# Series 340 prepASH 219, 229, 212

# Fully automatic ash determination and moisture analysis system



Serial Number S/N: Installation Date:

350-8127-000a15



# **Table of contents**

1	Introduction	
1.1	General Information about the Operating Instructions	
1.2	Representation and Symbols	
1.3	Safety instructions	4
2	Description of the Device	5
2.1	Proper Use.	
2.2	Structure and Function	
2.2.1	Structure of the Ashing Device	
2.2.2	Functions of the Ashing Device	
2.3	Technical data	
2.4	Controls and connections	
2.4.1	Front controls	
2.4.2	Rear controls and connections	8
2.5	Chamber	9
3	Commissioning	10
3.1	Unpacking	
3.2	Scope of delivery	
3.3	Choosing a suitable location	
3.4	Assembly and Connection	
3.5	Balance calibration.	
3.6	Gas Adjustment	
3.7	Mechanical calibration	
3.8	Inserting the turn-table	
3.9	Crucibles	
<b>4</b> 4.1	Software: First Steps	
4.1	Program Start	
4.2.1	Buttons	
4.2.1	Numerical Entry.	
4.2.3	Text entry	
4.2.4	External Keyboard / Barcode Reader	
4.3	prepASH User Interface	
4.3.1	Display bar	
	* *	
5	Create/Edit method	
5.1	Create a new method	
5.1.1	Parameter selection	
5.1.2	Calculation of losses and residues	
5.2	Events on completion of the program	
5.3	Edit method	
5.4	Method wizard	21
6	Program execution	
6.1	Analysis execution / Flow Chart	
6.2	Filename to store the data of analysis	24
6.3	Choose the method	
6.4	Sample list	
6.4.1	Tare mode	
6.4.2	Sample density	
6.4.3	Sample list	
6.4.4	Create a sample list	
6.4.5	Open an existing sample list	
6.5	Weighing in in prepASH and START	
6.6	Start after weighing in prepSTATION	
6.7	During the measurement	
6.8	Program end	
7	Calculation	36
8	Data transfer and prepDATA	37
J		
9	Control Buttons	38

10	SERVICE MODE / Configuration	41
10.1	Info 41	
10.2	prepASH ID	
10.3 10.3.1	prepASH Type	
10.3.1	Density correction	
10.3.2	Disk 43	42
10.5	User account	44
10.6	Gas adjustment	
10.7	Balance calibration.	
10.8	Mechanical calibration	
10.9	Temperature calibration and adjustment	
10.9.1	Temperature calibration	
10.9.2	Temperature adjustment	
10.10	Erase file	
10.11	Quit / access to windows	49
11	Error messages	50
11.1	Error 1: Interface board-Reset	
11.2	Error 2: Communication-error between Touch panel and Interface board	
11.3	Error 3: Balance error	
11.4	Error 4: Communication-error between Touch panel and Temperature-Module (7018)	
11.5	Error 5: Communication-error between Touch panel and Flow-Module(7017)	50
12	Integration into your IT environment	51
12.1	Installation of a printer on prepASH	51
12.2	Change the prepASH name of the domain	
12.3	Access to prepASH via network, LIMS connection	51
13	prepASH Software update	52
13.1	New prepASH software installation	
<b>14</b> 14.1	Maintenance and Service	
14.1	Housing Sample chamber	
14.3	Ventilation	
15	Appendix A: Carbon application	
15.1	Method	
15.2 15.3	Open method	
15.4	Edit method	
15.4.1	Step 1 + 2 (Moisture)	
15.4.2		
15.4.3	Step 5/6 (Ash)	
15.4.4	Additional	
15.5	Choose the method for the run	
15.6	During measurement	
15.7	Calibration	
15.7.1	Volatile correction	
15.8	Results	
15.9 15.9.1	Calculation Results	
13.9.1		
16	Appendix B: Scrubber application	
16.1	Scrubber connection	
16.2	Scrubber operation	64
17	Appendix C: Uninterruptible Power Supply UPS	64
18	Appendix D: Practical recommendations	65
18.1	Correct sample preparation	65
18.2	Optimization of result accuracy and work with very low ash content	65
18.3	Correct Crucibles handling & Weighing in	
18.4	Adapting to the current test procedure	
18.5	Possible causes of not accurate results and solutions	66
19	Accessories and Consumables	66

#### 1 Introduction

# 1.1 General Information about the Operating Instructions

The prepASH ashing and drying device is simple to use. Please read the operation manual carefully and thoroughly in order to understand and make the most optimum use of the many features and possibilities offered by prepASH in your daily routine.

This operation manual contains reproductions of the screen and switch surface icons, which are intended to make it easier to find information.

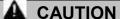
Dangers and notices are indicated in Chapter 1.2 "Representation and Symbols"

# 1.2 Representation and Symbols

Important safety-related instructions are highlighted visually at the appropriate points below:



Warning of a possible danger which may lead to death or to serious injury



Warning of a possible danger which may lead to minor injury or damage



Tips and important rules on how to use the ashing unit correctly.

# 1.3 Safety instructions

- When using the ashing device in surroundings with increased safety requirements, pay careful attention to the appropriate regulations.
- Only set it up in a room that is adequately spacious, dry and well ventilated.
- Only operate the device upright on its stand and on a stable, level base.
- Atmospheric contamination may only be non-conductive. Short-term conductivity caused by condensation may only arise occasionally.
- The device may not be operated or stored in a corrosive or explosive atmosphere (dust, steam, gas).
- Never operate the device with a faulty mains lead.
- Never lay the mains cable over sharp corners and edges or hot surfaces.
- If there is any reason to believe that it is no longer possible to operate the device safely, unplug it immediately from the electricity supply (pull out the mains cable) and secure it so that it cannot be operated inadvertently.
- During operation and maintenance work, take particular care with hot parts, in particular in the area of the furnace chamber (risk of burning).
- When the furnace chamber is opened whilst the machine is in operation or after the end of the trial, very hot air will escape, and the furnace chamber will radiate intense heat.
- Do not place or stand objects of any kind on the lid of the ashing device. Do not subject the lid to any mechanical pressure. Heat must be allowed to radiate freely from the machine during operation.
- Sample type: do not ash any explosive samples.
- Have an electrician check that the entire electrical system is working properly before the ashing
  device is first commissioned, after maintenance and servicing work on the electrical system and
  at intervals of at least every four years.

# 2 Description of the Device

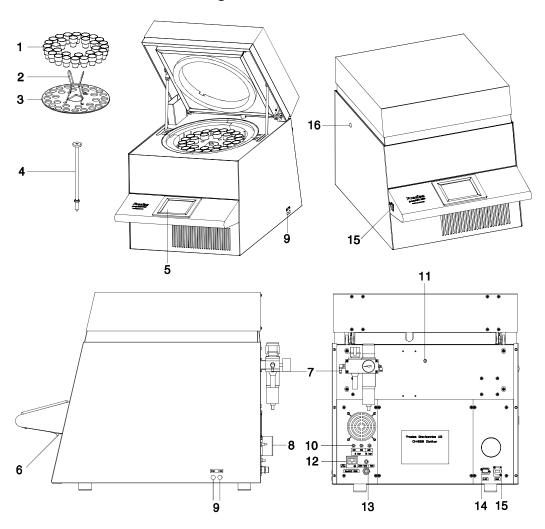
### 2.1 Proper Use

The ashing device should only be used for weighing, drying and ashing of solid materials and liquids. The max. Permissible load of the integrated balance should never be exceeded; otherwise the internal balance may be damaged.

If the ashing device is used in combination with other Precisa devices or with other makes of devices, it is imperative that you follow the instructions for the safe and proper use of the auxiliary devices.

#### 2.2 Structure and Function

#### 2.2.1 Structure of the Ashing Device



#### No. Description

- 1 Crucible
- 2 Plate holder
- 3 Turn table
- 4 Balance pan
- 5 Touch screen
- 6 Master switch
- 7 Maintenance unit with a compressed air connection
- 8 Exhaust gas nozzle

#### No. Description

- 9 Process gas flow control
- 10 Process gas and compressed air connections
- 11 Inlet for external temperature sensor
- 12 Electrical power outlet for connection of a scrubber or pump
- 13 Mains connection cable with connector CEE type 5pole male
- 14 LAN port
- 15 USB port
- 16 Mechanical connection for scrubber

#### 2.2.2 Functions of the Ashing Device

The prepASH ashing device is simple to operate. It facilitates the quick and reliable drying / ashing of liquid, porous or solid materials. The device works using thermo gravimetric principle.

The most important basic features of the ashing device include:

- Automatic drying, ashing and weighing of up to 29 samples.
- Simultaneous measuring and logging of all samples throughout the entire measurement procedure.
- Automatic compensation for lift.
- Integrated PC with touch screen for simple operation and clear display.
- Temperature range of 50 °C to 1000 °C.
- Access authorisation according to FDA21 part 11.

#### 2.3 Technical data

#### Mains connection

```
- Voltage: 230 VAC (+15/-20 %);
```

- Current: 25 A
- Frequency: 50 to 60 Hz
- Connector: CEEtyp, 5pole, 32 A, male (only N, L1 and PE connected)

#### Power consumption

- 5500 VA (3300VA, special model)

#### Balance system

- Weighing range: 120 g
- Resolution: 0.0001 g
- Unit: g
- Number of samples:

```
prepASH 219: 0 - 19
```

prepASH 229: 0 - 29

prepASH 212: 0 - 12

#### Heating system

- Ceramic infrared radiator
- Temperature range: 50 °C 1000 °C
- Temperature tolerance: <= +/- (1% + 2 °C)
- Temperature curves are individually configurable
- Auto stop: 0.1-100 (freely definable) mg/10min, mg/30min, mg/60min

#### Results

- Weight loss in %, ‰, g
- Weight loss to the pre-interval in %, ‰, g
- Residual weight in %, ‰, g
- Residual weight to the pre-interval in %, %, g

#### Monitoring

- On-line monitoring on your PC through ethernet

#### Gas requirements

- Oxygen: 99.5 % purity, 3 bar ( $\pm$  10 %), 0 9 l / min
- Nitrogen: 99.5 % purity, 3 bar (± 10 %), 0 9 l / min
- Compressed air, oil-free and without condensation, 6 bar (± 10 %)

#### Furnace atmosphere

- Oxygen, nitrogen or compressed air
- Flow rate is measured electronically

#### Interfaces

- USB ports for printer, USB stick, Barcode scanner or key board
- Ethernet (LAN) for online monitoring on a PC

#### Data outputs Data processing

- Direct print out at prepASH possible
- Data processing and report creation via prepDATA on your PC

#### Operation and display

- Color touch screen

- Menu-guided operation
- Language undepening icons

#### Printout via an optional printer

- Graphics
- Method
- Results and Statistics

#### • Weight and dimensions

- Weight 100 kg
- Dimensions H(H)xWxD / 620(980)x590x830 mm (H) Height with the sample chamber open

#### • Requisite footprint

- WxD / 590x830 mm (Requires a margin of 15 cm for safety)

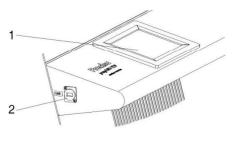
#### • Permissible ambient conditions

- Temperature: 5 °C 35 °C
- Relative humidity: 25 % 85 %, non-condensing

If you have questions regarding the technical data or requires detailed technical information about your ashing device, contact your local Precisa agent.

#### 2.4 Controls and connections

#### 2.4.1 Front controls



**1, 2** The touch screen (1) enables simple, menuguided control. All device settings can be changed easily via the touch screen display. USB port for key board, stick or barcode reader (2)



**3** The master switch is located on the underside of the control panel (3).

#### 2.4.2 Rear controls and connections

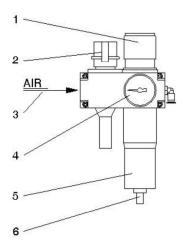
# ! DANGER

The ashing device is operated from a 230 V AC supply. The electrical wiring work should be carried out by a qualified electrician.

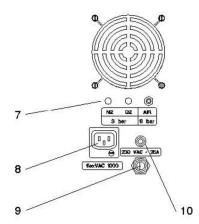
The exhaust gas hose must be fed outdoors in order to discharge harmful waste gases from the furnace chamber safely.

# **A** CAUTION

The pneumatic system for the prepASH must not be operated on pure oxygen (danger of a strong reaction between oxygen and oil).



- **1, 4** Use the controller (1) and the pressure gauge (4) to adjust the pressure in line with the pneumatic supply to the device.
- **2** The pneumatic system can be vented or disconnected from the compressed air network via the switch-on valve (2).
- **3** The oil-free compressed air 6bar +/-10% must be connected to the maintenance unit (3). You are advised to use a compressed-air hose with an internal diameter of 6 mm.
- **5, 6** Check the filter (5) at regular intervals for dirt and condensation. The filter must be emptied manually to ensure that no condensation enters the device (6).



**7** The process gases can be fed in via the terminal connections (7) with hoses (DxTh. / 4x0.75mm). The compressed air inlet (AIR) is connected to the maintenance unit in the factory.

**CAUTION** working with oxygen and nitrogen.

- **8** Electrical power outlet for connection of a scrubber (e.g. Büchi B-414) or pump
- ${f 9,\,10}$  mains cable 2 m with connector CEEtyp 5pole male whit only N, L1 and PE connected.

The miniature circuit breaker (10) causes the device to cut out in the event of an overload. Once the fault has been investigated and rectified, the miniature circuit breaker can be pushed back in.

- **11** The supplied hose is fitted to the exhaust gas nozzle (11) using the clip, and the other end is fed into an exhaust system.
- 12
- **12** LAN-connection (12) **13** USB-connection (13)

#### 2.5 Chamber

# **A** CAUTION

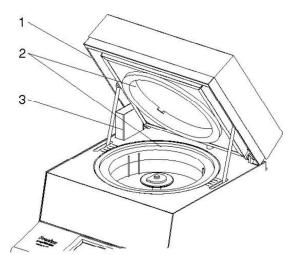
The parts inside the furnace chamber and the metal covers will become hot. Take care when opening and loading the furnace chamber.

Always use the supplied holder for removing the sample plate from the device. If you are removing individual crucibles, always use the supplied tongs (this will also help you to avoid rogue measurements).



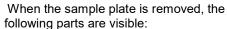
Handle the sample plate, crucible and balance pan with care. These parts are made from ceramic material and can be damaged easily.

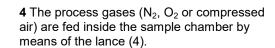
Take care not to damage the insulation when you load the device.

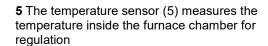


- **1** The circlip (1) prevents jamming when the lid is closed.
- **2** The entire furnace chamber is lined with insulating inserts (2).
- 3 The suction device (3) is in the back left.

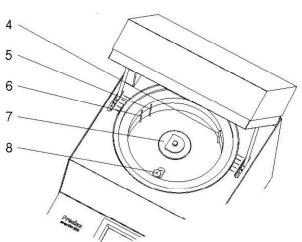
**CAUTION:** Watch out for hot parts when you are handling the device.







- **6** The temperature senor (6) protects from overheating in case of failure or wrong calibration of the regulating sensor.
- 7 The sample plate sits on the rotary axle (7).
- **8** The balance pan (8) is inserted into a protective pipe.

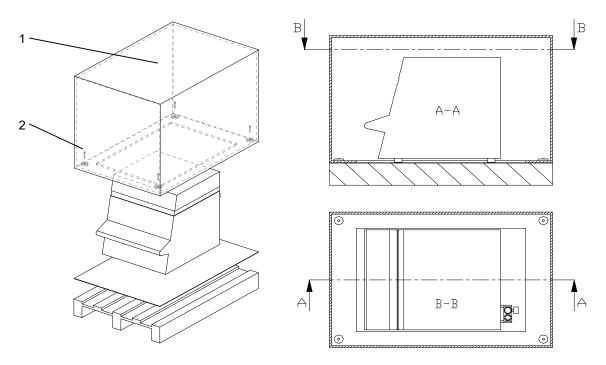


# 3 Commissioning

# 3.1 Unpacking

Observe the following points when you unpack the ashing device in order to avoid damage:

- Check the ashing device for any external visible signs of damage immediately after you unpack it. If you find that it has been damaged in transit, notify your Precisa service agent immediately.
- Read these Operating Instructions carefully before starting to work with the ashing device and pay particular attention to the safety instructions (see section 1.3 "Safety instructions")



#### Unpacking the prepASH

- 1. Open the box (1) at the top
- 2. Remove all the padding and accessories
- 3. Undo the 4 screws (2) on the base of the cardboard box
- 4. Lift off the cardboard box



Retain the packaging. If the device is delivered to the factory by a transport firm for repair after a lengthy period of use, this may only be done in the original packaging. If devices are not properly packaged, they may sustain considerable damage in transit.

# 3.2 Scope of delivery

Once you have unpacked all the parts, check that the delivery is complete.

Designation	present yes / no
Ashing device	
Turn table	
35 crucibles for the prepASH 229	
25 crucibles for the prepASH 219	
15 crucibles for the prepASH 212	
Balance pan	
2 sample holders	
Crucible tongs	
Mains plug	
Exhaust gas hose	
Clip for the exhaust gas hose	

# 3.3 Choosing a suitable location

In order to ensure that your ashing device functions properly and safely, select a location that complies with the permissible ambient conditions (see section 2.3"Technical data" on page 6 and also fulfils the following criteria:

- The room in which it is installed must be adequately spacious (minimum room capacity of 10 m<sup>3</sup>)
  and must be well ventilated.
- Observe the following safe distances from neighbouring devices, equipment and walls: space of at least 15 cm around the sides and at least 1.5 m from the ceiling.
- Put the ashing device on a solid, firm horizontal base, where it is not exposed to vibration.
- Ensure that the ashing device cannot be knocked over.
- Do not expose the ashing device to direct sunlight.
- Ensure that conditioning or ventilation is switched off during the weighing-in or at least does not influence the weighing process.



The ashing device is a precision instrument. Ensure that the device is not badly shaken. Vibrations from neighbouring devices can corrupt the ashing device measurements.

# 3.4 Assembly and Connection



All the parts must be attached without exerting undue force. Do not apply any undue force. If you have any problems, the Precisa customer service representative will be happy to assist.

The ashing device does not come fully assembled. Carry out the steps in the sequence outlined below:

1. Arrange to have an electrician assemble the plug:

 $L \rightarrow Phase 230 VAC$ 

E → Earth N → Neutral

- 2. Remove the protective stoppers from the requisite connections (process gases and waste gas nozzle) on the back of the device.
- 3. Attach the compressed air, the process gases, the mains cable, the waste gas hose and, if necessary, the alarm plug to the designated connection points (see section **2.4.2** "Rear Controls and Connections" on page 8).
- 4. Regulate the pressure of the compressed air supply using the attached maintenance unit (see section **2.4.2** "Controls and Connections on the Back" on page 8).
- 5. Refer to chapter 16.1 for any scrubber or pump connection.

# ! DANGER

The ashing device is operated with a 230 V AC supply. The electrical wiring work should be carried out by a qualified electrician.

Have the electrician check that existing electrical installations are in good working order and that fuses and cable cross sections are sufficient for the connection data on the rating plate for the ashing device.

The exhaust gas hose must be fed into an exhaust system in order to discharge harmful waste gases from the furnace chamber safely.

In order to continue the assembly process, you need to start the ashing device and open the furnace chamber.

- 6. Press the master switch on the front device (see section 2.4.1 Front controls on page 7)
- 7. The software starts up automatically.
- 8. Register as SUPERVISOR, press and enter the service mode by pressing User ID: PRECISA1 Password: PRECISA1

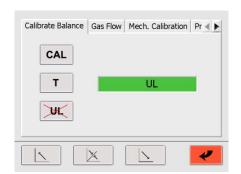


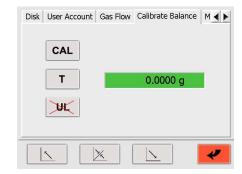




9. Choose "Calibrate balance" mode and open the lid from the touch screen, pressing







- 10. Remove the balance protection plug and slide in the weighing pan support.
- 11. The balance has to be re-started.
- 12. In the register balance calibration UL (under load) is displayed in the balance display. Confirm with the UL-button on the left. The balance will be re-started automatically. When the balance display shows a weight value (0.0000 g) you can quit the service mode by pressing.

#### 3.5 Balance calibration

Before use, the balance must be adjusted according to acceleration of the earth specific to the place of use. It is recommended to calibrate the balance regularly. The procedure is described in the section 10.7 page 39.

# 3.6 Gas Adjustment

The gas flow must be regulated after adjustment of pressure. This procedure is described in chapter 10.6 page 39.

#### 3.7 Mechanical calibration

Mechanical adjustment is made at the factory. The lifting and turning movements of the turntable are tested by the technician installing the device.

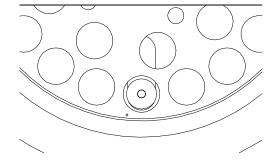
# 3.8 Inserting the turn-table

Carefully place the sample plate on the rotary axis.

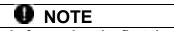


The small hole in the sample plate must lie to the left of the balance pan (refer to figure on the right).

The ashing device is now ready to be used.



#### 3.9 Crucibles



Glow the crucibles before using the first time

# 4 Software: First Steps

# 4.1 Program Start

The terminal is switched on by pressing the mains switch on the ashing device. After booting up, the login display appears. Enter your USER ID and the Password. Default setting when delivered:





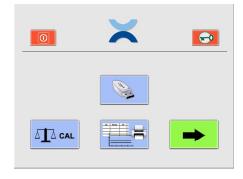
Level Supervisor: User ID: PRECISA1 Password: PRECISA1
Level User: User ID: PRECISA Password: PRECISA

Log in by touching the corresponding button.

The next window will appear in accordance with the given login and password.





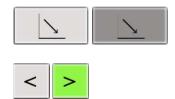


**User** (as from Firmware Release A1-1-2-04, previously saving to USB stick was restricted to supervisor)

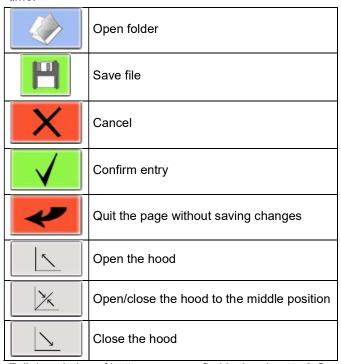
### 4.2 prepASH Controls

#### 4.2.1 Buttons

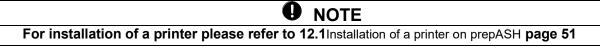
By touching the button the command is executed. The colour of activated icons is changed:



You can switch between the main menu pages in the prepASH user interface by touching the *arrow* buttons. Unless otherwise described, these program instructions move along one page to the right each time.



Full description of buttons you can find in the chapter 9 Control Buttons.



#### 4.2.2 Numerical Entry

Numerical values need to be entered at various points in the program.

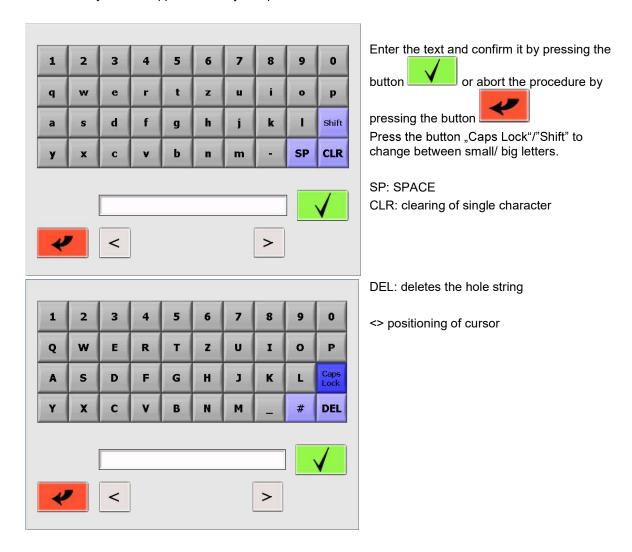
The Numerical Entry window appears when you tap the relevant field or number which needs to be changed. Incorrect entries can be corrected using the *DEL* button. The "-" at the top right is only active for the auto stop entry.



Enter the number and confirm it by pressing the button or abort the procedure by pressing the button

# 4.2.3 Text entry

The text entry window appears when you tap the relevant field.



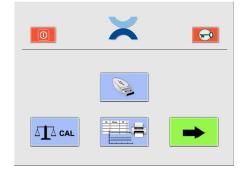
#### 4.2.4 External Keyboard / Barcode Reader

It is possible to use an external keyboard or a barcode reader to enter numbers and texts. Connect the key board or the barcode reader via USB port to the prepASH. The device is automatically recognized.

# 4.3 prepASH User Interface

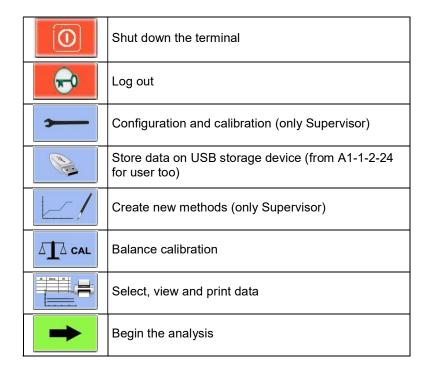
In the user interface you can calibrate the balance, print results and run a drying and ashing analysis. The supervisor additionally has the option to set the configuration and to perform several calibrations, to transfer the data onto a USB storage device and to create new methods.



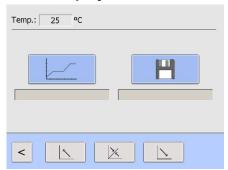


Supervisor

**User User (**as from Firmware Release A1-1-2-04, previously saving to USB stick was restricted to supervisor)



#### 4.3.1 Display bar



The display bar at the very top of the user interface shows the actual temperature.

It always remains visible on the main menu pages and during the analysis.

#### 5 Create/Edit method

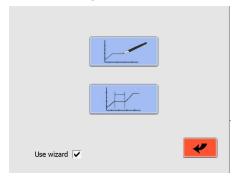
Methods can be created and edited only by the supervisor.

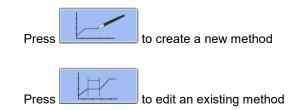






Click on the method button on the user interface.



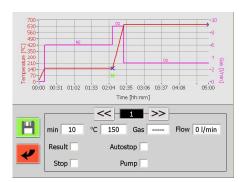


Make a tick in the "Use wizard" box to make/edit a method with the help of a wizard.

For editing, the wizard can only be used if the method is made with two temperatures (moisture and ash). For more information see section 5.4.

#### 5.1 Create a new method

Press the button to creat a new method and method interface will appear



Your method can consist of up to 10 steps by which the parameters may be defined.

Enter the parameter for each program step.

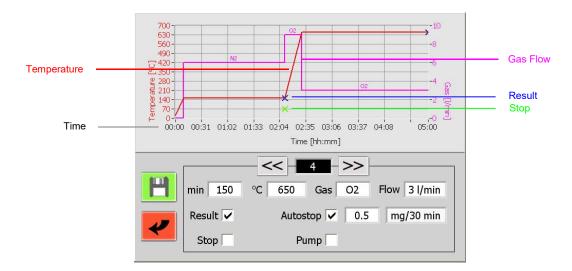
Switch from one step to another by touching the step buttons.

During the program sequence, prepASH processes all the steps in which parameter settings have been edited and then automatically switches to the cool-down step.

#### 5.1.1 Parameter selection

Every step contains at least the settings of the time and temperature.

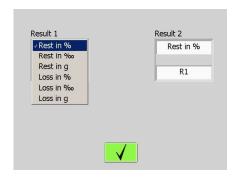
By touching the corresponding field the numeric entry window is opened and the value can be entered.



#### Labelling of the parameters

min 120	min = time/duration duration of the program step.
°C 550	°C Temperature Temperature [°C] at the end of the program step. This is achieved by a linear temperature gradient, starting from the end temperature of the previous program step, lasting the duration of the program step.
Gas O2	Gas (optional) - Flow Three gas connections (air, nitrogen, oxygen) enable the atmosphere to be set during the program sequence. The possible gas flow rates are 0, 3, 6 or 9 l/min.
Resultat 🗸	<b>Result</b> Set "Result" to select the program steps at the end of which the weights are accessible for residue calculation. Up to 4 results can be taken (the weighing in is available and does not have to be additionally marked). The weight is normally marked after the end of drying and ashing.
Stop T	Stop At the end of the step the program is interrupted and the hood is opened as soon as the chosen temperature has been arrived. The sample disk can be taken out of prepASH and ashing additives given to the sample.  The hood is closed automatically and the measurement is proceeded.
Autostop 🗸	Auto stop Activate/deactivate the auto stop with the button. The program step is ended once all samples have reached the selected constant weight. (weight [mg] per time [10 min, 30 min, 60 min] resp. % of weighing in per time [10 min, 30 min, 60 min]). The weight is stored for each individual sample at the time reaching the auto stop criteria. In the protocol you will find the time of auto stop for each sample. Choosing the "-" only the loss of weight is taken to calculate the AS. Without "-" losses and gains of weights are taken in account.
Pump	<b>Pump</b> Set "Pump" to switch on the external connected scrubber or pump in this program step. In the graph, switch on is showed with a P+ and switch off with a P
H	Save the new method.

#### 5.1.2 Calculation of losses and residues

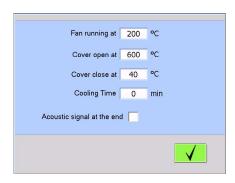


Define the way in which each result is displayed individually in the selection menu.

Each result can be calculated as residue or loss, in terms of per cent, per mil or as an absolute amount in grams.

The results may be relative to the starting weight (S, Sample) or to any earlier result (R1, R2, Residue).

# 5.2 Events on completion of the program



**Fan running at**: at temperatures above this temperature the exhaust is running and the lid is cooled. The fan has to be running before the decomposition of the organic compounds starts (Temp. < 200 °C).

**Cover open at:** Temperature, at which the lid is opened automatically after the run has been finished (half open position). The opening is only allowed at temperatures below 800 °C.

**Cover close at:** Temperature, at which the lid is closed automatically after the run has been finished.

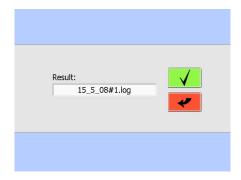
**Cooling Time**: only with ECD (enhanced cooling device), duration of active cooling after analysis

**Acoustic signal**: If you activate this field, prepASH will inform you about the end of analysis with a loud acoustic signal

Confirm your entry pressing the button



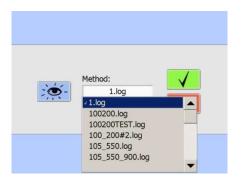
and next window to save the results will open.



pressing the button or abort the procedure pressing the button

#### 5.3 Edit method

Press the button to edit an existing method and Pull-Down-Menu will appear.

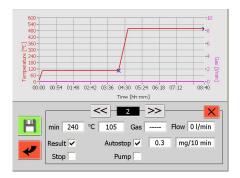


Choose the method in the Pull-Down-Menu

By pressing this button you can check the method but you can not change it



confirm your choice of method and proceed like creating a method.



Choose the program step to be changed and overwrite the parameters needed.

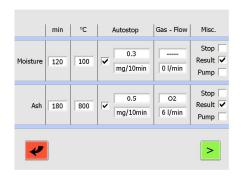
An edited method can be save under the same or a new name.

By pressing this button, you can delete the current program step.



save file

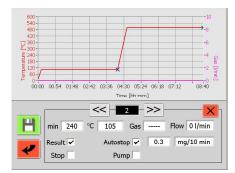
#### 5.4 Method wizard



Every step (moisture and ash) contains at least the settings of the time and temperature.

The ramp time is automatically calculated and can be seen/changed in the next screen.

Change to the next screen by pressing this button.

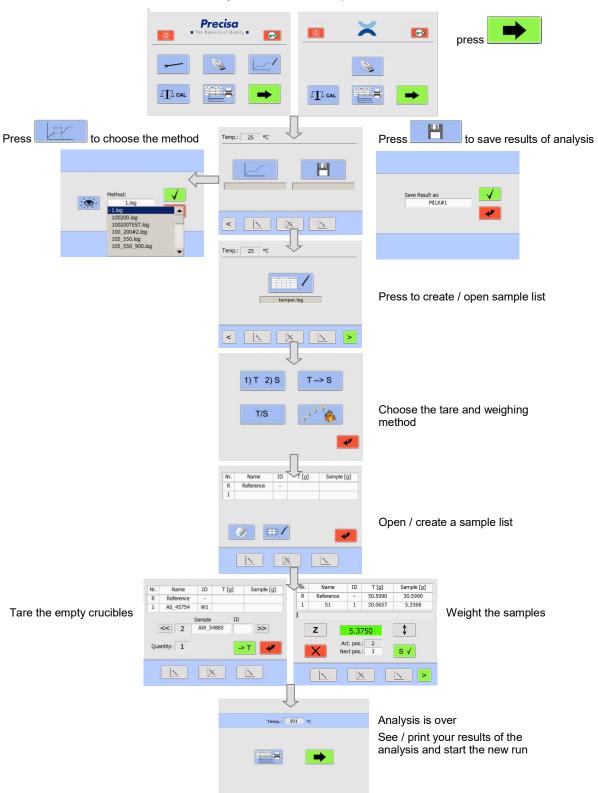


From here, you have the same possibilities as when you create a method without the wizard.

# 6 Program execution

# 6.1 Analysis execution / Flow Chart

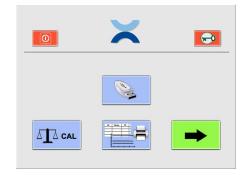
Start the analysis from the User / Supervisor interface



From the user interface you can go directly to the program execution.

Press to proceed to the program execution.





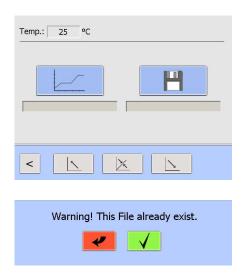
Supervisor

User

# 6.2 Filename to store the data of analysis

The data of analysis are stored automatically. The filename has to be entered before starting the run.

Press to store the filename.

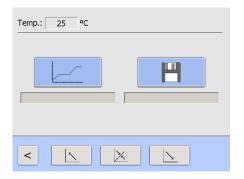






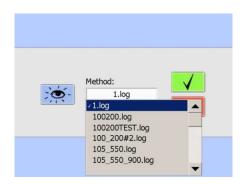


#### 6.3 Choose the method



Press to choose an existing method and a Pull-Down Menu will appear.

Choose the method in the Pull-Down-Menu.

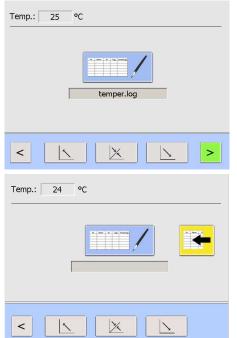


By pressing you can check the method but you can not change it.



Confirm your choice of method.

# 6.4 Sample list



You can create a new sample list (6.4.4 page 27) or open an existing sample list (6.4.5 page 28).

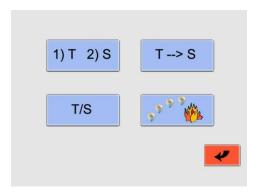
weighing in prepSTATION (-> manual prepSTATION) The sample list is created in prepDATA, weighings are preformed in prepSTATION.



Open the copleated list

#### 6.4.1 Tare mode

Press the button and the tare mode window is opening. You have several possibilities for sequences of taring the empty crucibles and weighing the sample.

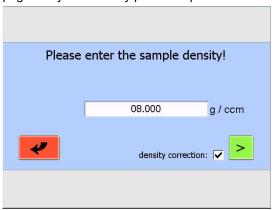


1) T 2) S	Tare all / weight individually	Step 1. Before taring the lid is closed automatically. All empty crucibles are weighed automatically and the tare weights are stored. Step 2. You will open the lid from the touch screen, give the weight each sample individually and store it.
T/S	Tare individually / weight individually	Weight the empty crucible and store its tare weight.  Then give the sample in the crucible and store the weight of the sample.  This procedure you need to carry out with each sample individually.  The lid will be stay open.
T> S	Tare all / weight all	Step 1. All empty crucibles are weighed and the tare weights are stored.  Before taring the lid is closed automatically.  Step 2. Open the lid and fill all the samples into the crucibles without weighing (e.g. pipette or graduated spoon).  The lid is closed and the weighing is done automatically.
Was I	Glow the crucibles	Glow the crucibles without any taring or weighing of any crucibles and samples.

#### 6.4.2 Sample density

From Firmware Release A1-1-2-04 and only if option sample density is activated in the service register (10.3.1 Density correction)

Please enter the sample density and activate the density correction. With the flash you turn to the next page. Only one density per run is possible



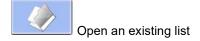
The entering of sample density is recommended for inorganic samples which have only a low loss on ignition and a large ash volume. The densities of these samples change only slightly during the analysis, the buoyancy change however is not negligible.

For organic samples with very low ash, this option is not needed, since the volumes of the ashes are very small and the buoyancy change is negligible. (The change of buoyancy of the crucible is always corrected effectively for each sample disc passage by reference crucibles).

#### 6.4.3 Sample list

After you have chosen the tare-mode you would need to create or open the sample list.







Create a new sample list

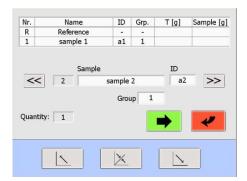
#### 6.4.4 Create a sample list



Enter the sample name and the ID into the entry field by clicking on it.

With >> , the data are transferred into the table and the entry fields of the next sample is opened.

With , you have the possibility to go backward and edit the entries.

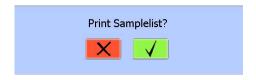


The field "Quantity" shows you the actual number of samples.

Nr.	Position of the crucible on the turn-table
Reference Empty crucible which is weighed for buoyancy correction at every measuring cycle	
Sample	Sample name (maximum 16 characters)
ID	Additional identification e.g. crucible number
Grp.	Group (19)
T [g]	Empty weight of the crucible
Sample [g]	Weight of the sample

After entering name and ID of all the samples (depend on the Tare-mode in use, see 6.4.1 page 26),

press the button to weight the empty crucibles. Follow the instructions on the screen.



You are requested to enter the file name and have the possibility to print the sample list.

Confirm the print of the sample list pressing the button



or abort the action by pressing the



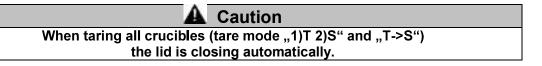
#### 6.4.5 Open an existing sample list



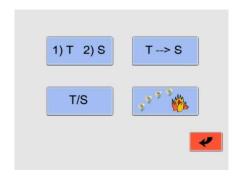


Choose the existing sample list from the Pull-Down Menu and confirm your entry

After opening the sample list, you can start to weight the empty crucibles or you can make changes in the sample list.



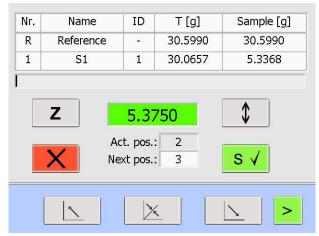
# 6.5 Weighing in in prepASH and START



Pressing the button with the sample list you open the tare mode window.

You have several possibilities for sequences of taring the empty crucibles and weighing the sample.

The weighing-in process and prepASH interface will depend on your tare mode (see Tare mode 6.4.1 page 26).





Weight for the current crucible is shown.

You have possibility to choose the next crucible, giving the number in the "Next pos." field.

If you made groups in the sample list, the procedure is made in order of the groups.

After you store the weight of the current crucible you will move to the next chosen position.

#### Description of important buttons:

Z	Zeroing the balance display without changing the stored tare value.
<b>\$</b>	If the crucible is take out of the samples disc to fill the sample this function facilitates the weighing. The crucible is re-centred by lifting the sample disc.
s√	The weighing is only accepted when stable (highlighted in green). The disc will be turned to the next position automatically.
X	Quit weighing
>	Press to start the analysis

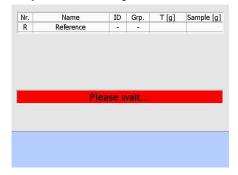
# 1) T 2) S

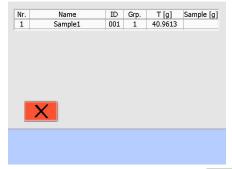
# Tare all / weight individually



Step 1. Open/create a sample list (see 6.4.4 and 6.4.5) and start the tare procedure by pressing

Step 2. Before taring, the lid closes automatically. The tare weight will be stored automatically.





Step 3. Open the lid. Give the weight of each sample individually and store it by pressing





Step 4. Press to start the analysis

# 2. T/S Ta

# Tare individually / weight individually

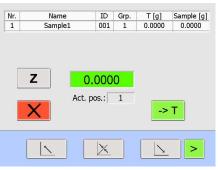
Step 1. Open/create a sample list (see 6.4.4 and 6.4.5) and start the tare procedure by pressing



Step 2. Weight the empty crucible and store its tare weight (press \_\_\_\_\_) . Give the sample in the

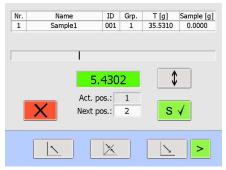
crucible and store the weight of the sample (press ). This procedure you need to carry out with each sample individually. The lid will stay in the middle position.

Tare



Step 4. Press to start the analysis

Weighing

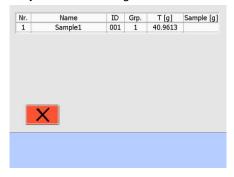


# 3. Tare all / weight all

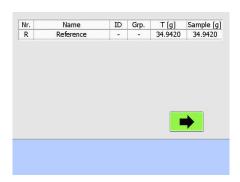
Step 1. Open/create a sample list (see 6.4.4 and 6.4.5) and start the tare procedure by pressing



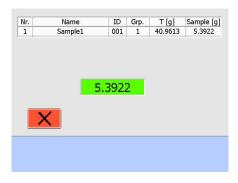
Step 2. The tare weight will be stored automatically (before taring the lid closes automatically).



Step 3. The lid opens automatically. Fill all the samples (e.g. pipette or graduated spoon).



**Step 4.** Press to weight all the crucibles with samples automatically.



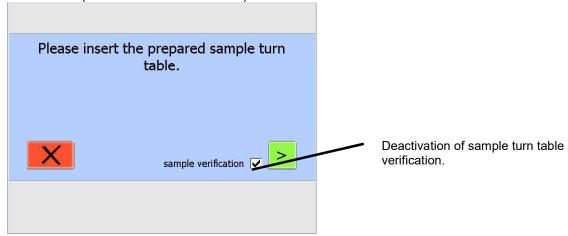
Step 5. Analysis starts automatically.

# 6.6 Start after weighing in prepSTATION



Open the compleated sample list (-> Manual prepSTATION)

This sample list is compared with the actual sample turntable in the prepASH. The verification can be desactivated (but this is not recommended).



After successful verification of the sample run can be started directly

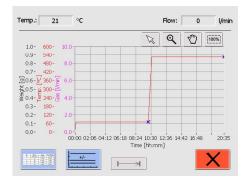


# 6.7 During the measurement

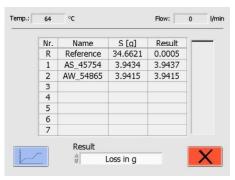
During the measurement you can switch between the graphic presentation and results in a table form. The calculation can be chosen for the actual display.



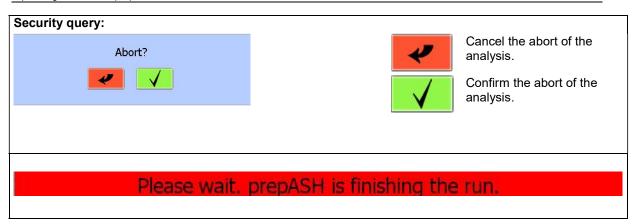






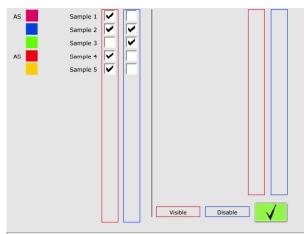


Q	Zoom
[100%]	100 % back to full-scale
	Move the graphic
K	Deactivate "zoom" and "move the graphic"
	Switch between graphic and table.
+/-	Add/remove plots from the graphic and disable samples
Result Loss in g	Select the calculation to be displayed. This will not change the calculation on the print out nor on the saved data.
<b>├</b> ────────────────────────────────────	Disable the active method step and go to the next step.
X	Abort of the analysis



#### Add/remove plots from the graphic and disable samples:





Clear (push on the box) the tick in the red field to make a sample invisible in the graph. The sample can be made visible by pushing on the box again.

Make a tick in the blue field to disable a sample. Now, this sample is ignored for the autostop criteria.

A disabled sample can't be enabled again!

An "AS" on the left side shows that the autostop criteria has been achieved.



Confirm your entry.

#### 6.8 Program end

# **A** CAUTION

The sample chamber hood opens automatically once the temperature selected in the program has been reached.

Do not place anything on top of the prepASH.

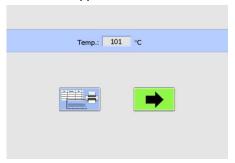
The parts inside the furnace chamber and the metal covers will become hot.

Be very careful to avoid contact.

Always use the supplied holder for removing the sample plate from the device. If you are removing individual crucibles, always use the supplied tongs.

The ventilation is running until the temperature chosen is reached.

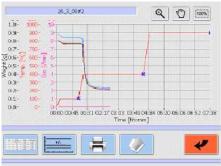
As soon as analysis is finished prepASH will switch off the heating system automatically and the following window will appear.

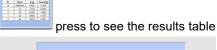


Result is displayed and can be printed

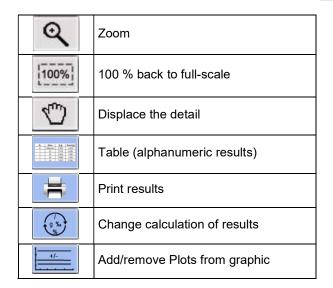
Next analysis can be started (This button is only available when the temperature is below the "lid-opening-temperature").











# 7 Calculation

Following results will be calculated during the analysis:

Residue [g] = End weight [g]

$$Residue[\%] = \frac{End \, weight}{Startingweight} \times 100\%$$

$$Residue[\frac{0}{00}] = \frac{Endweight}{Startingweight} \times 1000\frac{0}{00}$$

Loss[g] = Starting weight [g] – End weight [g]

$$Loss[\%] = \frac{Startingweight-Endweight}{Startingweight} \times 100\%$$

$$Loss[\sqrt[9]{_{00}}] = \frac{Startingweight - Endweight}{Startingweight} \times 1000\sqrt[9]{_{00}}$$

Average 
$$\overline{\mathbf{x}} = \frac{\sum_{i=1}^{n} \mathbf{x}_{i}}{\mathbf{n}}$$

Standard deviation 
$$\sigma = \sqrt{\frac{\sum\limits_{i=1}^{n} (x_i - \overline{x})^2}{n(n-1)}}$$

#### Data transfer and prepDATA 8

prepDATA is designed for Windows 2000 / XP / Vista / 7 / 8 and Windows 10 all with 32 or 64 bit. Windows Office (Excel) is required.

There are several ways to transfer data from prepASH to a PC. With prepDATA the results can be viewed, printed and saved in different formats.

### 1. Transfer the data via USB-Stick from the prepASH on your computer

With this mode, you are not able to have a remote on your PC during the run.

You can only get the results from the prepASH on your PC!

1. Switch in the prepASH "User Interface".









- 3. Choose the result that has to be saved from the Pull-Down Menu. If you don't have an USB-Stick inserted, security query will appear.
- 4. Confirm your entry pressing the button the result is saved on your USB-Stick in a folder named "prepASH\_Results".
- 5. Insert the USB Stick on your computer.
- 6. Start prepDATA, switch in the prepDATA menu-bar to "Measurement" and choose "Open from PC" to display the results on your PC.

#### 2. Connect prepASH with PC over the local network

With this mode, you are able to monitor the prepASH on your PC. And you can also see results that are saved on the prepASH.

- 1. Connect the prepASH with a network cable via LAN port to the local area network.
- 2. Connect your PC to the local area network.
- 3. Start prepDATA, switch in the prepDATA menu-bar to "Measurement" and choose "Open from prepASH" to display the results on your PC.

### 3. Connect prepASH with PC over a RJ45 cable

You can also connect the prepASH with your computer directly over a network cable. (In this case, it is important that you use a crossed network cable).

- 1. Connect the prepASH with the crossed network cable via LAN port with your computer.
- 2. Start prepDATA, switch in the prepDATA menu-bar to "Measurement" and choose "Open from prepASH" to display the results on your PC.



For further information about the connection prepASH with prepDATA check the prepDATA manual.



For installation of a printer please refer to 12.1 Installation of a printer on prepASH page 51

# 9 Control Buttons

< > << >>	You can switch between the main menu pages in the prepASH user interface by touching the <i>arrow</i> buttons. Unless otherwise described, these program instructions move along one page to the right each time.  Wih the help of double arrows you can switch between samples and user accounts.
△T∆ CAL	Balance calibration directory
20 1000 10	See and print results
	Print
CAL	Balance Calibration
	Weighing
<b>JUC</b>	The weighing range is below the range of the balance
	Log out
	Log in
<b>⊕</b> /	Give password
	Save results on the USB Stick
-	Access to the Service Mode
-	Start new analysis
	Start the analysis
H	Save
<b>_</b>	Confirm
X	Delete
0	Switch off
*	quit the page without saving changes
	Open folder
RST	Reset
	Open the hood
<u>×</u>	Open/close the hood to the middle position
	Close the hood
##/	Create a new sample list

Delete sample list  Edit existing method  Creat a method  Delete method  See the method	
Creat a method  Delete method	
Delete method	
See the method	
Air Air flow	
N2 Nitrogen flow	
O2 Oxygen flow	
3 I/min Gas flow 3 litres pro minute	
6 I/min Gas flow 6 litres pro minute	
Heating	
Zeroing the balance display without changing the stored tare value.	
1) T 2) S Tare all / weight individually	
Tare individually / weight individually	
T>S Tare all / weight all	
Glow the crucibles	
If the crucible is take out of the samples disc to fill the sample this function facilitathe weighing. The crucible is re-centred by lifting the sample disc.	ites
The weighing is only accepted when stable (highlighted in green). The disc will be turned to the next position automatically.	Э
-> T Tare the empty crucibles	
Zoom; with 100% back to full-scale	
Move the graphic	
Show 100% graphic	
Table (alphanumeric results)	
Add/remove Plots from graphic:	
Change results calculation	
Key board or Barcode reader entry	
Displays the audit trail	



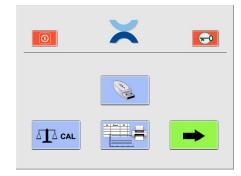
Switch on/ switch off fan

# 10 SERVICE MODE / Configuration

From the user interface you can go directly to the service mode.

Only Supervisor have access to the service mode.



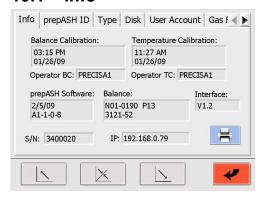


Supervisor

User

Enter into the calibration and configuration part from the user interface. Click on the register needed. (The balance calibration is accessible directly for the user interface for the user too. All other calibrations are reserved to supervisor and service).

#### 10.1 Info

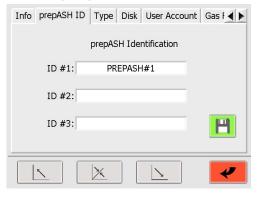


In this register you will find all information on identification and state of calibration of the system and software.

You have the possibility to print out this information

by pressing the button



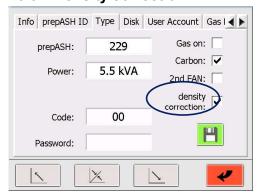


You have the possibility to add identifications to your prepASH (e.g. inventory number, number of test equipment, division).

This information will be shown on the reports of the analysis.

### 10.3 prepASH Type

### 10.3.1 Density correction



**Density correction:** if activated you will be asked to enter the sample density (see Sample density chap. 6.4.2)

Access for all other items only for service engineer. Password protected!

### 10.3.2 Audit Trail (Requirements of 21CFR11)

In 21 CFR part 11 FDA stipulates the requirements of electronic records and electronic signatures. In chapter 11.10 e is required:

Use of secure, computer-generated time-stamped audit trails to independently record the date and time of operator entries and actions that create, modify, or delete electronic records.

#### Implementation in prepASH

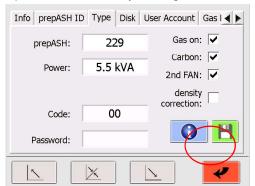
The prepASH software logs every change made to the device by the user / service engineer.

These changes are written in a special file and stored on the prepASH. To ensure that this file can not be manipulated, a checksum is additionally formed and stored in another file. This checksum is generated with an MD5 hash function

Each time the log file is read or written, its hash value is checked and the integrity of the log file is verified. If a manipulation is detected, it is noted in the log file and a new hash value is generated. This ensures that manipulations are always detected.

#### Description and display/printout

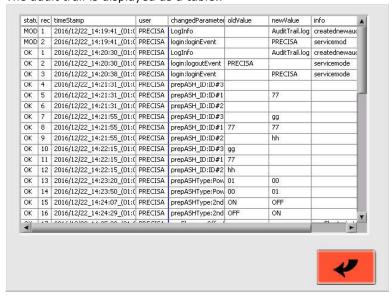
Open the audit trail by clicking on the info button in the service menu in register "Type".





Click on this icon to open the audit trail

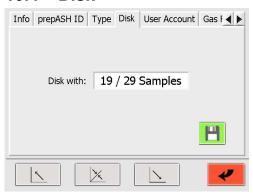
### The audit trail is displayed as a table.:



The individual columns have the following meanings:

column heading	description	
status	OK = file has not been modified, MOD = file has	
	been modified	
record number	number of the entry in the log file	
time stamp	time of entry in the log file	
user	registered user at the time of the entry	
changed parameter	changed settings	
old value	old value	
new value	new value	
info	Additional information about changes	

### 10.4 Disk



For the models prepASH229 and prepASH219 you can switch between the sample disks "19/29 Samples" and "12 Samples".

Exchange of the disc:

- Choose disk in the Pull-Down Menu
- 2. Save your entry by pressing
- 3. Follow the instructions on the screen

### 10.5 User account

Generate new "user" and "supervisor" in the register "User Account".

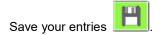




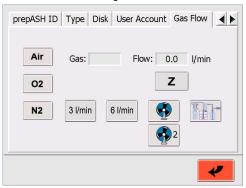
Press to enter the User Account mode

To create / change an "User Account" give an user ID, Password und Access Level (Supervisor or User)

To change from one "User Account" to another or to confirm an entry, press or .



### 10.6 Gas adjustment



Choose the gas and press corresponding button



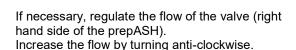
Open the valve (3, 6 or 3+6 l/min).

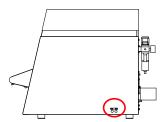
The effectively measured gas flow is indicated on the right in I/min.

(Inlet pressure: Air 6 bar, O2 und N2: 3 bar)









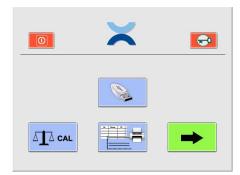
**Testing the exhaust fan** by pressing the Fan-button fan.

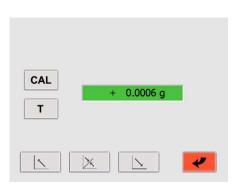


to switch on or switch off the exhaust

**Testing the external outlet for a Pump** by pressing the Pump-button to switch on or switch off the **Pump connected** to the **external outlet**, see also chapter 2.2.1, position 13.

### 10.7 Balance calibration







The calibration of the balance can be activated directly from the user interface that is accessible for the Supervisor and user by pressing the calibration button.

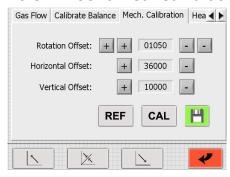
#### Calibration

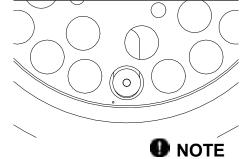
- 1. Tare the balance by pressing the T button.
- 2. Start calibration by touching the CAL button
- The device performs a zero point measurement. (--- 0 g ---) flashes.
- After the zero point measurement the display flashes the calibration weight\* (--- 50 g---)
- Place the calibration weight on the pan.
- The display flashes

Once the display stops flashing, this indicates that the calibration has ended. The value of the calibration weight is displayed.

\* With the help of Intelligent Calibration Mode (ICM), the device can detect the size of the calibration weight, which makes it possible to perform a calibration with different sized weights (in 10-gram increments).

### 10.8 Mechanical calibration





The small hole in the sample plate must lie to the left of the balance pan (refer to figure on the right)

# **A** CAUTION

Make sure that the motor positions are in the start position and that the sample plate is installed correctly (the easiest way is to restart the device).

Adjust the start position with,,+" and ,,-,. For that rotation the outer buttons are provided for a fast rough adjustment and the inner buttons for the fine-tuning.

Rotation Offset: Correct the rotation of the sample disk until the balance pan lies exactly under the

reference hole of the sample disk (without crucible).

Horizontal Offset Correct the position of the balance until the balance pan lies exactly under the

reference hole of the sample disk (without crucible).

Vertical Offset

Correct the height of the sample disk until the bottom of the crucible is positioned 3 mm above the balance pan (with crucible).





Press the CAL-Button to check the mechanical calibration. A calibration with the new offsets will be done.

Press the REF-Button to reset (set to zero) all the offsets. A calibration with this offsets will be done. From here you can start to new adjust.

### 10.9 Temperature calibration and adjustment

To perform a temperature calibration or adjustment you need a thermometer with a range up to  $1000 \,^{\circ}$ C and a tolerance of <= +/- (1% + 2  $^{\circ}$ C). The temperature sensor diameter must be less than 3.2 mm, the length at least 16 cm.

Before you start insert the temperature sensor from the back of the prepASH trough the sensor inlet into the sample chamber, see also chapter 2.2.1, position 11. The insertion depth into chamber is 155 mm.

### 10.9.1 Temperature calibration

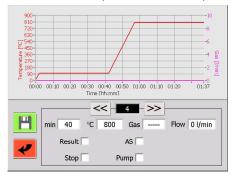
The temperature calibration is only to verify the temperature adjustment of the prepASH. In other words, to verify the prepASH is reaching the defined temperature for a measurement within a acceptable range of  $\leq +/- (1\% + 2 °C)$  of the defined temperature (e.g. +/-3 °C at 100 °C and +/-10 °C at 800 °C). If the values are out of this range you need to adjust the prepASH with a temperature adjustment.

The temperature calibration is performed similar to a normal drying and ashing procedure. Therefore you have to define a method with 2 temperatures where you like to verify the adjustment. The original factory adjustment and calibration has been made at 100 °C and 800 °C, so we recommend to use the same temperatures. But if you are working mainly with other temperatures, e.g. 105 °C and 550 °C you can modify the procedure accordingly. Make sure the prepASH has at least 30 minutes time to stabilize the temperature on each point.

#### 1. Define the method with 2 temperatures T1 and T2

The factory calibration method for T1=100 °C and T2= 800 °C is:

- 2 min to 100 °C, 40 min at 100 °C, 15 min to 800 °C and 40 min at 800 °C



#### 2. Start the calibration

After defining the method start the calibration like a normal measurement. You have to work without the turn table, crucibles and samples, therefore use the tare mode "Glow the crucibles" for the weighing in, refer to 6.4.1 for further information.

Make sure the external reference temperature sensor in inserted from the back of the prepASH.

#### 3. Measure the real temperature at T1

According to your method verify the temperature of the external sensor versus the set temperature of you method as follows:

- Wait until about 5 minutes before the end of the stabilization phase (not less than 30minutes) of the first temperature will finish.
- Read the temperature of the external thermometer as an average value over about 60 seconds and note this temperature as real temperature TR2.

#### 4. Measure the real temperature at T2

According to your method verify the temperature of the external sensor versus the set temperature of you method as follows:

- Wait until about 5 minutes before the end of the stabilization phase (not less than 30minutes) of the second temperature will finish.
- Read the temperature of the external thermometer as an average value over about 60 seconds and note this temperature as real temperature TR2.

#### 5. Verify the temperature T1 and T2 with the real temperature TR1 and TR2

The difference between T1 and TR1 as well as between T2 and TR2 should not exceed the specified limits of <= +/- (1% + 2 °C) of the temperature T1 or T2 (e.g. +/-3 °C at 100 °C and +/-10 °C at 800 °C)!

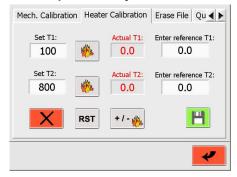
### 10.9.2 Temperature adjustment

The temperature adjustment has been performed during the manufacturing of the prepASH in the factory. Normally it is not necessary to adjust the prepASH out in the field!

### **A** CAUTION

We strongly recommend to make a temperature adjustment only if you have recognized big differences for the temperature settings during a temperature calibration. In this case perform a temperature adjustment to make sure the prepASH is reaching the defined temperature for a measurement within an acceptable range again.

#### The temperature adjustment screens looks as follows:



#### Performing the temperature adjustment in 2 steps as a 2 point adjustment

#### Step 1, at temperature "Set T1"

- Set the temperature "Set T1" for the lower calibration point e.g. drying temperature
- Press to start heating
- Wait 30 minutes after the actual temperature "Actual T1" has stabilized
- Read the temperature of the external thermometer as an average value over about 60 seconds. Enter this value as reference at "Enter reference T1".
- Press to stop the heating

### Step 2, at temperature "Set T2"

- Set the temperature "Set T"" for the higher calibration point, e.g. ashing temperature
- Press to start heating
- Wait 30 minutes after the actual temperature "Actual T2" has stabilized
- Read the temperature of the external thermometer as an average value over about 60 seconds. Enter this value as reference at "Enter reference T2".



Press

to stop the heating



• Save the new adjustment

# **A** CAUTION

It is absolutely necessary to perform after the temperature adjustment a temperature calibration to verify the adjustment has been successful.

If the temperature calibration is not according the limits repeat the temperature adjustment.

### Reset adjustment data

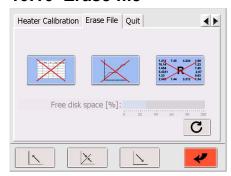
Use this button to reset the temperature adjustment. Only use this function (before temperature adjustment) if the temperature values are totally out of range. You don't need this function for a normal temperature adjustment. After reset, do the temperature adjustment as illustrated above.

#### Adjust heater



Access for Service Engineer only. Password protected!

### 10.10 Erase file





Delete sample lists







Update: free space is calculated







Press to confirm the file to delete

### 10.11 Quit / access to windows

Access to this function is protected by a password and restricted to service







Please find the service password on the supplement provided with the manual or is received from Precisa

### 11 Error messages



If any Error appears, the first thing you have to try is to restart the prepASH!

### 11.1 Error 1: Interface board-Reset

The "Interface board" has made a self-reset.

All the data are deleted → restart the prepASH and start a new measurement.

If the **Error 1** appears again after the restart → Contact the Precisa Service

# 11.2 Error 2: Communication-error between Touch panel and Interface board

There is no communication between the "Touch panel" and the "Interface board" possible. There could be different reasons for this problem:

- 1. The wire between the "Touch panel" and the "Interface board" is not properly connected.
  - → Reconnect the wire and restart the prepASH.
- 2. The wire between the "Touch panel" and the "Interface board" is damaged.
  - → The wire has to be replaced.
- 3. The Interface board was busy and couldn't receive the data from the prepASH.
  - → Restart the prepASH (If the Error appears again → contact the Precisa Service.)

#### 11.3 Error 3: Balance error

Restart the prepASH → Switch into the Service Mode → Go to the menu-tab to "Calibrate Balance" and see what is displayed on the screen. If the showed weight is about 0.000g, the balance is working properly and you can start a new run. If another information is shown on the display, the balance causes the error.

# 11.4 Error 4: Communication-error between Touch panel and Temperature-Module (7018)

There is no communication between the "Touch panel" and the "7018-Module" possible. There could be different reasons for this problem:

- The wire between the "Touch panel" and the "Module" is not properly connected.
   → Reconnect the wire and restart the prepASH.
- 2. The wire between the "Touch panel" and the "Module" is damaged. The wire has to be replaced.
- 3. The Module did shutdown autoaticaly.
  - → Restart the prepASH (If the Error appears again → contact the Precisa Service.)

# 11.5 Error 5: Communication-error between Touch panel and Flow-Module(7017)

There is no communication between the "Touch panel" and the "7017-Module" possible. There could be different reasons for this problem:

- 1) The wire between the "Touch panel" and the "Module" is not properly connected.
  - → Reconnect the wire and restart the prepASH.
- 2) The wire between the "Touch panel" and the "Module" is damaged.
  - → The wire has to be replaced.
- 3) The Module did shutdown himself.
  - → Restart the prepASH (If the Error appears again → contact the Precisa Service.)

# 12 Integration into your IT environment

### 12.1 Installation of a printer on prepASH

prepASH prints to the printer which is defined as standard printer in windows. This may be a local printer, a network printer or a PDF printer only creating a file e.g. a PDF Creator.

To install the printer to prepASH you need the specific printer driver for Windows 7. You will find it either on the CD or you have to download it from the website of printer manufacturer.

- 1) Close prepASH Software and enter Windows refer to 10.11 Quit / access to windows on page 49. Install the printer as usual on Windows 7:
- 2) Since prepASH has no CD drive you have previously to copy the printer driver on a USB stick.
- 3) Connect the USB-stick to prepASH.
- 4) Open USB-stick (→ "My Computer") and start printer installation.
- 5) After installation start the program up by restarting prepASH or by dobble click on the prepASH-symbol on the desktop.

### 12.2 Change the prepASH name of the domain

prepASH can be given any domain name on Windows system level.

1) Close prepASH Software and enter Windows refer to 10.11 Quit / access to windows on page 49. Change name as usual on Windows surface under Windows 7.



Pay attention to the new name in connection with prepDATA!

### 12.3 Access to prepASH via network, LIMS connection

For data backup or LIMS connection the prepASH can be reached via the network under its domain name or IP address.

1) To get information about the IP address or the domain name, go to the Windows level as described in 10.11 Quit page 49.

You are now on the Windows interface and can retrieve the information as usual on Windows 7



Detailed information about data storage as well as the approved user and password can be obtained from Precisa.

### 13 prepASH Software update

# 13.1 New prepASH software installation

- 1) Close prepASH Software and enter Windows refer to 10.11 Quit / access to windows on page 49.
- 2) Until version A1-1-0-17 Save the file "prepASH.exe" on an USB-Stick.
  - From version A1-1-0-19 unzip zip-file, copy folder "prepASH229 Setup Light" on a memory stick
- 3) Insert the USB-Stick in the prepASH.
- 4) Until version A1-1-0-17:
  - Open the USB-Stick ( → double click on "My Computer").
  - Replace the file "prepASH.exe" (that is on the desktop) with the "prepASH.exe" that is on the USB-Stick.
- 5) From version A1-1-0-19:
  - Remove old software: open windows start ->Control Panel -> Add or Remove Programs Control Panel -> Add or Remove Programs.
  - Open memory stick on prepASH desktop ( → double click on "My Computer)
  - Eexecute \prepASH229 Setup Light\Volume\setup.exe
  - Destination Directory -> Browse.. -> select
  - "C:\Documents and Settings\Administrator\Desktop\"



- Start Installation -> Next
- 6) Now, the software is updated. Either you restart the prepASH and the prepASH-software will start automatically, or you can start it by double-clicking the prepASH-lcon on the desktop.

### 14 Maintenance and Service

### ! DANGER

Disconnect the ashing device from the mains during all maintenance work (pull out the plug) and ensure that the device cannot be switched on inadvertently.

In order to guarantee operational safety, the ashing device must be inspected every 4 years at least by an electrician from your Service Centre to ensure that it is in good working order.

Other maintenance is generally limited to regular cleaning.

### 14.1 Housing

The housing can be cleaned on the outside with a damp cloth.

### 14.2 Sample chamber

Clean the sample chamber with a vacuum cleaner after taking out the parts inside (sample plate, crucible, balance pan). Take care of the sensitive insulating and heating radiators. Avoid damage with the suction tube.



The high temperatures may cause cracks to appear in the insulation inserts. This is normal.

### 14.3 Ventilation



### NOTE

The airing has to be cleaned every 6 months to insure sufficient airing of the furnace chamber.

#### 14.3.1.1 **Tools**

Pin type socket wrench (width across flats 7 mm) Phillips screwdriver

#### 14.3.1.2 Remove upper cover



- 1. Open the hood from the touchscreen software
- Unscrew the 2 hexagon bolts in the corners of the cover from below



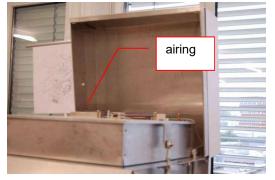


Close the cover from the touchscreen



**Important:** disconnect the prepASH from the mainsPull the upper cover slightly forwards and tilt it back.





5. The airing is accessible.

#### 14.3.1.3 Cleaning the airing

- 6. Unscrew the airing and clean it mechanically.
- 7. If needed also clean the prepASH side.
- 8. The ceramic tube may be cleaned mechanically



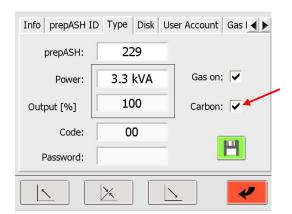


#### 14.3.1.4 Reinstall the upper cover

- 9. Tilt the upper cover down on to the hood.
- 10. Plug in the mains cable and turn on the instrument 11. Open the hood from the software.
- 12. Fix the upper cover on to the hood (hexagon bolts)
- 13. The device is now ready for use.

# 15 Appendix A: Carbon application

In this appendix are only the differences between a carbon and a normal measurement explained. Use the main manual for general prepASH information.



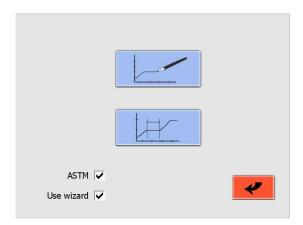
To use the carbon application, the function has to be activated in the service mode. Unlocking can only be made by a Precisa engineer.

See chapter 10 for more information about the service mode.

#### 15.1 Method

The ASTM method includes the steps for the moisture, volatile and ash. In the edit mode, you are not able to add/clear method steps, but you can change the time, temperature and gas in every step. In the steps where the results for the moisture and ash are taken, you can add an autostop criteria. The points for results calculation and lid opening (for cover on/off) are given and can't be changed.

For general information about methods see chapter 5. If you edit an ASTM method, the volatile calibration from the origin method is copied in the new method. For recalibration the method see chapter 15.5 / 15.7.



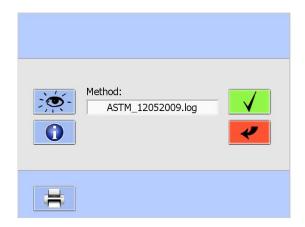
Important! Make a tick in the "ASTM" box to edit the ASTM methods.

Make a tick in the "Use wizard" box to make/edit a method with the help of a wizard.

Press to create a new method. A new method can only be made with the wizard.

Press to edit an existing method.

### 15.2 Open method

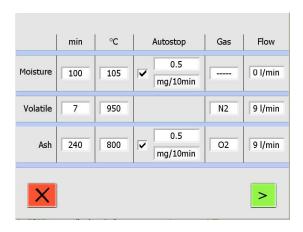


Choose the method in the pull-down-menu that you want to edit.

Press for information about the last volatile calibration.

Press to print out the method including the calibration information.

### 15.3 Wizard



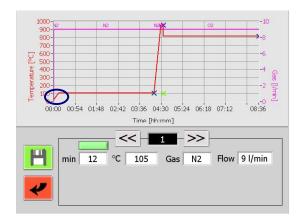
Every step contains at least the settings of the time and temperature.

The ramp time is automatically calculated and can be see/changed in the next screen (see section 15.4).

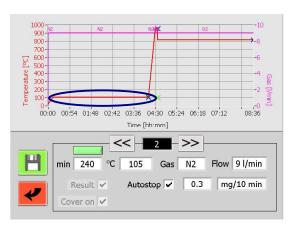
Change to the next screen by pressing this button.

### 15.4 Edit method

### 15.4.1 Step 1 + 2 (Moisture)



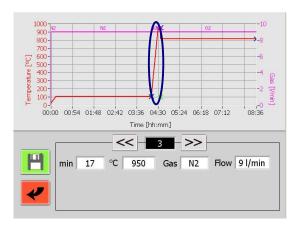
Step 1 is the ramp for the drying temperature. The ramp is limited to 50°C/min.



In Step 2 you can change the drying temperature and time. You are also able to add an autostop criteria. At the end of this step, the result is taken automatically and the lid is opening for adding the covers on the crucibles. *Don't put a cover on the reference crucible!* 

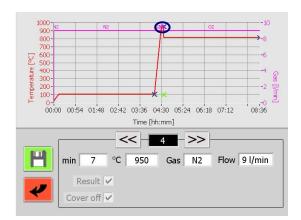
If you work with already dried samples, you have the possibility to skip steps 1 + 2. Just press the green button ( ). The button is getting red ( ). During measurement, the lid will open for adding the covers after one turn is finished. In this situation, the weigh-in is also saved as dry weigh. For this reason, be sure that the samples are really dry, on the other way there will be wrong results.

### 15.4.2 Step 3 + 4 (Volatile)



Step 3 is the ramp for the temperature where the volatile is taken.

The ramp is limited to 50°C/min.

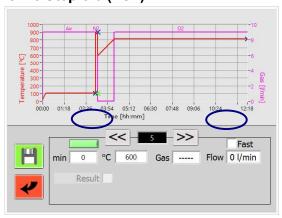


The time that you enter in step 4 is between achieving the temperature criteria (that is 2% under the set temperature) and the start of the volatile measurement.

After the volatile measurement is finished, the prepASH is cooling down. The lid is opening when the temperature is below the ashing-temperature (if the lid opening temperature – definied in the method – is lower, then this temperature is taken as reference).

The covers have to be removed from the crucibles.

### 15.4.3 Step 5/6 (Ash)



In step 5, you can enter the duration of the heating up to ashing temperature (from firmware release A1-1-2-04.)

If the system is still hot from the determination of the volatile components, is not actively cooled.

In previous versions of the software the temperature was increased as fast as possible to the ashing temperature. If you want to maintain this even after the software update, select "Fast" By clicking on the green button (turns red), you can perform an analysis without ashing step (from firmware release A1-1-2-04.)

In step 5 you can change the ashing temperature and time. You are also able to set an autostop criteria. The result is taken automatically at the end of the step.

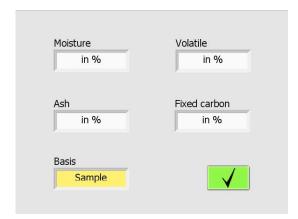


Save the method



Abort edit method

#### 15.4.4 Additional



Before saving, you are asked for the calculation of the results. In every method will the moisture, volatile, ash and fixed carbon be calculated. You are able to choose showing the results in [%] or [g] and the basis of the calculation (sample or dry mass).

Formula for calculation are in chapter 15.9.

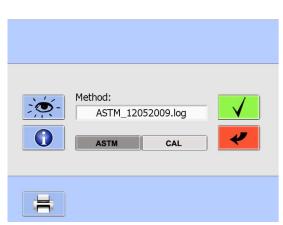


After confirming, you are asked for some addition information. For more see "Events on completion of the program" (chapter 5.2).

The method has to be saved with a different name then the edited method. The edited method can't be overwritten and is still available.

### 15.5 Choose the method for the run

For general information about program execution see chapter 6.



Press the "ASTM" button ( ASTM ) to see all the saved ASTM carbon methods in the pull down menu.

Each method has to be calibrated and can have only one valid calibration. So all data of the old calibration are lost when recalibrating a method.

Press the "CAL" button for calibration or recalibration. See chapter 15.7 for more information about calibration.

If you confirm a method that is not calibrated (and you also didn't choose to calibrate) then a message appears that the method is not usable. Calibrate the method for using.

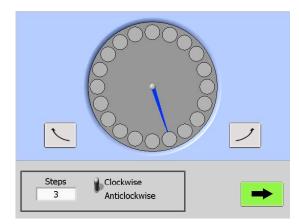
If you make a carbon measurement and not a new calibration, start from here like a normal measurement.

# 15.6 During measurement

After drying, the lid is opening automatically and you have to put the covers on the crucibles. Don't put a cover on the reference crucible!

At the second lid opening, remove the covers from the crucibles.





You can turn the sample table for better crucible handling during the lid opening.

The blue "arrow" shows the position of the reference crucible.

For single steps press the right-/left- turn button.



For bigger steps, press on the sample table in the middle of the screen. You can change the step number and the direction (clockwise, anticlockwise) in the menu at the bottom.

Press the green button to continue the measurement.

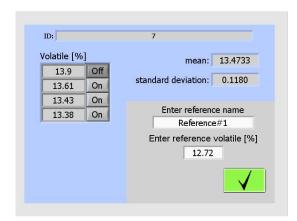


### 15.7 Calibration

As explained in chapter 15.5, the carbon methods have to be calibrated for use. If you choose to calibrate a method then start to prepare the sample list normally as explained in chapter 6.4. It doesn't matter which tare mode you choose.

You can make a calibration with 1 up to 29 reference samples (12 for 212, 19 for 219). For better results, we recommend to take more then one sample from every reference. For every sample with the same reference, you have to put in the same ID (choose the ID between 1 and 29) in the sample list. This is important for the reason that the software can only difference the samples over the ID. At the end of the calibration-measurement, you should still know the belonging between the ID and the reference. Write down or print out the sample list!

Start the calibration (as a normal run) after finishing the sample list.



At the end of the calibration-measurement, the adjoining screen appears.

For every used ID, a calculation with all reference samples for this ID is available. You have the possibility to deactivate (press the on-button right of the result) samples for a better standard deviation. Minimal you need one sample.

Enter the reference name and volatile (*in percent, calculated on sample dry basis*) for this ID.

Confirm if all data is correct for the actual ID. The program is going to the next ID.

After enter data for all ID the calibration is automatically saved on the prepASH. Now you are able to use the method for the measurement.

#### 15.7.1 Volatile correction

At the end of a carbon measurement, the volatile is adjusted automatically.

Make sure the calibration covers the hole range of expected volatile content. Between two calibration points the values are calculated linearely.

#### 15.8 Results

The result table is automatically adapted for the carbon application. The result is shown for moisture, volatile, ash and fixed carbon. For calculation see chapter 15.9.

You are able to change the calculation in matter of the basis and [%], [g].

### 15.9 Calculation

### **15.9.1 Results**

B = Weight of specimen after drying in moisture test [g]

C = Weight of specimen after heating in volatile matter test [g]

F = Weight of ash residue [g]

W = Weight of specimen used [g]

M = Moisture

V = Volatile

A = Ash

H = Fixed Carbon

### Sample basis value

$$M[\%] = (W-B)/Wx100$$

$$V[\%] = (B-C)/Wx100$$

$$A[\%] = F/Wx100$$

$$H[\%] = 100 - M[\%] - V[\%] - A[\%]$$

$$M[g] = (W-B)$$

$$V[g] = (B-C)$$

$$A[g] = F$$

$$H[g] = W - M[g] - V[g] - A[g] = C - F$$

### Dry basis value

$$M[\%] = 0$$

$$V[\%] = (B-C)/Bx100$$

$$A[\%] = F/Bx100$$

$$H[\%] = 100 - V[\%] - A[\%]$$

$$M[g] = 0$$

$$V[g] = (B-C)$$

$$A[g] = F$$

$$H[g] = B - V[g] - A[g] = C - F$$

# 16 Appendix B: Scrubber application

In this appendix are only the differences between a normal measurement and a measurement with an connected scrubber explained. Use the main manual for general prepASH information.

For details of the scrubber handling and maintenance refer to the scrubber manual.

### 16.1 Scrubber connection

To attach a scrubber to the prepASH we strongly recommend to use our **Condensation-Absorption-Connection-Set**, **340-9002** because all parts in this set are tested for this application.

The condensation-absorption-connection-set contains:

- Glass cooling coil
- High temperature tube, 1m
- Mains connection cable
- Drain termination, already assembled in prepASH
- Centering lead for glass cooling coil, already assembled in prepASH



Place the scrubber unit on the left hand side of the prepASH.





Connect the scrubber to the power outlet on the back side of the prepASH, see also chapter 2.4.2. Use the special Mains connection cable delivered with the scrubber connection set, 340-9002.

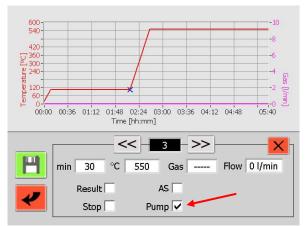
Insert the glas cooling coil through the hole on the left hand side of the prepASH into the centring lead.

Use the high temperature tube to attach the scrubber to the prepASH.

See chapter 2.4.2 for more information.

### 16.2 Scrubber operation

If the scrubber is connected direct to the external power outlet on the back side of the prepASH the scrubber can be switched on and off by the prepASH according the pump control setting in the parameters for the method.

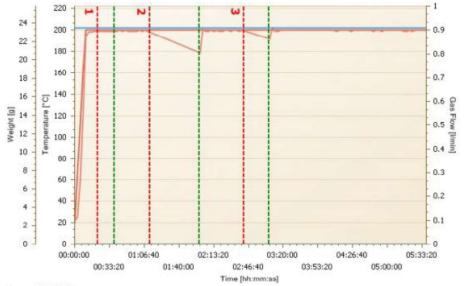


The scrubber is normally switched on during the ramp up to the higher temperature. Therefore activate the pump control in this phase only.

See chapter 5.1.1 for more information.

# 17 Appendix C: Uninterruptible Power Supply UPS

The optional uninterruptible power supply (UPS) prevents during mains power failure the loss of data and allows the automatic continuation of the analysis as soon as the power is restored. Heating is not possible with the UPS, the analysis will be interrupted for the time of the current interruption. Thanks to good insulation only low temperature losses are to be expected at short interruptions. In the protocol the start and end of the interruption is shown in the graph and the times are listed in a table. Also, the temperature decrease can be checked in the protocol. As long as the auto stop criteria is reached, the data can be used in the standard case. The evaluation and the responsibility if a repetition is necessary, rests with the operator.



#### Powerfail details:

Nr.	Start	Stop
1	0:21:49	0:38:23
2	1:12:10	1:59:37
3	2:42:34	3:06:14

# 18 Appendix D: Practical recommendations

### 18.1 Correct sample preparation

Prepare samples at a time for measurement. This prevents the sample exchanging moisture with the ambient surroundings. If a number of samples have to be taken simultaneously, they should be packed in air-tight containers so as to ensure that they do not change while they are in storage.

#### Solids:

Distribute powder samples into the crucible.

Make course samples smaller using a mortar or grinder. Do not expose the sample to any heat while you are grinding it as this will lead to a loss of moisture.

#### Liquids:

Pipette the samples directly into the crucibles. Make sure that no evaporation occurs before initial weighing.

# 18.2 Optimization of result accuracy and work with very low ash content

- A correct crucible treatment is essential. See also chapter 18.3 Correct Crucibles handling & Weighing in page 65.
- Work with largest possible amount of sample. Please note that this will increase the measuring time and possibly impair homogenous drying / ashing. See also large crucibles and adapters in Chapter 19 Accessories and Consumables page 66
- Use large crucibles to achieve faster and homogeneous drying / ashing. See also large crucibles and adapters in Chapter 19 Accessories and Consumables page 66
- Please note that at very small ash contents due to reproducibility of the measuring system sporadic negative residual weights may occur.

# 18.3 Correct Crucibles handling & Weighing in

- Use the same type of crucibles in one run.
   If you place crucible on the adapter, ensure that reference crucible and all other crucibles in the turn table will be of the same kind.
- To receive accurate and reproducible results always ensure that crucibles are clean and dry: Glow the crucible before each analysis for at least 30 minutes in prepASH or in the oven. Temperature for glowing the crucibles should be 100C° higher than test temperature. Keep you crucibles in the desiccator with silica gel. Place the crucibles on the turn-table with help of the pincers or tweezers and not with bare hands.
  - Place the crucibles on the turn-table with help of the pincers or tweezers and not with bare hands Ensure that no particles stick to the crucibles bottom or to the weighing pan.
- Carry out the weighing in as fast as possible, so that the sample will not exchange moisture with the ambient surroundings.
- Weight of the samples grouped to calculate the statistic should not vary on more than 10%.
- Powdery and plastic samples tend to develop static electricity, which may strongly influence
  performance of the analytical balance build-in the prepASH. Neutralize possible static electricity on
  your samples and containers before weighing with lonizer.

# 18.4 Adapting to the current test procedure

The moisture and ash analyzer is frequently used in place of other drying & ashing techniques (like the drying oven and muffle furnace), because it is easier to operate and offers shorter measuring times. Consequently, the conventional measuring process has to be adapted to the prepASH so that comparable results can be achieved.

- Perform a parallel measurement
- Vary the end point conditions
- Increase the sample weight, if necessary
- Adapting with the calibration curve of factor (e.g. ASTM coal and coke)

### 18.5 Possible causes of not accurate results and solutions

The weight display changes continuously

- The draft is too strong at the location of the instrument
- The instrument support is vibrating or fluctuating
- The weighing pan is touching a foreign body (check position of sample tray)
- The sample is absorbing moisture
- The sample is losing moisture
- Sample temperature not the same as surrouning temperature

Results of weighing are clearly incorrect

- Crucibles not correct cleaned or glowed
- The instrument has not been correctly tared
- The calibration is no longer correct

### 19 Accessories and Consumables

#### prepStation 340-9010

Station for the external weighing of the samples & EP120A balance with sensor draft-shield. prepStation assist in easy and error free external weighing of the samples for the prepASH analysis.



#### **ECD: Enhanced Cooling Device**

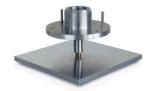
inbuilt cooling system to enhance the cooling betweens runs ex works: **340-8504** 

conversion set: **350-9004** (only for prepASH units after November 2013)



#### Turn-table holder 340-8012

Helpful to handle hot sample disk outside prepASH e.g. when adding sulfuric acid under a hood for sulfated ash



### **Condensation-Absorption-Unit (scrubber)**

For pre-incineration for sulfate ash determination Triple Scrub Nr. 114152330) Consists of: neutralization stage, adsorption stage, cold trap and receiving vessel.



#### Condensation-Absorption-Connection-Set, 340-9002

Exclusive connecting set to attach scrubber to prepASH.

#### Consumables:

Turn-table for 29/19 samples, SiC, 340-4065

Turn-table for 29/19 samples, SiC, 340-4065

Turn-table for 12 samples, SiC, 340-4072

Set of 5 crucibles (Porcelain glazed) **340-8037**, 35 ml use with turn- table 29/19 340-4065

Set of 5 crucibles for use with lid (Al2O3), **340-8032**, 26 ml use with turn-table 29/19 340-4065 and crucible - lid 340-8033

Set of 5 crucible – lids, (Al2O3), **340-8033** use with turn-table 29/19 340-4065 and crucible 340-8032

Set of 5 crucibles, (silicate ceramic), **340-8034**, 40 ml use only with turn-table 12 340-4072 and adapter 340-8035

Set of 5 adapters, (silicate ceramic), **340-8035** use only with turn-table 12 340-4072 and standard crucible 340-8034 or other crucible available from the market (Diameter: lower < 32 mm, upper < 55 mm))















