

# Fully automatic ash determination and moisture analysis system



Serial Number S/N: Installation Date:

350-8127-000a20



Precisa Gravimetrics AG Moosmattstrasse 32 CH 8953 Dietikon

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# 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 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	No.	Description
1	Crucible	11	Inlet for external temperature sensor
2	Plate holder	12	Electrical power outlet for connection of a scrubber or pump
3	Sample plate	13	Mains connection cable with connector CEE 3 pole male
4	Balance pan	14	LAN port
5	Touch screen	15	USB port
6	Master switch	16	Mechanical connection for scrubber
7	Maintenance unit with a compressed air connection	17	Optional fast cooling system
8	Exhaust	18	Adjustable feet
9	Process gas flow control	19	Extendable spirit level
10	Process gas and compressed air connections		

### 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: CEE Type, 3 pole, 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: 3 bar (± 10 %), 0 9 l/min , 99.5 % purity
  - Nitrogen: 3 bar (± 10 %), 0 9 l/min , 99.5 % purity
  - Compressed air: 6 bar  $(\pm 10 \%)$  with air turnover of 2-3 l/min
  - Oil-free and without condensation (ISO 8573-1:2010, at least standard 7.4.4
  - (solid particles 5-10 mg/m3, water vapor pressure dew point < + 3 °C, water liquid -, oil 5 mg/m3)

#### 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-condensent
- Exhaust gas hose (included in delivery)
  - Diameter inner/outer 63/68 mm, length 2 m, flow about 40m3/h, Temperature about 50°C, exhaust gases must be removed from the working room (hood)

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, levelling and connections

### 2.4.1 Front controls and levelling



**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 keyboard, stick or barcode reader (2)

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





- 4 Extendable spirit level
- 5, 6 Levelling device
- Loosen the lock nuts (5) on both levelling feet (SW13 open-ended spanner)
- Place the spirit level (4) horizontally
- Adjust both levels (6) (open-ended wrench SW15) until prepASH is perfectly levelled. The levelling foot has a right-hand thread. This means that if you turn it to the right (clockwise), the prepASH lowers.
- Tighten the lock nuts on both levelling feet.
- Place the spirit level back to it's home position.

### 2.4.2 Rear controls and connections

# \Lambda 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.

# 

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 inner/outer diameter of 6/8 mm or replace with any 1/8" connection.

**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 (outer  $\emptyset$  x thickness 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

**9, 10** mains cable 2 m with connector CEE Type 3 pole 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 using the clip, and the other end is fed into an exhaust system.

**12** LAN-connection **13** USB-connection



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.



When the sample plate is removed, the following parts are visible:

**4** The process gases ( $N_2$ ,  $O_2$  or compressed air) are fed inside the sample chamber by means of the lance (4).

**5** The temperature sensor (5) measures the temperature inside the furnace chamber for regulation

**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	
Sample plate	
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 7 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.

**W** NOTE 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  $E \rightarrow$  Earth  $N \rightarrow$  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 10).
- 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 10).
- 5. Refer to chapter 16.1 for any scrubber or pump connection.
- 6. Level the prepASH with help the adjustable front feeds and the inbuild spirit level.

# 

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.

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

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

Pre	ecisa
The Balan	Poce of Quality
User ID	Level
PRECISA1	Supervisor
***	

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

Calibrate Balance Gas Flow Mech. Calibration Pr	Disk User Account Gas Flow Calibrate Balance M
CAL	CAL
TUL	T 0.0000 g
	X

- 11. Remove the balance protection plug and slide in the weighing pan support.
- 12. The balance has to be re-started.
- 13. 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 sample plate

Carefully place the sample plate on the rotary axis.



The ashing device is now ready to be used.

### 3.9 Crucibles



# 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:

Precisa		<b>Precisa</b>	
The Balance of Quality		The Balance of Guality	
User ID PRECISA Password	Level User	User ID L PRECISA Password ******	User
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.



×	<b>~</b>
(a) (b)	
	<b>→</b>

Supervisor

#### 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 color 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.

	Open folder
	Save file
X	Cancel
$\checkmark$	Confirm entry
•	Quit the page without saving changes
	Open the hood
×	Open/close the hood to the middle position
	Close the hood

Full description of buttons you can find in the chapter 0



### 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.



#### 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 keyboard 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

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

Θ	Shut down the terminal
P	Log out
	Configuration and calibration (only Supervisor)
(A)	Store data on USB storage device (from A1-1-2-24 for user too)
	Create new methods (only Supervisor)
	Balance calibration
	Select, view and print data
	Begin the analysis

#### 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.

<b>Precisa</b> The Balance of Quality	The Balance of Quality
User ID Level Supervisor Password ******	
Click on the method buttor	n on the user interface.
	Press to create a new method
	Press to edit an existing method
Use wizard 🔽	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

### 5.1 Create a new method

Press the button

to creat a new method and method interface will appear

more information see section 5.4.



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	<i>min</i> = time/duration duration of the program step.				
°C 550	<b>°C Temperature</b> 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 🗌	<ul> <li>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.</li> <li>The hood is closed automatically and the measurement is proceeded.</li> </ul>				
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				
	Save the new method.				

### 5.1.2 Calculation of losses and residues



Result 1	Result 2
√Rest in %	Rest in %
Rest in ‱ Rest in g	
Loss in %	RI
Loss in ‰	
Loss in g	

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	200	°C				
Cover open at	600	°C				
covor opon at	000	Ū				
Cover close at	40	°C				
Cooling Time	0	min				
Acoustic signal at the end						
		$\checkmark$				

**Fan running 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). We suggest to set this not higher than 400 °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

Result: 15_5_08#1.log	<ul><li>✓</li><li>✓</li></ul>

and next window to save the results will open.

Enter the name of the method and confirm your entry

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	on.		
<b>Precisa</b> The Balance of Quality		×	
		() ()	
			-

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 H

to store the filename.

Temp.: 25 °C	
Warning! This File already exist.	

Save Result as:	✓
MILK#1	✓
Security query:	

Confirm overwriting



Cancel and save under a new name.

# 6.3 Choose the method



Choose the method in the Pull-Down-Menu.



# 6.4 Sample list

25 °C

Temp.:



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



Conf

Confirm your choice of method.

Image: second secon

You can create a new sample list (6.4.4 page 29) or open an existing sample list (6.4.5 page 30).

Weighing in prepSTATION (-> manual prepSTATION) The sample list is created in prepDATA, weighing's are performed in prepSTATION.



Open the completed list

#### 6.4.1 Tare mode

N. Sere 0. 132.5

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	<ul><li>Step 1. Before taring the lid is closed automatically.</li><li>All empty crucibles are weighed automatically and the tare weights are stored.</li><li>Step 2. You will open the lid from the touch screen, give the weight each sample individually and store it.</li></ul>
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	<ul> <li>Step 1. All empty crucibles are weighed and the tare weights are stored.</li> <li>Before taring the lid is closed automatically.</li> <li>Step 2. Open the lid and fill all the samples into the crucibles without weighing (e.g. pipette or graduated spoon).</li> <li>The lid is closed and the weighing is done automatically.</li> </ul>
9 <sup>9</sup> 9 9	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.

Please	enter the sample d	ensity!
	08.000	g / ccm
*	density correc	tion: 🗸 >

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.

Nr.	Name	ID	T [g]	Sample [g]	
R	Reference	-			
1					
		=/			
		H-7		~	
	<u>\</u>	X		2	
/					
2					-
		en ar	n existin	g list	

6.4.4 Create a sample list



create a new 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 <u>solution</u>, 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 sample plate			
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)			
Т [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 28), press

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

Press to open an existing 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.

#### 

When taring all crucibles (tare mode "1)T 2)S" and "T->S") the lid is closing automatically.

# 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 28).

Nr.	Name	ID	Т [g]	Sample [g]
R	Reference	-	30.5990	30.5990
1	S1	1	30.0657	5.3368
	Z	5.37	750	\$
	X Ac	t. pos. <t pos.<="" td=""><td>: 2 : 3</td><td>S √</td></t>	: 2 : 3	S √
				<u>&gt;</u>

5.3750				
Act. pos.:	2			
Next pos.:	3			

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.
\$	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

S √

S √

#### 1) T 2) S 6.5.1 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.

Name ID Grp. T [g] Sample [g]	e [g]	Nr.	Name	ID	Grp.	T [g]	Sample
Reference		1	Sample1	001	1	40.9613	
Diance whit	_						
Picase Wait	_	_					
			X				

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





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

-> T Step 2. Weight the empty crucible and store its tare weight (press . Give the sample in the

SV 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

Weighing



6.5.3 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).

Nr.	Name Sample1	ID 001	Grp. 1	T [g] 40.9613	Sample [g]
	X				

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

Nr.	Name	ID	Grp.	T [g]	Sample [g]
R	Reference	-	-	34.9420	34.9420

**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 completed sample list (-> Manual prepSTATION)



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

Please inser	t the prepared sample turn table.	
X	sample verification	Deactivation of sample plate verification.

After successful verification of the sample run can be started directly

Please wait for verification!

### 6.7 During the measurement

Temp.: 21 °C

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.

Flow: 0

l/min





8.0- 8.0- 90 4.0- 2.0- 0.0- 00	00 02:06 04:12 (	06:13 08:24 10:30 Time [rhu:	) 12:36 14:42 16: nm]	48 20:
- 8.0- - 98 4.0- - 2.0- - 0.0- 00	00 02:06 04:12 (	06:18 08:24 10:3 Time [hh:r	) 12:36 14:42 16: nm]	48 20:
- 2.0- - 2.0-	00 02:06 04:12 (	06:18 08:24 10:3 Time [hh:n	0 12:36 14:42 16: nm]	48 20:
-uu) seg 4.0- 2.0- 0.0- 00	00 02:06 04:12 (	06:13 08:24 10:30 Time [hh:r	0 12:36 14:42 16: nm]	48 20:
-8 4.0- - 2.0- - 0.0- 00	00 02:06 04:12 0	06:18 08:24 10:3 Time [hh:r	) 12:36 14:42 16: nm]	48 20:
- (8 4.0- - 2.0- - 0.0- 00	:00 02:06 04:12 0	06:18 08:24 10:30 Time [th:r	) 12:36 14:42 16: nm]	48 20:
- 2.0- - 0.0- 00	:00 02:06 04:12 (	06:18 08:24 10:30 Time [hh:m	) 12:36 14:42 16: nm]	48 20:
- 0.0-	00 02:06 04:12 (	06:18 08:24 10:30 Time [hh:r	0 12:36 14:42 16: nm]	48 20:
	:00 02:06 04:12 (	05:18 08:24 10:30 Time [hh:r	) 12:36 14:42 16: nm]	48 20:
	:00 02:06 04:12 (	06:18 08:24 10:30 Time [hh:n	) 12:36 14:42 16: nm]	48 20:
	+/			Х
	+/-	$  \longrightarrow \rangle$		X
	····	1 21		
54	°C		Flow:	0 l/r
da.	Newse	0.[-1	Decult	
vr.	Name	S[g]	Result	
RI	Reference	34.6621	0.0005	
1 /	AS_45754	3.9434	3.9437	
2 A	W_54865	3.9415	3.9415	
3				
4				
5				
6				
7		-		
/				J
	Result			
	a			X
	54 Nr. R 1 2 4 5 6 7	54 ℃ Nr. Name R Reference 1 AS_45754 2 AW_54865 3 4 5 5 6 7 Result	54         ℃           Ir.         Name         S [g]           R         Reference         34.6621           1         AS_45754         3.9434           2         AW_54865         3.9415           3         3         3           4         5         5           6         7         5           7         8         8	54         °C         Flow:           Ir.         Name         S [g]         Result           R         Reference         34.6621         0.0005           1         AS_45754         3.9434         3.9437           2         AW_54865         3.9415         3.9415           3         -         -         -           4         -         -         -           5         -         -         -           6         -         -         -           7         -         -         -

Q	Zoom
[100%]	100 % back to full-scale
	Move the graphic
$\bowtie$	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.
	Disable the active method step and go to the next step.
×	Abort of the analysis

Security query:		
Abort?		Cancel the abort of the analysis. Confirm the abort of the analysis.
Please wait.	prepASH is finishing the	run.

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

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").



Q	Zoom
[100%]	100 % back to full-scale
Ð	Displace the detail
N.         News         Viet         Install           5         Meens         500         500           4         0         500         100           3         0         500         100           4         0         500         100           4         0         500         100	Table (alphanumeric results)
	Print results
( 9%) 9%)	Change calculation of results
+/-	Add/remove Plots from graphic



press to see the results table

Nr.	Name	ID	S [g]	Residue 1 [g]	Residue [%]	Residue 2 [g]	Residue [%]	F
R	Reference	-	29.7337	29.7351	100.005	29.7352	100.000	T
1	PROBE1	1	0.0338	0.5726	1694.135	0.3360	58.680	T
2	PROBE2	1	0.9615	0.9464	98.425	0.7185	75.918	Т
3	PROBE3	1	0.9810	0.9413	95.955	0.7493	79.603	Т
4	PROBE4	1	1.2650	1.2416	98.147	1.0707	86.235	
5	PROBE5	2	0.5783	0.5685	98.308	0.4009	70.524	Π
6	PROBE6	2	2.1347	2.1254	99.564	1.9277	90.698	T
7	PROBE7	2	0.2440	0.2201	90.205	0.0001	0.046	Τ
8	PROBES	2	0.0000	0.0000	NaN	0.0000	NaN	Τ

# 7 Calculation

Following results will be calculated during the analysis:

Sample = (Tare + Sample) - Tare

Residue [g] = End weight [g]

$$Residue[\%] = \frac{End \ w \ eight}{Starting \ w \ eight} \times 100\%$$

$$\text{Residue}[\frac{0}{00}] = \frac{\text{End w eight}}{\text{Starting w eight}} \times 1000 \frac{0}{00}$$

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

Loss[%] = 
$$\frac{\text{Starting w eight} - \text{End w eight}}{\text{Starting w eight}} \times 100\%$$

$$Loss[\frac{0}{00}] = \frac{Starting w eight - End w eight}{Starting w eight} \times 1000 \frac{0}{00}$$

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

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

# 8 Data transfer with prepDATA

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".

Precisa The Balance of Guality	
	Result:

- 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.

### HINWEIS

How to install a printer on prepASH can be found in chapter 12.1 Installation of a printer on the prepASH.

# 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
<< >>	Wih the help of double arrows you can switch between samples and user accounts.
	Balance calibration directory
	See and print results
	Print
CAL	Balance Calibration
	Weighing
) HK	The weighing range is below the range of the balance
	Log out
	Log in
	Give password
( and the second	Save results on the USB Stick
~	Access to the Service Mode
	Start new analysis
-	Start the analysis
	Save
$\checkmark$	Confirm
X	Delete
Ο	Switch off
~	quit the page without saving changes
	Open folder
RST	Reset
<u> </u>	Open the hood
×	Open/close the hood to the middle position
	Close the hood
##/	Create a new sample list

	Delete sample list
	Edit existing method
	Create a method
$\mathbf{X}$	Delete method
-	See the method
Air	Air flow
N2	Nitrogen flow
02	Oxygen flow
3 l/min	Gas flow 3 litres pro minute
6 l/min	Gas flow 6 litres pro minute
we have a second	Heating
Ζ	Zeroing the balance display without changing the stored tare value.
1) T 2) S	Tare all / weight individually
T/S	Tare individually / weight individually
T> S	Tare all / weight all
****	Glow the crucibles
\$	If the crucible is taken out of the samples disc to fill the sample this function facilitates the weighing. The crucible is re-centered 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.
-> T	Tare the empty crucibles
Q	Zoom; with 100% back to full-scale
Ð	Move the graphic
100%	Show 100% graphic
N         Apple         Mail         Apple         Appl	Table (alphanumeric results)
+/-	Add/remove Plots from graphic:
g %	Change results calculation
	Keyboard or Barcode reader entry
$\bigcirc$	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.

<b>Precisa</b> The Balance of Guality	<b>-</b>		×	<b>~</b>
			(a)	
	•			-
Supervisor		User		



Enter 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

Info	prepASH ID	Туре	Disk	User Ac	count	Gas F < 🕨
Ba	lance Calibratio	n:	Ten	perature	Calibrat	ion:
0 0	3:15 PM 1/26/09		11: 01/	27 AM 26/09		
Op	erator BC: PREG	CISA1	Ope	rator TC:	PRECIS	A1
pre	epASH Software	Baland	e:		Int	erface:
2/ A:	5/09 L-1-0-8	N01-0 3121-	190 F 52	P13	Vi	2
S/N	3400020	IP:	192.16	58.0.79		#
	<u> </u>	×		7		*

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

## 10.2 prepASH ID

Info	prepASH ID	Type Disk	User Account	Gas F ┥ 🕨
		prepASH Ide	ntification	
	ID #1:	PREPASH	l#1	
	ID #2:			
	ID #3:			H
	<u> </u>	×	<u>\</u>	*

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

Info prepASH II	) Type Disk l	Jser Account Gas I
prepASH:	229	Gas on:
Power:	5.5 kVA	Carbon:
	(	density
Code:	00	correction:
Password:		
	X	<u>\</u>

**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.:

statu	rec	timeStamp	user	changedParameter	oldValue	newValue	info
MOD	1	2016/12/22_14:19:41_(01:0	PRECISA	LogInfo		AuditTrail.log	creatednewau
MOD	2	2016/12/22_14:19:41_(01:0	PRECISA	login:loginEvent		PRECISA	servicemod
OK	1	2016/12/22_14:20:30_(01:0	PRECISA	LogInfo		AuditTrail.log	creatednewau
ОК	2	2016/12/22_14:20:30_(01:0	PRECISA	login:logoutEvent	PRECISA		servicemode
ОК	3	2016/12/22_14:20:38_(01:0	PRECISA	login:loginEvent		PRECISA	servicemode
ОК	4	2016/12/22_14:21:31_(01:0	PRECISA	prepASH_ID:ID#3			
ОК	5	2016/12/22_14:21:31_(01:0	PRECISA	prepASH_ID:ID#1		77	
ОК	6	2016/12/22_14:21:31_(01:0	PRECISA	prepASH_ID:ID#2			
OK	7	2016/12/22_14:21:55_(01:0	PRECISA	prepASH_ID:ID#3		gg	
ОК	8	2016/12/22_14:21:55_(01:0	PRECISA	prepASH_ID:ID#1	77	77	
ОК	9	2016/12/22_14:21:55_(01:0	PRECISA	prepASH_ID:ID#2		hh	
ОК	10	2016/12/22_14:22:15_(01:0	PRECISA	prepASH_ID:ID#3	99		
ОК	11	2016/12/22_14:22:15_(01:0	PRECISA	prepASH_ID:ID#1	77		
OK	12	2016/12/22_14:22:15_(01:0	PRECISA	prepASH_ID:ID#2	hh		
OK	13	2016/12/22_14:23:20_(01:0	PRECISA	prepASHType:Pow	01	00	
ОК	14	2016/12/22_14:23:50_(01:0	PRECISA	prepASHType:Pow	00	01	
ОК	15	2016/12/22_14:24:07_(01:0	PRECISA	prepASHType:2nd	ON	OFF	
OK	16	2016/12/22_14:24:29_(01:0	PRECISA	prepASHType:2nd	OFF	ON	
04				EL 000 1			

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:

2.

1. Choose disk in the Pull-Down Menu



3. Follow the instructions on the screen

### 10.5 User account

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

Info prepASH ID	Type Disk Use	er Account Gas I
	$\sim$ /	
		<u> </u>
User ID	Password	Level
PRECISA	PRECISA	0
PRECISA1	PRECISA1	1
Jon	XYZ	
User ID	Thomas	
User ID <	Thomas 123	
User ID Password Level	Thomas 123 Superviso	>>>
User ID Password Level	Thomas 123 Superviso	or



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 . Save your entries

# 10.6 Gas adjustment



Choose the gas and press corresponding button

Air N2 O2
-----------

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

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

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



Press the "zero" button **Z** if the flow is not 0.0 l/min at closed valves.

If necessary, regulate the flow of the valve (right hand side of the prepASH). Increase the flow by turning anti-clockwise.

Testing the exhaust fan by pressing the Fan-button



to switch on or switch off the **exhaust** fan.



**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.

CAL

т

### 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

0.0006 g

Gas Flow	Calibrate Ba	lance	Mech	n. Calibrati	on	Hea ┥ 🕨
Rota	ation Offset:	+	+	01050	-	-
Horizo	ontal Offset:		+	36000	-	
Ve	tical Offset:		+	10000	-	
		RE	F	CAL		8
				<u>\</u>		*



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

# 

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).

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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 offset 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 °C and a recommended tolerance of <= +/- (1% + 2 °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 an acceptable range of  $\langle = +/-(1\% + 2 \degree C) \rangle$  of the defined temperature (e.g.  $+/-3 \degree C$  at 100  $\degree C$  and  $+/-10 \degree C$  at 800  $\degree C$ ). If the values are out of this range you need to adjust the prepASH with a temperature adjustment.

The temperature calibration is performed like 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 using 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 must work without the sample plate, 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 your 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  $<= +/-(1\% + 2 \degree C)$  of the temperature T1 or T2 (e.g. +/-3  $\degree C$  at 100  $\degree C$  and +/-10  $\degree C$  at 800  $\degree C$ )!

#### 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!

#### 

We strongly recommend making 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 look 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



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".



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 et al.
- 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".

Save the new adjustment

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

#### RST

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!

### 10.10Erase file







Update: free space is calculated

Security query:



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

Heater Calibration Erase File Quit	Wy Computer
	prodoli 1
Password:	
	рифАЭН
	<b>1</b>
	Recycle Bri
	∰ Start << 8:33 AM

**D** NOTE The service password can be found on the supplement to the operating instructions or can be obtained 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  $\rightarrow$  restart the prepASH and start a new measurement.

If the **Error 1** appears again after the restart  $\rightarrow$  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:

- The wire between the "Touch panel" and the "Interface board" is not properly connected.
   → Reconnect the wire and restart the prepASH
- The wire between the "Touch panel" and the "Interface board" is damaged.
   The wire has to be replaced.
- 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  $\rightarrow$  Switch into the Service Mode  $\rightarrow$  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.
- The wire between the "Touch panel" and the "Module" is damaged.
   → The wire has to be replaced.
  - The Module did shutdown autoaticaly.  $\rightarrow$  Restart the prepASH (If the Error appears again  $\rightarrow$  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:

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

# **12 Integration into your IT environment**

### 12.1 Installation of a printer on the 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. as PDF Creator.

To install the printer to prepASH you need the specific printer driver for Windows. 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 50. Install the printer as usual on Windows.
- 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 and start printer installation.
- 5) After installation start the program up by restarting prepASH or by double click on the prepASH-

symbol 🖂 on the desktop.

#### 12.2 Change the prepASH name in 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 50.
- 2) Change name as usual in Windows.



Pay attention to the new name in connection with prepDATA!

#### 12.3 Access to the prepASH over a network

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 50.
- 2) Retrieve the information as usual on Windows or ask your network administrator.

### 12.4 Access to the measured data on the prepASH over a network

The prepASH stores for each measurement a separate data file as ".log" in the Results directory on the prepASH what can be reached via the network under its domain name or IP address.

- Access over the network to the Results directory:
  - Address:
     \\prepash340xxxx\c\Results

     (prepash340xxxx means according to the prepASH serial number e.g. 3400099)

     User:
     prepash340xxxx means according to the prepASH serial number e.g. 3400099)

     Password:
     see note below

# 

The access password can be found on the supplement to the operating instructions. Support for data access can be obtained from Precisa.

## 12.5 Access to the LIMS data on the prepASH

The prepASH stores for each measurement a separate LIMS file as ".log" in the LIMS directory on the prepASH what can be reached direct or via the network under its domain name or IP address.

• Access over the network to the LIMS directory:

Address:	\\prepash340xxxx\c\Results\LIMS
	(prepash340xxxx means according to the prepASH serial number e.g. 3400099)
User:	prepash340xxxx\prepash
	(prepash340xxxx means according to the prepASH serial number e.g. 3400099)
Password:	see note below

#### 12.5.1 LIMS data file format

The LIMS data file is a tab separated text file with the extension ".log" as e.g. "Limsdata.log". Therefore the file can be opened with e.g. Excel and if the procedure to open is correctly done the data is split into different columns and looks like below.

Result	-Name	Lim	sdata.log						
Metho	d-Name	Lim	sdata.log						
Opera	tor	PRE	ECISA						
Start	11/29/201	6 9:31	:21 AM						
Stop	11/29/201	6 4:53	3:12 PM						
1	00:30:00	00:30	:00 300						
2	01:30:00	01:30	:00 550			On			
3	00:20:00	00:20	:00 800						
4	01:00:00	01:00	:00 800			On			
5	02:00:00	02:00	:00 800				1 - Sam	ple	
6	02:00:00	00:30	:00 800		0.30 n	ng/30min	2 - Sam	ple	
Nr.	Name	ID	T + S [g]	T [g]	S [g]	Residue 1 [g]	Residue [%]	Residue 2 [g]	Residue [%]
1	PP	-	42.1704	39.1461	3.0243	0.0006	0.020	0.0006 AS	0.020
2	PP	-	43.9326	40.8768	3.0558	0.0007	0.023	0.0006 AS	0.020
3	PP	-	41.4229	38.3368	3.0861	0.0010	0.032	0.0010 AS	0.032
4	PVC	-	42.5799	39.5417	3.0382	0.0006	0.020	0.0006 AS	0.020
5	PVC	-	43.9640	40.9439	3.0201	-0.0002	-0.007	-0.0001 AS	-0.003
6	PVC	-	42.2209	39.1985	3.0224	-0.0001	-0.003	0.0000 AS	0.000
7	Sample3	-	41.1640	38.1417	3.0223	-0.0002	-0.007	-0.0001 AS	-0.003
8	Sample3	-	41.6586	38.6491	3.0095	-0.0003	-0.010	-0.0004 AS	-0.013
9	Sample3	-	42.3145	39.2851	3.0294	-0.0001	-0.003	-0.0001 AS	-0.003

END

#### 12.5.2 Selection of the data for the LIMS programing

Which line contains the data of the first sample depends on the defined method, but always in the line after the line that starts with "Nr". This is most likely not line 13, as in this example!

The data of each sample is in the line at the tabulator positions as follows:

•	"Nr":	Line 13 at Tabulator 0
•	"Name":	Line 13 at Tabulator 1
•	"ID":	Line 13 at Tabulator 2
•	"Tare + Sample in g":	Line 13 at Tabulator 3
•	"Tare in g":	Line 13 at Tabulator 4
•	"Sample in g":	Line 13 at Tabulator 5
•	"Residue 1 in g":	Line 13 at Tabulator 6
•	"Residue 1 as in calculation set":	Line 13 at Tabulator 7
•	"Residue 2 in g":	Line 13 at Tabulator 8
•	"Residue 2 as in calculation set":	Line 13 at Tabulator 9
•	if more steps have been programmed:	
•	"Residue x in g":	Line 13 at Tabulator 10
•	"Residue x as in calculated":	Line 13 at Tabulator 11

"AS" means the final weight value of this programmed step (here 800°C for 30min.) has been taken because of the auto stop (here 0.30 mg/30min) as set for this step. If the "AS" is not appearing this means the prepASH has taken the value as it was at the end of the step after the time set (here 30min).

#### 12.5.3 Open a LIMS data file in Excel with "Open file"

• Open Excel. To open, select "All files (\*.\*)" so that files with ".log" are also displayed.

Dpen					×
$\leftrightarrow$ $\rightarrow$ $\uparrow$	« SERVER-SBS08 > Verkauf > PRODUKTE >	340_prepASH > _SAMP	PLE-DATA ~ (	> P Search_SAMP	LE-DATA
Organize 👻 New for bu				≣	- 🔳 😗
_Touch_U:	Na	Date modified	Туре	Size	1
🔁 _Zubehör	ASTM_c_libration.pdf	22.10.2013 16:24	Adobe Acrobat-D	23 KB	
<b>165</b>	Limsdata.log	23.08.2021 17:58	Textdokument	1 KB	
📒 230_Shim;	📭 Limsdata.xlsx	23.08.2021 17:58	Microsoft Excel W	12 KB	
260_Shink	Samplelist.log	23.08.2021 17:59	Textdokument	1 KB	
320	Test_20160407.log	07.04.2016 10:51	Textdokument	13 KB	
= 321 I G	📾 Test_20160407_Measurement_prepDATA	17.08.2018 16:00	Microsoft Excel 97	18 KB	
= 221 LS LV	Test_20160407_Methode_prepDATA.xls	7 08.2018 16:00	Microsoft Excel 97	9 KB	
521_L5+LA	Test_20160407_Remote_prepDATA.xls	17.08218 15:49	Microsoft Excel 97	13 KB	
321_Ruhrv	Test_20160407_Report_prepASH.pdf	21.06.2019 15:00	Adobe Acrobat-D	129 KB	
a30_Trock	Test_20160407_Report_prepASH_Extende	21.06.2019 13:06	Adobe Acrobat-D	69 KB	1
📒 340_prep/	Test_20160407_Report_prepASH_Extende	21.06.2019 13:07	Adobe Acrobat-D	71 KB	
alt_	Test_20160407_Report_prepDATA.pdf	17.08.2018 15:58	Adu e Acrobat-D	211 KB	
E _FOTOS	Fest_20160407_Result.xls	17.08.2018 15:53	Microsoft Scel 97	10 KB	
SAMPL	Test_Abort_Report_Extended_Appendix_p	06.11.2019 12:10	Adobe Acrobat	44 KB	
	Test Abort Report Extended prepASH.pdf	06.11.2019 12:09	Adobe Acrobat-D	49 KR	
File <u>n</u> a	me: Limsdata.log			<ul> <li>All Files (*.*)</li> </ul>	~
			Too <u>i</u> s	▼ <u>O</u> pen  ▼	Cancel

#### • Select "separated" and click "Next".

Text Import Wizard - Step 1 of 3		?	×
The Text Wizard has determined that your da If his is correct, choose Next, or choose the o Outginal data type	ata is Delimited. data type that best describes your data.		
Choose the file type that best describes you Delimited - Characters such as co Fixed width - Fields are aligned in	ur data: ommas or tabs separate each field. columns with spaces between each field.		
Start import at row: 1 File or	igin: 437 : OEM United States		~
My data has headers.	DDUKTE\340.prepASH\ SAMPLE-DATA\limsdata.log		
1 Result-NameLimsdata.log 2 Method-NameLimsdata.log 3 DperatorPRECISA 4 Start11/29/20169:31:21 AM 5 Stop11/29/20164:53:12 PM			I
4		Þ	
	Cancel < Back <u>N</u> ext >	<u>F</u> inisl	h

• Ensure "Tab" are included, to see the data split into columns and click "Next".

Text Import Wizard	- Step 2 of 3						?	×
his screen lets you review below. elimiters Iab Semicolon Comma Space Other: Data greview	set the delimite	rs your dat secutive d	ta contains. elimiters as (	You car one	n see how yo	our text is affecte	d in the	
Result-Name [j] Method-Name [j] Operator PF Start 1] Stop 1]	imsdata.log imsdata.log RECISA 1/29/2016 1/29/2016	9:31:21 4:53:12	AM PM		< Back	Next >	Fini	

## **12.6 Store a sample list on the prepASH**

You can load an existing sample list (see 6.4.5 page 30) what has previously stored on the prepASH direct or via the network under its domain name or IP address.

• Access over the network to the LIMS directory:

Address:	\\prepash340xxxx\c\$\Samplelists (Note the "c\$" instead of "c" only!)
	(prepash340xxxx means according to the prepASH serial number e.g. 3400099)
User:	prepash340xxxx\prepash
	(prepash340xxxx means according to the prepASH serial number e.g. 3400099)
Password:	see note below

#### 12.6.1 Sample list file format

The sample list file is a tab separated text file with the extension ".log" as e.g. "Samplelist.log". Therefore the file can be opened, edited and save with e.g. Excel. If the procedure to open and save is correctly done the data is split into different columns as below. See more about to open and save with Excel in 12.5.3 Open a LIMS data file in Excel with "Open file" on page 54.

R	Reference	-	-
1	PP	1-1	1
2	PP	1-2	1
3	PP	1-3	1
4	PVC	2-1	2
5	PVC	2-2	2
6	PVC	2-3	2
7	Sample3	3-1	3
8	Sample3	3-2	3
9	Sample3	3-3	3
10	-	-	1
11	-	-	1
12	-	-	1
13	-	-	1
14	-	-	1
15	-	-	1
16	-	-	1
17	-	-	1
18	-	-	1
19	-	-	1
20	-	-	1
21	-	-	1
22	-	-	1
23	-	-	1
24	-	-	1
25	-	-	1
26	-	-	1
27	-	-	1
28	-	-	1
29	-	-	1

#### 12.6.2 Selection of the data for the sample list

Keep the format as above with always the reference line "R" and 29 sample lines "1" to "29" but change at Tabulator 1 the names of the different samples, at Tabulator 2 the ID and at Tabulator 3 the group to calculate statistics. If no statistics is need set the group always as "1".

•	Line 2 for of Sample 1:	Tabulator 1: "Name of sample" here "PP"
		Tabulator 2: "ID of sample", here "1-1"
		Tabulator 3: "Group of sample" here "1"
•	Line 3 for of Sample 2:	Tabulator 1: "Name of sample" here "PP"
	-	Tabulator 2: "ID of sample", here "1-2"
		Tabulator 3: "Group of sample" here "1"
•	Line 4 for of Sample 3:	Tabulator 1: "Name of sample" here "PP"
		Tabulator 2: "ID of sample", here "1-3"
		Tabulator 3: "Group of sample" here "1"
•	Line 5 for of Sample 4:	Tabulator 1: "Name of sample" here "PVC"
		Tabulator 2: "ID of sample", here "2-1"
		Tabulator 3: "Group of sample" here "2"
•	Line 6 for of Sample 5:	Tabulator 1: "Name of sample" here "PVC"
	•	Tabulator 2: "ID of sample", here "2-2"
		Tabulator 3: "Group of sample" here "2"
•	and so on	



The access password can be found on the supplement to the operating instructions. Support for data access 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 50.
- 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
- 4) Insert the USB-Stick in the prepASH.
- 5) Until version A1-1-0-17:
  - Open the USB-Stick
  - Replace the file "prepASH.exe" (that is on the desktop) with the "prepASH.exe" that is on the USB-Stick.
- 6) 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
  - Execute \prepASH229 Setup Light\Volume\setup.exe
  - Perform installation, you may have to confirm "Next" several times.
- 7) 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-Icon 🖂 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.

Precisa

# 

The airing must 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
- 2. Unscrew the 2 hexagon bolts in the corners of the cover from below





- $\mathbf{\mathbf{Y}}$
- 4. A Important: disconnect the prepASH from the mains. Pull the upper cover slightly forwards and tilt it back.



3. Close the cover from the touchscreen



5. The airing is accessible.

#### 14.3.1.3 Cleaning the airing

- 1. Unscrew the airing and clean it mechanically.
- 2. If needed also clean the prepASH side.
- 3. The ceramic tube may be cleaned mechanically





### 14.3.1.4 Reinstall the upper cover

- 1. Tilt the upper cover down on to the hood.
- 2. Plug in the mains cable and turn on the instrument
- 3. Open the hood from the software.
- 4. Fix the upper cover on to the hood (hexagon bolts)
- 5. 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.

Info prepASH I	D Type	Disk	User	Account	Gas I ┥ 🕨
prepASH:	22	29			
Power:	3.3	kVA		Gas on:	
Output [%]	10	0		Carbon	
Code:	0	0			0.TD
Password:					
	×		7		•

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 0. 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.





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

	min	°C	Autostop	Gas	Flow
Moisture	100	105	0.5 mg/10min		0 l/min
Volatile	7	950		N2	9 l/min
Ash	240	800	0.5 mg/10min	02	9 l/min
X					>

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

Moisture	Volatile
in %	in %
Ash	Fixed carbon
in %	in %
Sample	
	V

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.



Confirm the entries.

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.

Method: ASTM_12052009.log	
#	

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.



The prepASH can be really hot! Use for handling the cover-tongs.



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.

ID:		7
Volatile [%]		mean: 13.4733
13.9	Off	
13.61	On	standard deviation: 0.1180
13.43	On	
13.38	On	Enter reference name
,		Reference#1
		Enter reference volatile [%] 12.72
		$\checkmark$

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 can 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 whole range of expected volatile content. Between two calibration points the values are calculated linearly.

#### 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 can change the calculation in matter of the basis and [%], [g].

# 15.9 Calculation

#### 15.9.1 Results

 $\begin{array}{l} B = \mbox{Weight of specimen after drying in moisture test [g]} \\ C = \mbox{Weight of specimen after heating in volatile matter test [g]} \\ F = \mbox{Weight of ash residue [g]} \\ W = \mbox{Weight of specimen used [g]} \\ M = \mbox{Moisture} \\ V = \mbox{Volatile} \\ A = \mbox{Ash} \\ H = \mbox{Fixed Carbon} \end{array}$ 

#### 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

# 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 to 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.



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 several 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 69.
- 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 on page 71.
- Use large crucibles to achieve faster and homogeneous drying / ashing. See also large crucibles and adapters in Chapter 19 Accessories and Consumables on page 71.
- 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 sample plate 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 100°C higher than test temperature. Keep you crucibles in the desiccator with silica gel.
   Place the crucibles on the sample plate 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 Ionizer.

## 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 surrounding temperature

Results of weighing are clearly incorrect

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

# **19 Accessories and Consumables**

# 19.1 Accessories

#### 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.

**3 inputs gas flow control unit for air plus 2 gases: 340-8502** Inbuilt gas flow control unit for air and 2 other gases like e.g. N2, O2

#### ECD - Enhanced Cooling Device: 340-8504

Inbuilt cooling system to enhance the cooling between runs ex works **340-9004**: Conversion set (only for prepASH units after November 2013)

#### Sample plate holder:340-8012

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

#### Condensation-Absorption-Unit (scrubber): 340-9001

For pre-incineration for sulphate 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.











# 19.2 Consumables

Sample plate for 29/19 samples, SiC, silicate ceramic: **340-4065** 

Sample plate for 12 samples, SiC, silicate ceramic: 340-4072

Set of 5 crucibles (porcelain glazed), 25 ml: **340-8037** use with sample plate 29/19 340-4065 APPROVED for applications with acid

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

Set of 5 crucible - lids, (Al2O3): **340-8033** use with sample plate 29/19 340-4065 and crucible 340-8032

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

Set of 5 crucibles, (porcelain glazed, SiC, ASTM-D-189-65), 40 ml: **340-8034** use only with sample plate 12 340-4072 and adapter 340-8035 APPROVED for applications with acid

Set of 5 crucibles, (porcelain glazed), 50 ml: **340-8038** use only with sample plate 12 340-4072 and adapter 340-8035 APPROVED for applications with acid
















## 20 Technical Data

Model	prepASH 229	prepASH 219	prepASH 212
Samples, Crucibles			
Samples	29	19	12
Sample plate for 19/29 samples	standard	standard	no
crucibles 25 ml	35	25	no
Sample plate for 12samples	option	option	standard
adapter	option	option	15
crucible 40 ml	option	option	15
crucible 50 ml	option	option	option
Weighing system	•	•	•
Weighing range	120 g	120 g	120 g
Readability	0.0001 g	0.0001 g	0.0001 g
Minimum sample weight	0.1 g	0.1 g	0.1 g
Heating control system	Ŭ	Ŭ	Ŭ
Temperature range	50 °C – 1000 °C	50 °C – 1000 °C	50 °C – 1000 °C
Temperature stability	+/- 2 %	+/- 2 %	+/- 2 %
Process control system			
Maximum program steps	10	10	10
Maximum total analysis time	36 h	36 h	36 h
	0.1-100 mg /	0.1-100 mg /	0.1-100 mg /
Auto stop	1min, 30min,	1min, 30min,	1min, 30min,
	60min	60min	60min
Atmosphere			
Oxygen, 0/3/6/9 l/min	option	option	option
Nitrogen, 0/3/6/9 I/min	option	option	option
Air, 0/3/6/9 l/min	option	option	option
Exhaust	yes	yes	yes
Absorption unit for sulphuric acid	option	option	option
Results			
Weight loss / Residual weight	%, ‰, g	%, ‰, g	%, ‰, g
Weight loss / Residual weight related	0/a 0/a a	0/ 0/ a	0/- 0/- <b>a</b>
to pre-interval	70, 700, Y	70, 700, Y	70, 700, Y
Repeatability	1 g / 0.02 %	1 g / 0.02 %	1 g / 0.02 %
Monitoring			
Remote on PC via Network	yes	yes	yes
Acoustic	yes	yes	yes
Operation			
Display	5.7" VGA color	5.7" VGA color	5.7" VGA color
Keyboard	touch screen	touch screen	touch screen
Menu-controlled	yes	yes	yes
PC independent operation	yes	yes	yes
Sample list, preparation and	1/00	1/00	1/00
evaluation on PC via network	yes	yes	yes
Sample preparation outside (with balance)	yes	yes	yes
Printout			
Graphic	yes	yes	yes
Table	yes	yes	yes
Method	yes	yes	yes
Memory capacity			
Methods	unlimited	unlimited	unlimited
Measured values	unlimited	unlimited	Unlimited

Model	prepASH 229	prepASH 219	prepASH 212
Data output, interfaces			
RS232 Interface for external weighing in	with USB converter	with USB converter	with USB converter
USB for Printer	yes	yes	yes
USB for Barcode Scanner	yes	yes	yes
USB for Memory stick	yes	yes	yes
USB for external weighing in	yes	yes	yes
Ethernet for Printer	yes	yes	yes
Ethernet for PC-Data-evaluation	yes	yes	yes
Ethernet for PC-Remote-observation	yes	yes	yes
Ethernet for PC-Remote-support	yes	yes	yes
Calibration			
Balance	2 points	2 points	2 points
Temperature	2 points	2 points	2 points
Connection			
Mains voltage	230 VAC (+15/-20 %)	230 VAC (+15/-20 %)	230 VAC (+15/-20 %)
Frequency	50 to 60 Hz	50 to 60 Hz	50 to 60 Hz
Current input	25 A	25 A	25 A
Weigh and dimensions			
Weight	99 kg	99 kg	99 kg
Dimensions (H) Height with cover opened	H (H) x W x D / 620 (980) x 590 x 870 mm	H (H) x W x D / 620 (980) x 590 x 870 mm	H (H) x W x D / 620 (980) x 590 x 870 mm