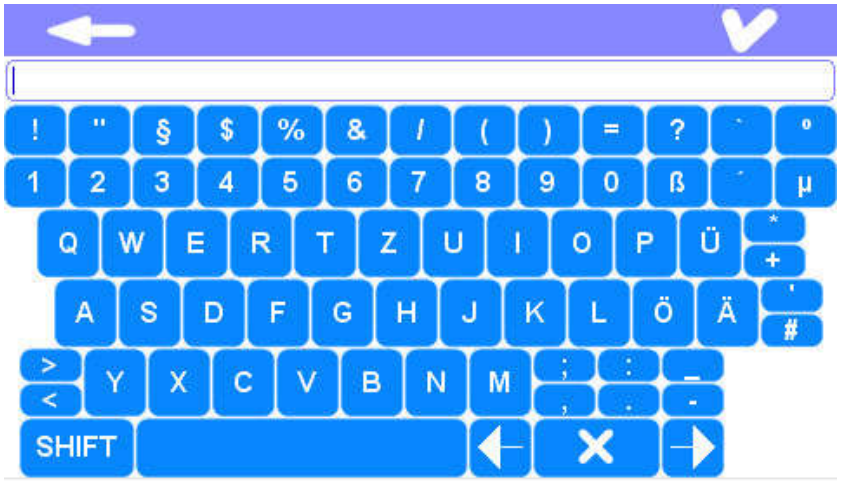
In several steps of the program it is necessary to enter numbers. Touching twice on the field or on the number to be changed, a numeric keyboard will appear



Numeric keyboard

6.1.2 Text input via touch-screen

For steps requiring a designation/description, a standard keyboard will appear by double-touching the touch-screen.

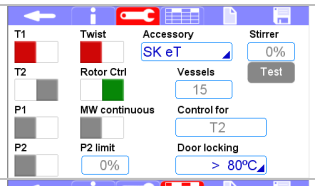
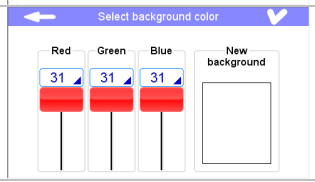


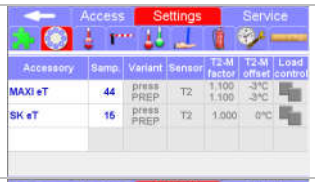
Standard keyboard

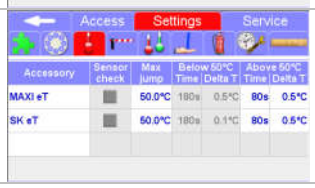
Generally used icons

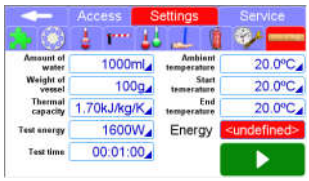
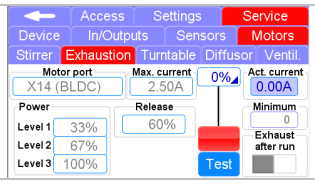
	General Menu
	Methods menu
	Select - OK
	Delete - Cancel
	Save
	New
	Back to previous page
	START button

Editable fields with numeric values and defined range

	• P2 limit: % • Door locking: °C
	• t (time): hh:mm:ss • MW (power): W • T2 (temperature 2): °C • P1 (pressure optional): bar • Cooling:min
	• Number of vessels loaded on the rotor plate: Number of vessels
  	• Background ○ Red ○ Green ○ Blue

	• Number of positions on the rotor plate: Samp.

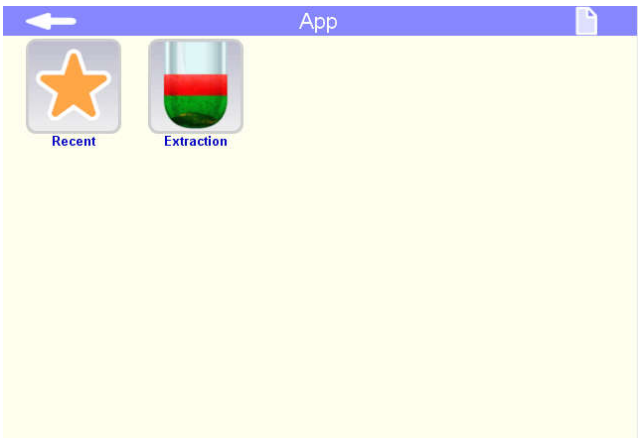
	• Max jump: °C
	• Above 50°C:
	  ○ Time: s
	  ○ Delta T: °C

	• Amount of water: ml • Ambient temperature: °C • Weight of vessel: g • Thermal capacity: kJ/Kg/K • Start temperature: °C • End temperature: °C • Test energy: W • Test time
	• Exhaust fan speed: %

Methods

6.1.3 Method selection from Milestone database

By touching the upper right icon, you can enter into the selecting menu of the applications. In each category, you can find a series of pre-installed methods.



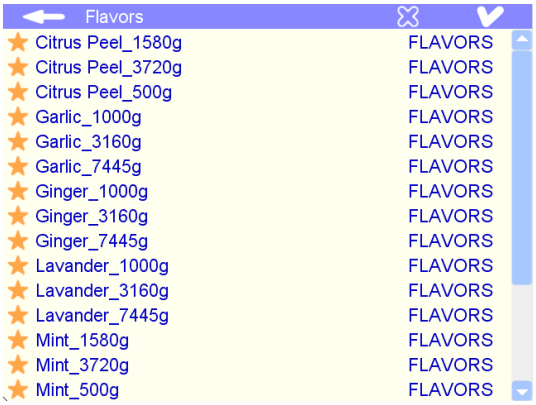
Applications

Categories

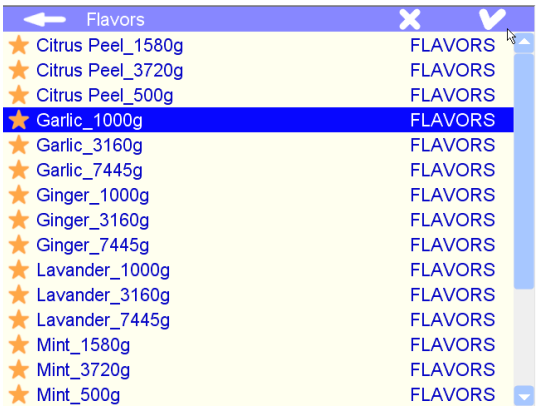
You require the Administrator access to open the categories (except for Recent).

Log in with your administration password.

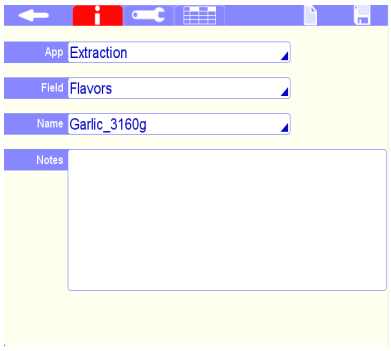
Here you can open and run several methods. The methods are optimized for each sample weight. For this reason, there are three different methods for each sample weight, which correspond to the three glass reactor sizes (Small, Medium and Large).

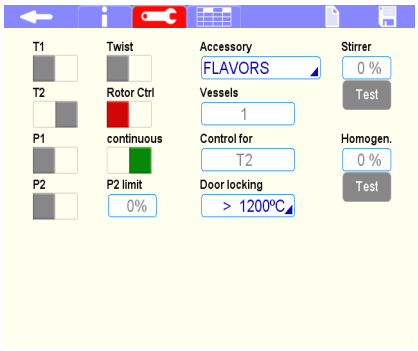


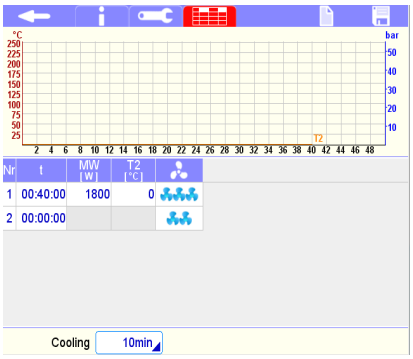
Select a method and click the upper right icon (check mark).



In the following registers, there are all method information and settings.





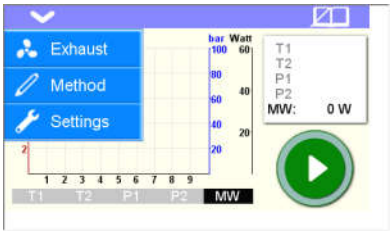


General information Setting Parameter

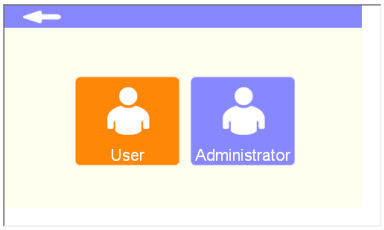
By clicking again the check mark, you can open and run the method.

6.1.4 Create new methods

Press the upper left check mark and select the icon methods.



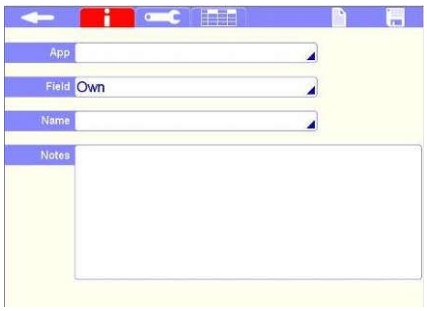
Log in as Administrator, in order to create a new method.

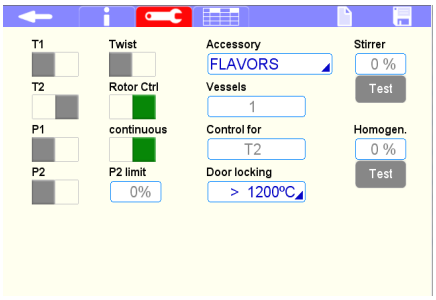


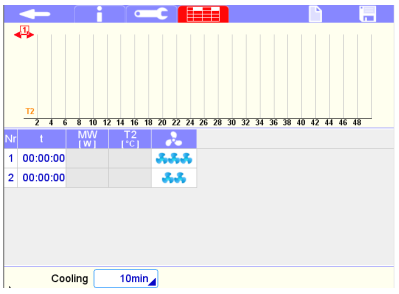
Log in with your Administrator password.

To create a new method, click on the icon shown in the upper right side of the screen.

In the following registers, there are all method information and settings.







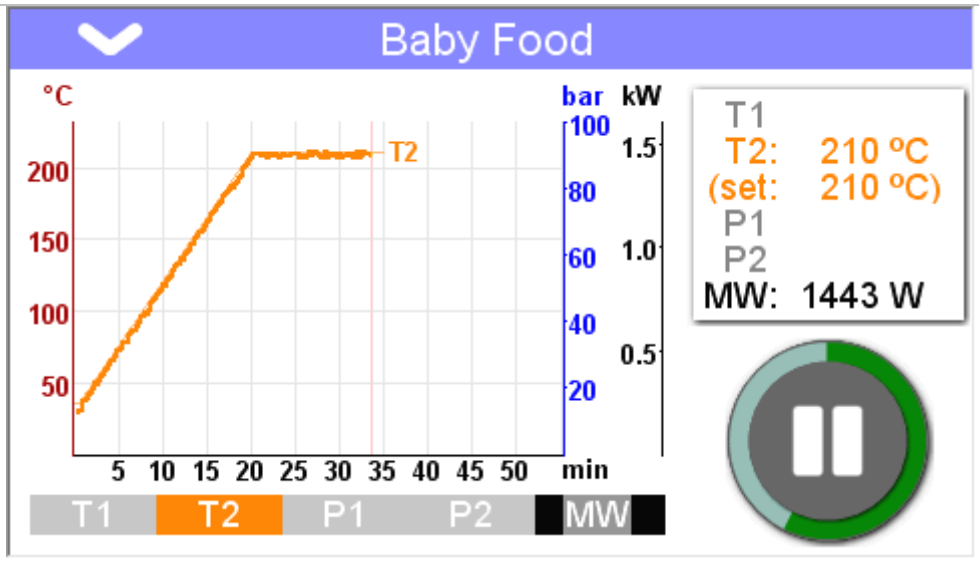
General information Setting Parameter

Many methods for fragrances require to work at constant power. In this case edit 0 in the T2 column and the desired time and power according to your development or to the application notes.

During the MW-cycle, the software records all data of control parameters. The gradients of temperature, pressure and power are displayed graphically in real time on graph.

The recorded curves of temperature (and pressure) appear in darker of the given curves.

After the program, the graph can be printed or saved on the USB pen.



Program sequence

PAUSE

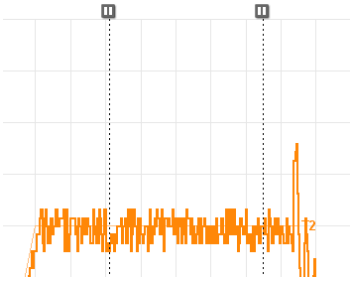
While the program is running, the pause icon appears to the right.

With the pause button the process can be interrupted (the timer is stopped and the microwave irradiation is stopped).

During the pause, the button changes to a split view. Now you can continue or stop the program.

In the graph, a marker appears from the point of interruption (at the top of the graph at the position where it was interrupted).

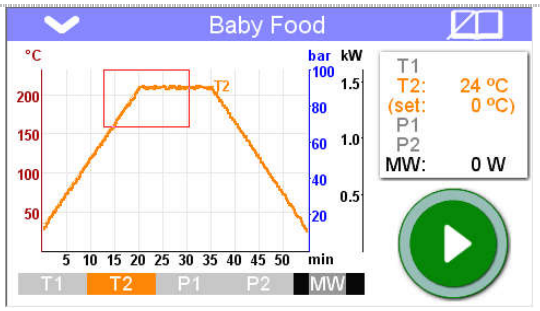
The opening of the chamber door has the same effect as pressing the pause button.



ZOOM

Touch the screen from the top left corner to the bottom right corner and you remain in the final position for about half a second before releasing.

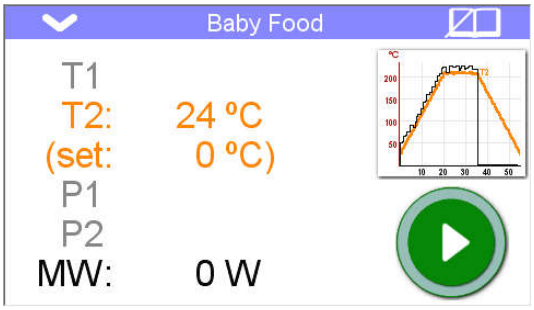
The section will be zoomed on screen



ZOOM-OUT

Below the graph the "Zoom Out" button appears. Tap this button to zoom out.

It's possible to follow the run selecting the window. Here there are all details about the actual internal temperature (set point and readed temperature), external temperature, pressure, microwave energy.



6.1.5 Graphical representation

The individual graphs can be displayed or hidden from the screen by pressing the corresponding button.

T1, RED

The curves (setpoint curve and measured curve) for the ATC (thermocouple) sensor, internal temperature.

T2, ORANGE

The curves (setpoint curve and measured curve) for the easyTEMP / Infrared temperature.

P, BLU

Curve of internal pressure

E, BLACK

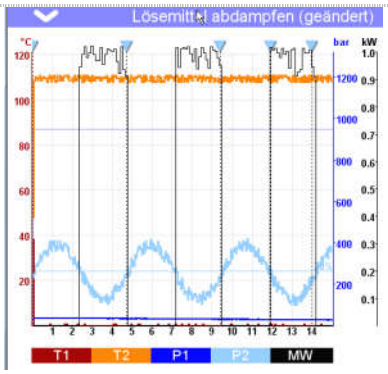
Curve of the Microwave emission

P2, LIGHT BLU

Curve of P2 (QP sensor) that detect venting of the vessels by detecting the amount of NOx or Cl_2 in the cavity.

SELECTION OF P2

When this button is activated, marks appears on graphic, if the P2-limit is exceeded. When pressing the button again, the P2 signal is shown as a line.



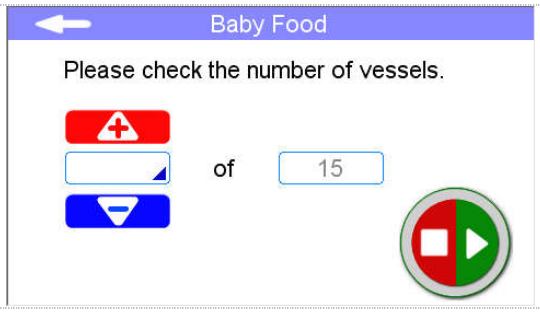
6.1.6 Load control function

For rotors which are directly controlled by the easyTEMP, the load control function is activated.

This function regulates the supplied microwave power depending on the number of vessels in the rotor to avoid temperature overshooting during the run.

According to the rotor configuration, when you start the digestion program, the software asks to indicate the number of vessels that you are using.

With a MAXI-44 rotor, a minimum of 10 vessels must be indicated to have a better functionality of the system.



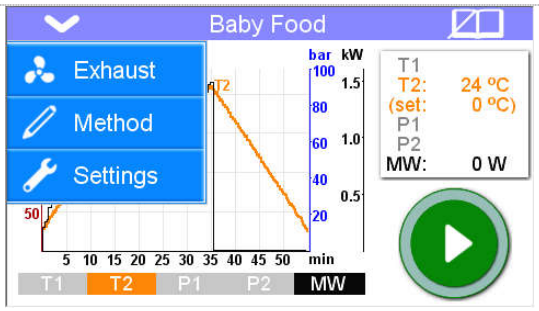
Setup

This part of software is dedicated for the setup of the system and only the Service login profile can change all parameters.

Administrator and Manager can change few parameters form this page.

To enter the Setup menu, click on the upper left icon of the screen and then on the settings icon in the menu.

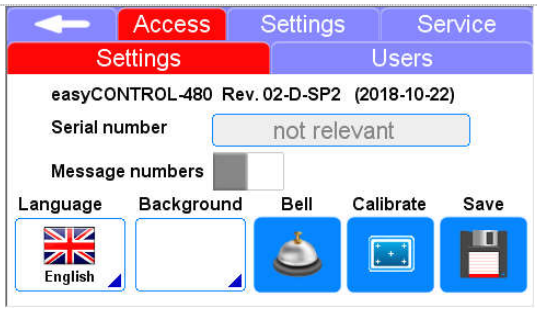
Here you have to log with the administrator password.



6.1.7 Access window

Settings

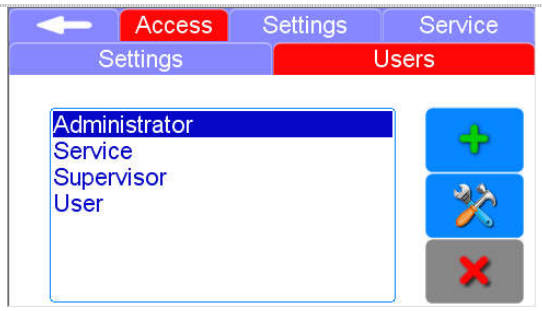
Here you can edit the general setting of the terminal.



Users

In this section you can :

- Create new users or administrators or edit existing ones;

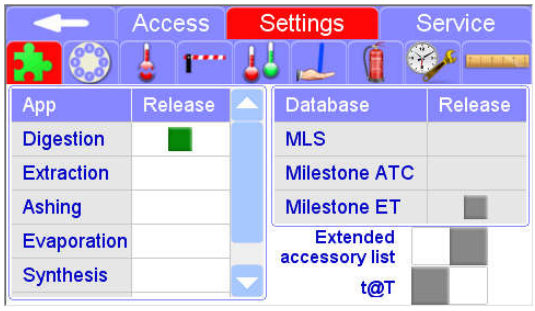


6.1.8 Settings window

In the following sections you can edit the system settings. The shaded settings can only be changed by the service. The Printer icon allows to print the setting.

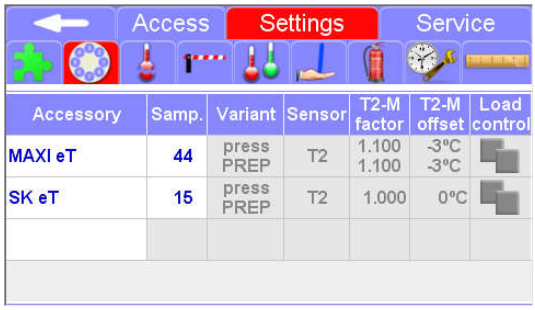
Applications

Here you have the possibility to select the application library according with your configuration.



Rotor

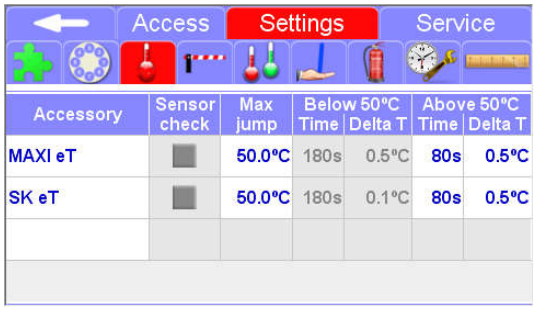
In this section you can set the number of vessels for each rotor type or add new rotor types.



Sensor Check

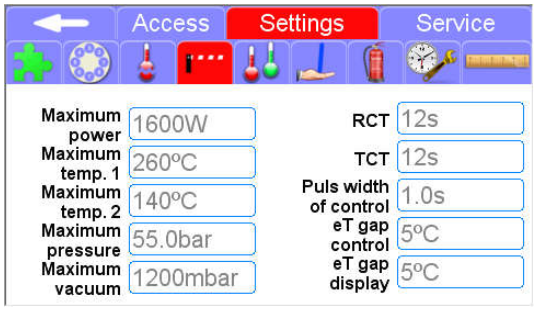
In this section, for each rotor type it is possible to set a different temperature and time range of the sensor check alarm.

Contact your service engineer for more details about these values.



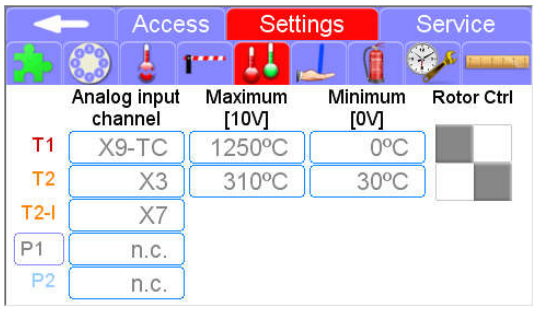
Maximum working conditions

These limits can only be set by Service.



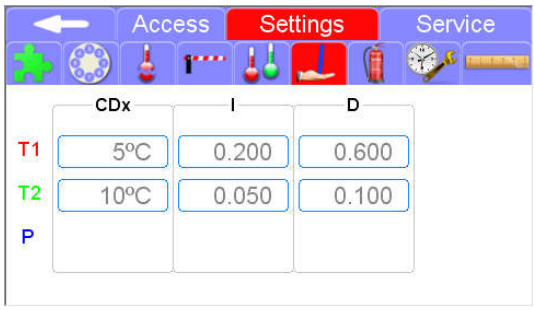
Sensor settings

These settings can only be edited by Service



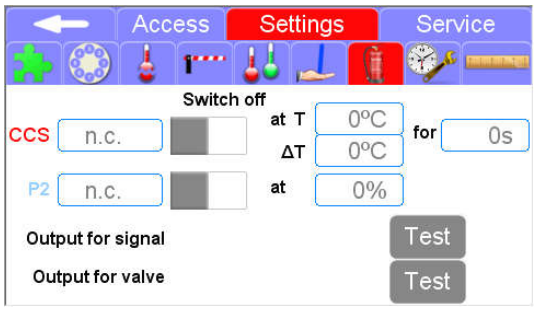
Control settings

These settings can only be edited by Service



Cavity control system (CCS)

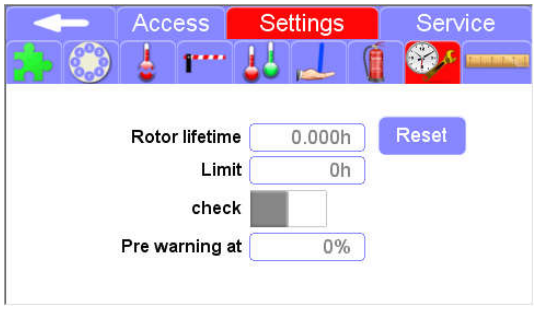
The settings of the cavity control system (CCS) can only be edited by Service.



Rotor lifetime

Here is an automatic countdown of the number of digestion cycles. at the end of the number of cycles, a message will appear to remind you to replace the consumable parts of the rotor.

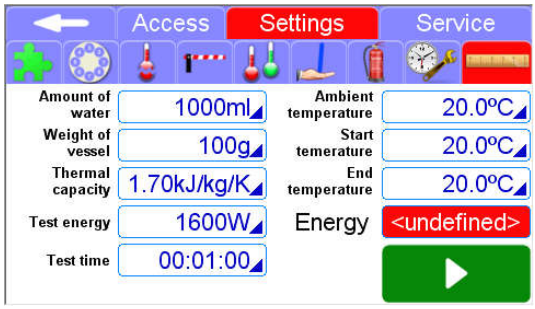
The administrator can at any time reset the countdown.



MW energy test

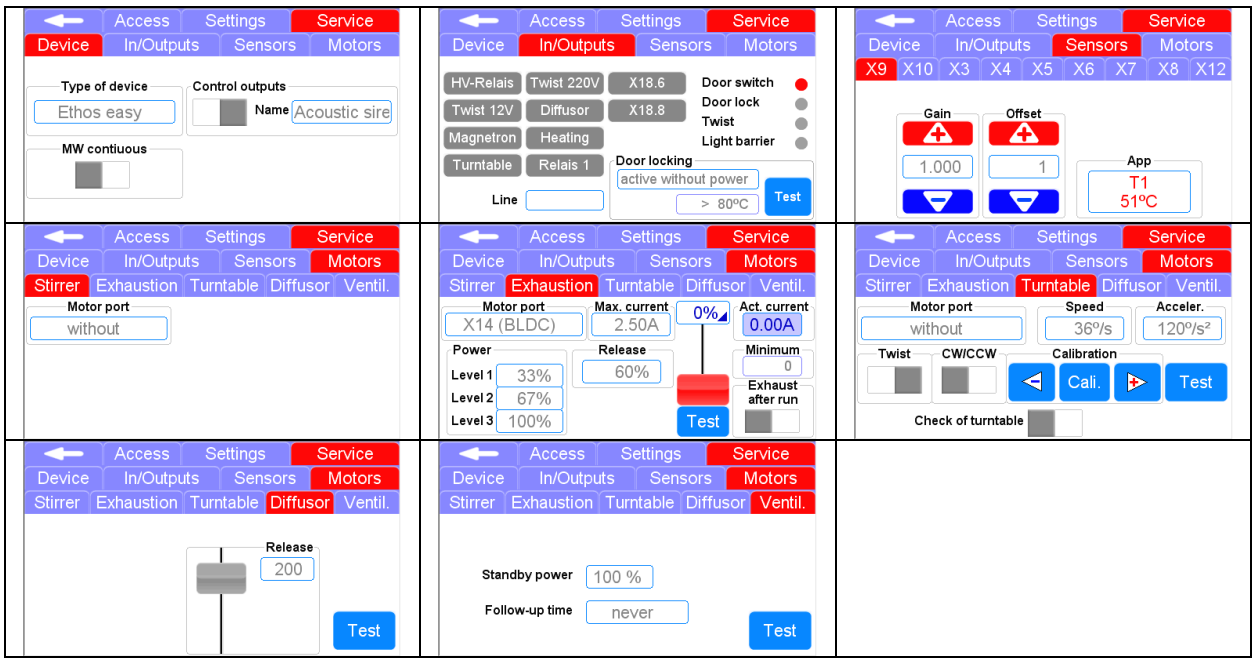
This window is for the microwave energy test.

Contact your service engineer for more details about the test.



6.1.9 Setup window

The below parameters can be modified only by a Milestone service engineer.

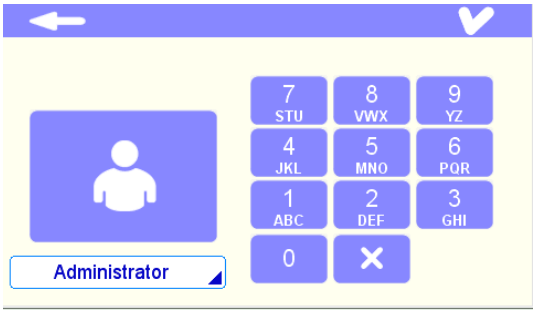


Create, Edit, Delete a User profile

6.1.10 First Steps

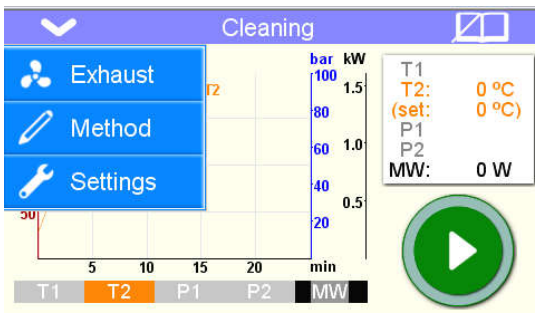
When the unit is turned on, the system will ask for the first access.

The first login MUST be done by the administrator of the lab and the default password is 123456.

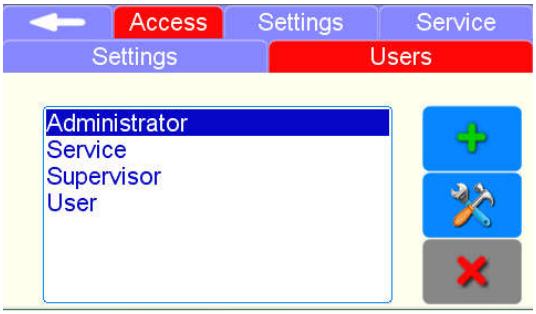


The default password MUST be changed after the first login. It's responsibility of administrator to change the default password with a new one. See the next chapters for more details on how to change the password.

Select the arrow at the top left of the screen and open the settings page.



Select the Terminal > Users window in order to get access to the Users list. From this page it is possible to Create, Edit or Delete a user profile.

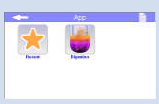


6.1.11 Overview of user profiles and access levels

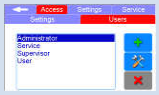
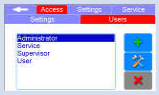
From the Users window, you can create a new login name. There are three different types of users with different levels of accessibility (privileges) that can be created.

Login type	General Description	Access level
User	Can only open files selected by the administrator. The user cannot modify any parameter of the method.	Low
Manager	Can create User and Manager profiles. Can open and modify the methods.	Medium
Administrator	Can create User, Manager and Administrator profiles. Can open and modify the methods. It have access to the time and data of the software and other technical options.	High

Here below a table with more details concerning the privileges according with the login type

	Open and Run a method from the Recent folder	Open and run a method from thedigestion Library	Create a new Method	Modify existing methods (not the one from the Milestone library)	Create a new Administrator account
					
User	☑	☒	☒	☒	☒

Manager	☑	☑	☑	☑	☒
Administrator	☑	☑	☑	☑	☑

	Accessories Setup (Rotor configurations)	Power test window	Sensor check settings	Create a new Manager account	Create a new User account
					
User	☒	☒	☒	☒	☒
Manager	☒	☑	☒	☑	☑
Administrator	☑	☑	☑	☑	☑

There are two additional login levels that cannot be modified by the Administrator.

Login Type	General Description	Access level
Service	This access is only for the Milestone Service Engineer. It gives access to the sensor calibration and others technical feature of the unit.	High
Supervisor	This access is for Milestone Producer for software optimization (engineering, updates, etc.).	High

6.1.12 Create a new ID user profile

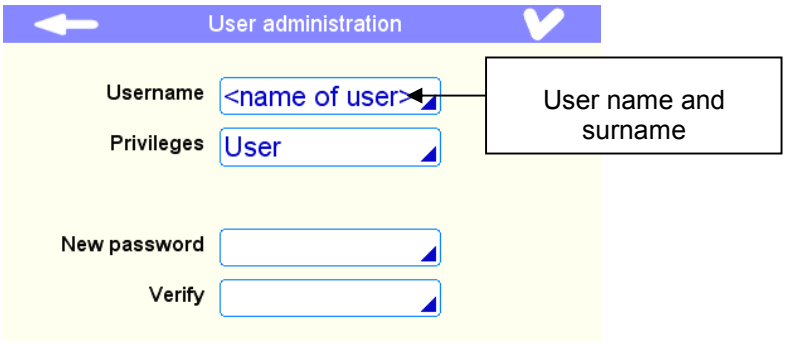
New ID profiles can be created by the Manager, Administrator, Service and Supervisor accounts.

Each user must have one single ID profile and each ID profile has to be associated to no more than one person.

It's responsibility of the user (with a Manager, Administrator, Service or Supervisor account) to guarantee the uniqueness of each account to every single person.

For the above reasons, Milestone suggests to:

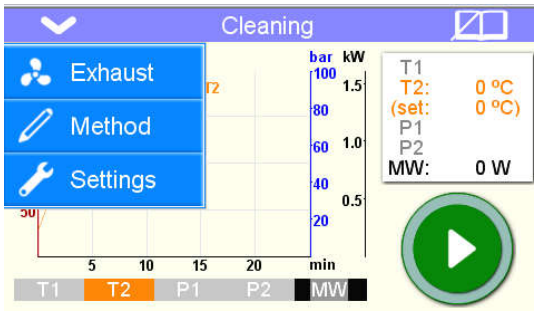
- Mandatorily use name and surname of each users as ID Username.



- Have a register (notebook or excel file) with list of all ID Username with the corresponding name and surname of users.

Here below all steps to create a new ID profile with the “CFR password” configuration activated.

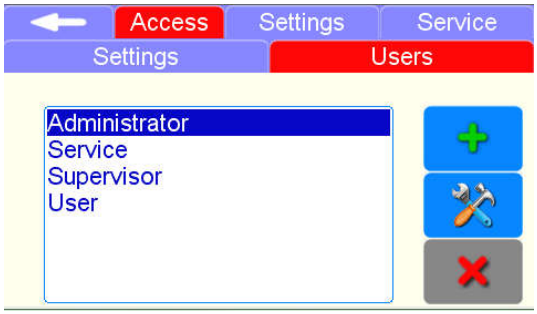
Open the scroll menu on the upper left side of the display and select “Settings”.



Select “Access” page and “Users” tab.

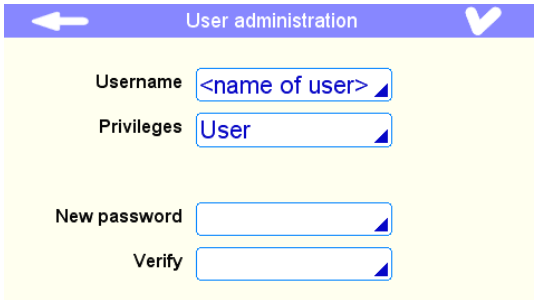
Create a new user changing the password.

Press to add new User

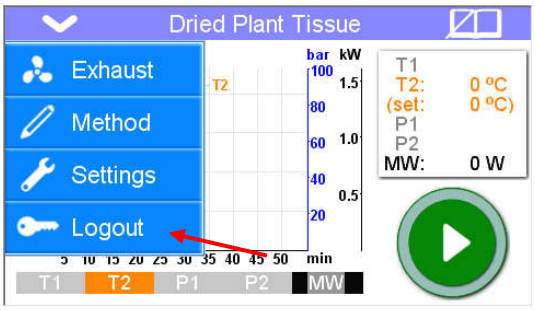


Insert Username, privileges and password.

The password must be at least 6 characters long

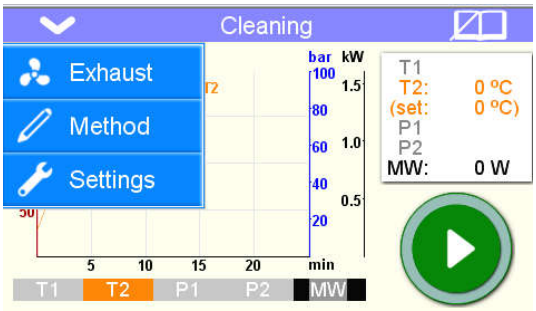


Go back to the Main page and log out



6.1.13 Edit or Delete an existing ID user profile

Open the scroll menu on the upper left side of the display and select “Settings”.

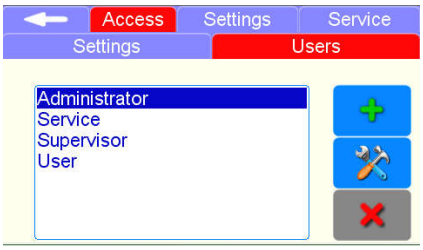


Select “Access” page and “Users” tab.

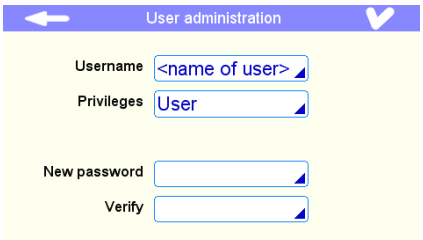
Select the user to edit or delete from the list.

1. Press to edit existing User

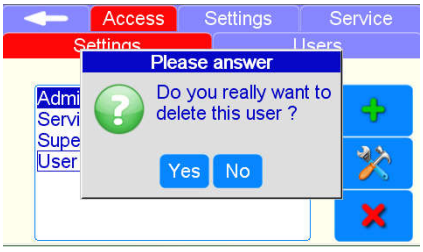
2. Press to delete existing User



Pressing the edit function you can change the privileges and the login password



Pressing the delete function a message will appear for confirmation. Press YES to remove the account.



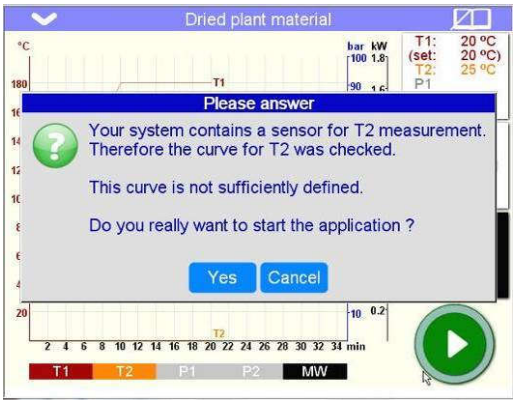
7 MESSAGES AND INQUIRES

Security query

To avoid accidental modifications to the method, the system performs security queries.

If the user starts a program which is not entitled or is not possible, a message appears.

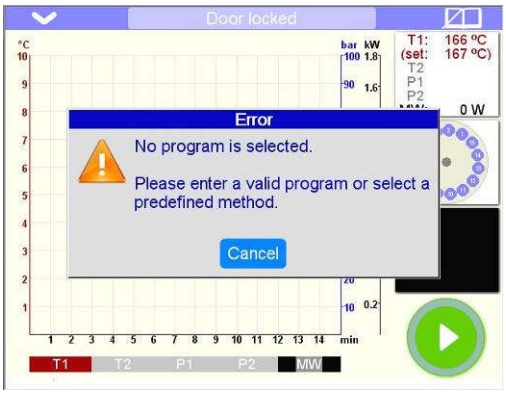
Messages are acknowledged by pressing the OK button.



Error messages

If an error is detected by the software, a window appears with the error message.

Error messages are acknowledged by pressing the Cancel button



List of queries and (error) messages

In this chapter, most queries and (error) messages and their meanings are listed. They are sorted by the following keywords:

- Login
- Sensors, Settings and Twist
- Program

7.1.1 Login

Text	Meaning/Comment
The password you entered is incorrect. Please repeat your entry or cancel the login.	The password you entered is invalid for the selected user.
You have no permissions for this device. Please contact your administrator.	The device has the login mode "with disk". The signature of the disk is part of a user who has no access to this device.

The registered name already exists. No name can be used twice. Choose a different name.	The name of the user must be different.

7.1.2 Sensors, Settings and Twist

Text	Meaning/Comment
In your system no sensor inputs are configured. Please contact your local service partner.	There are no configured sensors. The configuration of the sensors can be made only by the service (see Chapter sensor data).
Your system contains a sensor for pressure measurement. Therefore the pressure curve was checked. This curve is not sufficiently defined. Do you really want to start the application ?	The curve of a sensor is not completely defined. During the program the measuring of this parameter is not verified. This can lead to uncontrolled power output (see chapter description of the program parameters).
The control of the temperature sensors checked an error. The value of T1 (T2) has changed in one second of 25.0 ° C to 100.0 ° C and thus exceeded the limit of 50.0 ° C.	The sensor check has detected an error in the temperature measurement (see chapter Sensor Check). This message depends on the actual and nominal values and can vary according to the appearance. Check the temperature sensor and the placement of the sample (infrared sensor).
You have not entered a value for the element "Control for". Please select one of the available sensor inputs.	You do not have defined the parameter according to which the power has to be controlled. In the register Method \ Parameters you must specify which parameters you want to control (see Section parameters).
Twist function will be disabled. Temperature and pressure sensors can be damaged. Are you sure to deactivate the twist function ?	Security check when disabling the twist function (see also Chapter Twist)
Twist error A twist error occurred. Check the twist mode of your system.	No change of direction of rotation is detected by the system. Check the twist function or contact service.

7.1.3 Program

Text	Meaning / Comment
You have already executed a microwave application without storing these data. Do you want to ignore the recordings and start a new program ?	You have not saved the program file. During a restart, all unsaved data of the last run will be lost. The program file can be stored in the sequence register.
The program time of all segments is zero. Please specify a valid program or select a predefined method.	You have not yet defined a program. In the register method \ program allows you to create a program or load from disk (see Chapter program).
Your microwave program is longer than 2 hours. Is it correct ?	In all applications, an examination of the length of the program takes place. With a length of more than 2 hours, this prompt appears. The program is not limited to 2 hours.
You have already run a microwave program without saving the data. By leaving this page, all the data will be lost.	You have not saved the program file. When you exit the application pages all unsaved data of the last run will be lost. The program file can be stored in the sequence register.

Text	Meaning / Comment
Would you really want to exit this page?	
You have already executed a microwave application without storing these data. Do you want to ignore the recordings and load an old graphic file ?	The method and sample table are stored with the program file. When loading a sequence, the current method, sample table and chart the course will be overwritten.

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