VAPODEST 45

INSTRUCTION MANUAL

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

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

Vap 45

Order-No.: 764510 - Vapodest 45 complete with Titrator TL Easy + accessories
Order-No.: 7645 - Vapodest 45, Basic system without Titrator

gb 5045/00 *18.11.2001*

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Operation Manual

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Operational usage

With the Vapodest 45 you have purchased an automatic distillation system for the steam distillation of Kjeldahl digestions and associated distillations. Automatic addition of H_2O, NaOH and H_3BO_3 as well as removal of the sample remains after distillation by suction. The distillation system is also prepared to work with an external titrator.

Safety instructions

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

For repairs of electrical, electronic, or mechanical parts always contact your dealer or a qualified engineer!

Always switch off the apparatus at the mains and unplug before opening! Danger of electric shock!

The Vapodest 45 must not be operated in humid or hazardous locations. The maximum humidity allowed is 80%, the maximum ambient temperature must not exceed 40 °C (104 °F)!

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

The equipment must be operated according to this instruction manual. It is not permitted to change any part in order to modify its application.

Attention when handling acids and alkalis! Please observe your national safety regulations!

Use gloves when removing the glass digestion tubes as these can be very hot and there is danger of burning yourself! Also make sure you wear eye protection!

Be careful whenever handling glass and follow the national safety regulations concerning the handling of glass parts!

Gerhardt only guarantees a safe operation of the autosampler if Gerhardt Original-Glass parts are used!

The instrument must be operated by a skilled or specially trained staff!

1. Technical description

1.1. Warranty

Vapodest 45 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.

Please note that the natural wear- and -tear is excluded from warranty.

1.2. Technical data

Voltage: .....230 V AC, 50 Hz
Wattage: .....1600 W
Cooling water: .....about 3 liters per distillation minute
Cooling water pressure: > 0,5 bar
Pump capacity
- Diaphragm pump: ...about 13 ml/s
- Peristaltic pump: .....about 10 ml/s

Storage tanks: .....any size, recommended: KAN 40
Display: .....2 x 16LCD
Programs: 20
Dimensions: .....440 x 690 x 340 mm (B x H x T)
Weight: .....29 kg
Interface: .....2 x RS 485
.....1 x RS 232 (interface for external titrator)

1.3. Operating conditions

Vapodest distillation systems can be run under normal laboratory conditions. See also safety instructions on page 4.

For the connection to the tap (cold water) a permanent connection with an ½ inch thread is required.

2. Delivery

2.1. Check for transport damages

Before assembling the equipment please check it thoroughly! In case of any damage, please notify your carrier (mail, rail, road) immediately and obtain an expert's report!

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

2.2. Package list

- 1 x Distillationsystem Vapodest 45, complete

- Tubing set:
  1 x Water inlet tube 10/17 mm with connections of 1/2 inch and 3/4 inch, 2m
  1 x Verprene tube 8/12 mm, 2 m
  3 x PVC tube 4/7 mm, 2 m
  3 x PVC tube 8/12 mm, 2m
  3 x PVC pipe 6 x 1 mm, 420 mm
  1 x PVC pipe 10 x 1 mm, 420 mm

- 1 x Mains cable

- 1 x Kjeldatherm-digestion tube, 100 or 250 ml

- 1 x Instruction manual

Optional:

- 1 x Titrator Titro Line Easy

- 1 x pH-Electrode

- 1 x Electrode cabel DIN Vap 45

- 1 x Buffer solution pH 4, 250 ml

- 1 x Buffer solution pH 7, 250 ml

- 1 x KCL-Electrolyte solution, 250 ml

3. Description of the equipment

3.1. Front view Vapodest 45 - Version with Titrator

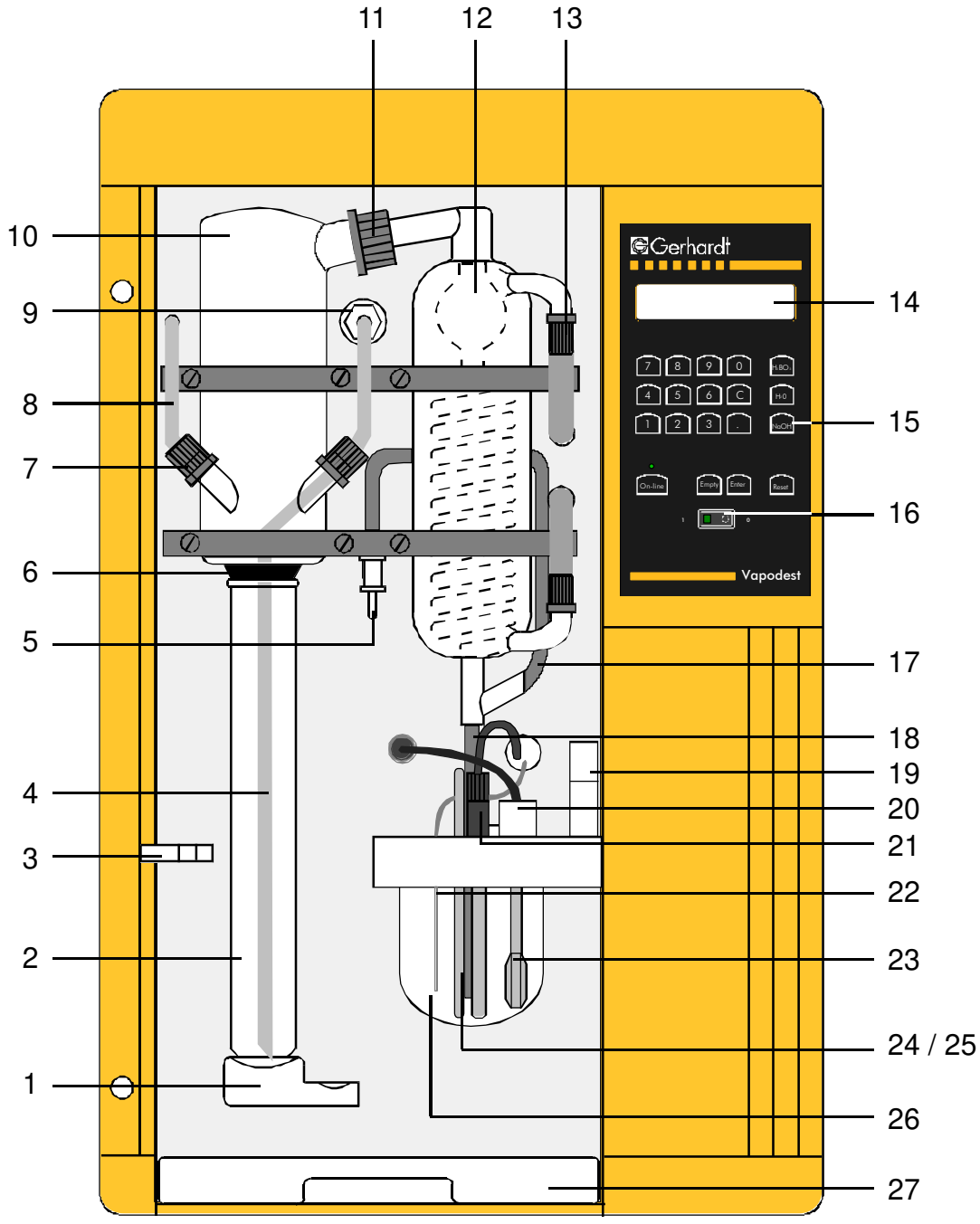


Figure 3.1: Vapodest 45 - Version with Titrator

3.2. Front view Vapodest 45 - Version without Titrator

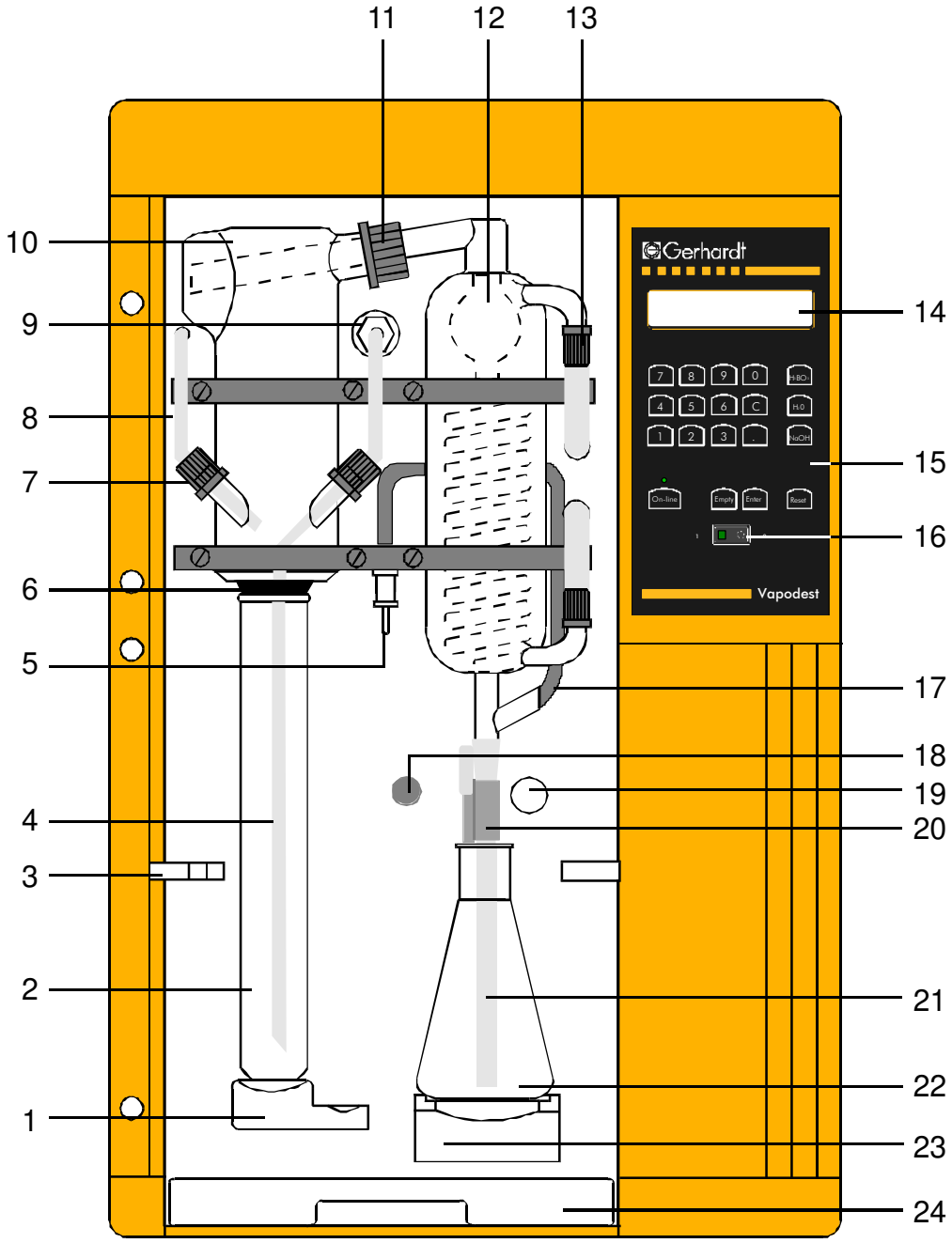


Figure 3.2: Vapodest 45 - Version without Titrator

3.3. Structural components and parts of front view

3.3.1. Front view Vapodest 45 - Version with Titrator

1 Quick clamping device with wedge
Digestion glass can be pressed to connection piece safely and securely. Press down the quick clamping device in order to insert digestion tube.

2 Kjeldatherm-Digestion tube
Special glass with cut
100/250 ml or flask with enlarged neck 250/500/750 ml or KDD 400/800 ml.

3 Holder for steam inlet tubing
If no digestion tube is attached the tubing can be fixed here instead.

4 PTFE-inlet tubing, steam - Order no. 11810
Steam, water for dilution, and the suction of the sample residues passes through here. Please regularly check that the opening of the PTFE-tube is not blocked by crystallisation.

5 Ventilation valve - Order no. 6474
During and after distillation the ventilation valve prevents suck back of the receiver solution into the condenser.

6 Viton-cone, macro or micro - Order no. 6470
The stopper ensures a perfect fit to the digestion tubes.

7 Screw cap GL 18 with silicone/PTFE seal - Order no. 16602

8 PTFE-inlet tubing, NaOH - Order no. 11811
Sodium hydroxide solution enters here.

9 PP-Distributor with PP-threaded joint - Order no. 50024

10 Distribution head - Order no. 7472
The inlet tubing for steam and sodium hydroxide solution pass through the distribution head. They are secured with screw caps (pos. 7). The distribution head prevents contamination of the distillate by the sample.

11 Screw cap GL 32 with silicone/PTFE seal - Order no. 7673

12 Distillation condenser - Order no. 7673
A combination of a bulb condenser and a coiled condenser acc. to Dimroth which provides in-ten-sive cooling capacity.

13 Screw cap GL 14 with plastic screw connection - Order no. 16601 + 16609

14 Display 2 x 16 LCD - Order no.
See chapter 6 „Keyboard“

15 Keyboard, chemical resistant - Order no. 11215
See chapter 6 „Keyboard“.

16 Mains switch, green, illuminated - Order no. 11750
Turns the equipment on or off

17 Tube for ventilation valve - Order no. 20903
Connection tube to the ventilation valve

18 Distillate outlet tubing, silicone 8/12 - Order No. 22704

19 pH electrode holder - Order no. 50034
The holder can be filled with potassium chloride solution 3.5 mol/l (included in the delivery). Here you can store the electrode when it is not in use to prevent the porous frit from drying out.

20 Sensor for level detection - Order no. 40870
The built-in sensor stops the program if the receiver vessel overfills during the distillation and titration.

21 pH-electrode (combined electrode) - Order no. 6602
Combined electrode with screw thread.
For further details please check the enclosed instructions by the electrode’s manufacturer.

22 Inlet tubing for titration acid

23 Stirring motor with propeller

24 Tubing for suction of the distillate - Order no. 20914
is behind the tubing for suction of the distillate
25 Inlet tubing for boric acid - Order no. 20290

26 Receiver vessel - Order no. 10214
For easy removal turn the glass through 90° and pull it downwards.
27 Drip tray - Order no. 50015
Condensate which might drip is collected in the drip tray. Make sure to clean the tray regularly.

** **Protection door (not illustrated) - Order no. 19017**
Whenever you run the distillation system make sure you shut the protection door!

3.3.2. Front view Vapodest 45 - Version without Titrator

Position 1 - 17 see page 8

18. Interface for stirrer motor / level detection

19. Wall duct for titration hose and electrode cable

20. Holder PVC

21. Destilation outlet tube, Silicone tubing 8/12 8/12 - Order no. 22704

22. Erlenmeyerflask

23. Platform - Order no. 17202
can be unscrewed if external titrator is used

24. Drip tray - Order no. 50015
Condensate which might drip is collected in the drip tray. Make sure to clean the tray regularly.

** **Protection door (not illustrated) - Order no. 19017**
Whenever you run the distillation system make sure you shut the protection door!

3.4. Rear view

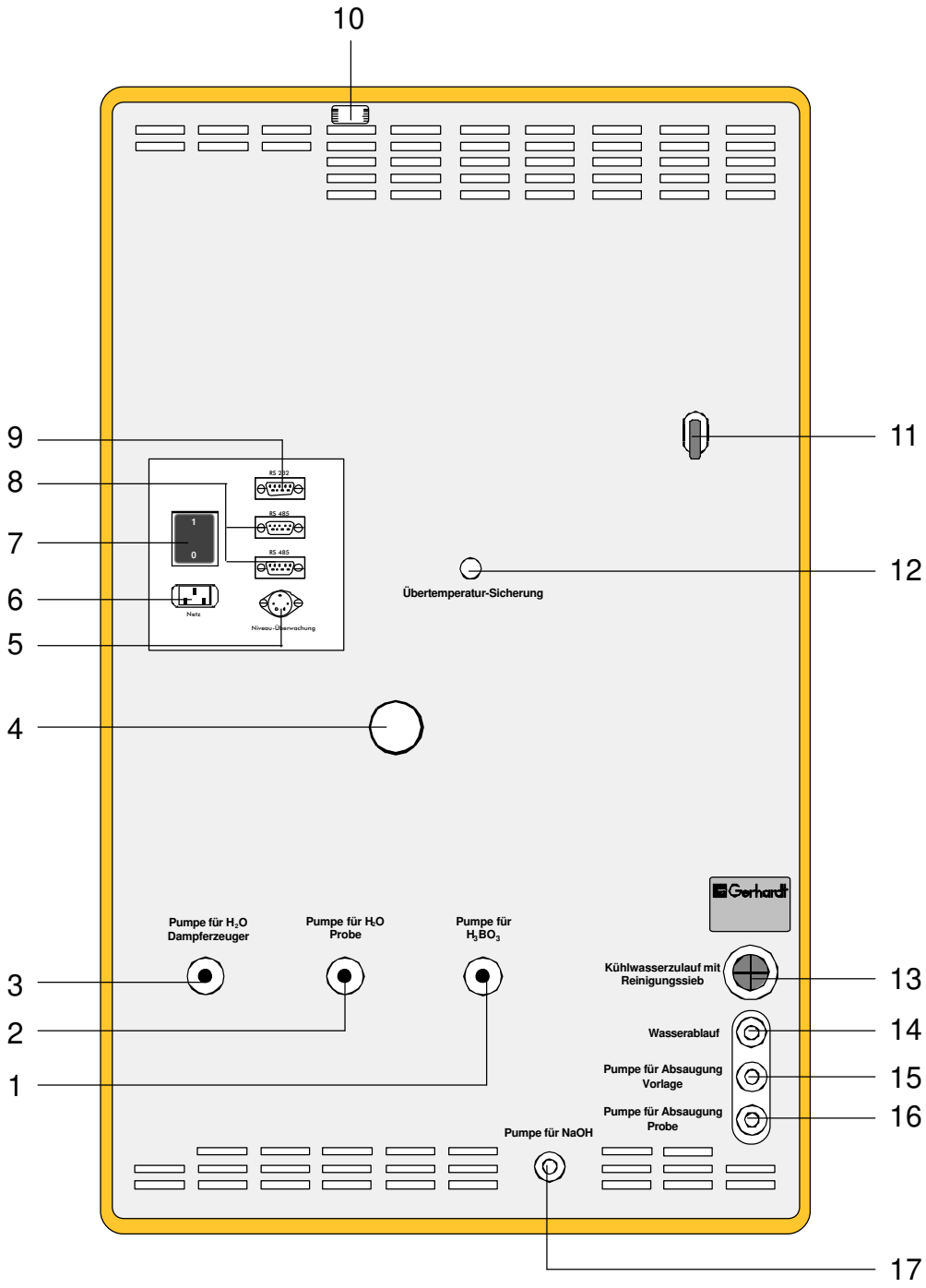


Figure 3.4: Vapodest 45 - Rear view

3.5. Structural components and parts of rear view

1 Diaphragm pump for H_3BO_3 - Order no. 10310/1
Connection pipe for PVC-tubing 4/7, connection to the storage tank "*H_3BO_3*"

2 Diaphragm pump for H_2O sample - Order no. 10310/1
Connection pipe for PVC-tubing 4/7, connection to the storage tank "*H_2O*"

3 Diaphragm pump for H_2O steam producer - Order no. 10310/1
Connection pipe for PVC-tubing 4/7, connection to the storage tank "*H_2O*"

4 Wall duct
Tube through which hose for the titration liquid as well as the cable for the electrode

5 Level detector
Connector for diode plug, 3-pin (level detector for set of storage tanks KAN 40)

6 Appliance plug with mains cable
The enclosed mains cable is plugged in here

7 Excess current switch, 10A
Equipment is cut off automatically if there is excess current

8 Interface RS 485
Interface RS 485 guarantees an ISO/GLP conform quality control. Up to 32 different units (also of different brands) can be connected to each other.

9 Interface RS 232
Prepared to take the external titrator.

10 Outlet ventilation tubing

11 Excess pressure - steam outlet
Safety device to let steam escape in case of an overpressure in the boiler.

12 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.

13 Cooling water inlet with sieve
Connection thread ¾ inch for water inlet tubing 10/17

14 Water outlet
Connection pipe for PVC-tubing 8/12, connection to water outlet

15 Peristaltic pump for receiver - Order no. 10360
Connection pipe for PVC-tubing 8/12, connection to water outlet

16 Peristaltic pump for sample suction - Order no. 10360
Connection pipe for Verprene-tubing 8/12, connection to store tank "*Sample waste*"

17 Diaphragm pump for NaOH - Order no. 10310/3
Connection pipe for PVC-tubing 8/12, connection to storage tank "*NaOH*"

4. Assembly and Installation

4.1. Set up of equipment

Please observe the local water and waste regulations and those of your public water supply enterprise!

Please note that the length of the inlet and outlet tubing is restricted to 2 meters.

The equipment should be located on a fixed laboratory bench, close to the cold water connection and the drain.

The water pressure must be at least 1.3 bar in order to activate the integrated pressure detector.

There should be sufficient space for the set of tanks below the work bench.

4.2. General information

The VAPODEST 45 distillation system comes fully pre-assembled. Please unpack the equipment with care !

1. Place the equipment on the work bench. Ideally, the equipment should be set up under constant light conditions. This ensures that the contrast of the display does not have to be adjusted all the time. Be aware that direct sunlight on your screen has a negative influence on the quality of your display.

2. Unpack accessories.

4.3. Tubing connections

When connecting the tubings please observe the inscription on the connection pipes at the back of the equipment (see also chapter 3. "Rear View" and chapter 11. "Tubing Diagram").

1. Connect the pump tubes to the PVC-pipes of the store tanks:
   - Inlet tubing sodium hydroxide (PVC-tubing 8/12)
   - Inlet tubing for distilled water steam generator (PVC-tubing 4/7)
   - Inlet tubing for distilled water sample (PVC-tubing 4/7)
   - Inlet tubing for H_3BO_3 (PVC-tubing 4/7)

2. Connect water inlet tubing (pressure proof tissue tubing 10/17) to cooling water inlet and laboratory water supply.

3. Connect outlet tubing:
   - Connect water outlet tubing (PVC-tubing 8/12) to corresponding pipe at the rear of the equipment and place in the drain.
   - Connect sample waste outlet (Verprene tube 8/12) to corresponding pipe at the rear and direct to sample waste tank.
   - Connect outlet (PVC-tubing 8/12) to corresponding pipe at the rear of the equipment and place in the drain (only for models with external titrator)

4.4. Connect Storing Tanks

1. Set up store tanks for distilled water, sodium hydroxide solution, boric acid solution, and sample waste under the work bench. You also have to provide a vessel for the titration acid. See also chapter 11 "Tubing Diagram".

Use of the set of tanks KAN 40 (optional):

1. Connect the diode plugs of the level detectors to the distribution box and connect to the sokket level detection (chapter 3.4, pos.5).

4.5. Mains connection

Please check the nominal voltage on the identification plate before connecting to the mains.

1. Mains connection of the Vapodest:
   - Make sure that the mains switch at the front of the equipment is off (i.e., in position "0")
   - Insert appliance plug in rear outlet
   - Connect mains cable to shock proof socket

2. Excess current switch:
   Check if excess current release at the rear of the equipment is in position "1" (see chapter 3.4. pos. 7). This switch turns the equipment off when it takes too much current.

4.6. Connection of the External Titrator

Please, make sure to study the enclosed instruction manual before using the titrator!

1. **Titrator:**
   - Unpack and place titrator next to Vapodest

2. **Connect Titration Tubes**
   Insert the titration tube from the rear of the Vapodest through the wall duct to the titration cell and put it into the opening. The end of the tube has to be inserted into the receiving vessel.

3. **Connect the interface cable with the interface RS 232 of the Vapodest.**

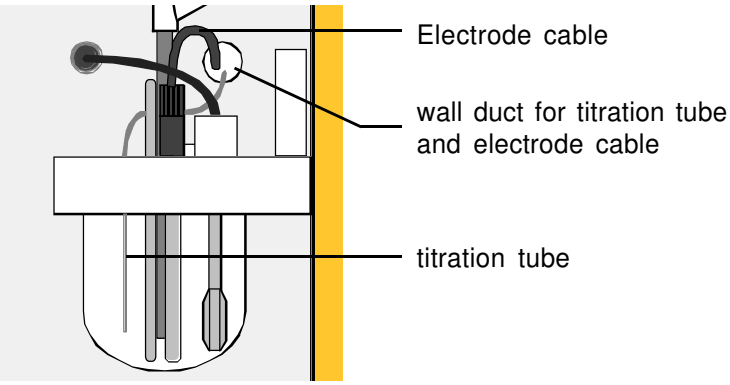
4. **Provide mains connection for the titrator**

4.7. Connection of the electrode

Please, make sure to study the enclosed instruction manual before using the electrode!

1. Unpack electrode with utmost care and connect it with the electrode cable
2. Insert the electrode carefully through the opening into the titration cell till the limit stop.
3. The electrode cable has to be guided through the wall duct from the front of the instrument. Then, connect it to the previewed connection for the titrator (also see instruction manual of titrator).

   Hints for the usage of the electrode will be found on the enclosed manual.



5. Starting operation

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

Always close the protection door before operating the distillation unit!

1. Fill tanks with chemicals:
   - H_2O distilled or demineralized
   - NaOH: 32%
   - H_3BO_3: 2-4%
   - Titration acid

2. Turn on tap. Make sure you turn on tap completely in order to activate the integrated pressure detector of the cooling water valve.

3. **Attention!**
   It is of utmost importance for the communication of the Vapodest 45 with the titrator to turn on the titrator first. If this is not done, the titrator cannot be initialized.

4. Start Vapodest by turning the mains switch on.

5. After switching on the unit the pump to fill the steam generator starts immediately. During booting sequence, the display shows the number of the current software version. Then, the display shows the main menu from whitch all major functions can be selected.

   ```
   Low water
   Press Enter
   ```

   Enter

   ```
   Filling
   Steam generator
   ```

   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.

6. Depending on the pre-settings (with or without titrator), there will be a short notice about the present version of the softwar and then the following message will be displayed:

   Operation without Titrator

   ```
   Distillation = 1
   Programming  = 2
   ```

   Operation with Titrator

   ```
   Dest.=1  Prog.=2
   Result=3
   ```

The Vapodest 45 is now ready for operation.

Turn off the instrument, if you don't intend to use it for a longer period of time.

6. Keyboard

The VAPODEST 45 can be programmed and controlled through the keyboard of the control panel. Please note, that the contrast adjustment of the display depends on the light conditions in the laboratory. The unit should be placed under constant lightning conditions and the display should not be exposed to direct sunlight as this can reduce the contrast of the display so making it unreadable. The functions can be activated by gently pressing the corresponding sections of the panel.

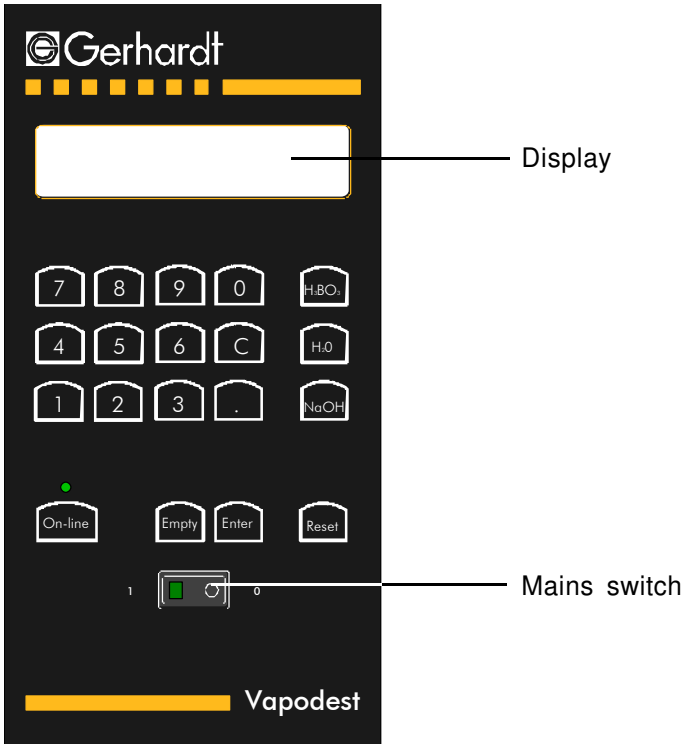


Figure 6.1: Keyboard - Display

Keys	Description
Enter	Confirm or store programs or specific settings
1 9	Numeric keys to set the desired values
Reset	Interrupt a program, return to starting position
C	Go to previous step
On-line	Press to light „On-line“: Control-LED illuminated, Interface is activated
.	Point / Comma Key, Press combined with key „On-line“ to enter synchro mode

Keys	Description
H_3BO_3	Manual addition of H_3BO_3
H_20	Manual addition of H_2O
NaOH	Manual addition of NaOH
Empty	Manual suction of sample
. + Empty	Manual suction of receiver

7. Programming

Each Vapodest undergoes a pre-delivery performance test in the quality assurance department before leaving C.Gerhardt. On arrival the machine is supplied with the test program.

After switching on the Vapodest 40, the display shows the actual version of software,

After that the display shows the standby-mode:

Operation without Titrator

```
Distillation = 1
Programming  = 2
```

Operation with Titrator

```
Dest=1  Prog=2
Result=3
```

From this mode all programming and operation is performed, which is explained in the following.

7.1. Special functions

Selecting language

1. Enter the language selection menu by pressing the key "C".

```
Select language
D=1 GB=2 F=3 E=4
```

D = German GB = English
F = French E = Spanish

2. Select your desired language by pressing the keys "1", "2", "3" or "4", confirm with "Enter".
3. The display changes to the „contrast adjustment“-menu.

7.2. Setting the contrast

```
<1-Contrast-3>
```

1. Pressing the key „1” decreases, pressing the key „3” increases the contrast of the display, according to the surrounding light conditions.
   Note:
   The adjustment range can be further increased by pressing the keys „1” or „3” at the end of each scale.
2. Confirm the set value by pressing the key „Enter”.
3. The display changes to the „chemical sensor“- menu.

7.3. Setting the chemical sensor

The chemical sensor is used in conjunction with the „KAN 40“ set of tanks to monitor the chemical level inside the tanks.

```
Chem. sensor
On=1 Off=2
```

- Press key „1”, if KAN 40 is used.
- Press key „2”, if other tanks are used.
- The display changes to the initial „Standby“-mode.

7.4. Selection of the Titrator

Here, you can select the option of using the Vapodest with the external titrator via the RS 232 interface.

```
Use Titrator
Yes=1  No=2
```

When choosing 2 you get back into the operating mode.

When choosing 1 you get the following message which has to be confirmed by "enter".

```
Titro Easy =1
Titro alpha=2
```

The Vapodest 45 can work with titrators by Schott, Titroline Easy and alpha.

Be careful! The Titroline Easy needs a special software. In case you are already using this titrator, please the local agent of C. Gerhardt or Schott.

Confirm selection by 'enter' and you will be back in the operating mode.

7.5. Programming the distillation system

General remarks
The Vapodest 40 is provided with 10 programs (0-9) where the programs 0-8 are intended for the distillation of KJELDAHL digestions, free ammonia and the nitrogen determination acc. to Devarda. The chemicals to be added in program 9 are not specified.

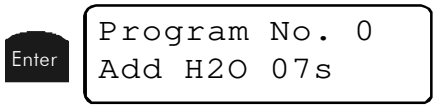
The example used assumes a sulphuric acid digestion leaving approx. 10 ml of free unbound concentrated sulphuric acid.

The display shows the standby-mode:

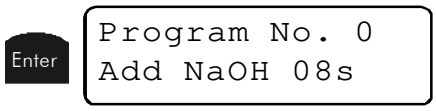
Operation without Titrator	**Operation with Titrator** (For operation with TitroLine Alpha see also additional instruction manual)
2 → `Distillation = 1` `Programming = 2`	2 → `Dest=1 Prog=2` `Result=3`
- Press key „2" to enter the program mode	- Press key „2" to enter the program mode
Enter → `Program No. 0`	Enter → `Program No. 0`
- The apparatus always comes up with program 0. For a different program enter the desired program number. - Press the key "Enter" to confirm the desired program	- The apparatus always comes up with program 0. For another program enter the desired program number. - Press the key "Enter" to confirm the desired program
Enter → `Program No. 0` `Add H3BO3 06s`	Enter → `Program No. 0` `Add H2O 07s`

Operation without Titrator

- The time varies acc. to the chosen program. Enter 6 seconds for a 300 ml Erlenmeyer flask.
  When you change this value, remember that the distillate outlet tubing must always dip into the receiver solution.
- Adjustment range 0-99 s



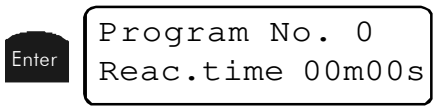
- It is necessary to have a 6 fold dilution of the residual acid after digestion in this case 70 ml = about 7 sec. of H_2O.
- Adjustment range 0-99 s



- Again, It is necessary to have a 6-7* fold dilution of the residual acid after digestion in this case 70 ml = about 8 sec. of NaOH.

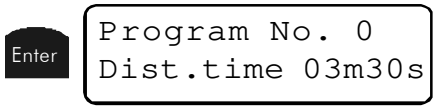
* There must always be an excess of NaOH.

- Adjustment range 0-99 s



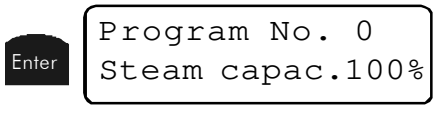
- This allows the beginning of distillation to be delayed so that a cold reaction can occur, e.g. for Devardas. For KJELDAHL this is not relevant, so a time of 0 is entered.
- Adjustment range 0-99 min

- Adjustment range 0 - 59 s



- The distillation time must be chosen so that approx. 90 ml of distillate is collected. On setting 100 % for the steam power 3 minutes, 30 seconds is sufficient.
- Adjustment range 0-99 min

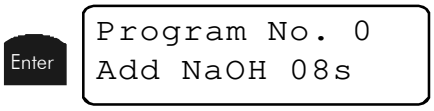
- Adjustment range 0 - 59 s



- Adjustment range 40-100%
  Each 10 % interval is epuivalent to 150 watts of heating power. e.g.: 40 % = 600 W, 50 % = 750 W, 100 % = 1500 W
- **Note:** with micro tubes 100 ml the maximum capacity that can be used is 80 %

Operation with Titrator

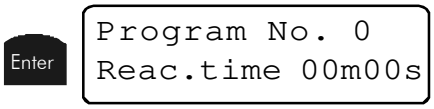
- It is necessary to have a 6 fold dilution of the residual acid after digestion in this case 70 ml = about 7 sec. of H_2O.
- Adjustment range 0-99 s



- Again, It is necessary to have a 6-7* fold dilution of the residual acid after digestion in this case 70 ml = about 8 sec. of NaOH.

* There must always be an excess of NaOH.

- Adjustment range 0-99 s



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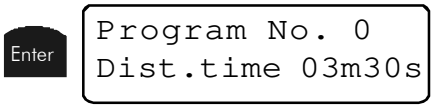

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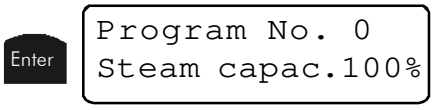
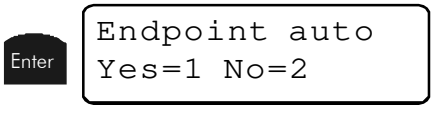
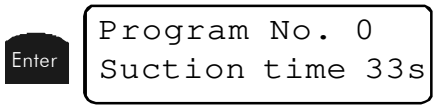


- Adjustment range 40-100%
  Each 10 % interval is epuivalent to 150 watts of heating power. e.g.: 40 % = 600 W, 50 % = 750 W, 100 % = 1500 W
- **Note:** with micro tubes 100 ml the maximum capacity that can be used is 80 %

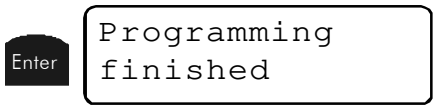


- The pH-value of the boric acid solution can be read automatically at the beginning of an analysis and taken as the endpoint or it can be a set value at the titrator. In order to compensate even the slightest fluctuatins of the pH-value, the automatic endpoint is recommanded.

Operation without titrator

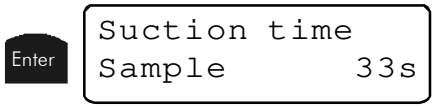


- The suction time for a 10 ml solution is 1 s
- Adjustment range 0-99 s

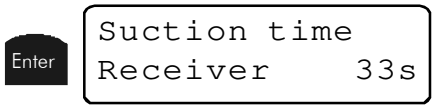


- Press the key „Enter“ to return to the „standby“-mode.
- Now you can start programmed distillations or write more programs.
- The set values remain stored in memory until they are changed again.

Operation with titrator

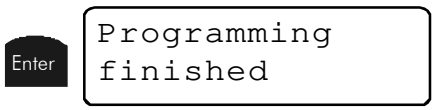


- The suction time for a 10 ml solution is 1 s
- Adjustment range 0-99 s



- The suction time must be long enough so that the receiver vessel is emptied completely. Residual drops do not influence the subsequent analysis.
- Adjustment range 0 - 99 s

- The porous frit of the electrode must be completely immersed into the receiver solution in order to ensure a stable pH measurement.
  Adjustment range 0 - 99s



- Press the key „Enter“ to return to the „standby“-mode.
- Now you can start programmed distillations or write more programs.
- The set values remain stored in memory until they are changed again

7.6. Settings of the Titrator

7.6.1. Schott Titrator Type TL Easy

- If the TL Easy is used with the Vapodest 45 there will only be the consumed volume shown on the display of the Vapdest 45.
- It the option "Endpoint auto" = yes is selected during the programming of the Vapodest 45, then the titration will go back to the pH-value of the boric acid receiver at the beginning of the distillation. the mode of the titrator should be set also on "endpoint titration"
- We recommend the mode EP (endpoint determination) if during the programming of the Vapodest 45 'endpoint auto' = no has been chosen. The pH endpoint can only be set at the titrator. At the end of the distillation, the titration will go to this pH-value.
- A selection of languages in general is possible.
- Titrations to the equivalent end point are not feasable.
- Calculations are not possible.
- During the distillation and titration procedure the titrator cannot be manual operated.

8 Operation

8.1. Safety instructions

Attention when handling acids and alkalies! Please observe your national safety regulations!

Allways wear protective goggles and protective clothing!

Use gloves when removing the glass digestion tube as this will be very hot, and there is danger of burning!

Take care when handling glass parts and follow the national safety regulations concerning the handling of glass parts!

8.2. Priming the system

Please make sure that the system is fully primed. This must be performed before for the initial start, after longer working inter-missions (holidays etc.) and each time after the inlet and outlet tubes have been removed.

1. Check the chemical tanks.
2. Turn on the tap.
3. Insert the inlet tube into an empty digestion tube.
4. Lower the quick clamping device and insert the digestion tube.
5. Ensure there is a tight fit between the tube and the Viton cone.
6. Insert the distillate outlet tube into an empty Erlenmeyer flask, and place the flask into position.
7. Close the protection door, and switch on the distillation system.
8. Keep the key „H_2O“ pressed until the water runs into the digestin tube
9. Keep the key „NaOH“ pressed until the sodium hydroxide solution runs into the digestion tube.
10. Keep the key „H_3BO_3“ pressed until the boric acid runs into the receiver
11. To empty the contents in the digestion tube press the key „Empty“
12. Remove the digestion tube and the Erlenmeyer flask.

The distillation system Vapodest 45 is now primed.

8.3. Test run

Each day before starting to distill samples you should do a distillation without sample.

Don’t be disturbed by noises deriving from the approx. 105 °C hot steam being led into the digestion tube. If you use the C. Gerhardt diges-tion tubes, there is little danger of broken glass.

8.4. Distilling a sample

This program can be interrupted any time by pressing the key „Reset“.

Operation without Titrator

1. Place an Erlenmeyer flask into position
2. Place the steam inlet tube into the sample tube filled with sulphuric acid digestion
3. Place the quick clamping device and place the digestion tube into position
4. Check the tight fit of the tube against the Viton cone.
5. Close the protection door and switch the instrument on.
6. The display shows the standby mode

```
Distillation = 1
Programming  = 2
```

7. Press key „1“ to access the program mode


```
Program No.0
```

8. Enter the desired program number (key 0...9)


```
Program No. 0
Run=Enter
```

9. On running a sample the display shows the distillation sequence of chemical additions, distillation time and suction sample. During the reaction time (if used) and distillation period both the programmed time and countdown time are displayed. With the chemical additions only the countdown time is displayed.
10. Once the program has finished the program remove the digestion tube by lowering the quick clamping device. **Attention**, the digestion tube is extremely hot after the distillation. Make sure to wear protective glasses as well as gloves!
11. Place the steam inlet tube into the holder.
12. Clean the H_3BO_3 distillate outlet tubing with H_2O-distilled, then remove the Erlenmeyer flask with the distillate and continue the determination.

Operation with Titrator

Attention!
For the communication between the Vapodest and the titrator it is important to turn on the titrator first. If this sequence is not observed, then the titrator cannot be initialized.

Calibration
In order to reach a perfect pH measurement the electrode has to be calibrated at regular intervals. A two-point calibration is carried out. The zero point deviation is determined with the buffer solution pH 7, the slope with buffer solution pH 4 or pH 9. The selection of the second buffer solution depends on the pH range to be worked with.

See also original Schott instruction manual for more details about the titrator..

1. Place the steam inlet tube into the sample tube filled with sulphuric acid digestion
2. Place the quick clamping device and place the digestion tube into position
3. Check the tight fit of the tube against the Viton cone.
4. Close the protection door and switch the instrument on.
5. The display shows the standby mode

```
Dest=1  Prog=2
Result=3
```

6. Press key „1“ to access the program mode


```
Program No.0
```

7. Enter desired program (Key 0-9) and confirm with "Enter".
8. The following insert is shown, depending on what option was selected - yes or no during the programming of 'Endpoint auto':


```
Program No. 0
Run=Enter
```

9. The display shows this message if the

Operation without titrator

If you wish to keep this program and distill further samples, please press the key „Enter“. If you wish to change the program, reprogram the system. If you have finished all distillations, switch the unit off.

Operation with titrator

'Endpoint auto' option 'no' was selected.
The program is started by 'enter'. Go to 11.

10. If the option "endpoint auto" has been answered with "yes", then the pH-value of the receiving solution is shown after a short period of measuring.

```
Prog.0 pH 00.000
pH measurement
```

```
Prog.0 pH 4.123
Run=Enter
```

Now, the program can be restarted by pressing "enter".

11. All steps are shown. During the addition of the chemicals and during the suction the remaining amount of time is shown, whereas during the distillation and at a given reaction time the amount of time which has been programmed is shown as well.

12. After the distillation the titration takes place.

```
Titration
EP pH 00.000
```

Display showing option 'endpoint auto'

```
Titration
```

Display without option 'endpoint auto'

13. Sample and receiver are automatically removed after titration. The receiver is filled again with boric acid.
Then, the titration result is shown

```
Prog.0 finished
4.123pH 12.123ml
```

14. After the program is ended press "Enter", then push down the quick clamping device and remove digestion glass.
Attention, the digestion glass is extremely hot after the distillation. Make sure to wear protective gloves and glasses!

15. Insert the inlet tubing into the holding device

16. By pressing key '3', the result can be shown again.

17. Should you wish to continue with the same program setting, please press 'enter'.
Should you wish any changes, please press "Reset".

8.5. Cleaning

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:

Operation without titrator

Addition H_3BO_3	00 s
Addition H_2O	13s
Adition NaOH	00 s
Reaction time	00 m 00 s
Distillation time	07 m 00 s
Steam power	100 %
Suction time sample	20 s

Operation with titrator

Addition H_2O	13 s
Addition NaOH	00 s
Reaction time	00 m 00 s
Distillation time	07 m 00 s
Steam power	100 %
Endpoint auto	Yes
Suction time sample	20 s
Suction time receiver	20 s
Adition H_3BO_3	06 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.

9. Error messages

The micro-processor continually surveys all the functions of the distillation system. As soon as there is an error, it will be shown on the didplay and an acoustic signal is given.

9.1. General error messages

Error message	Action	Key
No cooling water	Cooling water pressure < 0,5 bar. Check cooling water inlet. Make sure tap is open completely.	Enter
No sample tube	Insert sample tube	Enter
Check chemicals	Check set of storage tanks	Enter
Low water Press Enter	Turn off equipment, check tank for H_2O, turn on again. If failure persists consult technical support.	Enter
Filling Steam generator	This message disappears as soon as steam generator is filled.	
After the above mentioned errors are corrected, the following message is displayed. Stop Prog.No. X Comtinue=Enter	Enter = continue of interrupted program Reset = Standby mode	Enter or Reset
Wait for steam	Message disappears as soon as stand-by is reached	
Add sol.>1min Continue=Enter	Check programming Enter = continue of interrupted program Reset=Standby mode	Enter or Reset
Program undefined	Check programming	Reset
Excess steam pressure	Switch the system off and call service	
Sensor error	Switch the system off and call service	

9.2. Error messages Vap 45 with titrator

Error message	Action	Key
Receiver overfilled	Remove receiver manually press ENTER	Enter
Titrator not ready	Check connection of titrator - further steps please see instruction manual TITRATOR	Enter
Titration time > 15 min	Check connections of titrator, check programming!	Enter
Hardware errror	Turn off equipment - call service	Enter

10. Instructions for the upgrade of the Vapodest 45 to be operated with external titrator

In case you have ordered the Vapodest 45 without titrator and now, wish to upgrade your unit to be used with an external titrator, we kindly ask you to follow the instructions enclosed:

1. Remove platform

2. Push the titration cell on the holding device and attach using the enclosed screw (M3 x 20)

3. Prepare plug in connection:
   insert the 5-pin turn-lock fastener plug.

4. Take off protection cap from tubing connection and insert Verprene tube for suction.

5. Insert silicone tubing for boric acid addition as well as the silicone distillate outlet tubing through opening into titration cell (cut the distillate outlet tubing for the right lenght).

6. Connect electrode
   Attention: make sure to read enclosed instruction manual prior to operation of electrode!
   - Carefully unpack the electrode and connect with electrode cable
   - Using utmost care, insert the electrode into the opening of the titration cell completely.
   - The electrode cable has to be inserted from the front of the instrument through the wall duct. Then, plug it into the previewed ope ning of the titrator (also see manual of titrator)

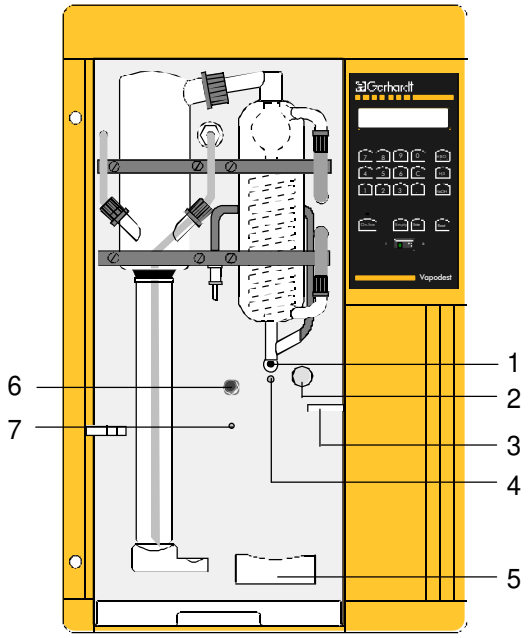
7. Setting up of titrator
   Attention: make sure to read enclosed instruction manual prior to operation of titrator!
   - Unpack titrator and put it next to Vapodest
   - Connect titration tubings:
     Insert the titration tubing from the rear of the instrument through the wall duct into the titration cell. Then, plug it into the previewed opening. The end of the tubing has to be immersed into the receiver vessel.

8. Connect the interface cable to the interface RS 232 of the Vapodest.

9. Connect titrator to the mains.

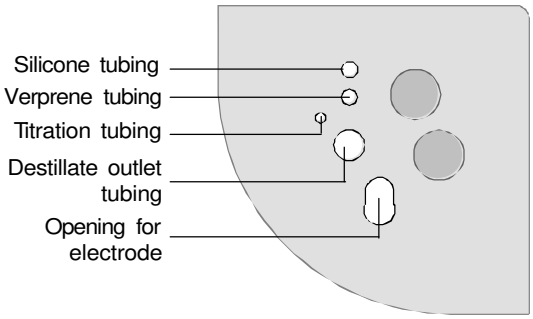
10. Correction of software: To make sure that the Vapodest is aware that an external titrator has been connected the software has to be changed accordingly. Please observe the following steps:

Front view Vapodest



1. Tubing connection for verprene tube (with protection cap)
2. Wall duct for titration tubing and electrode cable
3. Holding device for titration cell
4. Silicon tubing
5. Platform
6. 5-pin connection
7. Thread for screw M3 x 20

Top view titration cell



- Turn the instrument of and then on again.
- Push key "C" four times to get in the titrator menu
  Here, you can select the option of using the Vapodest with the external titrator via the RS 232 interface.

```
Use Titrator
Yes=1   No=2
```

When choosing 2 you get back into the operating mode.

When choosing 1 you get the following message which has to be confirmed by "enter".

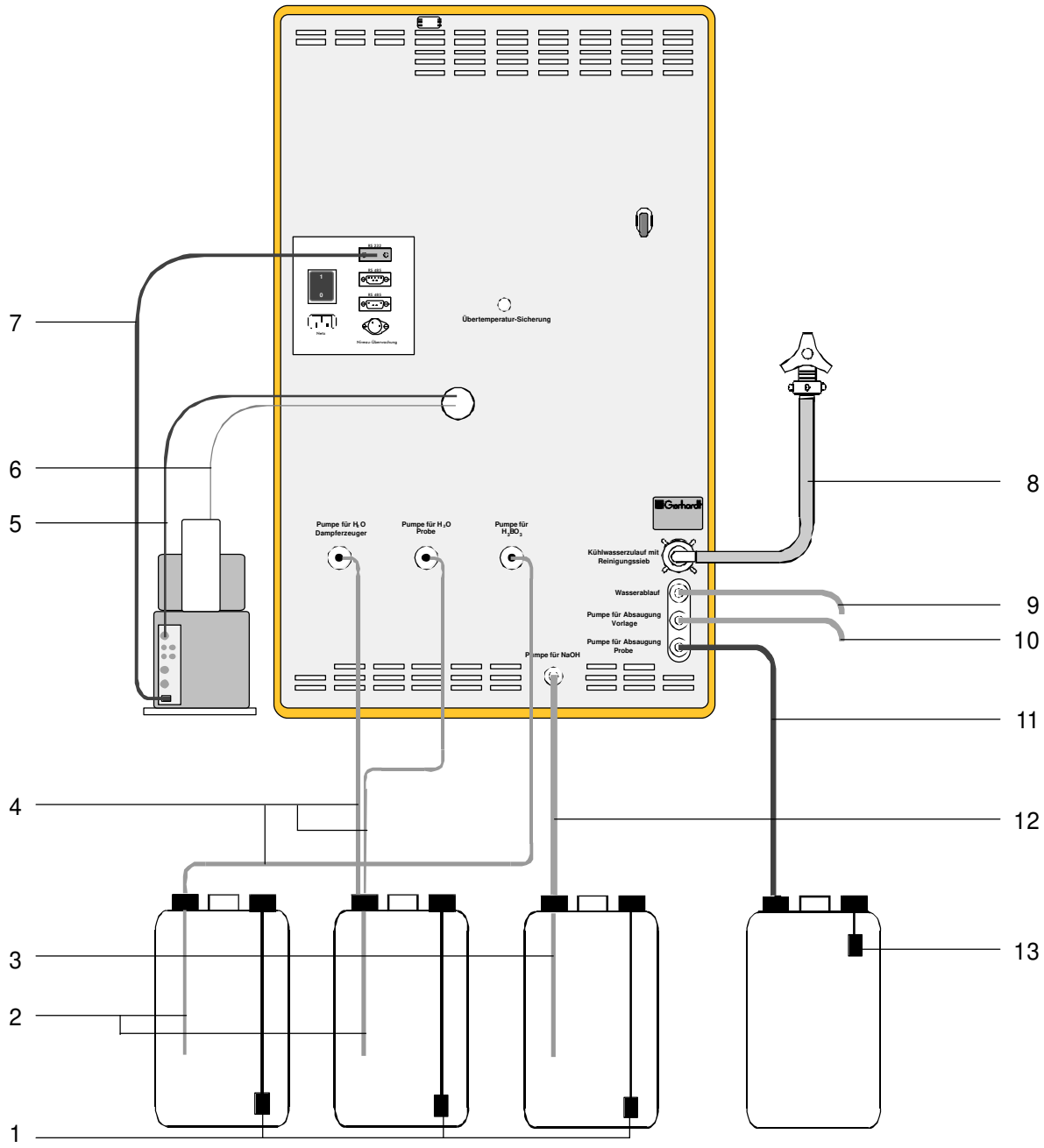
```
Titro Easy =1
Titro alpha=2
```

The Vapodest 45 can work with titrators by Schott, Titroline 96 and alpha.

Be careful! The Titroline 96 needs a special software. In case you are already using this titrator, please the local agent of C. Gerhardt or Schott.

Confirm selection by 'enter' and you will be back in the operating mode.

11. Tubing diagram



11.1. Structural components and parts of tubing diagram

1 ..... Level sensor with plug for H_2O, H_3BO_3 and NaOH-tank, complete
2 ..... PVC-pipe 6 x 1, 420 mm
3 ..... PVC-pipe 10 x 1, 420 mm
4 ..... PVC-tubing 4/7
5 ..... Electrode cable (incl. in delivery of titrator)
6 ..... .PTFE-titration tubing (incl. in delivery of titrator)
7 ..... Interface cable (for TL 96 = TZ 3098, for Alpha TZ 1599)
8 ..... Water inlet tubing 10/17
9 ..... PVC outlet tubing 8 / 12
10 ..... PVC outlet tubing 8 / 12
11 ..... Verprene-tubing 8 / 12
12 ..... PVC-tubing 8 / 12
13 ..... Level sensor with plug for „Sample Waste“ tank, complete

12. Maintenance

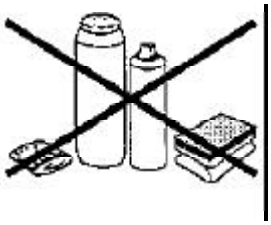
In case of replacements of parts make sure that only original C. Gerhardt products are used!

12.1. Spare parts and accessories

Parts description	Order no.
Distillation condenser	7673
Plastic screw connection for distillation condenser	16609
Ventilation valve	6474
Distribution head, glass	7472
Vitone cone	6470
PTFE-Inlet tubing steam	11810
Teflon sieve for 11810	30698
PTFE-Inlet tubing, NaOH	11811
PP-distributor	50024
PP-tube joint	17542
Holder for pH-Electrode	50034
Level decoder in the receiver	40870
pH-Electrode (Einstabmeßkette)	6602
Propeller	50037
Receiver	10214
Screw cap GL 14	16601
Screw cap GL 18	16602
Screw cap GL 32	16604
Silicone-/PTFE-Seal GL 18	16606
Silicone-/PTFE-Seal GL 32	16607
Silicone seal with connection, straight, for DK/Vap	16609
Plexiglas protection door	19017
door hangle	18251
Door hinges, 1 Pair	18250
Rubber foot GF21, self adhesive	17945
Drip tray, PP	50015
Mains switch green, illuminated	11750
Keyboard	11215
Excess current switch, 10A	15378
Tubular heating element	12354
Steam generator	40850
Excess pressure valve for steam generator	20604
Ventilation valve WMF	20620
Mains cable	1311
Magnetic valve with pressure control	17109
Semiconductor relay WG-A5-6D25	15350
Micro switch for quick clambing device	13308
Pinch-solenoid valve steam and shut-off	10385
Ventilation glass	40272
Diaphragm pump PML 1140-ND100	10310
Peristaltic pump	10360
Water inlet tubing 10/17, 2 m	22802

Parts description	Order No.
Silicone-tubing 6 x 10	20903
Silicone tubing 8 x 12	22704
Silicone tubing 4 x 7	20914
Silicone tubing 16 x 8	22705
Novoprene-tubing 4,8 x 1,6	20919
Verprene-tubing 4 x 8	20920
Verprene-tubing 8 x 12	20921
PTFE-tubing 4 x 6 x 135	21902
Tubing reduction, PP 51 x 10 x 10	50022
Tubing reduction, PP 51 x 10 x 5	50023
PVC-tubing 4/7, 2 m	22601
PVC-tubing 8/12, 2 m	22604
PVC-tube 6 x 1, 420 mm	25450
PVC-tube 10 x 1, 420 mm	25451
Accessories	
Titrator Titro Line Easy, special model with Gerhardt Software	7689
Titrator Titro Line Easy, regular Schott model	7687
Titrator set of acessories, needed for operation with Vap 45 with titrator 7689 respectively	7686
Electrodecable DIN Vap 45	11119/2
Buffer solution pH 4, 250 ml	11125
Buffer solution pH 7. 250 ml	11126
KCL electrolyte solution, 250ml	11127
Set of tanks KAN 40	7649
Level sensor with plug for H_2O, H_3BO_3 and NaOH-tank	11320
Level sensor with plug for „Sample Waste“ tank	11321
Level sensor with plug for titration tank	11323
RS 232/485 Adapter ARS	4260
Data cable DK 42 for RS 485, 2 m	4261
Data cable DK 45 for RS 485, 5 m	4262
Data cable DK 22 for RS 232, 2 m	4264
Terminator AST	4265
Upgrade kit Micro	
Viton cone VK, micro, for 100 ml tubes KMT	7671

12.2. Service and cleaning



Always wear gloves before cleaning any parts of the instrument.

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.

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

Make sure to follow cleaning instructions in chapter 8.5. "Cleaning program".

To minmize malfunctions of the Vapodest 45, we recommend to have it checked by authorized service personnel regularly.

12.3. Trouble shooting

The excess current switch cuts off in case of excessive current consumption. Switch on the excess current switch again. If this problem reoccurs, call service!

Make sure to observe the error messages given in Chapter 13.

In case of breakdown or failure of your distillation unit, **please contact your local dealer or:**

C. Gerhardt GmbH & Co. KG
Bornheimer Straße 100
D-53119 Bonn
Tel.: + 49 (0) 228 / 98179-17 Sales/Office
+ 49 (0) 228 / 98179-16 Service
Fax: + 49 (0) 228 / 98179-60
e-mail: Info@Gerhardt.de
http://www.Gerhardt.de

Subsidaries:

C. Gerhardt UK Ltd.
Unit 5, Avonbury Court, County Road, Brackley,
Northants NN13 7AX
Tel.: 0044-1280-706772
Fax: 0044-1280-706088
e-mail: Info.Gerhardt@pop3.hiway.co.uk.

C. Gerhardt France s.a.r.l.
Email: eric.fourest@gerhardt.fr
9, rue du 11 novembre
78690 Les Essarts le Roi
Tel.: 0033-1-30464100
Fax: 0033-1-30464101

13. Other

13.1. On-line mode

The RS 485 interface allows quality assurance conforming to ISO/GLP. It enables up to 32 different instruments (including other makes) to be connected between each other.

The on-line mode provides the following possibilities:

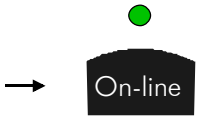
1. Connection of personal computer for controlling and monitoring in conjunction with the GerLab® user program.

2. Connection of PC for controlling and monitoring in conjunction with a customerised terminal program, i.e., Telix (MS-DOS), Terminal (for Windows). With the assistance of the interface a program, based on the customers' needs, can be written, too.

3. Synchronous working of various equipment. The programming and controlling is effected via a system, which has been appointed semi master.

The working instructions for the synchronous mode are as follows:

– Connect all instruments with the interface cables RS 485
– Provide the last unit connected with the terminator AST
– Activate the On-line key of each instrument (control LED illuminated)



– Determine the semi master by pressing the keys „." (keep pressed) and „On-line" (control LED flashes)

By pressing this key-combination once more you can return to the simple On-line position (Control LED illuminated).

– Programme the semi master as described in the previous paragraph.
After having confirmed program step 5 the following is shown on the display:

```
Data transfer
= Enter
```

– Press the key „Enter" to initiate the data transfer from the semi master to the connected instruments

– After the termination of the data transfer the program-ming mode automatically ends, and the display shows the stanby-mode.

Further information about GerBus® and GerLab® can either be obtained from C. Gerhardt GmbH & Co. KG or our appointed dealer.