

Vapodest 50s carousel

Rapid distillation system


with control via PC

Instruction manual



Please read this instruction manual with care before you start operating the system!



Please observe the safety instructions of this manual marked with  in order to avoid any dangerous handling!



LAB Online Exhibition



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1 Important notes



1.1 How to use this instruction manual

Please make sure that the persons working with the Vapodest have read and understood this manual in order to observe all the instructions.

This instruction manual must be accessible to all users at any time and is a part of the Vapodest system!

The instrument will be called 'Vap 50s carousel' in the entire document.

This instruction manual only explains the technical details necessary for the operation of the instrument but is NOT a complete technical description.


The security advises given in this manual have to be observed under all circumstances! The symbols at the border of the text emphasize the dangers cited in the text.


Symbols at the instrument warn of possible dangers and will refer you to this manual!


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1.2 Explanation of symbols


	Attention! Advance notice of danger spots, carefully read instruction manual!
---	--


	Attention! Read instruction manual!
---	--


	Attention! Hot surface!
---	----------------------------


	Attention! Use eye protection!
---	-----------------------------------

	Attention! Mains voltage!
---	------------------------------

	Attention! Use protective gloves!
---	--------------------------------------

	Attention! Beware of hot liquids and vapours!
---	--

	Notice! Important information!
---	-----------------------------------

	Attention! Advance notice of substances which might be a health risk or cause irritation!
---	--

2 Safety

2.1 Operation as directed

With the Vap 50s carousel you have purchased a highly automated distillation system for the distillation of Kjeldahl digestions and associated distillations. Automatic addition of H₂O, NaOH, H₃BO₃ and titration acid as well as the removal of the sample remains after distillation by suction are features of this new system. Vapodest 50s carousel is controlled externally via PC or Laptop which needs to have the software "Vapodest Manager" installed.



Make sure to use the instrument according to our instructions, especially:

- all instructions of this manual
- the regulations in place in the country the instrument is used

All other usage is not recommended!



C. Gerhardt GmbH & Co. KG is not liable for any damages caused due to non recommended usage

- no modifications are allowed for safety reasons
- repairs of electric, electronic or mechanical components may only be done by authorized personnel!

2.2 Safety instructions



The Vap 50s carousel corresponds to the technical standard used at this date and observes the safety rules and regulations in place.

It is understood that the user has to observe

- the accident prevention regulations in place
- the general accident prevention regulations
- EU-regulations or other country specific instructions



Indicating label and danger symbols must not be taken off!



All safety measurements foreseen by Gerhardt must be observed and must not be taken out of operation.



Make sure that no liquid gets into contact with cable connections or the interior of the electrical parts of the unit!

- Danger of electric shock!



In case of an emergency disconnect from mains immediately!



Always pull the mains plug before opening!

- Danger of electric shock!



Prior to each start of the Vap 50 s carousel, make sure that the protection door and the carousel protection cap is closed tightly. Danger of burns and splashing liquids!



Make sure to wear protective gloves when taking the digestion tubes out and wear protective glasses as well.

The digestion tubes are very hot and there is the danger of burns!



Attention when handling chemicals! Please observe the manufactures safety instructions (see label) and your national safety regulations!

2 Safety



2.3 Work bench / Authorized user

The Vap 50s carousel must not be run in damp or hazardous location. The maximum humidity allowed is 80 %, the maximum room temperature must not exceed 35 °C!



The equipment must not be exposed to aggressive vapors of acids, alkalis, or solvents.



The Vap 50s carousel has to be placed on a horizontal and stable footprint.



The Vap 50s carousel may only be operated by trained staff or specially trained persons.

The user has to:

- read, comprehend, and observe the operation manual
- wear suitable clothes for laboratory work
- deny access to any unauthorized personnel



In order to operate the Vapodest-Manager, basic knowledge in the operation of a PC with Windows has to be present.

2.4. Danger spots at the instrument



- Attention, danger of burns on the surface of the digestion tubes!



- Attention, danger of splashing hot or caustic liquids at the tubing connections at the back of the instrument!



- Attention, danger of burns when taking out the hot glass container!
Use gloves when removing the glass digestion tubes!

3 Technical description

3.1. Warranty conditions

Vapodest 50 carousel is designed and fabricated following the high quality guidelines of DIN EN ISO 9001. On the basis of the C. Gerhardt conditions of warranty our products are guaranteed for 1 year as long as the equipment is used according to the instructions in this manual. Also fill out the "**Report for the installation and initial operation**" and send back to C. Gerhardt.

Please note that the following parts are excluded from warranty.

- Digestion tube, Order-no.: 12-0301
- Vitone cone, Order-no.: 12-0351

3.2. Technical data

Nominal voltage: 230 V AC 50 Hz oder 115 V 60 Hz oder
230 V 60 Hz

Nominal wattage: 1830 W

Cooling water consumption: about 3 liters per distillation minute

Cooling water pressure min. 0,5 bar, max. 6 bar

Pump capacity (read chapter 7.9. calibration dosing pumps)

- Diaphragm pump about 15 ml/s with water
about 10 ml/s with NaOH
- Peristaltic pump about 10 ml/s with water

Storage tanks: 2 x 20 Liter, 2 x 10 Liter, 1 x 5 Liter

Display: LED with illumination

Number of programs: unlimited

Dimensions: 1010 x 670 x 1655 mm (B x T x H)
(1290 mm with board attached on the side)

Weight: 190 kg

Recovery rate: > 99,5 %

Reproducibility: +/- 1 %

Detection limit: 0,1 mg N

Interfaces:

- Vapodest 2 x RS 485 interface: Connection for GerBus®
1 x 5-5-pin connector for level sensor
- ComBox 2 x USB, 1 x RS 485 for GerBus®,
1 x RS 232 connection for balance

Micro dosing pump: Deviation of flow rate < 0,2 %

PC-requirements: IBM compatible PC with Windows XP (recomm.) or Vista
Min. 512 MB RAM - Min. 1 Ghz Prozessor -
Min. 20 GB harddisk - Min. 1 USB port

4 Description of the equipment

4.1 Front view Vap 50s carousel



4 Description of the equipment

4.1.1. Description Front view Vap 50s carousel

Fig. 4.1.1. a
Drawer with tanks open



1 Adjustable feed (Important, fix adjustable feed), Bestellnr. 1001891

The adjustable feet have to be screwed in completely in order to ensure absolute stability. Also use included rubber caps.

2 Drawer with set of tanks and compressor

Please open the drawer in order to fill the tanks. See Chapter 5.5. for the description of how to fill the tanks. The instrument should not be operated with an open drawer!

- H₂O-tank 20 l, NaOH-tank 10 l, H₃BO₃-tank 10 l, Sample Waste tank 20 l
- Compressor JunAir

Important: after emptying the sample waste tank you should make absolutely sure to have the plug for the level sensor put in correctly. Otherwise, the software cannot check the level in the tank; an error message will be displayed.

3 Hinged handle (lockable)

To be able to open the drawer the button of the hinged handle has to be pushed in so that the handle will fold down. To close the drawer the handle has to be pushed inwards again.

4 Carousel- also see Chapter 10.1.

The carousel can take the original Gerhardt sample tubes. Depending on the desired configuration the carousel can take the following sample tubes:

- Carousel 20-place, 20 x 250 ml
- Carousel 16- place, 16 x 400 ml (KDD- or BS-400)
- Carousel 12- place, 12 x 800 ml

5 Carousel protection hood with handle

To change the carousel or clean it, gently lift the protection hood and pull it out sideways (also see chapter 10.2.1.)

6 Loading flap

Open the loading flap for loading and unloading of the carousel. The instrument cannot be operated with an open loading flap.

7 Turn-lock fastener for carousel

To lift off the carousel, unscrew the knob; after inserting the carousel the turn-lock fastener has to be screwed on tight again.

8 Upper Part Distillation System

For further description of the Distillation System please see Chapter 4.3. "Front view Vapodest upper part".

9 Service Drawer

For loading and unloading of the carousel the switch has to be touched gently, the carousel will then move to the next loading position. Should the switch be hit longer so that a signal is heard and the service drawer opens automatically (see Chapter 13).

10 Sensor Switch for Carousel, maintenance-free

Switch with dual function

In order to load or un-load the carousel, press it shortly.

If you want to open the service drawer, press the switch for a longer period (ca. 5 sec)

Fig. 4.1.1. b
Service drawer open



Fig. 4.1.1. c
Side extension

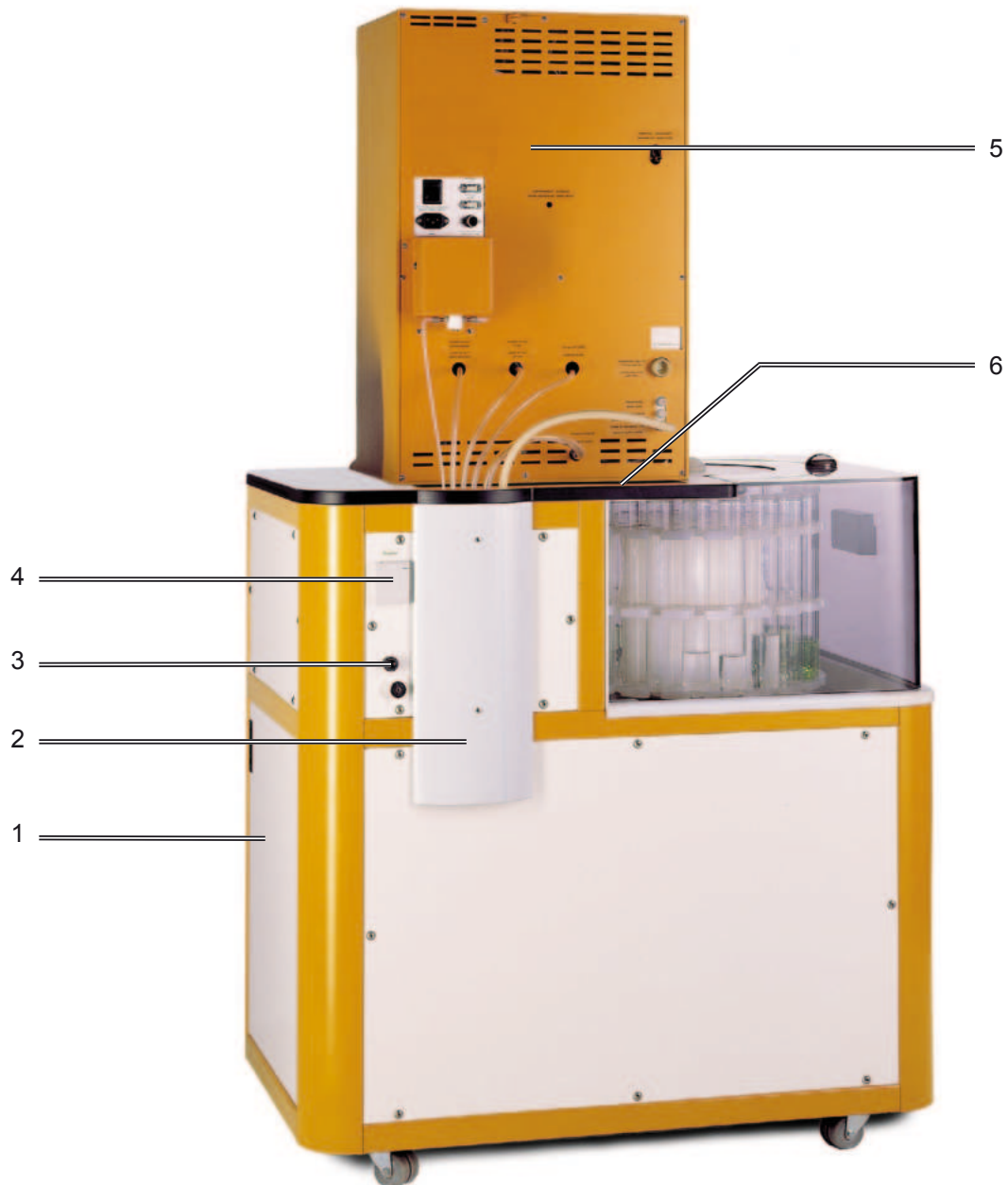


Side extension

The side extension is included in the shipment and you can easily attach it on the side. You can e.g. use it to put your laptop there. In order to attach the side extension please use the enclosed Allen key.

4 Description of the equipment

4.2 Rear view Vap 50s carousel



4 Description of the equipment

4.2.1 Description rear view Vap 50s carousel

1 Side door for titration tank

Behind the door there is the tank for the titration acid. To open the door, push in the handle and pull the loop.

2 Tubing duct

For the protection of the tubes

3 Connection for external compressed air connection

If available, the in-house compressed air supply can be connected here, if the compressor included in the shipment is not connected.

4 Socket at the instrument

For the peripheral instruments (e.g. balance or laptop). When turning the Vapodest on, the current supply is turned on as well.

5 Upper Part of Distillation System

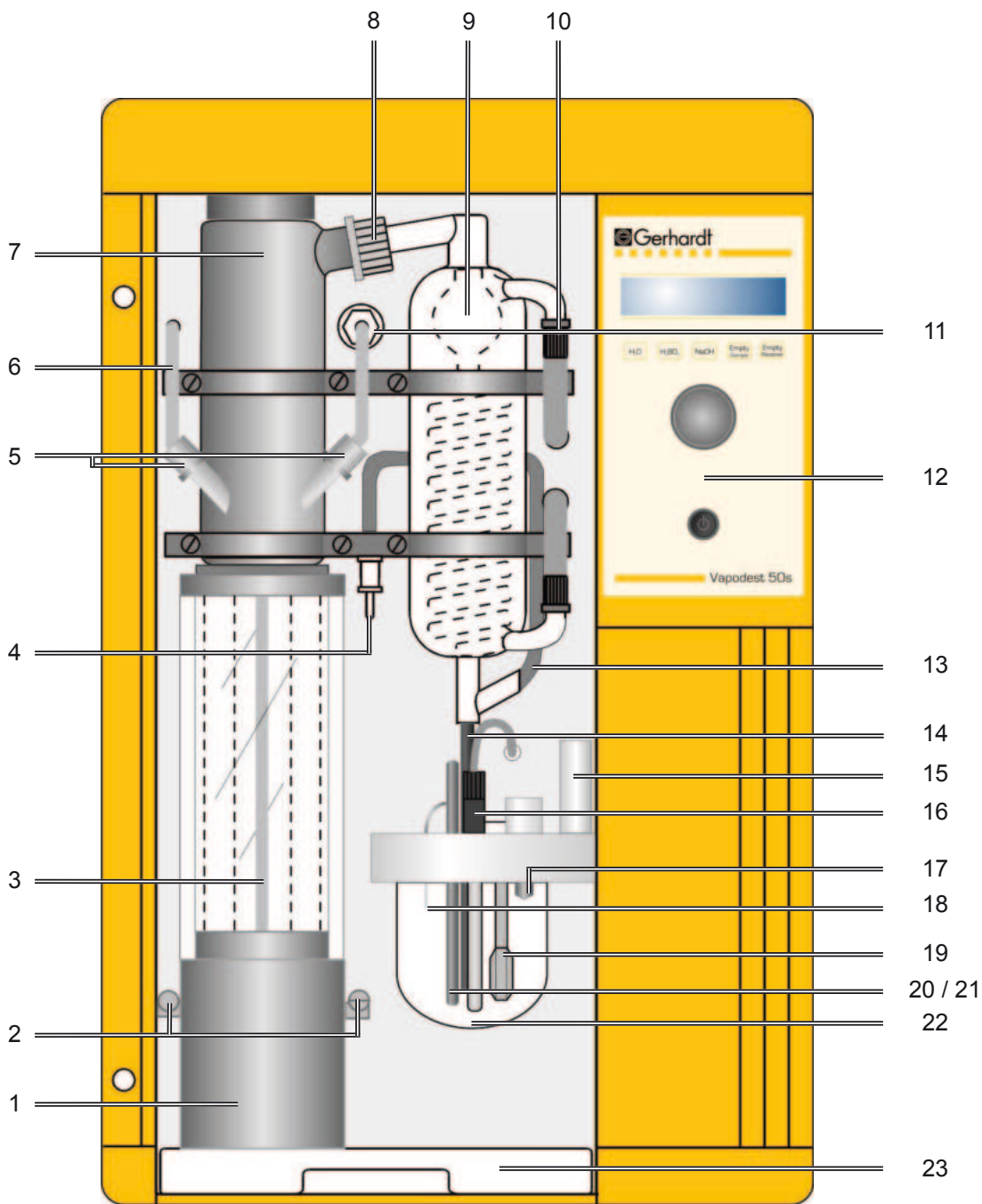
Further description of the upper part can be found in chapter “4.4. Rear View, upper part Vapodest”.

6 Dripping tray

Collects any drops of chemicals. If necessary, make sure to clean it.

4 Description of the equipment

4.3 Front view Vapodest upper part



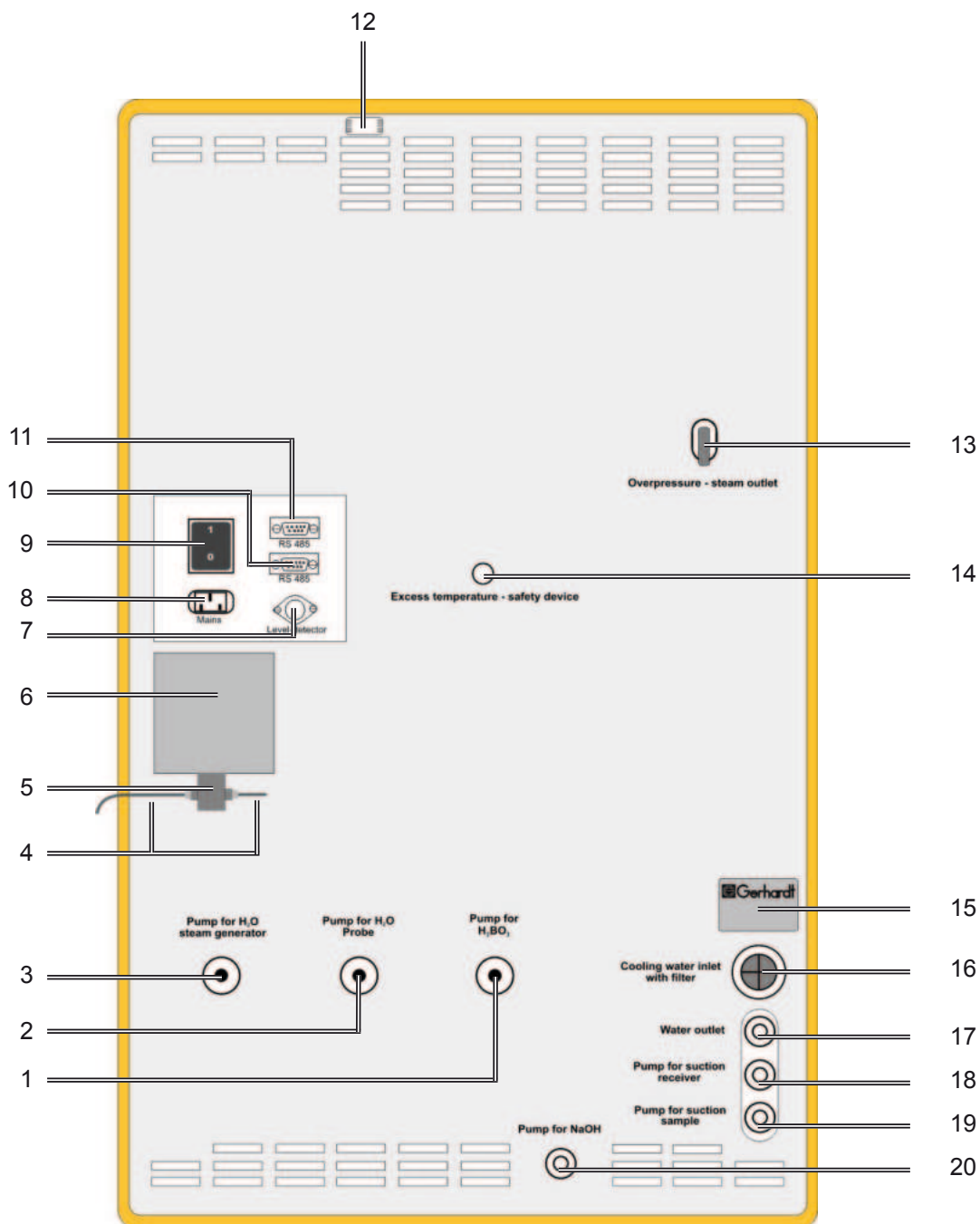
4 Description of the equipment

4.3.1. Description front view Vapodest upper part

- 1 Basket (3 versions for different size digestion tubes are available)**
Takes the sample tubes. It should only be taken off to be cleaned or exchanged (see also chapter 10.2.2.).
- 2 Tension Fork, Item-No. 1001575**
Facilitates the fast and safe clamping of the basket. Push the tension fork down to insert or lift the basket.
- 3 PTFE-inlet tubing, steam, Item-No. 1002178**
Steam, water for dilution, and the suction of the sample residues passes through here. Please check regularly that the opening of the PTFE-tube is not blocked by crystallisation.
- 4 Ventilation valve, Item-No. 1000036**
During and after distillation the ventilation valve prevents suck back of the receiver solution into the condenser.
- 5 Screw cap GL 18, Item-No. 1004321**
with silicone/PTFE seal
- 6 PTFE-Inlet tubing, NaOH, Item-No. 1003401**
Sodium hydroxide solution enters here.
- 7 Distribution head PP, Item-No. 1000056**
The distribution head prevents that any sample residue gets into the receiver.
- 8 Screw cap GL 32, Item-No. 1004322**
With silicone/PTFE seal
- 9 Distillation condenser, Item-No. 1000059**
Combined ball- and coil condenser with high cooling power.
- 10 Screw cap GL 14, Item-No. 1004320**
With plastic screw connection
- 11 PP-Distributor with PP-threaded joint, Item-No. 1000538**
- 12 Control panel Vapodest**
See chapter "6.2 Control Panel"
- 13 Tube for ventilation valve, Item-No. 1000484**
Connection tube to the ventilation valve
- 14 Distillate outlet tubing, silicone 8/12, Item-No. 1000496**
- 15 pH electrode holder, Item-No. 1001257**
The holder can be filled with potassium chloride solution 3.5 mol/l. Here you can store the electrode when it is not in use to prevent from drying out.
- 16 pH-electrode (combined electrode), Item-No. 12-0355**
Combined electrode with screw thread. For further details please check the enclosed instructions by the electrode's manufacturer.
- 17 Sensor for level detection, Item-No. 1001265**
The built-in sensor stops the program if the receiver vessel overfills during the distillation and titration.
- 18 Inlet tubing for titration acid, Item-No. 1001672**
- 19 Stirring motor, Item-No. 1001266 with propeller, Item-No. 1001260**
- 20 Tubing for suction of the distillate, Item-No. 1000498**
- 21 Inlet tubing for boric acid, Item-No. 1000188**
Is behind the tubing for suction of the distillate
- 22 Receiver vessel, Item-No. 1001269**
For easy removal turn the glass through 90° and pull it downwards.
- 23 Drip tray for upper part, Item-No. 1001586**
Condensate which might drip is collected in the drip tray. Make sure to clean the tray regularly.

4 Description of the equipment

4.4 Rear view Vapodest upper part



4 Description of the equipment

4.4.1. Description rear view Vapodest upper part

- 1 Diaphragm pump for H₃BO₃, Item-No. 1001736**
Connection pipe for PVC-tubing 4/7, connection to the storage tank "H₃BO₃"
- 2 Diaphragm pump for H₂O Probe, item-No. 1001736**
Connection pipe for PVC-tubing 4/7, connection to the storage tank "H₂O".
- 3 Diaphragm pump for H₂O Dampferzeuger, Item-No. 1001736**
Connection pipe for PVC-tubing 4/7, connection to the storage tank "H₂O".
- 4 Inlet and outlet tubings for titration acid**
Bubbles must be avoided under any circumstances in the inlet tubings in order to guarantee precise titration results (also see instructions in Chapter 7.7. Rinsing dosing pump).
- 5 Micro dosing pump (Deviation of flow rate < 0,2 %), Item-No. 1001650**
Micro dosing pump made of chemically inert ceramic components.
- 6 Box for micro dosing pump**
Protection for micro dosing pump
- 7 Level detector**
Connector for diode plug, 3-pin (level detector for external storage tanks)
- 8 Appliance plug for mains cable**
The enclosed mains cable is plugged in here
- 9 Excess current switch, 10A**
Equipment is cut off automatically if there is excess current
- 10 Interface RS 485, ComBox**
Connection for data cable RS-485 ComBox.
- 11 Interface RS 485,**

- 12 Outlet ventilation tubing (protected)**
Ventilation for steam generator, here hot steam can escape
- 13 Excess pressure - steam outlet**
Safety device to let steam escape in case of overpressure in the boiler.
- 14 Excess temperature fuse**
Safety measurement to turn off heating if the water level in the steam generator drops so low that the heating cartridge is not covered any more. See also error message in chapter 12. 1 "NO H₂O FOR STEAM GENERATOR".
- 15 Identification plate with serial number of the instrument**
Please make sure to always refer to the serial number in any case of service problem.
- 16 Cooling water inlet with sieve, Item-No. 1000363**
Connection thread $\frac{3}{4}$ inch for water inlet tubing 10/17
- 17 Water outlet**
Connection pipe for PVC-tubing 8/12, connection to water outlet
- 18 Peristaltic pump for receiver, Item-No. 1000084**
Connection pipe for PVC-tubing 8/12, connection to water outlet
- 19 Peristaltic pump for sample suction, Item-No. 1000084**
Connection pipe for Verprene-tubing 8/12, connection to store tank "Sample waste".
- 20 Diaphragm pump for NaOH, Item-No. 1001738**
Connection pipe for PVC-tubing 8/12, connection to storage tank "NaOH"

5 Assembly and installation

5.1. Check for transport damage

Before assembling the apparatus please check if the content of the box is complete and intact!

In case of any damage, please notify your carrier (post, rail, road) as well as your supplier!

5.2. Unpack apparatus and set up

You need a connection (cold water pipe) with 1/2 inch thread to connect it to the water supply.



The VAPODEST must not be run in damp or hazardous location. The maximum humidity allowed is 80 %, the maximum room temperature must not exceed 40 °C!

Please observe the local water and waste regulations and those of your public water supply company! Please note that the length of the inlet and outlet tubing is restricted to 2 metres.

Make sure to take a faucet, which can be connected to the inlet tubing permanently. The water pressure must be at least 0,5 bar in order to activate the integrated pressure detector.

The VAPODEST 50s carousel distillation system comes fully pre-assembled. Please unpack the equipment with care !

1. **Important!** Set the adjustable feet (see also chapter 4.1. no.1) to secure the instrument. Prior to that put the rubber caps under the feet.
2. Unpack accessories

The exact contents of the delivery can be checked by the following list.

5 Assembly and installation

5.3. Package list

- 1 x Distillation system Vapodest50 carousel,
complete mounted with carousel, compressor, and storage tanks
- 2 x Rubber caps D 80 (are put on the adjustable feet), Item-No. 1001891
- 1 x Side extension (for Laptop or display), Item-No. 1002656
- 1 x Allan key, size 4, item-No. 1002069
(to attach the side extension)
- 2 x Keys (to lock the drawer for the set of tanks), item-No. 1002805
- 1 x Electrode with screw plug connection, Item-No. 12-0355
- 1 x Set Digestion tubes, depending on the type of carousel:
 - for carousel 20-place = 20 x 250 ml- tubes, Item-No. 12-0301
 - for carousel 16-place = 16 x 400 ml- tubes
Type KDD 400, Item-No. 12-0310 or BS 400, Item-No. 12-0308
 - for carousel 12-place = 12 x 800 ml- tubes, Item-No. 12-0311
- 1 x Buffer solution pH 4.00, 250 ml, Item-No. 1000176
- 1 x Buffer solution pH 7.00, 250 ml, Item-No. 1000177
- 1 x KCl Electrolyte solution 250 ml, Item-No. 1000178
- 1 x Water inlet tube 10/17 with connections of $\frac{1}{2}$ and $\frac{3}{4}$ inch, 2 m
Item-No. 1000394
- 2 x PVC-tube 8/12, 2 m (transparent), Item-No. 1000047
- 1 x Verprene-tube 8/12, 2m (white), Item-No. 1000499
- 1 x Mains cable, Item-No. 10-0058
- 1 x Oil-bottle, instruction manual and cap for compressor, Item-No. 1004457
(are located in the drawer for the tanks next to the compressor)
- 1 x CD-ROM Vapodest Manager, Item-No. 1004160
- 1 x ComBox with RS 485, USB and interface for balance, Item-No. 1004313
- 1 x Set data cable:
 - USB-cable, 5 m, Item-No. 1004365
 - Data cable DK 45, 5 m, Item-No. 10-0193
 - CD with USB/serial RS232 converter driver, Item-No. 1004367
- 1 x Instruction manual Vapodest 50s carousel
- 1 x Test report
- 1 x Report for the installation and initial operation

5 Assembly and installation

5.4. Tubing connections

When establishing the tubing connections, make sure to observe the labeling of the connection at the rear of the instrument. Always make sure that the connections are sitting tight.

1. Connect the water inlet tubing for cooling water (pressure resistant hose 10/17) with the cooling water inlet at the rear of the instrument and water supply.
2. Connect water outlet tubing (PVC-tubing 8/12, transparent) to corresponding pipe at the rear of the equipment and place in the drain.
3. Connect receiver outlet tubing (PVC-tubing 8/12, transparent) to corresponding pipe at the rear of the equipment and place in the drain.
4. Verprene-Tube 8x12 (can be used alternatively to get rid of the sample waste if it is possible to direct it directly into the waste water system). Otherwise, the manufacturer has prepared the interface for directing the sample waste into the sample waste tank.

All other tubing connections have been done by the manufacturer. However it is recommended to check the proper fitting of the tubings and the corresponding connection piece (see also chapter "14 Tubing diagram").

5.5. Fill storage tanks with chemicals



Be careful when working with acids and alkalis! Make sure you observe the safety instructions concerning work with hazardous materials!



- **Open drawer with tanks**
- Loosen the lock and unscrew the lid of the tank
- Fill tank H₂O with distilled or demineralized H₂O
- Fill tank NaOH with NaOH 32% - 40 %
- Fill tank H₃BO₃ with H₃BO₃ 2-4 %
- After the filling has been completed screw on the lid of the tank and close the lock (you have to hear the ,click'!)
- Close drawer with tanks



- **Fill tank with titration acid**
- The tank for the titration acid is behind the door on the right side
- Open door (push in handle and grab the loop)
- Unscrew lid of the tank
- Fill storage tank with titration acid
- Screw on lid and close side door

5.6. Connection of the electrode

Make sure to read the enclosed instructions for electrode

1. Take off the upper screw cap and the protection cap. Now, connect the electrode to the cable and carefully introduce it in the receiver vessel (see chapter 4.3, pos. 16)
2. Fill the holder for the pH-electrode up to the marked level with potassium chloride solution 3.5 mol/l. You should store the electrode in here in order to prevent it from drying out.

5 Assembly and installation

5.7. Connection of PC / Laptop

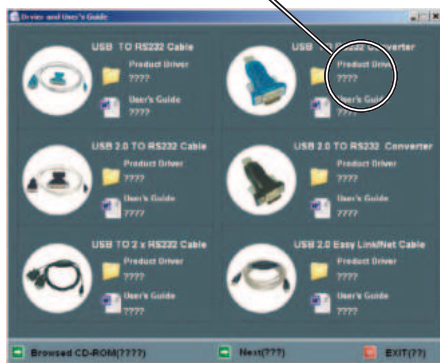
The data exchange between the Vapodest and the PC is done using the Gerhardt Com-Box. The customer needs a USB-interface for connecting GerBus to the PC. The ComBox is part of the instrument and cannot be combined with other Vapodest-instruments.

Please note!

To avoid problems during the installation, please make sure that the USB-driver is installed on the PC PRIOR to connecting it to the ComBox!

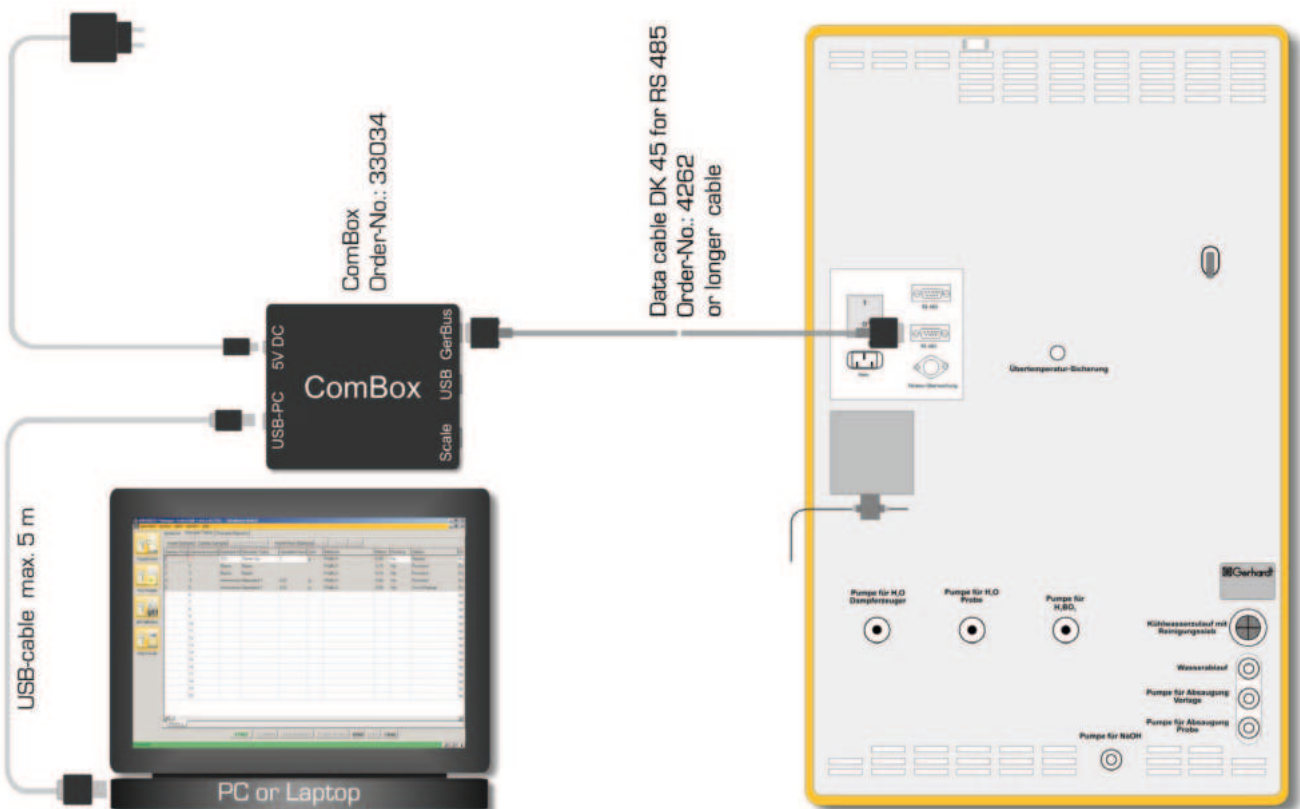


Fig 5.6.
choose this USB-driver



1. Switch on PC or Laptop
2. **Install USB-driver for the ComBox.**
Insert the CD-ROM "Driver and user's guide" and Install the driver "USB to RS 232 Converter" according to the instruction of the manufacturer.
3. Connect mains cable with ComBox and the mains connection.
4. Connect USB-data cable (max. 5m) with USB-interface of the ComBox and the USB-interface at the PC.
5. Connect data cable DK 45 (5m) with the RS 485 interface of the Vapodest and the RS 485 for the ComBox.

Note: It is possible to connect a longer data cable with e.g. a length of 100 m or longer. This has the advantage that the Vapodest and the PC don't have to be in the same room.



5 Assembly and installation



5.8. Mains connection

Please check the nominal voltage on the product information plate before connecting to the mains. Should the nominal voltage deviate for more than +/- 10%, then you must contact your service engineer.

- Make sure that the mains switch at the front of the instrument is in position "0"
- Connect the mains cable with the rear appliance plug and the shockproof socket
- Check if excess current release at the rear (chapter 4.2., pos. 9) of the equipment is in position „1“. This switch turns the equipment off when it takes too much current.

5.9. Connection to the balance

Set up the balance according to the instruction manual enclosed. Connect balance to Vapodest using the data transfer cable (The respective data cables can be obtained directly from the manufacturer of the balance). A balance with a standard RS 232 interface can be connected directly with the ComBox.

In chapter '7.1.3. Type of balance' you are told according to which criteria to choose the correct type of balance.

If there are any problems with your balance please contact the manufacturer of the balance or C. Gerhardt.

5.10. How to install the software

Vapodest Manager can be installed on a PC/Laptop with Microsoft Windows XP® or Windows Vista®.



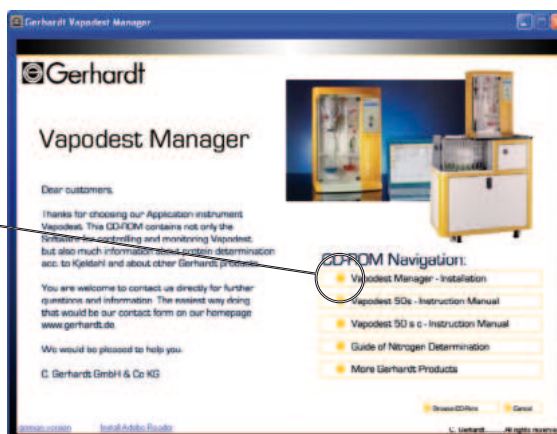
Important windows settings

To avoid any malfunctions of the Vapodest Manager, please make sure to **switch off all energy saving options incl. screen saver** in the Windows control panel. If you work with different user accounts please make sure to log in as administrator before starting the installation.

5.10.1. How to install Vapodest Manager

1. Insert CD-ROM and select "Setup.exe" (if auto start function is not activated).

Vapodest Manager installation



5 Assembly and installation

2. After selecting the language, select "**Vapodest Manager Installation**" from the menu. The automatic installation routine will start. Please be kind enough to follow the instructions on the screen and confirm the individual steps.
3. The installation is terminated successfully by pressing the key 'Finish'. There will be an automatic shortcut to your desktop.

5.11. Turn on apparatus

Always close the protection door before operating the distillation unit!

1. Turn on tap completely.
2. Start Vapodest by turning the mains switch on..

In the Vapodest display there will be info about the current software version. Then the info about the present status of the instrument will be displayed.

After switching on the unit the pump to fill the steam generator starts immediately. The steam generator is filled with water up to the pre-set level, then the heating starts. In order to run an analysis you have to wait for about 5 minutes till the steam generator has reached its operating pressure. All other functions are available right away.

3. Turn on the PC and start the program Vapodest-Manager by double-clicking on the corresponding desk top symbol.
4. At the first start after the initial installation, Vapodest-Manager will check the following settings.
 - Confirm selection of COM-Port with "**OK**" (see chap. 7.1.2. Select interface).
 - Select language. Choose your language from the menu bar. Press button "**Now**" to enter further system settings (see chapter 7.1. Configuration) or press button "**Later**" if you like to make further system settings at a later time (recommended).
 - Run pH-calibration (see chapter 7.6. pH-calibration). An analysis can only be run after a successfully done pH-calibration.
 - Define methods (see chapter 8 Create a method)
5. Now you can enter further system settings or start an already entered program. Make sure to read the next chapter!

Vapodest 50s
01 Aug 2007

Vapodest 50s
Firmware 1.17

Vapodest 50s
Standby



6 Operation

6.1. General information / Main functions

The operation of the Vapodest is done using a PC with the controller software Vapodest-Manager. Using the operator button, various operations, like e.g. the pH-calibration or rinsing of the pumps, can be started directly at the distillation unit. The bi-directional data exchange between PC and Vapodest allows a permanent monitoring of the status directly at the monitor of the PC. In case of any problems, there will be an error message.

Vapodest Manager offers you the following main functions:

1. System setting

Functions for e.g. selection of language, basic functions, calibration of the pumps, pH-calibration, titration functions, statistics about the instrument, and many more features.

Make sure to also read chapter 7

2. Create a method (Programming)

Determine program parameter for the various methods, store and administer them. All parameter necessary for controlling the distillation/titration are defined as well.

Make sure to also read chapter 8

3. Sample input

Defining the series and input of sample data like e.g. the sample name, weight, kind of sample etc. A series can have up to 20 samples, which can be different sample types, blank samples and standards.

Make sure to also read chapter 9

4. Execution of method

Start analysis, monitor status of analysis etc

Make sure to also read chapter 10

5. Table of results

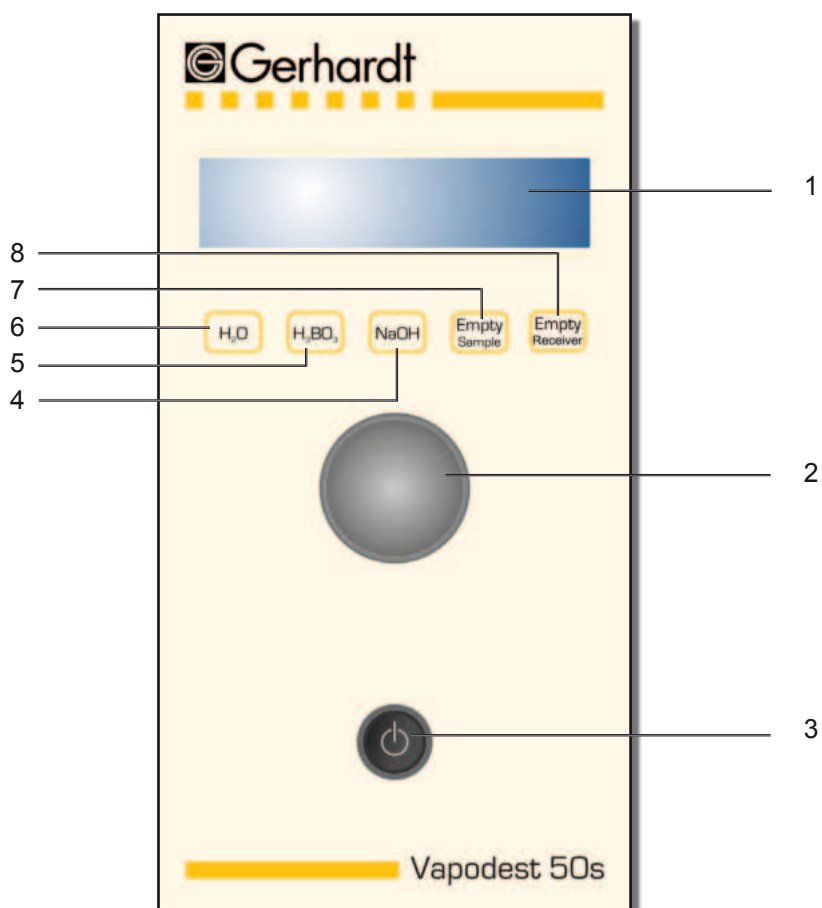
Display of the results, administration of results, information about running of analysis, in- and export of table with results, and many more.

Make sure to also read chapter 11

6 Operation

6.2. Operator panel Vapodest

Fig. 6.2.A:
Operator panel
Vapodest 50s

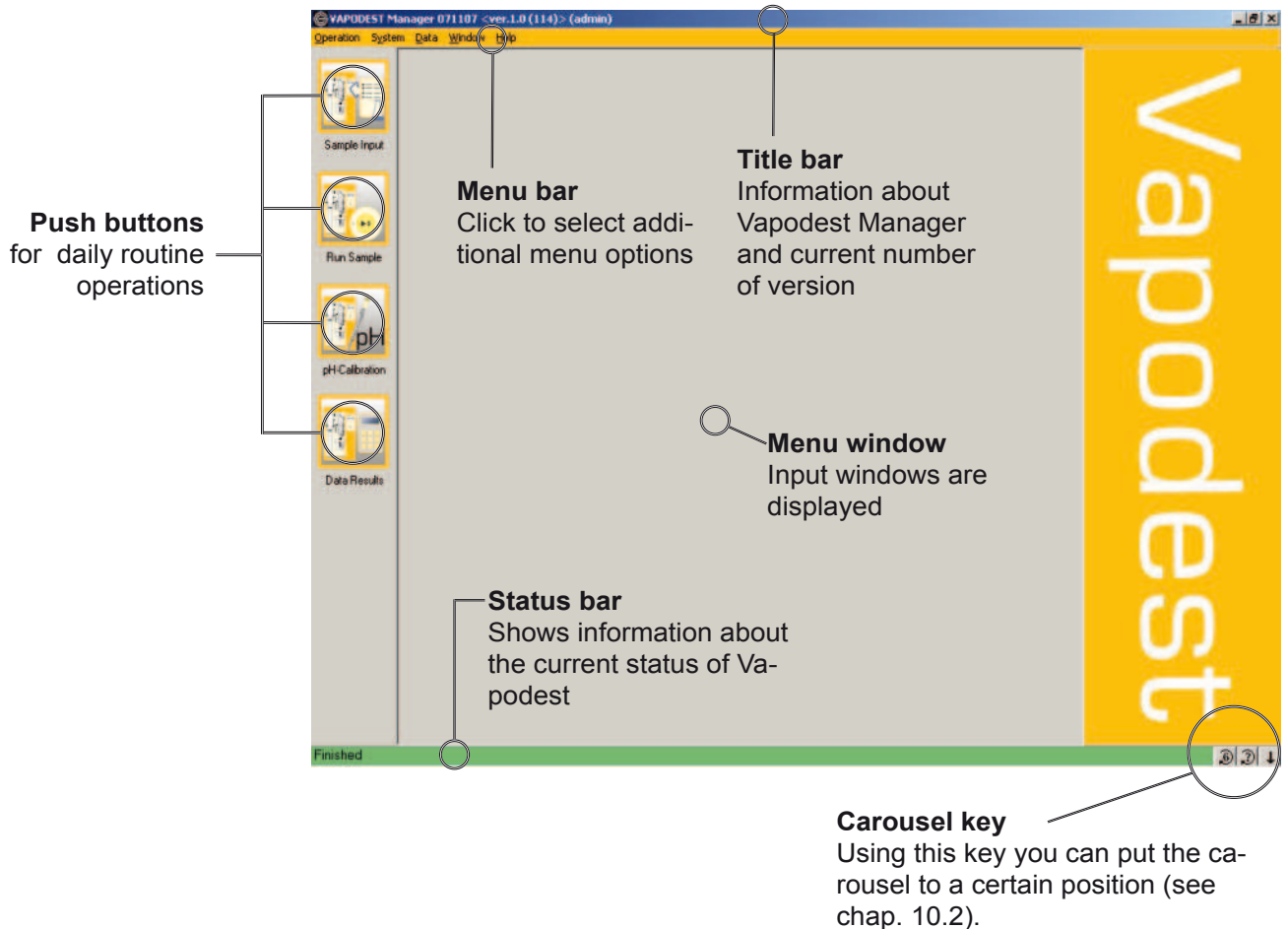


- | | |
|---|---|
| 1. Display | Shows the current status of the instrument |
| 2. Operator button | Key button with dual function
Turn it= contrast is increased or decreased
Press it = store contrast settings
and confirm procedure |
| 3. Mains switch | Turn instrument on or off |
| 4. Key "NaOH" | Manual addition NaOH |
| 5. Key "H₃BO₃" | Manual addition H ₃ BO ₃ |
| 6. Key "H₂O" | Manual addition H ₂ O |
| 7. Key "Empty Sample" | Manual suction sample tube |
| 8. Key "Empty Receiver" | Manual suction receiving vessel |

6 Operation

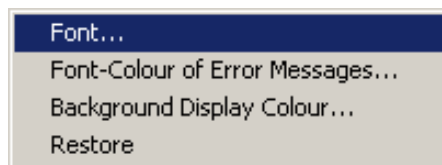
6.3. Operator panel Vapodest Manager

After starting Vapodest Manager, the following window will be visible:



Additional operator panels

Use your right mouse key to click on the program window to open the menu context:



You can determine fonts, colour for error messages and background of screen. The selection of fonts and colours is done in the typical style of Window.

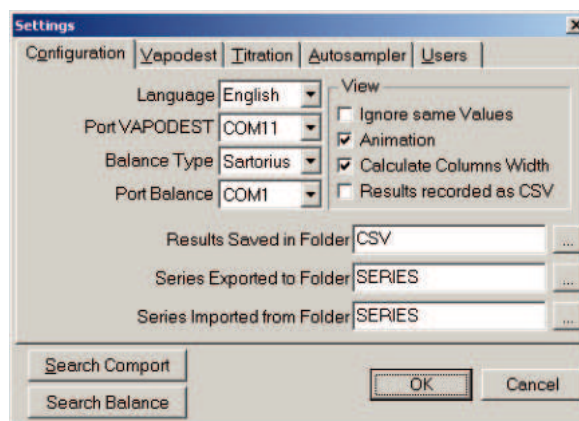
7 System settings

In this chapter the basic functions for Vapodest are explained. For standard procedures it is sufficient to determine these functions once when the instrument is taken into use for the first time.

Press the key "OK", to confirm changes. Press key "Leave", to abandon menu field without any changes.

7.1. Configuration

In the menu bar, select **System > Settings**, in order to open the following window.



7.1.1. Language

At any time, you can select a language. It is not necessary to start the software again to do so.

7.1.2. Select interface- Port Vapodest

Vapodest Manager automatically takes COM 1 as available interface. If the distillation unit is connected to another port, this one has to be selected in the list of options. Press key "Search Comport", to select a free interface.

7.1.3. Type of balance


Here, you select the type of balance you are working with. Should it not be listed here, contact C. Gerhardt.

7.1.4. Select interface for balance - Port balance


Select the relevant interface of the balance from the list of options (see also chap. 5.9).

7.1.5. Determine the locations of storage for results, in- and export


- Saving the data of results in file ...

Press the key , to define a memory for your analysis results. The results will be stored in CLV format, so that they can be imported by most spreadsheet programs. Each of the results is stored in its own file.

- Storing of data of series in folder ...

Press the key , to define a memory for the export of your series data. The entire series is stored in its own file.

- Series of files is imported ...

Press key , to determine a search path for the import of the data for the sample series.

7 System settings

7.1.6. View

Ignore identical values

This option is for the presentation of data in a logbook-table. Should several values be identical within one column, they will be shown by a space holder.

Animation

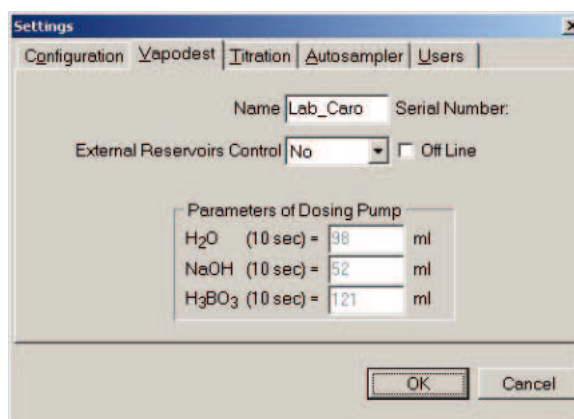
Activate this option so that during the analysis a simple animation is shown.

Width of column automatically

Relates to the presentation of the texts in the columns of the table. Activate this option to automatically adapt the column width to the length of the text automatically.

7.2. Vapodest

In the menu bar select **System > Settings > Vapodest**, to open the following window.



7.2.1. Name

Enter any name for Vapodest. This name will be on all print outs of the system.

7.2.2. Using an external set of tanks

Please answer with 'yes' only if you are using the set of tanks KAN 50 with level sensor.

7.2.3. Pump volume of the dosing pumps

Information about the delivery volume of the respective dosing pumps. The calibration of the dosing pumps is described in chapter "7.9. Calibrate dosing pumps".

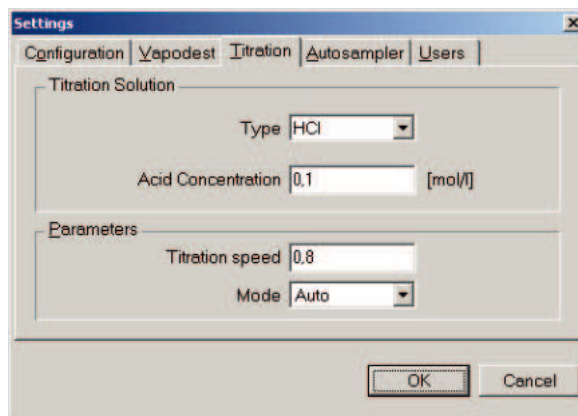
7.2.4. Off line (demo mode)

Activate this option to start Vapodest Manager in self-sufficient demo mode (without distillation system), e.g. for presentations, trainings and many more.

7 System settings

7.3. Titration

In the menu bar select **System > Settings > Titration**, to open the following window.



7.3.1. Functions titration solution

Selection of the titration acid and its concentration [mol/l].

- **Default: HCL, H2SO4 and other**

7.3.2. Parameters

- Titration speed

The titration speed can be influenced by this factor.

It considers the various pH-changes for various concentrations of the titration solutions and boric acids. If the factor is set to 1, a titration is done fitted to a 2% boric acid, which is titrated with a respective (0.1 mol/l) titration solution. If another titration solution is selected, e.g. with a concentration of 0.5 mol/l. there is the danger of uncontrolled over dosing with same titration functions. Here, the titration speed can be adapted with the help of the factor.

Rule is: Factor > 1, titration speed is reduced
Factor < 1, Titration speed is increased

Calculation of titration speed

$$S = \frac{\text{pHx} - 6,1668}{c \times 6,66}$$

pHx = start continuous pumping in distilling mode

c = Concentration in mol/l

S = Titration speed

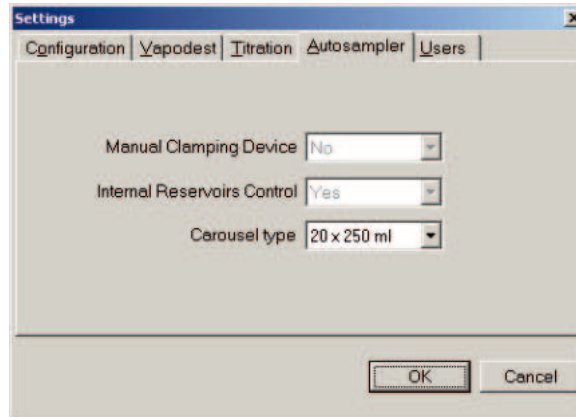
- Mode

You can chose between 3 different tables or by selecting "Curve" work without tables. Detailed information on how to work with titration tables is available in chap. '7.8. Titration table'.

7 System settings

7.4. Autosampler

In the menu bar select **System > Settings > Autosampler**, to open the following window.



7.4.1. Manual clamping device

Only when working with Vapodest 50 without the carousel (autosampler), you have to enter "yes" here (also see chap. 10.6. Manual change of samples).

7.4.2. Internal reservoir control

Only press 'yes' when using the internal set of tanks for the carousel otherwise make sure to press 'no' always.

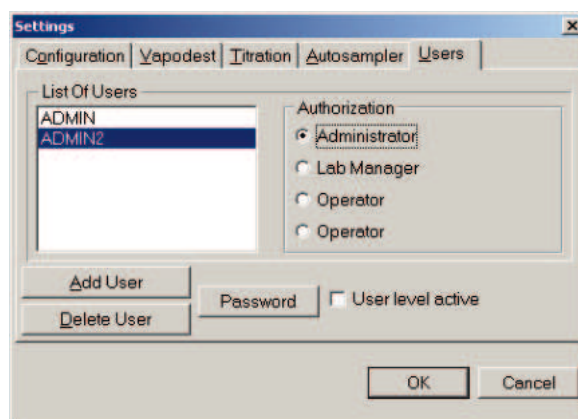
7.4.3. Carousel type

Select the type of carousel you want to use:

- 20 x 250 ml (standard)
- 16 x 400 ml
- 12 x 800 ml

7.5. How to manage user

In the menu bar select **System > Settings > User**, to open the following window.



Registration as administrator

To be able to use the following functions, you have to register as "Administrator". Mark the field "User area active" and press button "Password".



7 System settings

Enter a new pass word in the field **"Enter password"** and confirm password by re-entering it in field **"Repeat password"**. Then press **"OK"**.



Make sure to remember your pass word to be able to register again as administrator.

7.5.1. Add user

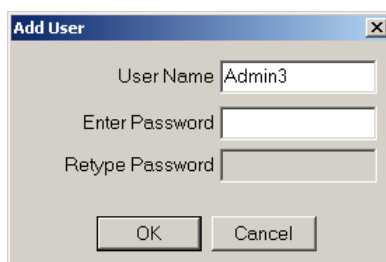
Press key **"Store user"** and enter the user name and the respective authorisation. Then press **"OK"**.

7.5.2. Delete user

Select a name from the user list and and press key **"Delete user"**. Then press **"OK"**.

7.5.3. Administration of password

Select a name from the user list and press the key **"Password"**.



Assign new password

Enter a new pass word in the field **"Enter password"** and confirm pass word by re-entering it in field **"Repeat pass word"**. Then press **"OK"**.

Delete password

Press key **"Delete"**, to delete an existing pass word.

7.5.4. Authorisation (Assign user rights)

Select a name from the user list and then this name is assigned a user right from a selection of the list **"Authorisation"**.

- **Administrator**
Unlimited user rights.
- **Lab Manager**
Extensive user rights, no assigning of pass word, but system functions, setting up methods, input of samples, running of analysis, pH-calibration possible.
- **Operator**
Limited user rights, setting up methods, input of samples, running of analysis. pH-calibration possible.
- **Lab technician**
Very limited user rights, running of analysis, input of sample data, pH-calibration possible.

7 System settings

7.6. pH-Calibration

7.6.1. General reference notes for pH-calibration

Combined electrodes are subject to a regular wear and tear and thus, should be calibrated regularly. The pH-drift is compensated at the start of the analysis when the automatic reading of the pH-value is compensated. However, as soon as the reaction time of the electrode is > 30 s a check or a replacement of the electrode is advisable.

A two point calibration with buffer solution pH 4 and pH 7 is done. The deviation from zero is obtained with buffer solution pH 7 and the steepness with buffer solution pH 4.

The values obtained are shown in [mV/pH] and [%], values accepted are a zero point of 0 mV and a steepness of 59 mV/pH (100 %). The tolerance range of the deviation from zero is +/- 20 mV. The steepness is accepted between 50 mV/pH and 70 mV/pH. The ideal value with buffer solution pH 4 is at 177 mV and at a pH-value of 7 at 0 mV.

Another criterion for the state of the electrode is the reaction time. If the electrode reacts slowly during the calibration and if there is no stable measurement within 30 s, the calibration is not accepted. Should one of the tolerance ranges be exceeded during the calibration, there is a respective error message and the calibration has to be started again with a new buffer solution or with a new electrode.

Ideal temperature for buffer solution is 20 °C!

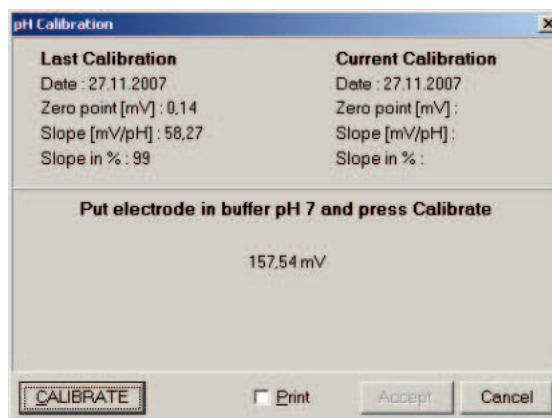
Reference note:

Usually the life span of the electrode is 1 year. If there is a slow measuring, make sure to exchange the electrode. In order to guarantee a perfect function of the instrument make sure to use original electrodes from C.Gerhardt only.

Make sure to read the reference notes in the instruction manual enclosed which informs you about handling and care of the electrode.

7.6.2. Run pH-calibration

From the menu bar select **System > Titrator > pH-calibration** or press the push button "**pH-Calibration**", to open the following window.



Electrode
in Buffer pH7

Follow the instructions on the screen and immerse the electrode, which has been rinsed with water and dried carefully (don't rub it dry!!) in buffer solution pH7. Start procedure by pressing key "**Calibration**" or by pressing of the Vapodest-function button. The tension measured and the 30s stabilisation time counted backwards are displayed. As soon as a measurement is stable for 2s, there is an automatic switch to the second buffer solution.



7 System settings

Electrode in Buffer pH4

Clean the electrode again with water and dry carefully, then immerse it into the buffer solution pH 4. Press key **"Continue"** or the Vapodest-Function button, to continue measuring. The tension measured and the 30s stabilisation time counted backwards are displayed. As soon as a measurement is stable for 2s, the calibration is terminated.

The data obtained after the calibration can be accepted by pressing key **"Accept"** and if wanted also printed out. There is the possibility to repeat the calibration by pressing key **"Calibration"** without storing the values.

7.7. Rinse micro dosing pump

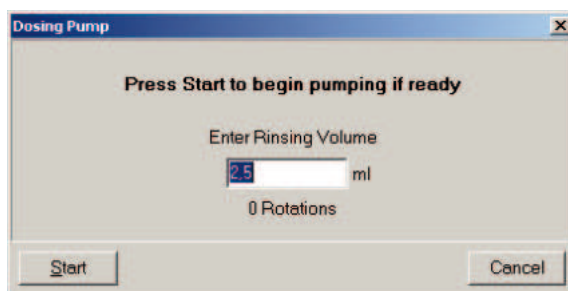
The rinsing program cleans the micro dosing pump and the inlet tubings. Please note, that if air is in the inlet tubing, the results can be falsified. Thus, check the inlet tubings regularly to see whether air bulbs are present.

The following situations make a rinsing necessary:

- When the titration liquid is exchanged
- There is air in the inlet tubing
- When the instrument is taken into operation for the first time
- The instrument has not been used for a while

7.7.1. Run rinsing process

From the menu bar select **System > Titrator > Rinsing micro dosing pump** to open the following window.



In such a case, enter the rinsing volume. In most cases a rinsing volume of about 10 – 15 ml is sufficient. The dosing pump delivers theoretically 0.05 ml per turn. Minor deviations are possible. These are balanced off by a calibration done by the manufacturer (also see chap. 7.9.3. Titration calibrate dosing pump).

The rinsing process is started by pressing key **"Start"**.

Below the insert window the number of the turns of the pump head is displayed. Should there still be air bulbs in the insert tubing after the rinsing, check whether the tubing connections are sitting tightly on the micro dosing pump and the titration tank. Then, repeat rinsing process.

Press key **"Cancel"**, to finish rinsing process.

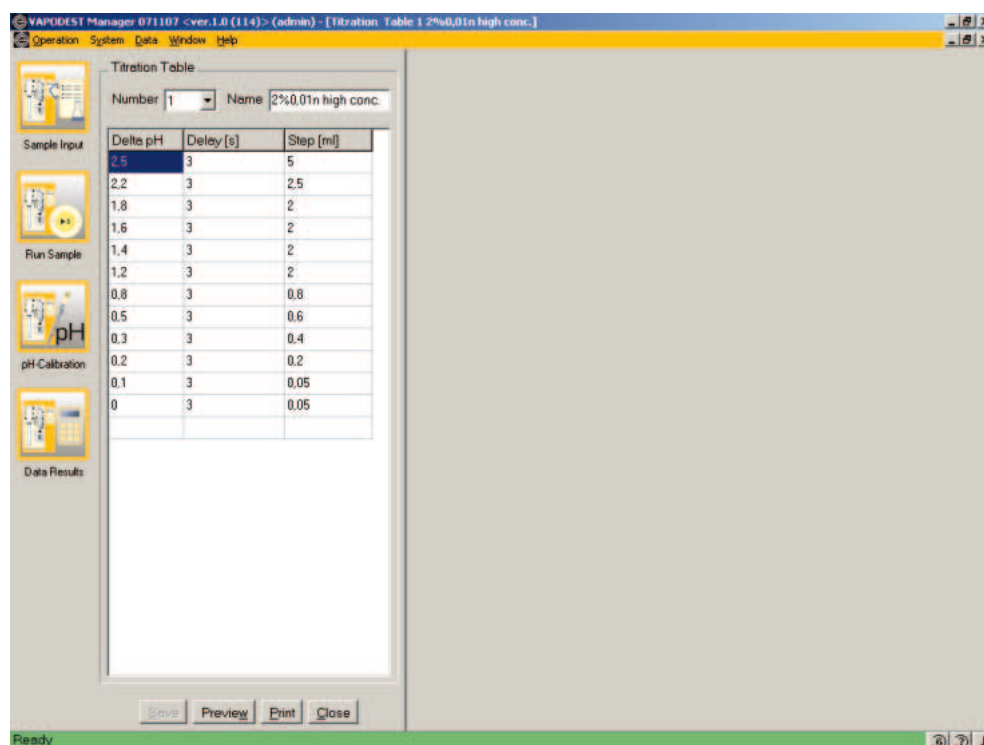
7 System settings

7.8. Titration table

In the Vapodest system a pH-end point determination for the detection of the measuring value. The functions in the titration table have an influence on the titration speed. The lower the N-content of the sample the more careful the additional amount of titration solution has to be set. The concentration of the boric acid used is an additional influence factor. Depending on the method, you have to test several functions, to obtain optimal results. Please check the three sample tables for various boric acid concentrations which are located at the end of this chapter.

7.8.1. Set titration table

From the menu bar select **System > Titrator > Titration table**, to open the following window- Titration speed.



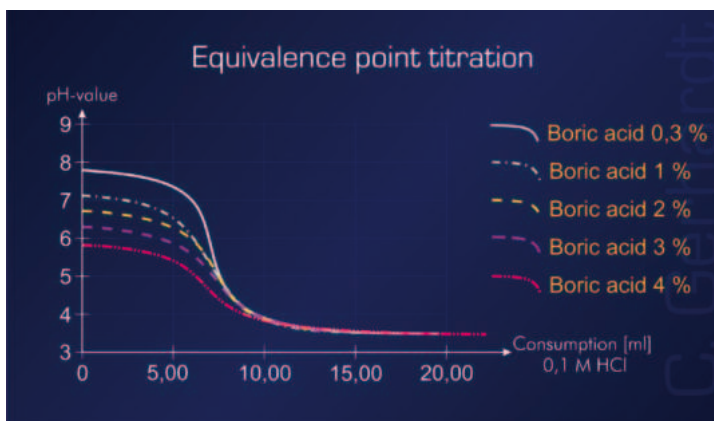
- **Number**
You can define table 1 - 3
- **Name**
Enter a name for the table
- **Delta pH**
Indication of the pH-difference - given
- **Time for blending (Delay)**
During this time a stable pH-measurement has to be done till the next dosing step (choose between 1 - 99 s).
- **Step**
Additional amount of titration solution can be chosen between 0,05 - 9,95 ml

Press key "**Store**", to accept functions. In chapter "**8. Create a method**" you are informed how the titration table can be allocated to the various methods.



7 System settings

7.8.2. Titration curve when using different boric acid concentrations



7.8.3. Sample titration tables

We recommend the following tables for different boric acid concentrations:

Delta pH	Delay [s]	Step [ml]
2.5	3	5
2.2	3	2.5
1.8	3	2
1.6	3	2
1.4	3	2
1.2	3	2
0.8	3	0.8
0.5	3	0.6
0.3	3	0.4
0.2	3	0.2
0.1	3	0.05
0	3	0.05

Concentration
2 % 0,01 N

Delta pH	Delay [s]	Step [ml]
2.5	3	1.2
2.2	3	0.5
1.8	3	0.4
1.6	3	0.2
1.4	3	0.1
1.2	3	0.1
0.8	3	0.1
0.5	3	0.05
0.3	3	0.05
0.2	3	0.05
0.1	3	0.05
0	3	0.05

Concentration
2 % 0,1 N

Delta pH	Delay [s]	Step [ml]
2.5	3	0.5
2.2	3	0.3
1.8	3	0.2
1.6	3	0.1
1.4	3	0.1
1.2	3	0.05
0.8	3	0.05
0.5	3	0.05
0.3	3	0.05
0.2	3	0.05
0.1	3	0.05
0	3	0.05

Concentration
4 % 1 N

Please find more informations about titration settings in chapter "7.3. Titration".

7 System settings

7.9. Calibrate dosing pumps H₂O and NaOH

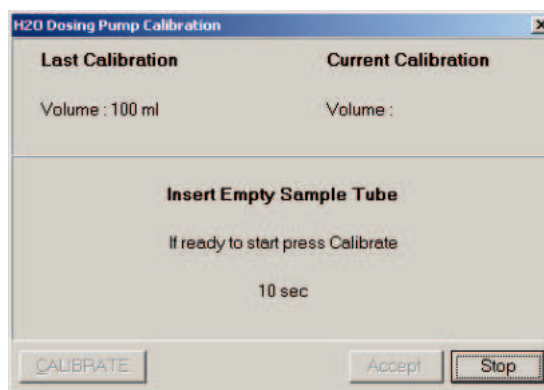
The delivery rate of the pump depends on the viscosity of the chemicals used, the delivery height and further other conditions. Thus, you must calibrate the dosing pumps prior to the first operation or whenever the chemicals used are changed.

Reference note

In order to run the following functions, you have to register as an "Administrator".

7.9.1. Calibrate dosing pump NaOH and H₂O

In the menu bar select **System > Service > Adjustment > NaOH dosing pump** or **"H₂O Dosing pump"** to open the following window.



Comment:

The calibration of the pump can only be done if you put in an empty digestion tube. In order to do so you have to take out the glass guiding cage and to put in the clamping lever for the manual glass addition.

Also read chap. "10.6. Manual change of sample". Make sure that the tanks are filled with the respective chemicals.

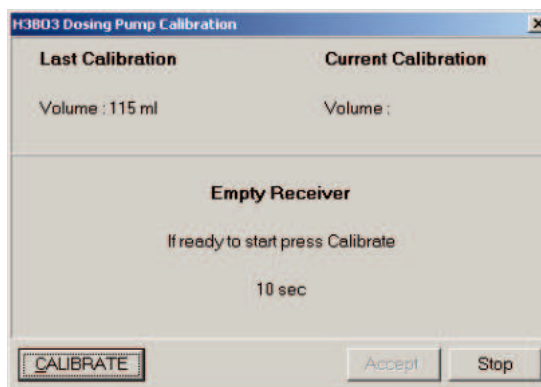
1. Push quick clamping device down and put in empty digestion tube.
2. Aerate tubing. Press Vapodest-key "H₂O" or "NaOH" till liquid accumulates in the digestion tube.
3. Empty digestion tube and then put it in again.
4. Press key "CALIBRATE", to start process. The pump runs for about 10 s and fills the digestion tube.
5. Then take out digestion tube and fill the content in a measuring cylinder to determine the delivery volume.
6. Enter the volume in the dialogue field and press key "Continue".
7. Press "Accept", to finish process or run another calibration.
8. After the calibration of the pump you have to take out the quick clamping device and insert the test tube guide cage.



7 System settings

7.9.2. Calibrate dosing pump H_3BO_3

In the menu bar select **System > Service > Adjustment > H3BO3 Dosing pump** to open the following window.



Comment:

Make sure that the tank is filled with the respective boric acid.

1. Aerate tubing. Press Vapodest-key "**H3BO3**" till liquid accumulates in the receiving tube.
2. Empty receiving tube – in order to do so, turn glass and pull under towards you. Then insert receiving tube again.
3. Press key "**CALIBRATE**" to start process. The pump runs for about 10 s and fills the digestion tube.
4. Then take out the receiving glass and put the content into the measuring cylinder, to determine the delivery volume.
5. Enter the volume in the dialogue field and press key "**Continue**".
6. Press "**Accept**", to finish process or run another calibration.
7. After the calibration of the pump you have to take out the quick clamping device and insert the test tube guide cage.

7 System settings

7.10. Service-System functions

The following system functions are only used for service by an authorized customer service or the administrator. Usually, no changes of these functions are necessary.

7.10.1. Calibrate titration dosing pump

From the menu bar select **System > Service > Adjustment > Calibrate titration dosing pump**

The delivery volume of the micro dosing pump is set here. The calibration is done by C.Gerhardt so that a calibration is not necessary.

In the following cases, we recommend a calibration:

- The results are always too high
- The results are always too low
- the volume to the pump has been altered

You can check the pump capacity with the help of the rinsing program as described in chapter "7.7. Rinse micro dosing pump".

7.10.2. Pressure sensor

From the menu bar select **System > Service > Adjustment > Pressure sensor**
- Measurement amplifiers for steam generator is calibrated

7.10.3. pH-Measurement amplifiers

From the menu bar select **System > Service > Adjustment > pH-Measurement amplifiers**
- The pH-measurement amplifiers are calibrated

7.10.4. Firmware Update

From the menu bar select **System > Service > Firmware Update**
- Vapodest Firmware is updated

7.10.5. Check the in- and outlets

From the menu bar select **System > Service > Adjustment > Check in-& outlets**
- The inlets and outlets are checked.

7.10.6 Statistics data

From the menu bar select **System > Service > Statistics**

Here you can call up details of the statistics of the instrument:

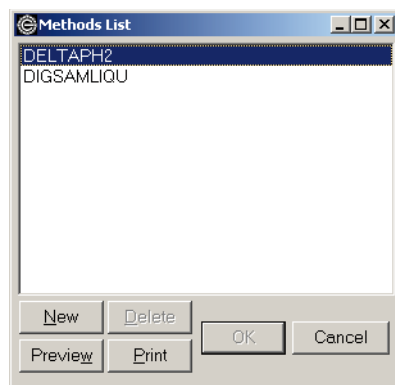
- Entire running time of instrument
- Total of turning on and off
- Last service
- Next service due on

8 Create a method (Programming)

In this chapter all parameter necessary for the control of the analysis are explained.

8.1. Open list of methods

From the menu bar select **Operation > Method**, to open the following window.

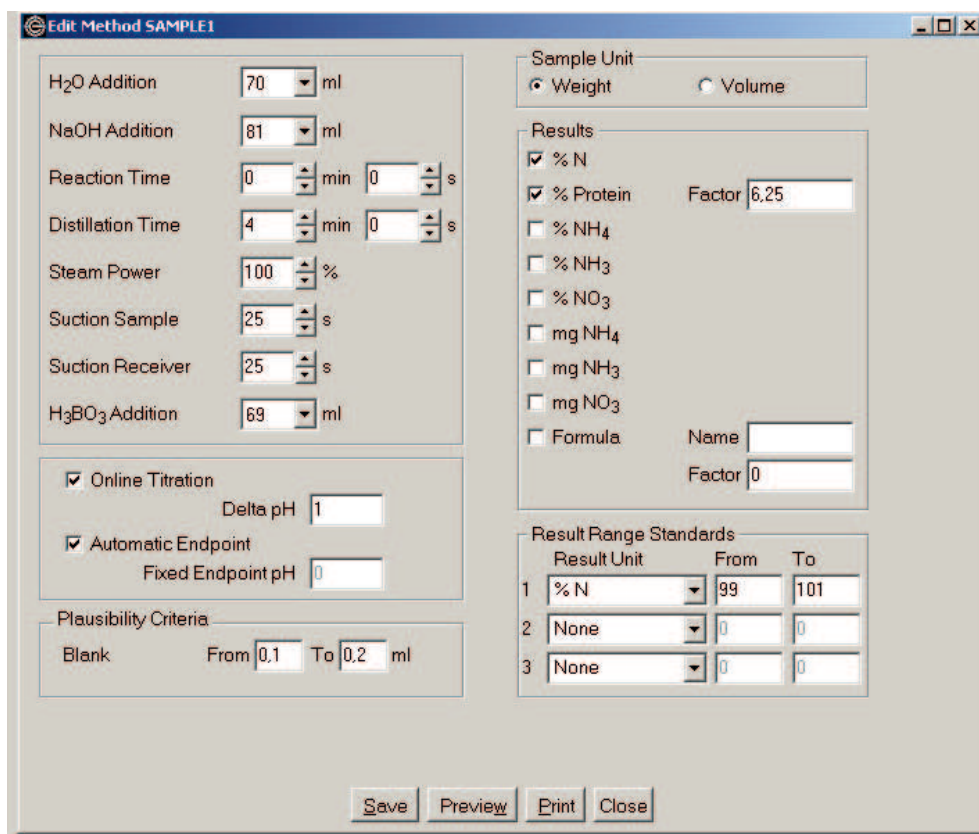


A list with the methods already defined is shown. Now, you can create a new method or delete, edit, view and print out an existing method.

8.1.2. Create a new method

Press key "New", and enter a method name in the dialogue window. Then press key "OK", to open the method window. The programming example refers to a sulphuric acid digestion acc. to Kjeldahl, during which 20 ml H₂SO₄ were used.

The remaining amount of acid after the digestion is important for the programming. It depends on the type of sample, the digestion time as well as the digestion temperature.



8 Create a method (Programming)

- Addition H₂O

A sufficient dilution of the concentrated sulphuric acid reduces the energy which is set free during the alkalisation process. The addition of H₂O depends on the amount of the free (unbound) sulphuric acid at the end of the digestion. In general the 5fold amount of water is sufficient.

Setting range 0 - 190 ml - the specification of the dosing steps depends on the calibration of the pumps.

- Addition NaOH

In order to be able to distil ammonia over it is important to create a strong alkaline environment. When catalysts containing copper sulphate are used, a colour change of the sample takes place if it is alkaline. When Selen is used as a catalyst, an indicator can be added to the sample to have a visual check. The addition of NaOH depends on the amount of free (unbound) sulphuric acid at the end of the digestion. In general, the quadruple amount is needed for a 32% NaOH.

Setting range 0 - 190 ml - the specification of the dosing steps depends on the calibration of the pumps.

- Reaction time

The reaction time is especially for the nitrate nitrogen determination when Devarda's alloy is used (e.g. for the analysis of fertilizers). For Kjeldahl analysis this amount of time is not necessary.

Setting range 0 - 99 min and 0 - 59 s

- Distillation time

The distillation has to be set so that about 100ml distillate is produced. If the steam output is set to maximum this corresponds to 3 min 30 s to 4 min. In the water analysis, a phosphate buffer is used instead of NaOH for the determination of the ammonium-nitrogen. In such a case, 200 ml have to be distilled over.

Setting range 99 min und 59 s

- Steam power

Usually, maximum power is used for the Kjeldahl analysis. If the power is reduced, the distillation time is increased at the same time to obtain the same amount of distillate. For some determinations, e.g. for the determination of ammonium, a certain amount of distillate is required for a given amount of time. 100 % correspond to 1500 Watt heating power, 50 % 750 Watt etc.

Attention, when micro glasses with 100 ml are used, the max. heating power should be 80 %.

Setting range 40 % - 100 %

- Suction sample

If the distilled sample is not longer needed for further analysis, it can be sucked off using the built-in tubing pump.

Setting range 0 - 99 s

- Suction receiver

The duration of the suction is calculated so that the receiving vessel is completely empty. Should there be any remaining single drops, they won't have any influence on the following analyses.

Setting range 0 - 99 s

8 Create a method (Programming)

- **Addition H₃BO₃**

The diaphragm of the electrode must be completely immersed in the receiving solution, so that a stable pH- measurement is ensured.

Setting range 0 - 999 ml

- **Online-Titration**

When an online-titration is done, then the titration is already done during the distillation. Thus the entire time needed for the analysis is reduced. In the on-line-titration mode, you have to enter the pH – difference to the end point, which you are titrating to during the distillation. The difference is titrated after the distillation is finished.

Delta pH = pH-Difference

- **Plausibility criteria (for the blank value)**

If a sample is measured as a blind value, the result is valued according to these criteria and the series of analysis is interrupted with an error message if necessary.

Setting range 0 to max. 1 ml (Additional amount Triersäur

- **Measuring unit sample**

In the program a selection is done, whether the calculation is done in weight or volume.

- **Calculation of results**

Select the units desired, which should be calculated later on. If you select protein, then you also have to enter the protein factor. All units shown can be combined in any way. For the calculation of the units also see formula sheet in the attachment.

"Formula"

A free definition of the unit (name) incl. factor is possible in the field 'Formula'.

This formula is calculated as follows:

$$\text{Result weight} = \frac{\text{Factor} \times \text{Acid concentration} \times (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Weight [g]}}$$

$$\text{Result volume} = \frac{\text{Factor} \times \text{Acid concentration} \times (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volume [ml]}}$$

- **Standard – Tolerance range**

If you work with one standard during the series, then you can set a tolerance range for the result. If the result goes below this range or if it is exceeded there will be an error message during the analysis and in the print out of the result.

There is the option of selecting 3 different standards with various contents.

Press key "**Store**", to store the program created under the program name selected.

You can print out the program parameter for checking and documentation purposes. In order to do so, press key "**Print preview**", or, if you want to print without preview, key "**Print**".

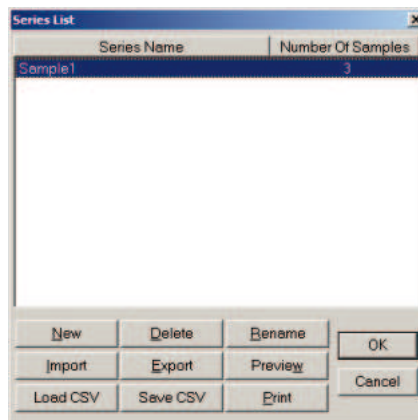
Press key "**Close**", to close the window create a method

9 Sample input

In this chapter entering the sample data in a series is described. A series contains a maximum of 20 samples and can include samples, blank samples and standards. Each type of sample can be assigned an individual program. Make sure that the carousel has to be loaded with sample glasses in the correct order to obtain correct results (also see chap. "12.2.2. Loading of carousel").

9.1. Open sample series list

From the menu bar select **Operation > Sample input**, or press push button „Sample input" to open the following window.

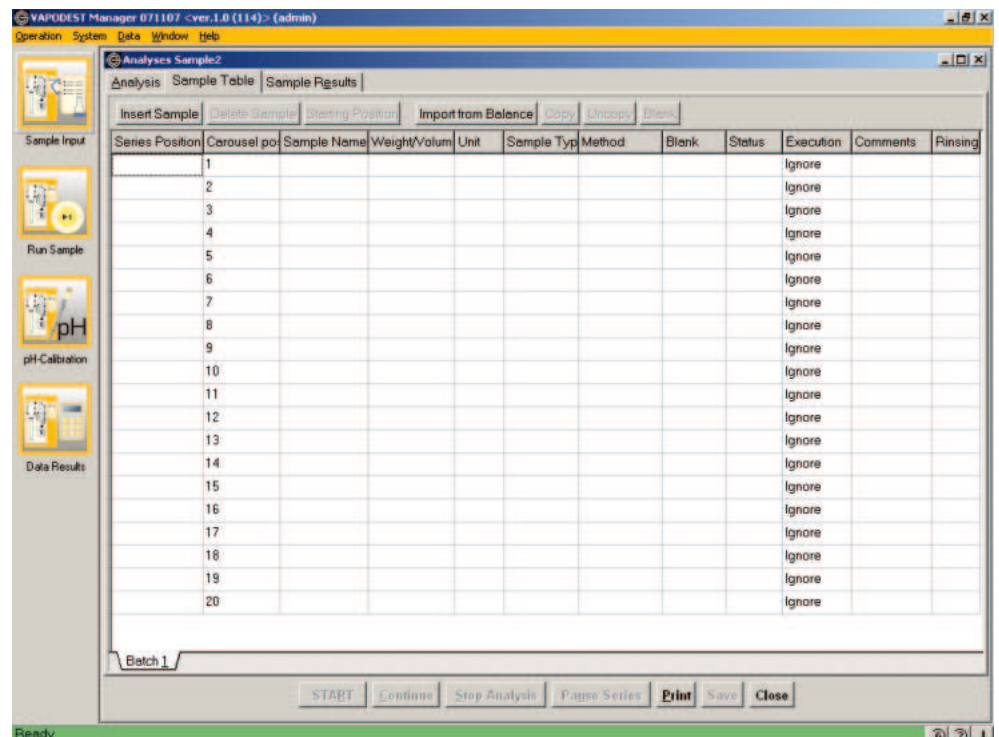


A list with pre-defined series is shown. You can create either a new series or edit, delete or print existing series. Using keys "Import" and "Export" you can import or export data in CSV-format. (Also see chap. 7.1.4. Where to store in- and export data).

Red Text = Series has been processed
Black Text = Series has not been proceeded or only partially proceeded

9.1.1. Set up new sample series

Press key "New", and enter a name for the series in the dialogue window. Then press key "OK" to open a sample series table.

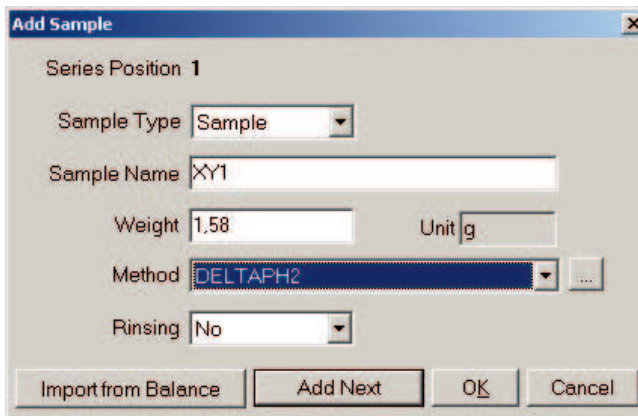


The table consists of lines and columns and each line is assigned a sample type. A series can have a maximum of 20 lines (Samples).

9 Sample input

9.1.1.1 Enter sample

Press key "Enter sample", to open the window for the sample input.



Sample type

Open the selection list to determine the type of sample:

Sample

A sample with defined weight, volume is analysed.

Standard 1, 2 and 3

In this position a standard is used.

Blank

In this position a blank value should be determined

No Titration

A distillation without further titration is done. If needed, the titration can be done manually at a later point.

Cleaning

This position is used to clean the Vapodest, e.g. after working with an excessively foaming sample. Put an empty, clean digestion tube in this position.

Sample name

Enter a sample name here.

Weight/Volume

Enter the weight/volume of the sample here or press key „Import Balance” to transfer the weight directly from the balance.

Method

Select a method from the method list, with which this type of sample should be analyzed. (See chap. 8 Create a method).

Rinsing

Contrary to the procedure explained above, "Sample type - Cleaning" the digestion tube is not exchanged after the analysis. After finished rinsing the next sample is processed.

If additional sample types are entered, press key "Additional Sample". Press key "OK", to accept task and to close the window.

9.1.1.2. Print sample table

Press key "Print" to open the print window. Here, you can execute further functions. Select "Sample Input" and press key "Print" to start the printing job.

9 Sample input

9.1.1.3. Delete sample

Highlight a field in the line, which you would like to delete and press key "Delete Sample".

9.1.1.4. Define position for the start of a series

Usually, the first sample is defined as the start of a series. This position is marked red in the column "Carousel position". If you want another sample as the start position then you have to high light the respective line and print the key "Start Position".

9.1.1.5. Import balance

Press this key if you want to enter the weight of the sample directly into the Vapodest.

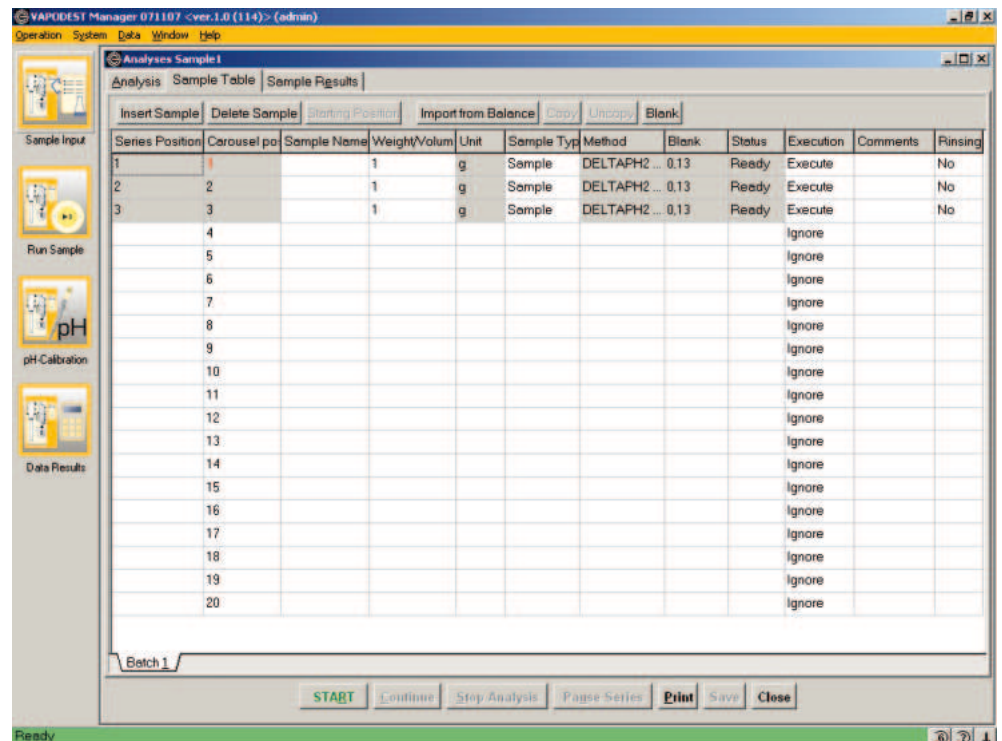
9.1.1.6. Print sample table

Press key "Print" to open the print window. Here, you can execute further functions. Select "Sample Input" and press key "Print" to start the printing job (also see chapter "11.3. Print Data" to get more informations about this menu)

9.1.1.7. Store table

Press key "Store" to store data.

9.1.2. Description of the columns and rows



Series Position	Carousel po	Sample Name	Weight/Volum	Unit	Sample Typ	Method	Blank	Status	Execution	Comments	Rinsing
1	1		1	g	Sample	DELTAPH2 ...	0.13	Ready	Execute		No
2	2		1	g	Sample	DELTAPH2 ...	0.13	Ready	Execute		No
3	3		1	g	Sample	DELTAPH2 ...	0.13	Ready	Execute		No
4									Ignore		
5									Ignore		
6									Ignore		
7									Ignore		
8									Ignore		
9									Ignore		
10									Ignore		
11									Ignore		
12									Ignore		
13									Ignore		
14									Ignore		
15									Ignore		
16									Ignore		
17									Ignore		
18									Ignore		
19									Ignore		
20									Ignore		

- Rows in general

A double click on the white fields of a sample row corrects the values. Grey fields cannot be corrected.

- Column "Serial position"

Samples are numbered from 1 to max. 20

9 Sample input

- **Column 'Auto sampler'**
Only in use when the automatic auto sampler 'Carousel' is in use. The position of the sample tubes in the sample carousel are defined.
Red number = this sample is processed next.
- **Column 'Method'**
Anzeige des Methodenprogrammes, mit dem die Probe analysiert werden soll. Durch Anklicken der 3 Punkte "..." in diesem Feld können Sie die Methodeparameter einsehen.
- **Column 'Blank value'** – gives you the blank value obtained.
- **Column 'Rinsing'**
You can determine whether the sample tube should be rinsed after the analysis another time.
- **Column 'Sample name'** - You can define a sample name
- **Column 'Sample type'**
You can enter the type of sample, e.g. blank value, sample, standard, etc
- **Column 'Weight/volume'**
Enter weight/volume of sample.
- **Column 'Unit'** - weight is entered in g, volume in ml
- **Column "Status"**

Ready -	The sample can be analysed, all data necessary for the analysis are present
Finished -	Sample has been analysed
Aborted -	Analysis has been aborted without result
- **Column "Execution"**
Highlight a field in this column, then you can open a list with the following options:

Execution:	Sample is analyzed within the series.
Later:	Sample table is not deleted at the end of the analysis so that these sample can be analyzed at a later date as well (e.g. if not all the data was present at the start of the series).
Ignore:	Sample cannot be analyzed and will be skipped in the next series.

As soon as the sample table is stored, you can start the analysis. In order to do so, press key **"Start"**. Further information for running an analysis is located in chapter **"10 Execution of methods"**.
Press key **"Close"** to leave window sample table.

10 Execution of method

This chapter describes the execution of an entire series determination. However, we can only give you a general overview how to work with Vapodest in this manual. Should you need any specific methods we will send you the respective application on request. These data sheets contain detailed info about the sample preparation and programming.



Make sure to always wear protective clothes (gloves, protective glasses, lab coat) when working with Vapodest.



When you take out the digestion tube, make sure to wear protective glove or have glass tongs at hand and wear protective glasses. The glass container is very hot and there is the danger of burns!



Be careful when handling acids and alkalis! Please observe the safety regulations according to the current laws regarding handling of dangerous goods!



We can only guarantee a trouble free workflow of the auto sampler if original Gerhardt-glass parts are used!

10.1. Prior to the analysis

Test run

In order to make sure to obtain reproducible results, we kindly ask you to run a distillation without sample every day prior to the first analysis. In order to do so, enter the sample type as 'Cleaning' (see chapter 9.1.1.1. Enter sample)

Check set of tanks

Check the level of reservoirs and the connections at regular intervals.

Turn on faucet

The system is only working when the faucet is turned on.

Run pH-calibration

After the initial installation of the Vapodest Manager, a pH-calibration is necessary. Otherwise, the analysis cannot be started. We recommend that you calibrate the pH-electrode regularly as it is subject to normal wear and tear.

Enter sample

You have to define a series of samples with at least one sample prior to the start of the analysis. (see chap. 9 Sample Input)

10 Execution of method

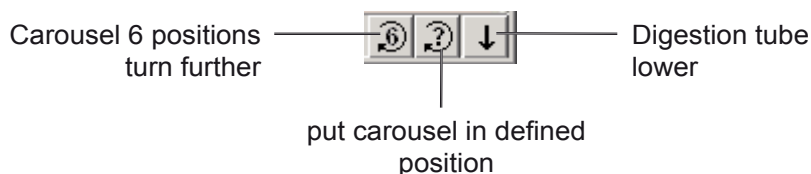
10.2. Load sample into carousel

Make sure that the carousel is loaded in the order which is defined in the sample table. Otherwise the results of the analysis are not correct.

Put sample carousel in the loading position

You can run the carousel either use the key carousel of the Vapodest-Manager or by tapping on the sensor switch directly on the Vapodest (see fig. left).

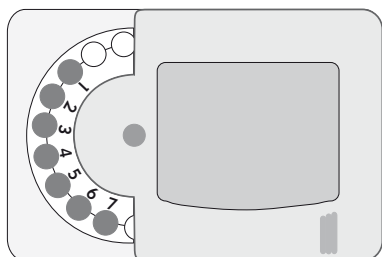
Tap on sensor switch



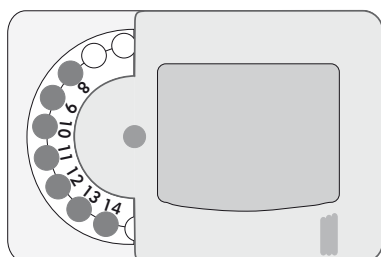
Put carousel in loading position A (see ill. below), open loading hatch and then put in the first glasses. Close loading hatch again and go the next loading position B and then to C. During the analysis, a built in sensor automatically checks whether a position of the carousel has not been loaded. There is also a message on the display.

Loading position A, B, C, example with carousel 20-fold

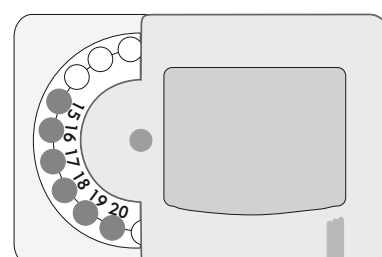
Loading position A



Loading position B



Loading position C



10 Execution of method

10.3. Working with various sizes of digestion tubes

If you want to work with different sizes of glass, you have to change the carousel and the guidance cage.

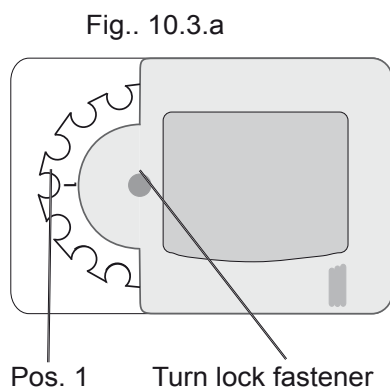


During a series it is not possible to work with different sizes of glasses.

10.3.1. Change carousel

There are three types of carousels available:

- 20-fold for 20 x 250 ml tubes
- 16-fold for 16 x 400 ml tubes (KDD und BS 400)
- 12-fold for 12 x 800 ml tubes



1. Empty the carousel completely.
2. Take off protection cap: in order to do so, pull handle upwards and then towards the side.
3. Turn carousel manually so that position 1 is on the very left (also see ill. 10.2.a). that way, the carousel can be easily inserted again at a later time.
4. Open the turn-lock fastener for the carousel lösen and then pull out completely..
5. Take out carousel.
6. Insert the carousel again and turn till it is fixed in the groove of the receiver for the carousel. Make sure that, the carousel sits straight.
7. Insert turn lock fastener and pull till the stop.
8. Put on protective cap again.
9. In the menu "System functions > Carousel", the software has then to be set to the correct type of carousel. This is important as the software needs the info that a new type of carousel is used (also see chap. 7.4. Selection of carousel)

10.3.2. Change guidance cage

There are three different types of test tube guide cages available:

- Guidance cage for glasses 250 ml – Standard ausführung
- Guidance cage for glasses 400 ml (KDD and BS 400)
- Guidance cage for glasses 800 ml

How to take out the guidance cage:

Press clamping device down (chapter 4.3, pos. 2) and pull guidance cage together with the drip tray forward (chapter 4.3, pos. 23).

How to insert the guidance cage:

Put PTFE-insert tubing (chapter 4.3, pos. 3) in the guidance cage and pull drip tray slightly forward. Press clamping lever downwards and insert the guidance cage slightly diagonally until you hit the upper stop. Then, make sure that the fixing pins of the guide cage are locked into place in the quick clamping device. Should the guide cage not be inserted correctly there will be an error message in the display.

10 Execution of method

10.4. Start determination of series

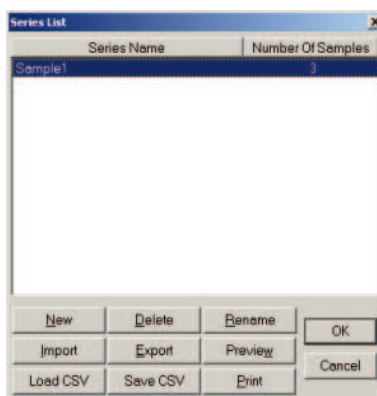
Serial determination means that a series of samples has already been stored in the main menu "Sample input" and that they can be analyzed one after the other.

Load carousel as described in chapter "10.1. How to load samples in carousel" and close the door and all drawers.

In the menu bar select **operation > Run sample**, or press the push button "Run sample" to open the list of series.



Fig. 10.4.a
Series list



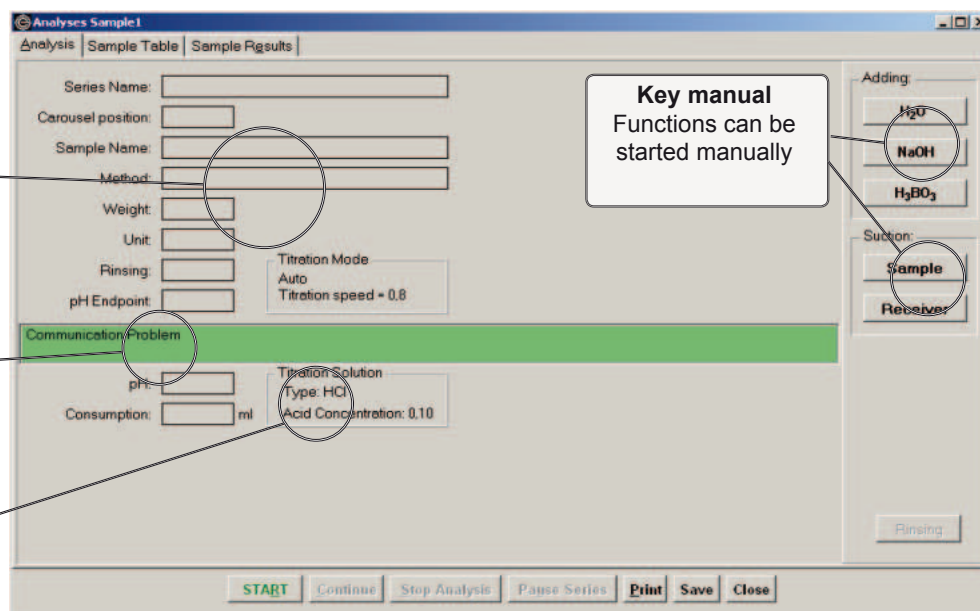
Select a series from the list and press the key "OK". Then the table of the sample series opens.

Fig. 10.4.b
Analysis window

Information fields
Facts about the type of sample and method

Status display
Facts about the status of the instrument + error messages

Status Titration
Facts about the pH-value and consumption titration acid



In this window you have the best possible control of the current analysis.

Press key "Start" to switch into the standby mode.

As long as the analysis is not running, you can start the following functions using the "Key manual":

- Suction receiver, Addition H₂O. Addition H₃BO₃

10 Execution of method

As soon as the message **"System is ready"** is shown in the status display, you can start the determination of the series by pushing key **"Continue"**.

The various program steps are shown in the display. Each automatic change of sample is displayed by "Change of position".

If the analysis is interrupted due to an error, the program is automatically continued after the correction of the error.

During an analysis in process, the analysis window cannot be closed. Vapodest Manager can only be closed after the program is terminated.

After the last sample is terminated, the message **"Analysis terminated"** is shown in the status display. By pressing key **"Enter"** you get back to the start picture of the series determination. Then, you have the option to enter a new series or leave the analysis with the key **"Esc"**.

10.5. Functions during an analysis in process

During the analysis in process, you have the option to influence the procedure manually, to enter new sample series and to read the results of the current analysis.

10.5.1. Key 'Pause'

The analysis of the current sample is aborted and can be continued at a later time.

10.5.2. Key "Abort Analysis"

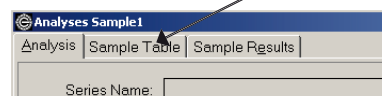
The analysis of the current sample is aborted, but the remaining samples of this series can be analyzed at a later point. After pressing this button you have the choice between the following options:

- Execute Suction+Rinsing On Broken Sample
Abort with suction of receiver and rinsing of the system
- Suction
Abort with suction of receiver but without cleaning of the system
- Cancel
Abort without suction and rinsing

10.5.3. Key "Pause Series"

If necessary, you can abort the series in process and continue at a later date..

In the sample series table you can view info about the status of the individual samples. In order to do so, activate the window **"Sample table"**.



10.5.4. Key "Stop"

The analysis of the current sample is aborted, the remaining samples of this series can be analyzed at a later point

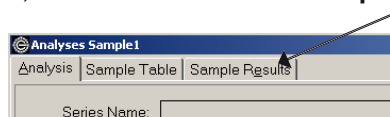
10 Execution of method

10.5.5. Key "NaOH"

For the manual addition of NaOH keep this key pushed down.

10.5.6. View results of the analysis

You can view the results of the current sample series in the window "**Sample results**". In order to do so, activate the window "**Sample results**".



10.5.7. Input samples series during an analysis in process

During an analysis in process you can enter additional sample series, which can be started after terminating the current series (see chapter "9 Sample input").

10 Execution of method

10.6. Manual change of sample - Single determination

The sample glasses of a series can also be fed manually. This option is useful when glasses are used whose size are not accepted by the carousel.

10.6.1. Modifications for single determination

Prior to starting with the single determination you have to modify the instrument in the following way:

1. Take out the guidance cage (see chapter 10.2.2 Change guidance cage).
2. The manual clamping lever is enclosed in the delivery of the Vapodest. It is located in the tank drawer and is attached to the protection hood for the Z-axis. To exchange it, pull the quick clamping device towards you (chapter 4.3, pos. 2) and then, insert the manual clamping lever to the stop in the opening. The digestion tube has to sit tightly after inserting it. Should this not be the case, then you must push manual tension device deeper .

10.6.2. Start single determination

1. **Turn instrument off and on!**
2. Press down quick clamping device and put in digestion tube. Make sure to that you insert the inlet tubing 'steam' into the digestion tube at the same time.
3. Check tight fitting of the glass and close protective door.
4. Set level sensor control of the tanks to "inactive" (see chapter 7.2.2. Using external set of tanks), otherwise an error message will be displayed.
5. Open sample series list and start analysis.
6. After terminating each individual analysis press tension lever downwards, take out sample glass and insert new sample glass.

After the last sample has been determined the message "Analysis over" is shown on the display. You can now either start a new series or terminate the analysis.

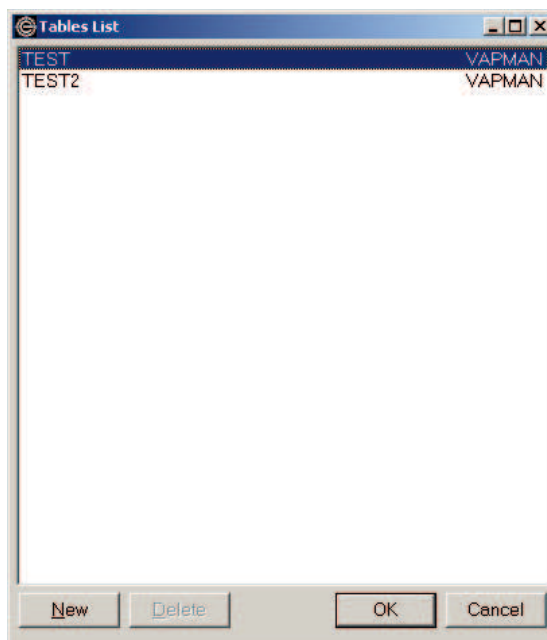
11 Table of results

In the sample table, all records about all analysis data, actions during the program, results and many more info are presented in table format. Thus Vapodest Manager offers vast logging- and documentation functions, thus functions to support quality control. The table format offers a well defined view of all analysis data e.g. all data of the month of May 2007 can be viewed in a table 'May 2007' sorted according to various criteria.



11.1. Open sample table

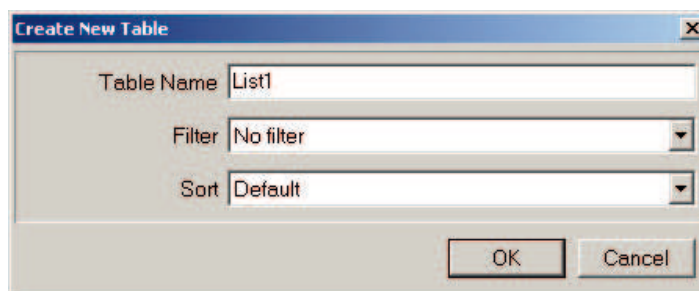
In the menu bar select **Data > Results** or press the push button "Result table", to open the following window.



A list with already defined table is shown. Now you can set up a new table or open or delete an existing table

11.1.1. Set up new table

- Press key „New“ or ALT+N. Now, the following window is opened:



- Enter a table name (max. 255 characters, no special characters or blanks)
- You can assign a filter from an existing table to this table. (see chapter „11.2.3. Filter data“).

11 Table of results

- Furthermore, you can assign the sorting function from an existing table. (see chapter 11.2.2. Sorting data). **"Pre-setting"** is an ascending sorting according to date and time.
- Press key **"OK"** or **"Enter"** to confirm new table with the options entered. Then, a new table window will open.

11.1.2. Delete table

In order to delete a table you have to proceed as following:

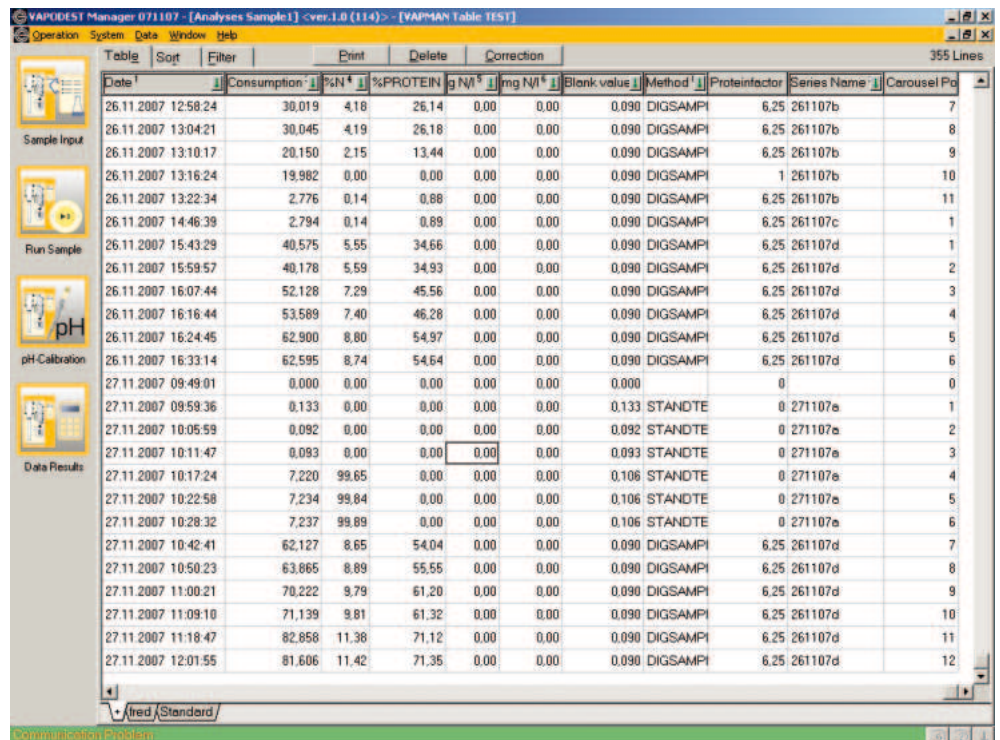
- From the menu bar select **"Table"**.
- With your mouse or the key ABOVE and DOWN select the table you want to delete and press the key **"Delete"**..
- Confirm procedure with key **"OK"**.

11.1.3. Open table

Select a table from the list of tables and confirm with key **"OK"**. If there is no table listed, you have to define a new table (see 11.1.1. Setting up new tables). Also you can open two or more tables simultaneously.

11.2. How to work with the table of results

In the table of results, the data of the analysis as such are shown. The tables offer the options sorting, filter and presenting the data in any way desired. One table may contain several work sheets. However, each of these sheets may contain a different configuration of rows and/or columns. Please note that the filter- and sorting functions are always valid for all work sheets.



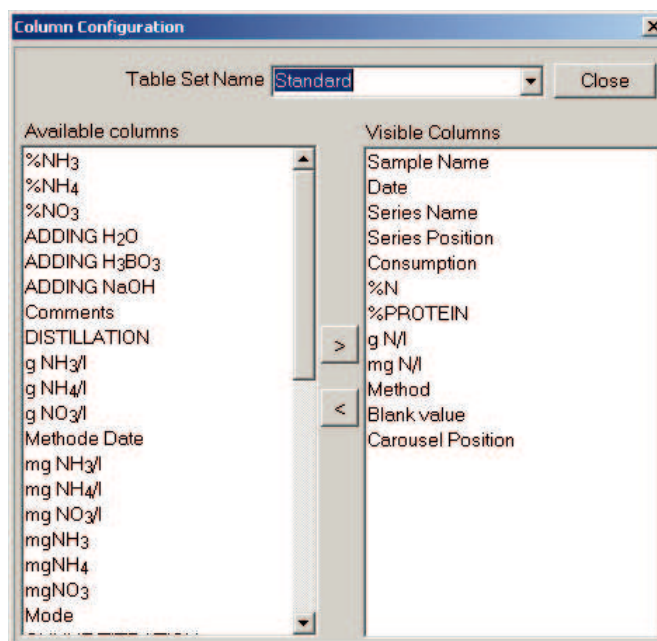
Date	Consumption	%N	%PROTEIN	g N/l	mg N/l	Blank value	Method	Proteinfactor	Series Name	Carousel Po
26.11.2007 12:58:24	30,019	4,18	26,14	0,00	0,00	0,090	DIGSAMPI	6,25	261107b	7
26.11.2007 13:04:21	30,045	4,19	26,18	0,00	0,00	0,090	DIGSAMPI	6,25	261107b	8
26.11.2007 13:10:17	20,150	2,15	13,44	0,00	0,00	0,090	DIGSAMPI	6,25	261107b	9
26.11.2007 13:16:24	19,982	0,00	0,00	0,00	0,00	0,090	DIGSAMPI	1	261107b	10
26.11.2007 13:22:34	2,776	0,14	0,88	0,00	0,00	0,090	DIGSAMPI	6,25	261107b	11
26.11.2007 14:46:39	2,794	0,14	0,89	0,00	0,00	0,090	DIGSAMPI	6,25	261107c	1
26.11.2007 15:43:29	40,575	5,55	34,66	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	1
26.11.2007 15:59:57	40,178	5,59	34,93	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	2
26.11.2007 16:07:44	52,128	7,29	45,56	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	3
26.11.2007 16:16:44	53,589	7,40	46,28	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	4
26.11.2007 16:24:45	62,900	8,80	54,97	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	5
26.11.2007 16:33:14	62,595	8,74	54,64	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	6
27.11.2007 09:49:01	0,000	0,00	0,00	0,00	0,00	0,000		0		0
27.11.2007 09:59:36	0,133	0,00	0,00	0,00	0,00	0,133	STANDTE	0	271107a	1
27.11.2007 10:05:59	0,092	0,00	0,00	0,00	0,00	0,092	STANDTE	0	271107a	2
27.11.2007 10:11:47	0,093	0,00	0,00	0,00	0,00	0,093	STANDTE	0	271107a	3
27.11.2007 10:17:24	7,220	99,65	0,00	0,00	0,00	0,106	STANDTE	0	271107a	4
27.11.2007 10:22:58	7,234	99,84	0,00	0,00	0,00	0,106	STANDTE	0	271107a	5
27.11.2007 10:28:32	7,237	99,89	0,00	0,00	0,00	0,106	STANDTE	0	271107a	6
27.11.2007 10:42:41	62,127	8,65	54,04	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	7
27.11.2007 10:50:23	63,865	8,89	55,55	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	8
27.11.2007 11:00:21	70,222	9,79	61,20	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	9
27.11.2007 11:09:10	71,139	9,81	61,32	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	10
27.11.2007 11:18:47	82,858	11,38	71,12	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	11
27.11.2007 12:01:55	81,606	11,42	71,35	0,00	0,00	0,090	DIGSAMPI	6,25	261107d	12

11 Table of results

11.2.1. How to configure columns

In this menu you can determine which columns should be shown in the table.

- With the right mouse key click on the table to open the context menu and select **"Configure columns"**.



- On the left side you see all available columns. These columns can be selected individually and by pressing the key ">" they can be shifted to the right side into "Visible columns" and vice versa.
- In the field "Table sheet denomination" you can assign a name to store the functions.
- Press key "Close", to store the functions.
- For each setting, an individual table sheet is set up. Several table sheets can be shown in one table. These table sheets can be selected by simply clicking on them.

11.2.2. Sorting of data

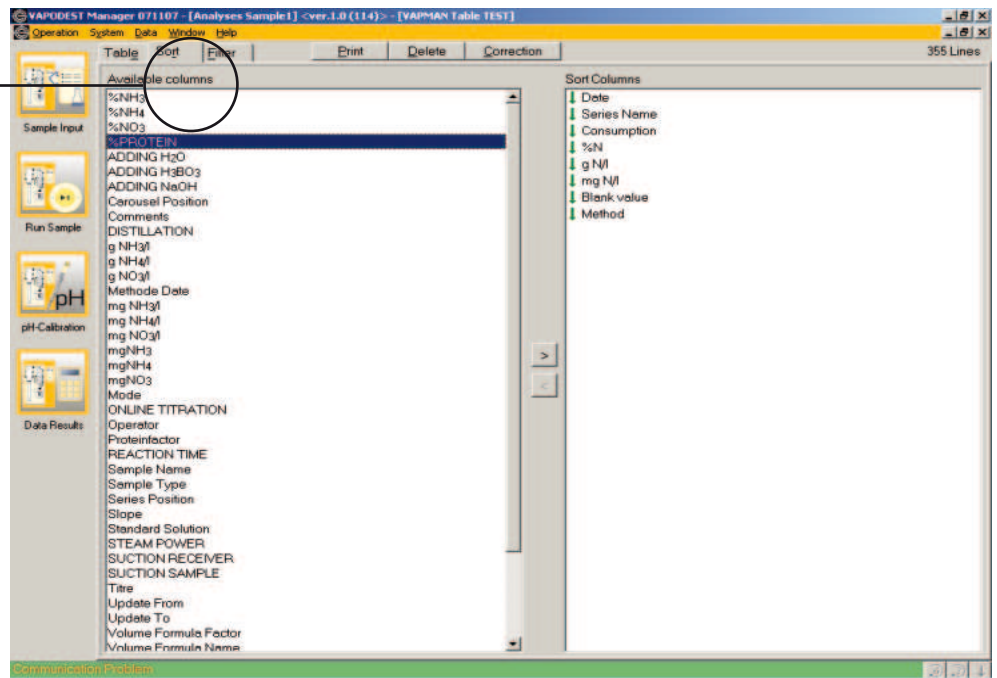
You can sort all data within the table window in descending or ascending order.

The sorting function is activated by clicking on the head line of the individual columns. There will be a green arrow, which shows whether the data are sorted in descending or ascending order. You have to press the green arrow to change the direction. Furthermore, digits are displayed which indicate in which priority (order) the columns are sorted.

The table window "Sorting" can also be used for sorting the data. Here, the selection of the columns to be sorted is done using the keys "<" and ">".

11 Table of results

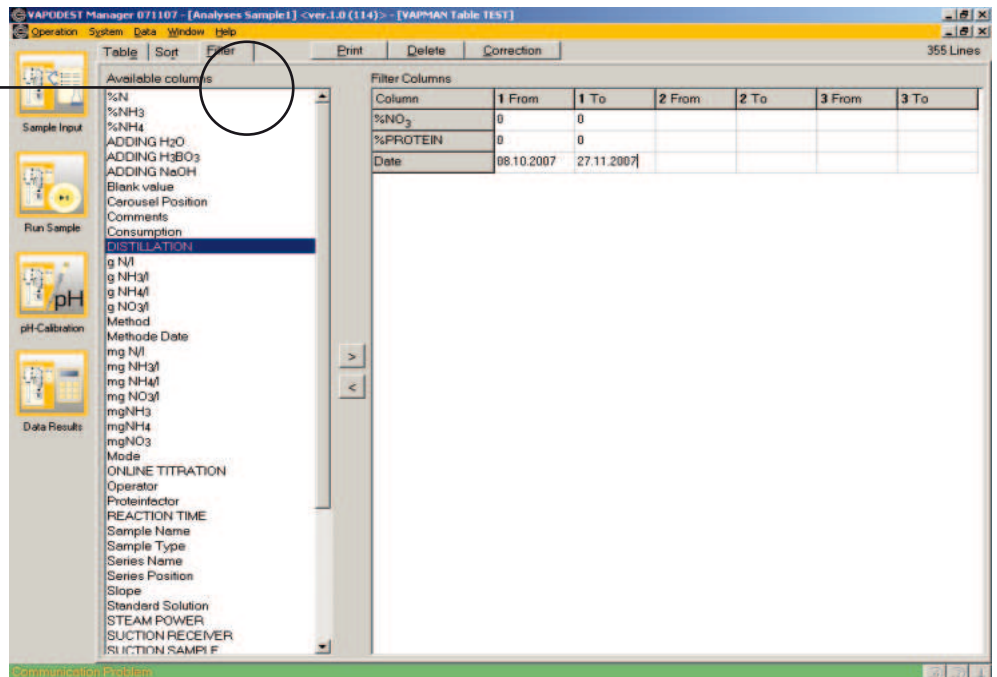
Fig. 11.2.2.a
Sort data



11.2.3. Filter data

In the table window "Filter" you determine the display of your data in the table. In order to define the filter criteria of the tables or to alter them, you use the keys "<" and ">".

Fig. 11.2.3.a
Define filter



Each table can have its own filter. A filter is assigned to each column, whereas up to 15 filters can be defined. You can assign up to 3 conditions to one filter. A condition is defined by the range of the value which is entered in the fields "from" and "to".

11.2.4. Search for data

11 Table of results

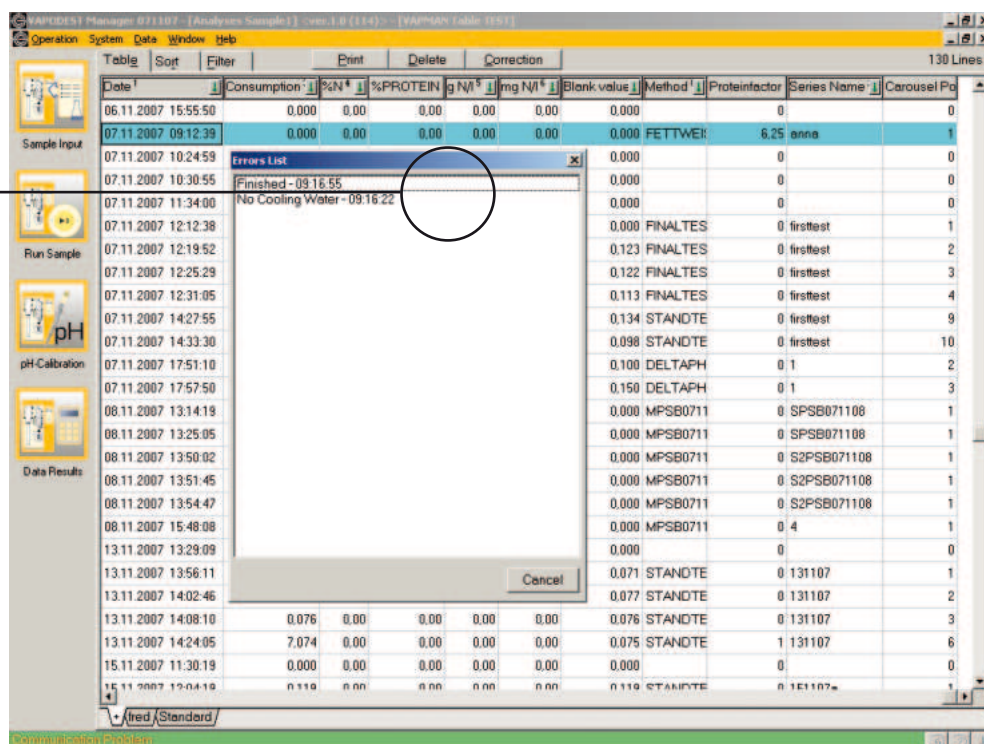
In order to find specific data in a column of the table, observe the following procedure:

- Activate one line in the table column, where the wanted data are located.
- With the right mouse key click on the table-card and select **"Find"** from the menu or you press STRG+F. The window with the search parameter opens up.
- Enter the search term and press key "Find" or ALT+F.

11.2.5. Display program records

All incidents which occur during the analysis are recorded in the result table together with date and time. A double click on a program line opens an "Error-List", which records all incidents.

Fig. 11.2.5.a
Errors list



Date	Consumption	%N*	%PROTEIN	g N/l*	mg N/l*	Blank value	Method	Proteinfactor	Series Name	Carousel Po
06.11.2007 15:55:50	0,000	0,00	0,00	0,00	0,00	0,000				0
07.11.2007 09:12:39	0,000	0,00	0,00	0,00	0,00	0,000	FETTWEI	6,25	anna	1
07.11.2007 10:24:59						0,000				0
07.11.2007 10:30:55						0,000				0
07.11.2007 11:34:00						0,000				0
07.11.2007 12:12:38						0,000	FINALTES		0 firsttest	1
07.11.2007 12:19:52						0,123	FINALTES		0 firsttest	2
07.11.2007 12:25:29						0,122	FINALTES		0 firsttest	3
07.11.2007 12:31:05						0,113	FINALTES		0 firsttest	4
07.11.2007 14:27:55						0,134	STANDTE		0 firsttest	9
07.11.2007 14:33:30						0,098	STANDTE		0 firsttest	10
07.11.2007 17:51:10						0,100	DELTAPH		0 1	2
07.11.2007 17:57:50						0,150	DELTAPH		0 1	3
08.11.2007 13:14:19						0,000	MPSB0711		0 SPSB071108	1
08.11.2007 13:25:05						0,000	MPSB0711		0 SPSB071108	1
08.11.2007 13:50:02						0,000	MPSB0711		0 S2PSB071108	1
08.11.2007 13:51:45						0,000	MPSB0711		0 S2PSB071108	1
08.11.2007 13:54:47						0,000	MPSB0711		0 S2PSB071108	1
08.11.2007 15:48:08						0,000	MPSB0711		0 4	1
13.11.2007 13:29:09						0,000				0
13.11.2007 13:56:11						0,071	STANDTE		0 131107	1
13.11.2007 14:02:46						0,077	STANDTE		0 131107	2
13.11.2007 14:08:10	0,076	0,00	0,00	0,00	0,00	0,076	STANDTE		0 131107	3
13.11.2007 14:24:05	7,074	0,00	0,00	0,00	0,00	0,075	STANDTE		1 131107	6
15.11.2007 11:30:19	0,000	0,00	0,00	0,00	0,00	0,000				0
15.11.2007 12:04:19	0,119	0,00	0,00	0,00	0,00	0,119	STANDTE		0 1E1107	1

11.2.6. Export data in CSV

You can export data into CSV-format. When exporting into the CSV-format filter- as well as sorting functions are taken into account.

- Open the result table and select the desired table sheet in the respective table.
- Set filter- and sorting functions
- Press the right mouse key..
- Select **"Export"** from the context menu.
- Enter a name for the file and press key **"Store"**.

The exported data can now be opened in an excel sheet.

11 Table of results

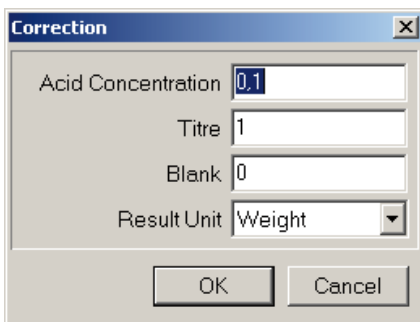
11.2.7. Delete results

You can delete individual lines or several lines at the same time.

- With the left mouse key, highlight the line which you want to delete (should you want to delete several lines press the Strg-key simultaneously).
- Press key "Delete" and confirm the dialogue field with **"OK"**.

Comment

Prior to deleting the data, you can archive them using the CSV-export in an excel program (see chapter 11.2.6. CSV-export).



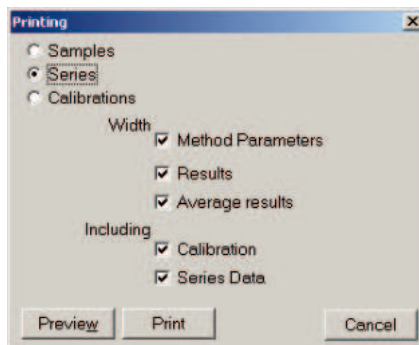
11.2.8. Correct results

You can correct single results later on.

- Highlight the line, where you want to make corrections with left mouse key (to highlight several lines press the strg-key simultaneously).
- Press key "Correct" and enter the individual corrective data
- Press key "OK" to confirm

11.3. Print data

You can print data of single samples or series. Press key **"Print"** to open the print menu.



In order to do so, highlight one single or several lines in the table of results. To highlight several lines, hold the Strg-key pressed down and click on the lines desired.

Samples

The data of the highlighted sample is printed.

Series

The data of the entire series belonging to the highlighted sample are printed.

Calibration

Only the calibration data belonging to the highlighted sample are printed. Depending on the selection, you can chose further options for the print, like e.g. method parameter, results, mean value (of same samples), calibration and series data.

Press key **"Preview"** to obtain a print preview. Press key **"Print"** to start printing process.

12 Error messages

The functions of the distillation system are monitored continuously. As soon as an error occurs, it is shown in the Vapodest-display, in the status bar of the analysis window and with an acoustic signal.

After solving the error you can continue the analysis in most cases. In order to do so, you have to press the key "Continue" in the analysis window.

12.1. General error messages

Error message	Trouble shooting
Sensor error steam generator	Turn instrument off, then turn it on again. Should the problem not be solved, call customer service.
Excess steam pressure	Turn instrument off, and then turn it on again after 15 minutes. Should the problem reoccur, call customer service.
Error waterlevel steam generator	Turn instrument off, check reservoir for "H2O", and then turn it on again. Should the problem not be solved, call customer service.
Error hardware. Switch power off	Turn instrument on and off. Should the problem not be solved, call customer service.

12.2. Error messages pH-Calibration

Error messages	Trouble shooting
Stabilization time > 30 s	Check electrode (see instruction leaflet of the producer of the electrode) and if necessary get a new one.
Measuring range zero point > +/- 15 mV	Repeat calibration with fresh buffer solution or check electrode.
Slope / pH = x. tolerance range: <50-70 mV/pH>	Repeat calibration with fresh buffer solution or check electrode.
Measuring range 180 mV +/- 15 mV	Repeat calibration with fresh buffer solution or check electrode.

12 Error messages

12.3. Error messages during the analysis

Error message	Trouble shooting
No cooling water	Cooling water pressure < 0,5 bar. Check cooling water inlet. Make sure that the faucet is turned on. By pressing the key „Continue“ the analysis is continued. By pressing "Stop" the analysis is aborted.
Receiver overfilled	The analysis is aborted. Take out receiving vessel and empty it. (take out glass or start suction receiver manually).
No sample tube or door open	Put in guidance cage or close the door. Attention, should the analysis be in process, it is aborted!
Wait for steam	Message disappears, as soon as the steam generator has reached the correct pressure.
Check chemicals	If the Vap 50 carousel is used as a Vap 50 (manual sample feeding), the level sensor control has to be set on „Inactive“ (see chapter 7.4. carousel“).
No NaOH No H ₃ B O_3 No H ₂ O No Titrant	Check content of the reservoirs and if necessary, fill them up. Check contacts of level sensor. The distillation in process is finished. After solving the problem the series can be started again.
Waste full	Check tank for sample residues and if necessary, empty it. The distillation in process is finished. After solving the problem the series can be started again.
Blank value is out of range	Select a bigger tolerance range or check whether there is a contamination of the analysis system, e.g. rinsing residue in the digestion tube, carry over of the sample, contamination of the chemicals, etc
Titration time out	End point is not found. <ul style="list-style-type: none"> - Check electrode and if necessary calibrate. - Check titration liquid. - Check, whether there is air in the titration hose (e.g. after changing the titration liquid) - Check whether micro doing pump is running.

12 Error messages

12.4. Error messages communication

Error messages	Trouble shooting
Incompatible Vapodest data on PC. Copy data to PC.	Saved data of the ComBox are written on the PC. Press key 'OK' to start the process.
No device found	Check the cabling (see chap. 5.7. Connect PC/Lap-top)
Autosampler Communication Problem	Vapodest Manager is looking for the auto sampler set of tanks. If no auto sampler is used, you have to enter under System>Settings>Carousel and then - Using internal set of tanks - 'No' (see chapter 7.4. Carousel).

12.5. Error messages during programming

Error messages	Trouble shooting
No method available	Define a method (See chap. 8 Create a method).
No rinsing method	Define a rinsing method (See chap. 8 Create a method)
There are undefined methods	If the series is called up, which uses a program which had been deleted after the sample input; it has to be defined again. Call up main menu "Create a method" and check programming..

If the input is not correctly defined or not allowed, an error message is displayed during the respective program step; the corresponding message is displayed. Please correct your input accordingly.

12 Error messages

12.6. Error messages carousel

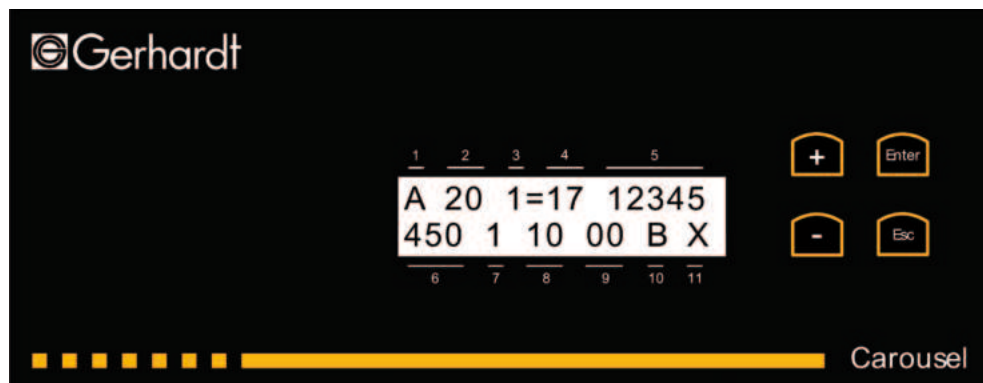
Error messages	Trouble shooting
Air pressure to low	Check compressed air, check whether compressed air connections are sitting tight and check voltage supply of the compressor.
Air pressure to high	Check compressed air, check whether compressed air connections are sitting tight and check voltage supply of the compressor.
Problem with the drive of the carousel	Turn off instrument and call customer service.
Sensor error Z-axis	Turn off instrument and call customer service.
Wrong working position	Check whether original-Gerhardt-glasses are used. Maybe the glass feeding is blocked.
Tube carriage not lowered	Check whether the throw is blocked. Turn off the instrument and call customer service.
Check protection cap.	Check whether the loading flap is closed. Check whether the protective hood of the carousel is fitting tightly.
Carousel rack error	Check functions for selection of carousel (see also chapter 7.4. carousel) or check defined series.
Carousel / Z-axis error	Turn off instrument, call customer service.

13 Service display of the carousel

Sensor key
for opening
the drawer



The service display helps to detect errors fast. Furthermore, the current operating status is shown and can be transmitted directly to the customer service desk of your dealer. In order to open the service drawer, please touch the sensor for about 5 seconds. After opening the service drawer, the service display shows the current number of the version in use and then, switches automatically to the main menu.



13.1. Set the contrast

While being in the main menu, push the key "-" once and confirm with "Enter". Now, the contrast can be changed by using either "+" or "-". After setting the desired contrast, press "Esc". The settings are stored. By pressing "+" you get back to the main menu.

13.2. Autosampler test

By using the test for the autosampler all functions of the carousel are tested automatically. From the main menu press the key "-" twice. Press "Enter" to start process. The program can be interrupted at any time by pressing "Esc".

13.3. Description of the various positions in the main menu

In the main menu 11 positions are displayed to indicate the available functions and the current operating status of the carousel.

1. Status of the Carousel (1-digit display)

L = Loading; the autosampler is working on its own and is not yet controlled by the program of the Vapodest 50s.

A = Auto, the program of the Vapodest 50s controls all functions of the autosampler; the analysis has been started.

T = Test, a test program can be called up for a simple test of the autosampler in order to simulate an entire work process for the selected carousel.

2. Type of Carousel (2-digit display)

Shows the type of carousel selected. The display has to correspond to the type of carousel and the setting of the software.

20= carousel 20-place

16= carousel 16-place

12= carousel 12-place

3. Super sonic (1-digit display)

Check by ultrasound whether there is a tube in the current state of the analysis.

1 = Tube

0 = No tube

13 Service display of the carousel

3.1 =, >, < (1-digit display)

The position of the carousel is checked permanently. The 360° of the carousel can be divided into 1024 positions. The lift of the Z-axis can only be activated if the display shows the exact positioning of the carousel.

4. Position of the tube (2- digit display)

Shows the current position of the tube.

5. Tank (5-digit display)

An error concerning the tank is shown by a flashing display. If ,!' is displayed the minimum level (respectively the maximum level in the tank sample waste) has been reached!

- 1 = H₂O-tank
- 2 = NaOH-tank
- 3 = H₃BO₃-tank
- 4 = Titration acid-tank
- 5 = Sample waste-tank

6. Pressure Control (3- digit display)

Shows the current pressure in kPa. Standard pressure about 450 kPa.

7. Protection hood and loading flap (1- digit display)

- 1 = closed
- 0 = open, or protection hood not available

8. Pneumatic sensors (2- digit display)

Shows the current position of the lifting cylinder. The left display shows the lower sensor, the right display the upper sensor.

- 1 = Sensor active
- 0 = Sensor non-active

9. Air valves (2- digit display)

The status of the compressed air valves is displayed with "0", respect. (↑ ↓). Only one valve at a time can be addressed.

- 0↑ = Display is shown during the upward movement of the carriage for the tubes and the distillation
- ↓0 = Display shown during the downward movement of the carriage for the tubes
- 00 = No valve is addressed

10. Stepping motor (1- digit display)

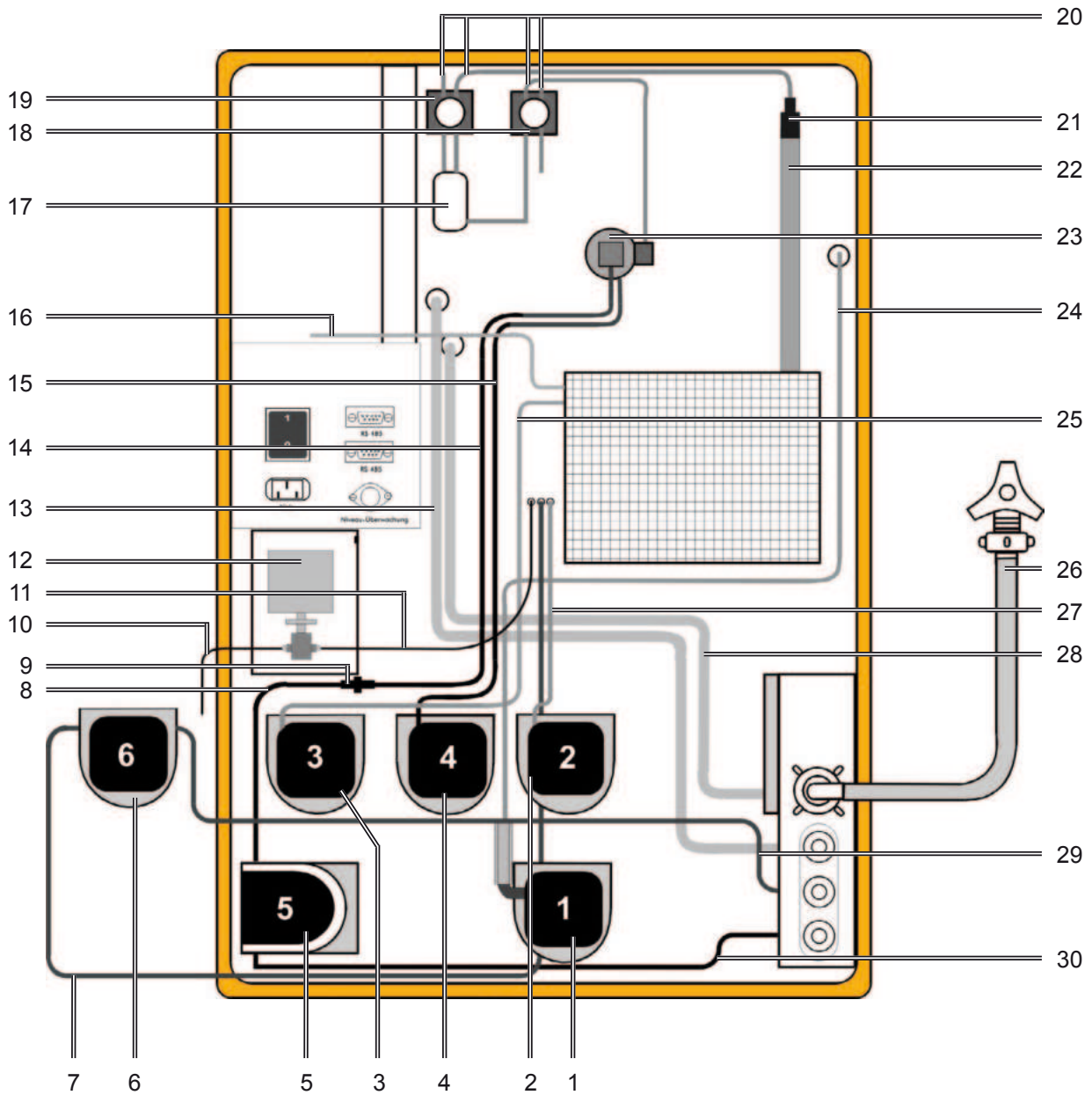
- R = Right, sense of rotation of the motor
- L = Left, sense of rotation of the motor
- S = Stop, the motor is free of tension, the carousel can be turned manually
- B = Break, the motor is kept in the current position

11. Loading position (1- digit display)

- A = 1st loading position
- B = 2nd loading position
- C = 3rd loading position
- X = No loading position

14 Tubing diagram

14.1. Upper part Vapodest with open rear wall



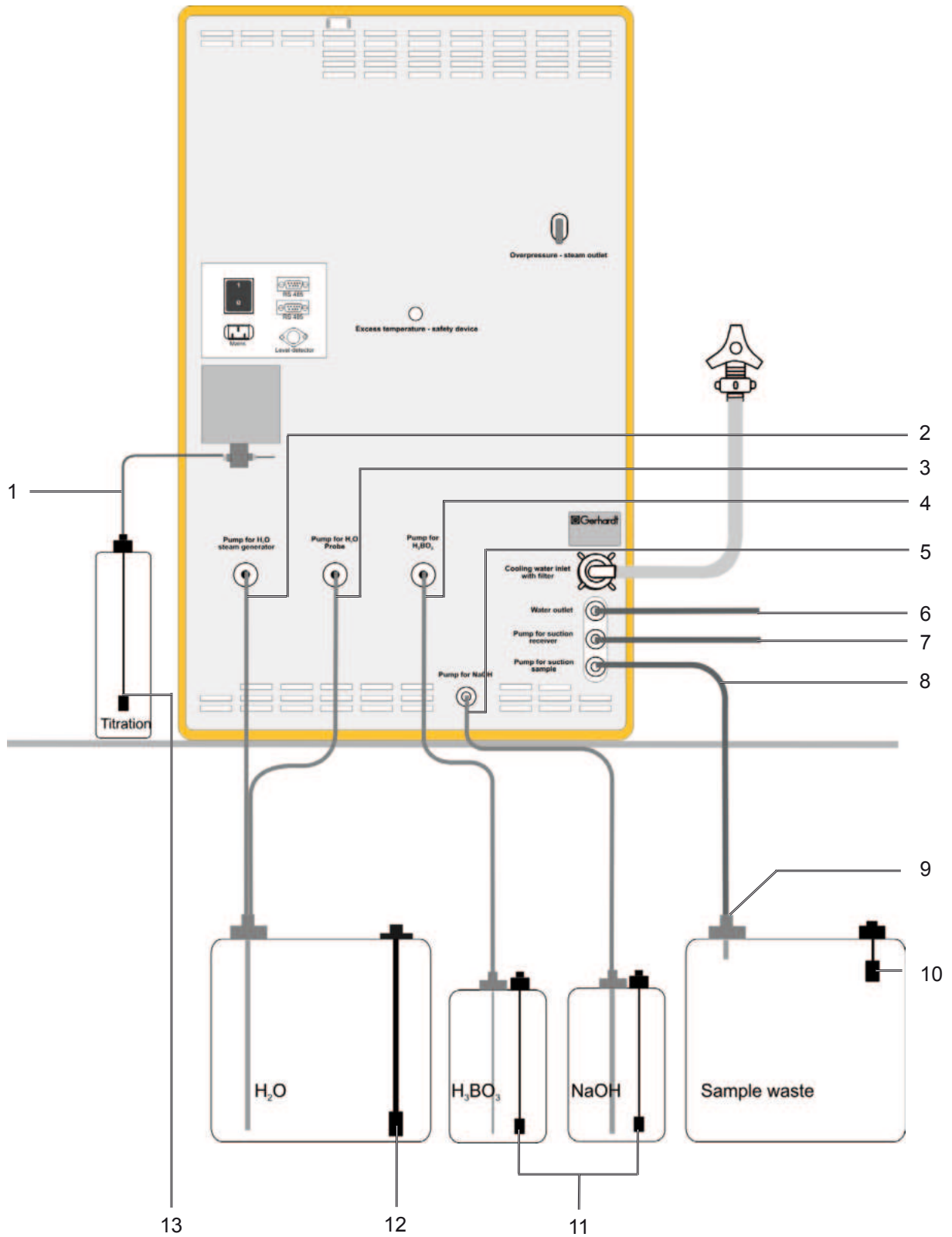
14 Tubing diagram

14.1.1. Description of the tubing diagram

Pos.	Description	Item-No.
1	Diaphragm pump NaOH	1001738
2	Diaphragm pump H ₃ BO ₃	1001736
3	Diaphragm pump H ₂ O steam generator	1001736
4	Diaphragm pump H ₂ O sample	1001736
5	Peristaltic pump suction waste	1000084
6	Peristaltic pump suction receiver	1000084
7	Verprene-tubing 4/8	1000498
8	Verprene-tubing 4/8	1000498
9	Tubing reduction, PP 51 x 10 x 5	1000398
10	PTFE-tube suction for titration liquid 1,6 x 3,2 x 1600 mm incl. fitting	1001724
11	PTFE-tube with stretched point for titration acid 1,6 x 3,2 x 500 mm incl. fitting	1001672
12	Micro dosing pump	1001650
13	Silicon tubing 8 x 16, Outlet condenser	1000497
14	Verprene-tubing 8 x 12	1000499
15	Verprene-tubing 4 x 8	1000498
16	Silicone-tubing 4 x 7	1000488
17	Ventilation glass	1000048
18	Pinch-solenoid valve, shut-off	1000090
19	Pinch-solenoid valve, steam	1000090
20	Novoprene-tubing 4,8 x 8	1002289
21	Tubing reduction	1000397
22	Silicone-tubing 6 x 10	1000484
23	PP-distributor with PP-thread	1000538
24	PTFE-Inlet tubing, NaOH	1003401
25	Silicone tubing 4 x 7	1000488
26	Water inlet tubing 10 x 17	1000394
27	Silicone-tubing 4 x 7	1000488
28	Silicone-tubing 8 x 16 for cooling water outlet	1000497
29	Verprene-tubing 4 x	1000498
30	Verprene-tubing 4 x 8	1000498

When you order additional tubing, please make sure to also give the desired length!

14 Tubing diagram



14 Tubing diagram

14.2.1. Description of the tubing connections

- 1 PTFE-tubing suction for titration liquid
- 2 PVC-tubing 4/7, 2,85 m
- 3 PVC-tubing 4/7, 2,55 m
- 4 PVC-tubing 4/7, 2,55 m
- 5 PVC-tubing 8/12, 2,60 m
- 6 PVC-outlet tubing 8/12, 2,00 m
- 7 PVC-outlet tubing 8/12, 2,00 m
- 8 Verprene-tubing 8/12, 1,9 m
- 9 Snap lid for tubings (for all tanks)
- 10 Level sensor with plug for "Sample waste" tank, complete
- 11 Level sensor with plug for chemicals reservoir tank, complete
- 12 Level sensor with plug for water tank, complete
- 13 Level sensor with plug for titration tank, complete

15 Maintenance

Please make sure that only original C. Gerhardt spare parts are used.

To minimize malfunctions of the VAPODEST, we recommend having it checked by authorized service personnel annually.

15.1. Service and cleaning

Make sure to wear gloves at all times when cleaning the components.



To avoid damage by corrosion, leaking liquids (especially acids and alkali) must be wiped off and neutralized immediately to prevent damage to casting, lacquer or housing.

Never use abrasive cleaners! In most cases it will be sufficient after each use to clean the plates using a moist cloth and some household washing-up liquid. Subsequently rub dry.

Condensate that might escape is collected in the drip tray. Please clean the drip tray regularly.

To clean the carousel tray take out the carousel as described in chapter 10.3.1. and clean the tray.

Regularly check the state of the tubing and tubing connections and exchange them if damaged.

To minimize malfunctions of the Vapodest 50s carousel, we recommend to have it checked by authorized service personnel regularly.

15.1.2. Cleaning program

Glass parts and suction pump should be cleaned before long periods of non-usage (i.e. holidays). This way blockages caused by crystalline deposits are avoided.

The following program should be run:

Addition H_3BO_3	0	ml
Addition H_2O	120	ml
Addition NaOH	0	ml
Reaction time	0	s
Distillation time	7	min
Steam power	100	%
Suction time	20	s

Place an empty digestion tube and an Erlenmeyer flask into position, and start the program. In case of extreme deposits in the glassware you can clean the system by putting about 10 ml of sulphuric acid into the digestion tube.

15 Maintenance

15.2. Spare parts

Upper part, distillation system

Distillation condenser, glass	1000059
Plastic screw connection for distillation condenser	1000358
Ventilation valve	1000036
Distribution head, PP	1000056
Vitone cone	12-0351
PTFE-inlet tubing, steam	1002178
Teflon sieve for item-no. 1002178	1000630
PTFE-inlet tubing, NaOH	1003401
PP-distributor	1000538
PP-tube joint	1000250
Holder for pH-electrode	1001257
Level recorder in the receiver	1001265
pH-Electrode	12-0354
Propeller	1001260
Receiver made of glass	1001269
Screw cap GL 14	1000350
Screw cap GL 18	1000351
Screw cap GL 32	1000353
Silicone-/PTFE-seal GL 18	1000356
Silicone-/PTFE-seal GL 32	1000357
Plexiglas protection door	1001263
Door handle	1000392
Door hinges, 1 pair	1000391
Drip tray with hole for upper part	1000399
Mains switch	1004331
Control panel	1004119
Knob for operator button	1004325
Excess current switch, 10A	1001358
Tubular heating element	1000259
Steam generator 230 V	1000232
Excess pressure valve, steam generator	1000483
Mains cable	10-0058
Magnetic valve with pressure control	1000363
Micro switch for quick clamping device	1000290
Pinch-solenoid valve steam and shut-off	1000090
Ventilation glass	1000048
Diaphragm pump for NaOH	1001738
Diaphragm pump for H ₃ BO ₃ or H ₂ O	1001736
Peristaltic pump	1000084

Tubes

When you order additional tubing, please make sure to also give the desired length!	Water inlet tubing 10/17, 2 m	1000394
	Silicone-tubing 6 x 10	1000484
	Silicone-tubing 8 x 12	1000496
	Silicone-tubing 4 x 7	1000488
	Silicone-tubing 16 x 8	1000497
	Novoprene-tubing 4,8 x 8	1002289
	Verprene-tubing 4 x 8	1000498
	Verprene-tubing 8 x 12	1000499

15 Maintenance

PTFE-tubing 4 x 6	1000559
PVC-tubing 4 x 7	1000500
PVC-tubing 8 x 12	1000047
PTFE-tube suction for titration liquid (with fitting)	1001724
PTFE-tube with stretched point for titration acid (with fitting)	1001672
Tubing reduction, PP 51 x 10 x 10	1000397
Tubing reduction, PP 51 x 10 x 5	1000398

Lower part, carousel

Tank 10 liter for H ₃ BO ₃ and NaOH	1001981
Tank 20 liter for H ₂ O and waste sample	1000341
Tank 5 liter for titration acid	1001980

Snap coupling



Snap lid



Quick lock for **tank lid H₂O and H₃BO₃** consists of:

1 x Snap coupling with cut-off	1001982
1 x Snap lid plug with cut-off	1001984



Quick lock for **tank NaOH** consists of:

1 x Snap coupling with cut-off	1001983
1 x Snap lid plug with cut-off	1001985



Quick lock for **tank Sample Waste** consists of:

1 x Snap coupling without cut-off	1002173
1 x Snap lid plug without cut-off	1002174

Protection hood for carousel	1001908
Drip tray for carousel	1001905
Round belt 4 x 960 for carousel drive	1001921
Side extension (for Laptop or printer)	1001896
PVC-pipe 6 x 1, 300 mm	1002027
PVC-pipe 10 x 1, 230 mm	1002028
PVC-pipe 6 x 1, 230 mm	1002029
Level sensor with plug for "Sample waste" tank, complete	1002121
Level sensor with plug for chemicals reservoir tank, complete	1002122
Level sensor with plug for water tank, complete	1002120
Level sensor with plug for titration tank, complete	1002123

Electronic

Sensor switch for service drawer and control of the loading positions	1001874
Power-board	1001881
Control-board	1001880
Keyboard-Service display Carousel	1002001

15.3. Accessories

Sets for adaption

for tubes 250 ml:

Basket 250 ml	1001797
Carousel 20-place for 20 x 250 ml tubes	1001950

for tubes 400 ml:

Basket 400 ml	1001804
Carousel 16-place for 16 x 400 ml tubes (KDD and BS 400)	1001959

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for tubes 800 ml:

Basket 800 ml	1001807
Carousel 12-place for 12 x 800 ml tubes	1001963

Set of adaption Micro (only Vap 50)

Vitone cone VK, micro, for 100 ml tubes KMT	12-0352
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More accessories

Tubes

250 ml, KTG 250 standard version	12-0301
400 ml, KDD 400 standard version	12-0310
800 ml, KDD 800 standard version	12-0311
400 ml, BS 400 special version, diminished	12-0308

EDV

Data cable DK 42 for RS 485, 2 m	10-0192
Data cable DK 45 for RS 485, 2 m	10-0193
Terminator AST for RS 485	10-0196
RS-Adapter	10-0190

Further accessories and information about the Vapodest program can be found in the product catalogue or at your local dealer!

15.4. Trouble shooting

The excess current switch cuts off in case of excessive current consumption. Switch on the switch again.

Make sure to handle error messages as described in chapter "12. Error Messages".

In case of more problems or failures of your Vapodest 50s carousel, please contact your local dealer or:

C. Gerhardt GmbH & Co. KG
Cäsariusstr. 97
D-53639 Königswinter
Phone: +49 (0)22 23 - 29 99 0
Fax: +49 (0)22 23 - 29 99 99
info@Gerhardt.de
www.gerhardt.de

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15.5. Disposal

The disposal of the packaging and the parts used has to be done according to the rules and regulations which are valid in the country of installation. Should the product itself be disposed of, make sure to observe the local rules and regulations..

15.5.1. Information for the Disposal of Electric and Electronic Instruments within the European Union



The disposal of electrically operated instruments is settled within the European Union by national regulations, which are based on the EU-directive 2002/96EC about electric and electronic used-instruments (WEEE). Thus, all instruments delivered after August 13th 2005 must not be disposed into the communal domestic waste.

Since the regulations about disposal of waste might vary from one country to the other within the European Community, we kindly ask you to contact your supplier or dealer.

In Germany, this obligation for identification will be valid as of March, 23rd 2006. As of this date, C. Gerhardt will take back all instruments delivered after August, 13th, 2005 without charges and will dispose of them according to the regulations, or Gerhardt will come to an agreement with the last user of the unit. For all instruments delivered prior to August 13th, 2005 the last user will be held responsible for the proper disposal. The only crucial factor accepted, for the chronological placement, is the serial number at the back of the instrument.

15.5.2. Ban on Materials according to ROHS Regulation 2002/95/EG

Ban on materials from the ROHS regulation 2002/95/EG is not valid for the electro- and electronic instruments of category 8 and 9 and thus not for those instruments described in this instruction manual. However, we want to draw your attention to the fact that we feel obliged to observe the regulations for the RoHs for all our products. Please be kind enough to contact us if you have any further questions.

15.5.3. Transfer

We kindly ask you to always make sure to add this instruction manual to the product in case of transferring it to another party.

16 Other

Calculation of the results

c = nominal concentration of the standard acid solution

f = correction factor of standard acid solution (titer)

- 1) $\% \text{ N} = \frac{1,4007 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Content [g]}}$
- 2) $\% \text{ Protein} = \frac{\text{Protein factor} \times 1,4007 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Content [g]}}$
- 3) $\% \text{ NH}_3 = \frac{1,7031 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Content [g]}}$
- 4) $\% \text{ NH}_4^+ = \frac{1,8039 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Content [g]}}$
- 5) $\text{mg NH}_4^+ = c \times f (\text{Consumption [ml]} - \text{Blank value [ml]}) \times 18,039$
- 6) $\text{mg NH}_3 = c \times f (\text{Consumption [ml]} - \text{Blank value [ml]}) \times 17,031$
- 7) $\% \text{ NO}_3^- = \frac{6,2005 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Content [g]}}$
- 8) $\text{mg NO}_3^- = 62,005 \times c \times f (\text{Consumption [ml]} - \text{Blank Value [ml]})$
- 9) $\text{mg N/l} = \frac{14007 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volume [ml]}}$
- 10) $\text{g N/l} = \frac{14,007 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volume [ml]}}$
- 11) $\text{mg NH}_3/\text{l} = \frac{17031 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volume [ml]}}$
- 12) $\text{g NH}_3/\text{l} = \frac{17,031 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volume [ml]}}$
- 13) $\text{mg NH}_4^+/\text{l} = \frac{18039 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volume [g]}}$
- 14) $\text{g NH}_4^+/\text{l} = \frac{18,039 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volume [ml]}}$
- 15) $\text{mg NO}_3^-/\text{l} = \frac{62005 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volume [ml]}}$
- 16) $\text{g NO}_3^-/\text{l} = \frac{62,005 \times c \times f (\text{Consumption [ml]} - \text{Blank value [ml]})}{\text{Volumen [ml]}}$

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