Metrohm

METROHM AG CH-9101 Herisau (Switzerland)

708 Sampling Unit

Series 01 ...

Instructions for use 8.708.1003

91.12 Ti/mm

Instructions for Use of 708 Sampling Unit

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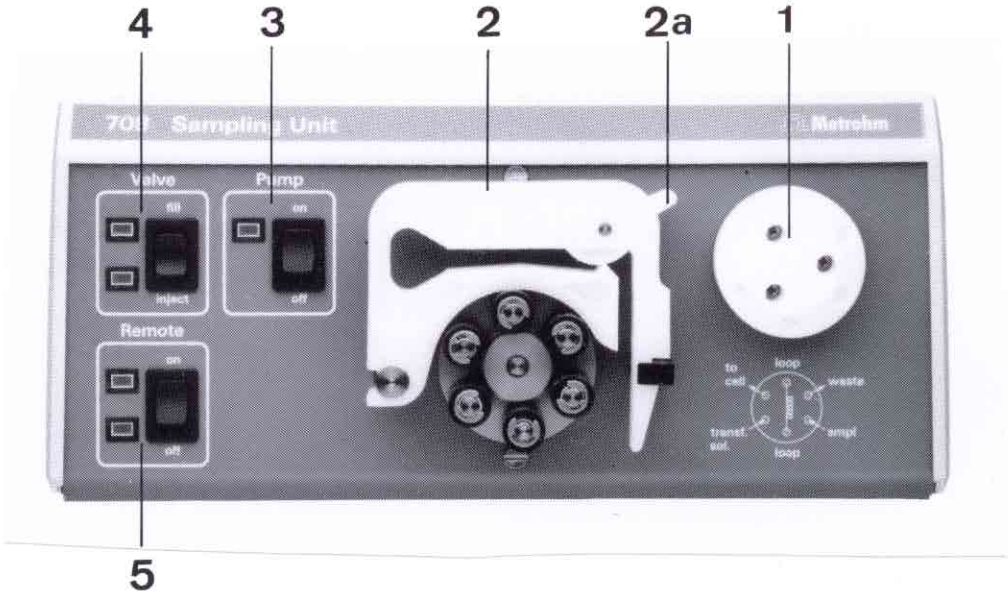
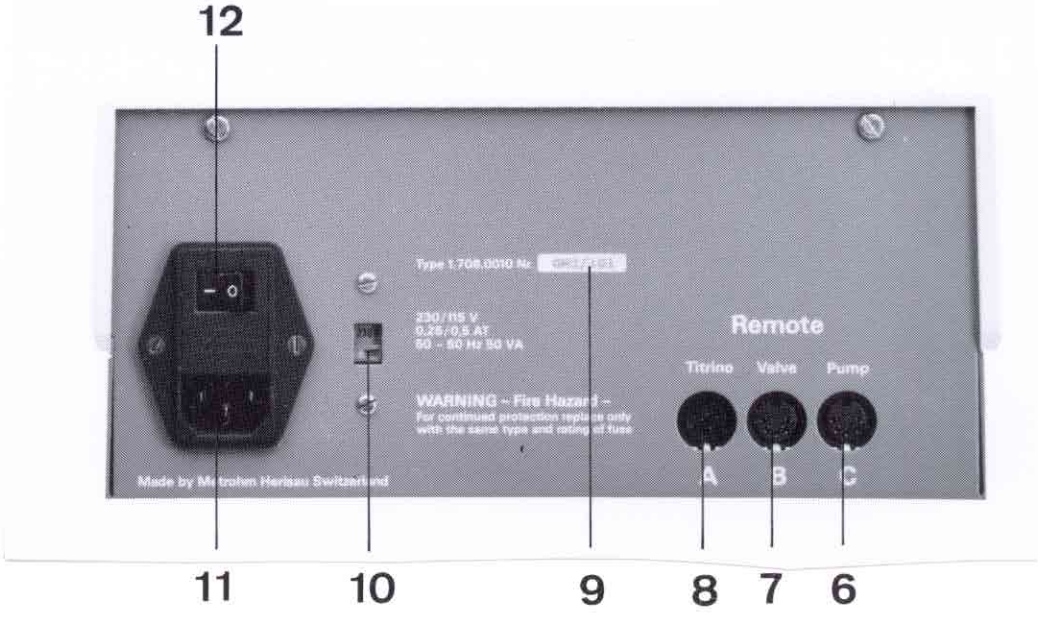

4
3
2
2a
1
Sampling Unit
Metrohm
Valve
fill
inject
Pump
on
off
Remote
on
off
to cell
loop
waste
transf. sol.
ampl
loop
5


12
Type 1.708.0010 Nr.
230 / 115 V
0.25 / 0.5 AT
50 - 60 Hz 50 VA
Remote
Titrino
Valve
Pump
WARNING – Fire Hazard –
For continued protection replace only
with the same type and rating of fuse
Made by Metrohm Herisau Switzerland
A
B
C
11
10
9
8
7
6

1. Overview

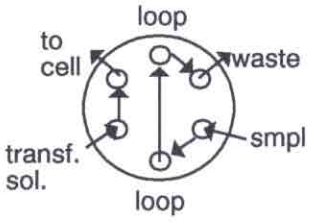
Front

(1) **Valve block**
For the following tubing connections:

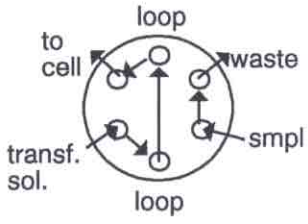
"waste"	Waste tubing.
"smpl"	Sample feed.
"loop"	Sample loop, metering system.
"transf.sol."	Transfer solution. Transfers the sample from the sample loop to the titration vessel.
"to cell"	Tubing leading to titration vessel.

Flow path

"fill" position

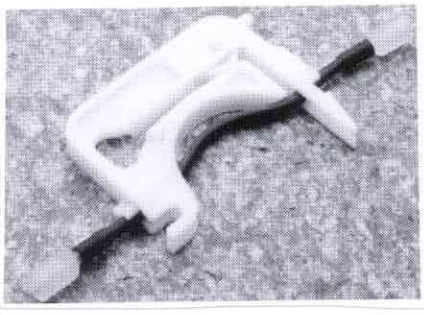


"inject" position



(2) **Tubing cassettes**
in working position. If the pump is switched off for a longer duration, the tubing cassettes should be raised. This measure prolongs lifetime of the pump tubings.
2a) Setting the contact pressure, i.e. control of the flow rate.
Lever up: highest pressure = highest flow rate.
Lever down: lowest contact pressure = lowest flow rate.
Important: An excessive contact pressure shortens the lifetime of the pump tubing. This must be especially noted in KF titrations.

Inserting the pump tubing in the tubing cassette:



(3) **On/off switching of pump**
Manual operation of the pump. Must be "on" when the pump is switched on and off by remote control.

(4) **Switching the valve**
Manual switching of the valve, for the flow path see ①.
Warning: Do not press key if a remote control cable is attached to the socket "Valve"! (The valve switching could be damaged.)

Rear

(5) **On/off remote control of pump**
Must be "on" if the pump is switched on and off by remote control.

(6) **Socket for on/off switching of pump**
Switching via remote control. Switches "Remote" and "Pump" must be "on".

(7) **Socket for valve switching**

(8) **Socket for connection to Titrino**
Ensures that a start is made only when the titration vessel is conditioned.

(9) **Rating plate**
with manufacturing, serial and instrument number.

(10) **Display of the set mains voltage**
Before switching the apparatus on for the first time, ensure that the set mains voltage matches the voltage of your supply system. If not, disconnect mains cable and alter the voltage setting.

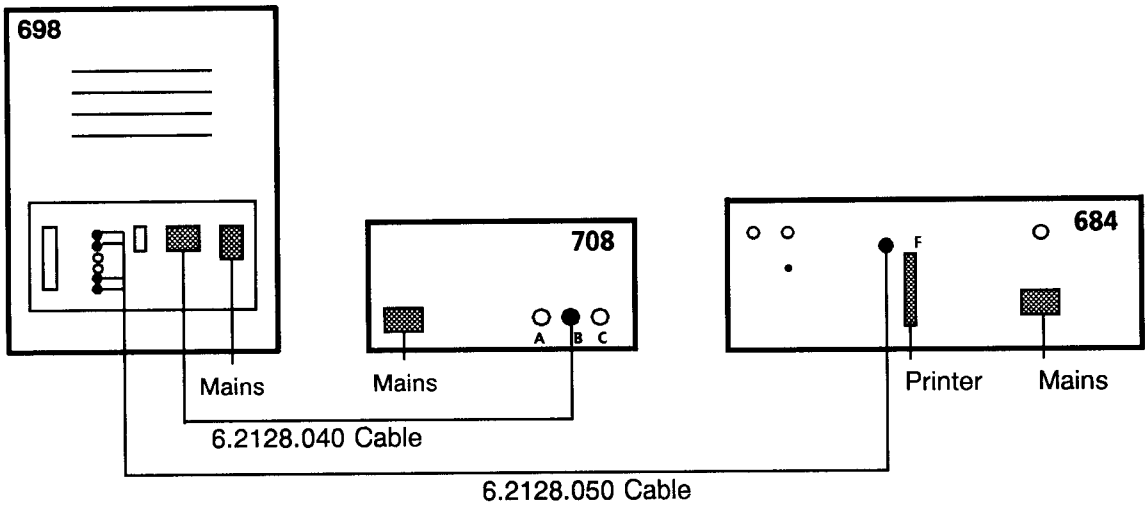
(11) **Mains connection**
With supply systems in which severe HF disturbances are superimposed on the mains voltage, the Sampling Unit should be operated via an additional mains filter, e.g. Metrohm model 615.
When the apparatus is opened or parts removed from it, certain components may be live if it is connected to the supply system. It is thus essential to disconnect the mains cable before altering certain settings or replacing parts.

(12) **Power switch**

2. Working with the 684 Coulometer and 698 Autosampler

2.1 Cable connections

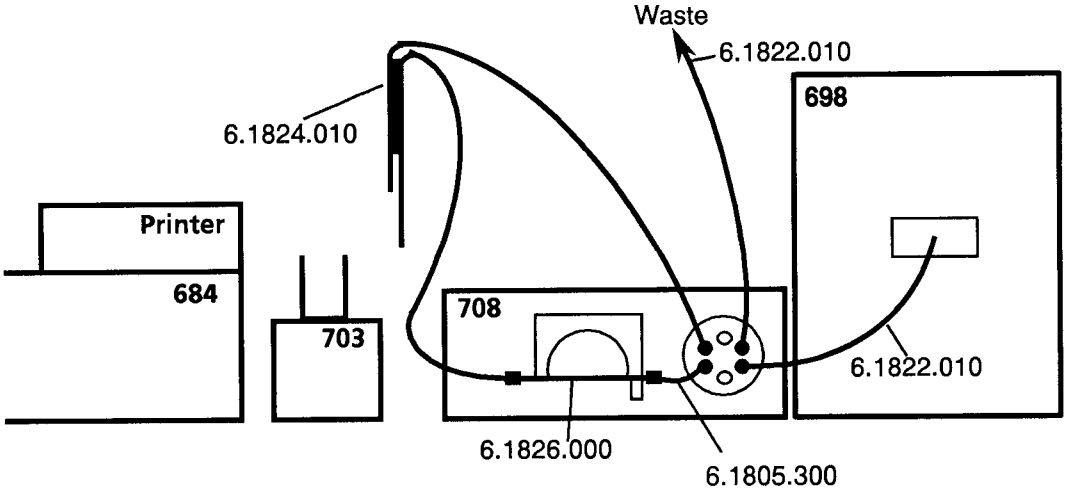
The instruments are interconnected as follows:



Configure the 3.540.2441 RS232 Interface of the 684 KF Coulometer so that calculations are performed without confirmation of the sample weight: DIP switch 8 of interface to ON.

2.2 Tubing connections when 703 Ti Stand is used

The following setup is recommended (see cover picture):



Tubing connections

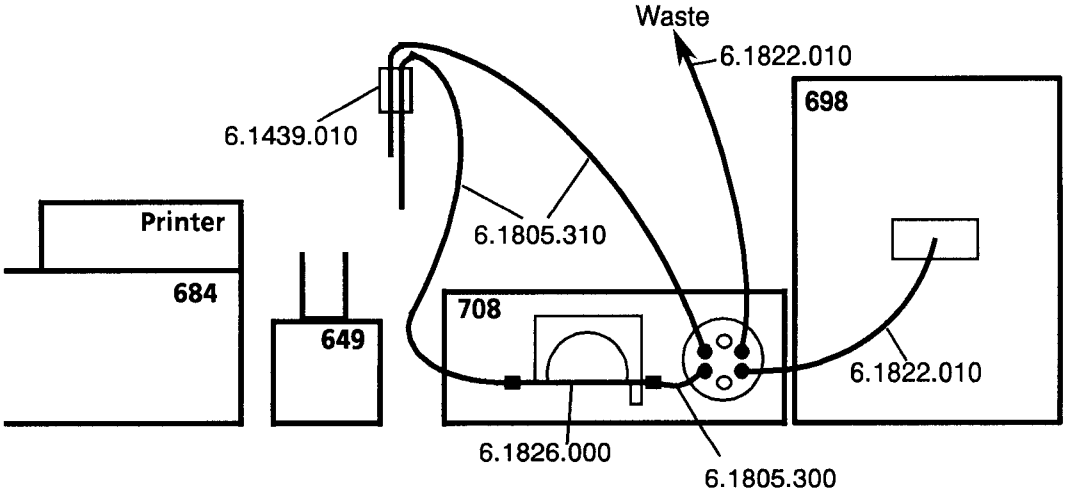
- Connection 698 Autosampler - "smpl" of the valve block: This connection should be as short as possible (loss of sample!). With the recommended setup, a length of ca. 40 cm is sufficient. Shorten 6.1822.010 tubing to ca. 40 cm. Attach the 6.2744.000 Compression Fitting from the accessories of the 698 Autosampler to the free end of the tubing.
  Connect tubing between 698 Autosampler and "smpl".
- Attach 6.1822.010 waste tubing to "waste" of the valve block and route to waste bottle.

- Fit 6.1808.020 Tubing Connectors to both ends of 6.1826.000 pump tubing. Hold the tubing with a cloth to facilitate insertion of the tubing connectors. Keep a pump tubing on hand as a replacement. You should always have spare pump tubing in reserve.
- Remove both tubing cassettes from the holder of the 708 Sampling Unit. You need only one tubing cassette.
  Lay pump tubing in the guide of the cassette so that the stopper is on the left (see page 2).
  Replace cassette in the holder ensuring the pump tubing is free from kinks.
- Connect right end of the pump tubing using 6.1805.300 tubing to "trans.sol" of the valve block.
- Connect left end of pump tubing to the 6.1824.010 Twin Burette Tip.
- Connect twin burette tip to "to cell" of the valve block. Shorten this end of the burette tip somewhat so that the solvent flow can be observed.
  Fix the twin burette tip in the titration vessel upper half with the 6.1446.060 Stopper and 6.2730.030 Screw Fitting.
- Attach the desired 6.1825.XXX Sample Loop to the two "loop" connections of the valve block. For work with the 698 Autosampler, sample loops with a volume ≤ 250 µL can be used.
- Attach 6.2021.020 Electrode Holder to the stand rod of the 703 Ti Stand and use 6.1228.000 Holder for storage of the aspiration tube. During aspiration, the twin burette tip can be stored in the 6.1228.000 Holder.

Function check
Switch the pump on. The flow rate should be regulated by means of the contact pressure (see page 2) to ca. 5 - 7 mL/min ("fast drop rate"). In case of problems, see page 19.

2.3 Tubing connections when 649 Magnetic Stirrer is used

The following setup is recommended:



Tubing connections
- Connection 698 Autosampler – "smpl" of the valve block: This connection should be as short as possible (loss of sample!). With the recommended setup, a length of ca. 40 cm is sufficient.
  Shorten 6.1822.010 tubing to ca. 40 cm. Attach the 6.2744.000 Compression Fitting from the accessories of the 698 Autosampler to the free end of the tubing.
  Connect tubing between 698 Autosampler and "smpl".
- Attach 6.1822.010 waste tubing to "waste" of the valve block and route to waste bottle.
- Fit 6.1808.020 Tubing Connectors to both ends of 6.1826.000 pump tubing. Hold the tubing with a cloth to facilitate insertion of the tubing connectors. Keep a pump tubing on hand as a replacement.
- Remove both tubing cassettes from the holder of the 708 Sampling Unit. You need only one tubing cassette.
  Lay pump tubing in the guide of the cassette so that the stopper is on the left (see page 2).
  Replace cassette in the holder ensuring the pump tubing is free from kinks.

- Connect right end of the pump tubing using 6.1805.300 tubing to "trans.sol" of the valve block.
- Connect left end of pump tubing with the aid of the 6.1805.310 tubing to the longer piece of the 6.1439.010 Dispensing/aspiration Tube of the coulometry cell.
- Take the second 6.1805.310 tubing, pull a length of ca. 11 cm out of the nipple at one end: Cut off brown seal, then cut tubing diagonally to a length of ca. 1 cm. Slide the seal over the tubing with the white part facing the tubing end. Hold tubing with flat-nose pliers and move seal ca. 11 cm up the tubing. Finally, cut tubing off level using a razor blade.
  Connect this end to the shorter part of the dispensing/aspiration tube. The tubing runs through the tube. Attach other end of 6.1805.310 tubing to "to cell" of the valve block.
- Attach the desired 6.1825.XXX Sample Loop to the two "loop" connections of the valve block. For work with the 698 Autosampler, sample loops with a volume ≤ 250 µL can be used.

Function check
Switch the pump on. The flow rate should be regulated by means of the contact pressure (see page 2) to ca. 5-7 mL/min ("fast drop rate"). In case of problems, see page 19.

2.4 Operation

1. Prepare 684 KF Coulometer: Add reagent to coulometry cell, condition cell (press <GO>). Switch on send and enter sample weight (<sample>) in mg, see also page 7.

2. Switch on the pump of the 708 Sampling Unit. Check flow rate ("fast drop rate"). If need be, move pump tubing a little to the right. In case of problems, see page 19.

3. Fill the sample vials of the 698 Autosampler with ca. 0.9 mL sample and press in the cover fully with the opening downwards. Load sample turntable of 698 Autosampler with vials. The sample vials should be dried for 1 h at 105°C and stored in a desiccator.
   Warning: The samples must not contain any solids! (danger of blockage)

4. "Program" 698 Autosampler:

<STOP>	Press stop key
VIAL	Display
XX <ENTER>	Enter first sample vial
THRU	Display
XX <ENTER>	Enter last sample vial

5. Starting 698 Autosampler:

<START>	Press start key
INIT V	Display
XX <ENTER>	Enter position of first sample vial
RINSE	Rinse cycle between samples (0 = no)
0 <ENTER>	Enter 0
LAST V	Position of the last sample vial
XX <ENTER>	This starts the first determination

   A start is made as soon as the drift measured by the 684 KF Coulometer is less than 100 µg/min. If you wish to start at lower drift values, you will have to wait until these are reached. A good drift value when the 708 Sampling Unit is running is ca. 12 µg/min.

Switching off
If the 708 Sampling Unit is switched off, lift up tubing cassette. This measure prolongs the life of the pump tubing!
If the 708 Sampling Unit is not used for some time, the wet system should be rinsed with methanol: Before switching off, siphon in methanol "as a sample" during ca. 3 min.

2.5 Calibration of the sample loop

The exact volume of the sample loop is determined with, e.g. a solution of water in methanol (standard methanol).

- Using a microlitre syringe or a Dosimat, inject a known volume V_1 of your solution into the coulometry cell. Perform, e.g. 5 determinations and calculate the mean value of the titrated water → t_1.
- Now add the same solution to the sample vials of the 698 Autosampler and start so that the solution is injected via the sample loop of the 708 Sampling Unit.
  Again perform 5 determinations and determine the mean value of the titrated water → t_2. Same conditions and same titration parameters as above!
- Calculate the volume of the sample loop V_{loop} using the following formula:

$$V_{loop} = \frac{V_1 * t_2}{t_1}$$

- Write the volume on the rough surface of the small plate of the sample loop.
- Conversion of the volume into the sample weight:
  Weigh an exactly known volume V_0 of your sample → m_0. The weight is calculated using

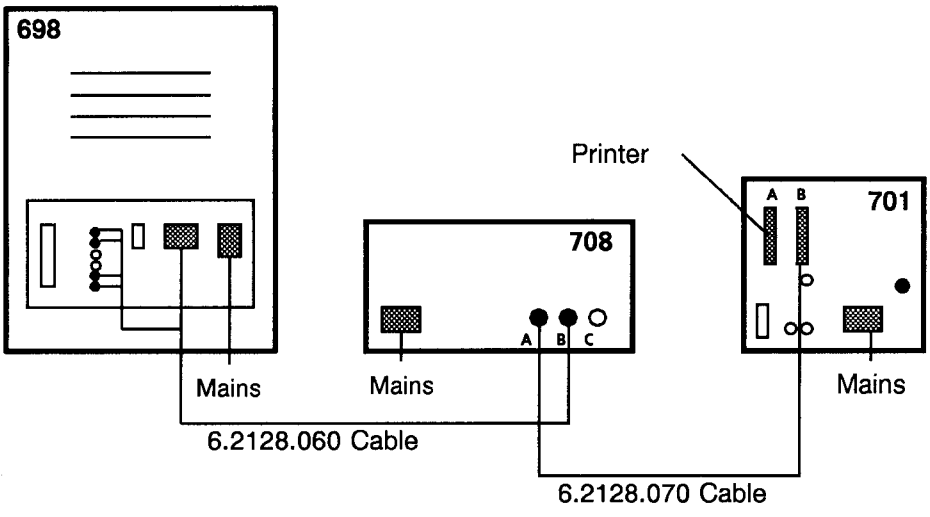
$$m_{sample} = \frac{m_0 * V_{loop}}{V_0}$$

3. Working with the 701 Titrino and 698 Autosampler

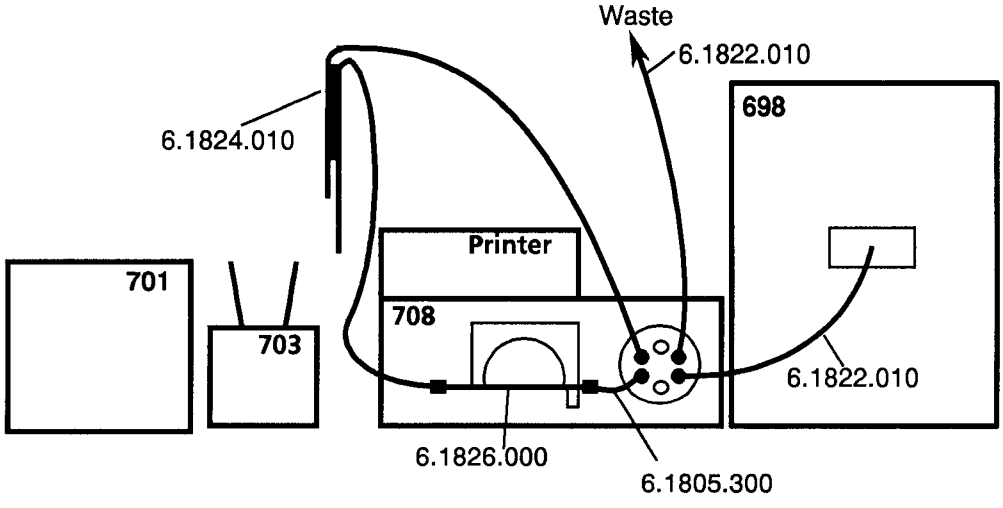
It is also possible to use the 702 SM Titrino in place of the 701 KF Titrino for work in the SET mode with conditioning.

3.1 Cable connections

The instruments are interconnected as follows:



3.2 Tubing connections



Tubing connections

- Connection 698 Autosampler - "smpl" of the valve block: This connection should be as short as possible (loss of sample!). With the recommended setup, a length of ca. 40 cm is sufficient. Shorten 6.1822.010 tubing to ca. 40 cm. Attach the 6.2744.000 Compression Fitting from the accessories of the 698 Autosampler to the free end of the tubing.
  Connect tubing between 698 Autosampler and "smpl".
- Attach 6.1822.010 waste tubing to "waste" of the valve block and route to waste bottle.
- Fit 6.1808.020 Tubing Connectors to both ends of 6.1826.000 pump tubing. Hold the tubing with a cloth to facilitate insertion of the tubing connectors. Keep a pump tubing on hand as a replacement. You should always have spare pump tubing in reserve.

- Remove both tubing cassettes from the holder of the 708 Sampling Unit. You need only one cassette.
  Lay pump tubing in the guide of the cassette so that the stopper is on the left (see page 2).
  Replace cassette in the holder ensuring the pump tubing is free from kinks.
- Connect right end of the pump tubing using 6.1805.300 tubing to "trans.sol" of the valve block.
- Connect left end of pump tubing to the 6.1824.010 Twin Burette Tip.
- Connect twin burette tip to "to cell" of the valve block. Shorten this end of the burette tip somewhat so that the solvent flow can be observed.
  Fix the twin burette tip in the titration vessel upper half with the 6.2730.050 Stopper.
- Attach the desired 6.1825.XXX Sample Loop to the two "loop" connections of the valve block.
  For work with the 698 Autosampler, sample loops with a volume ≤ 250 µL can be used.

Function check
Switch the pump on. The flow rate should be regulated by means of the contact pressure (see page 2) to ca. 5 - 7 mL/min ("fast drop rate"). In case of problems, see page 19.

3.3 Operation

1. Prepare 701 KF Titrino: Add solvent and condition titration cell (press <START>). Select the desired report and enter sample size.
   After the start, the titration method of the 701 KF Titrino should not contain any inquiries for identification or sample size.
2. Switch on the pump of the 708 Sampling Unit. Check flow rate ("fast drop rate"). If need be, move pump tubing a little to the right. In case of problems, see page 19.
3. Fill the sample vials of the 698 Autosampler with ca. 0.9 mL sample and press in the cover fully with the opening downwards. Load sample turntable of 698 Autosampler with glass vials. The sample vials should be dried for 1 h at 105°C and stored in a desiccator.
   Warning: The vials must not contain any solids! (danger of blockage)
4. "Program" 698 Autosampler:

<STOP>	Press stop key
VIAL	Display
XX <ENTER>	Enter first sample vial
THRU	Display
XX <ENTER>	Enter last sample vial

5. Starting 698 Autosampler:

<START>	Press start key
INIT V	Display
XX <ENTER>	Enter position of first sample vial
RINSE	Rinse cycle between samples (0 = no)
0 <ENTER>	Enter 0
LAST V	Position of the last sample vial
XX <ENTER>	This starts the first determination

   A start is made as soon as the 701 KF Titrino is conditioned.

Switching off
If the 708 Sampling Unit is switched off, lift up tubing cassette. This measure prolongs the life of the pump tubing!
If the 708 Sampling Unit is not used for some time, the wet system should be rinsed with methanol: Before switching off, siphon in methanol "as a sample" during ca. 3 min.

3.4 Calibration of the sample loop

The exact volume of the sample loop is determined with, e.g. a solution of water in methanol (standard methanol).

- Using a microlitre syringe or a Dosimat, inject a known volume V_1 of your solution into the titration cell. Perform, e.g. 5 determinations and calculate the mean value of the titrant consumption → t_1.
- Now add the same solution to the sample vials of the 698 Autosampler and start so that the solution is now injected via the sample loop of the 708 Sampling Unit. Again perform 5 determinations and determine the mean value of the titrant consumption → t_2. Same conditions and same titration parameters as above!
- Calculate the volume of the sample loop V_{loop} using the following formula:

$$V_{loop} = \frac{V_1 * t_2}{t_1}$$

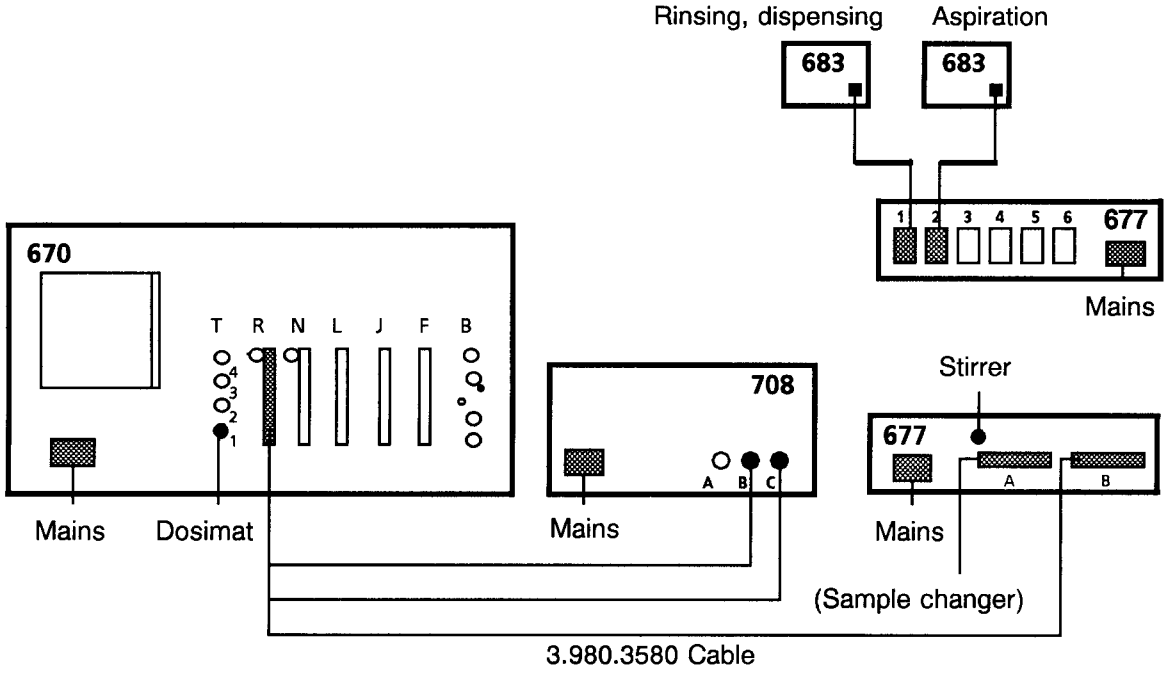
- Write the volume on the rough surface of the small plate of the sample loop.
- Conversion of the volume into the sample weight:
  Weigh an exactly known volume V_0 of your sample → m_0. The weight is calculated using

$$m_{sample} = \frac{m_0 * V_{loop}}{V_0}$$

4. Working with the 670 Titroprocessor and 677 Drive Unit

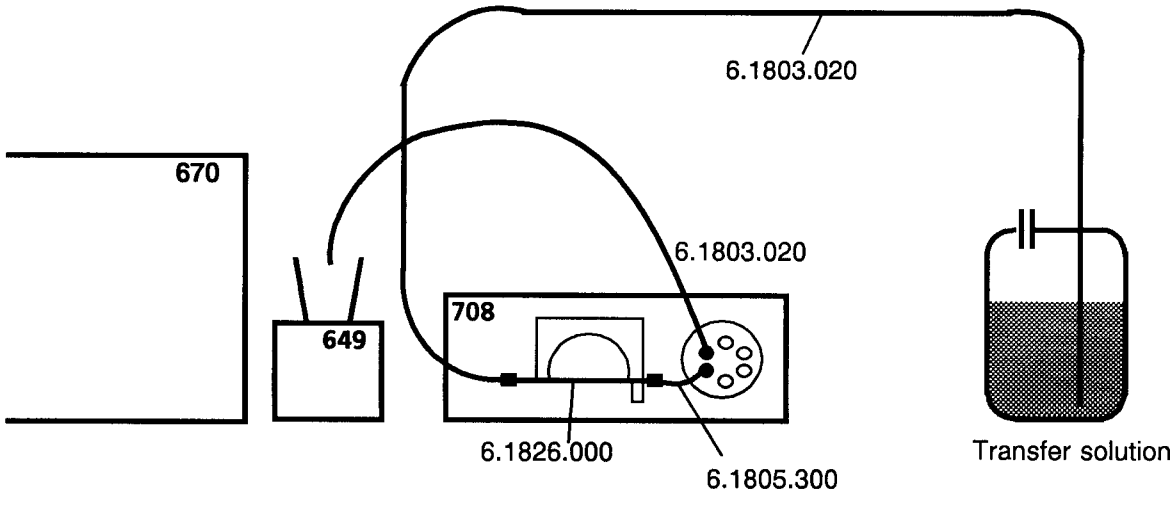
4.1 Cable connections

The instruments are interconnected as follows:



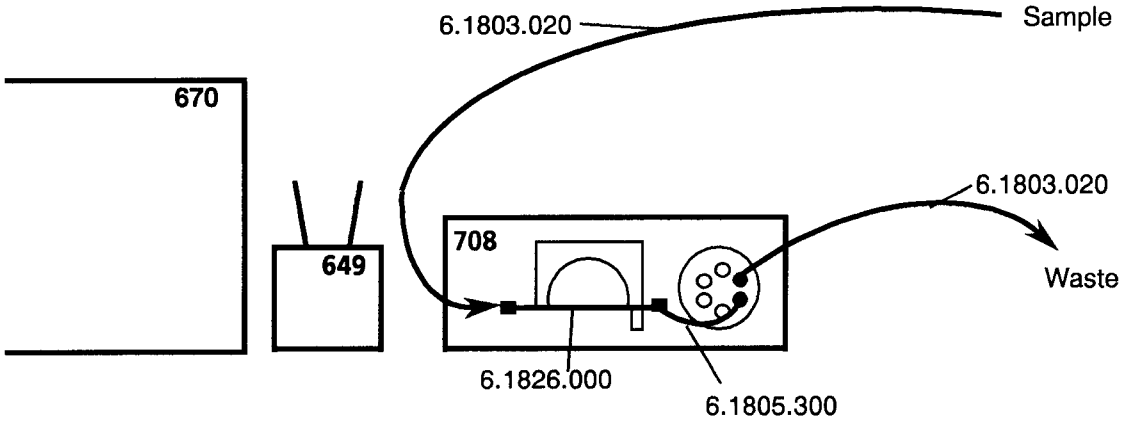
4.2 Tubing connections

- Fit 6.1808.020 Tubing Connectors to both 6.1826.000 pump tubings. Hold the tubing with a cloth to facilitate insertion of the tubing connectors. Keep a set of pump tubings on hand as a replacement.
- Remove both tubing cassettes from the holder of the 708 Sampling Unit.
- Setting up 1st pump circuit:



. Lay pump tubing in the guide of a cassette so that the stopper is on the left (see page 2). Replace cassette in the holder ensuring the pump tubing is free from kinks.
. Connect right end of the pump tubing using 6.1805.300 tubing to "trans.sol" of the valve block.
. The left end of the pump tubing leads to the transfer solution.
Cut 6.1803.020 tubing using a sharp cutting tool (e.g. razor blade) to the correct length. Attach 6.1811.020 Nipple to other end of tubing: Slide nipple with thread facing end of tubing over the tubing. Cut tubing diagonally to a length of ca. 1 cm. Slide seal with the white part facing the end of the tubing over the tubing. Hold tubing with flat-nose pliers and move seal. Finally, cut off tubing level with seal using a razor blade.
Connect nipple to left end of pump tubing and route to transfer solution.
. Fit 6.1811.020 Nipples to both ends of a ca. 60 cm length of 6.1803.020 tubing. Make connection between "to cell" of valve block and burette tip in the titration vessel and position burette tip in the titration vessel using 6.1446.030 Stopper.

- Setting up 2nd pump circuit:



. Insert second pump tubing in the tubing cassette and place the cassette in the holder of the 708 Sampling Unit.
. Use 6.1805.300 tubing to connect the right end of the pump tubing to "smpl" of the valve block.
. The left end of the pump tubing leads to the sample.
Attach a 6.1811.020 Nipple to another piece of 6.1803.020 tubing. Connect this to the left end of the pump tubing and route to sample.
If you supply the samples using a sample changer, also fit a 6.1811.020 Nipple to the other end of the 6.1803.020 tubing. Attach a 6.1543.060 Burette Tip to this and insert it in the sample head of the sample changer.
. Use another piece of 6.1803.020 tubing for the waste tubing and fit a 6.1811.020 Nipple to one end. Connect this tubing to "waste" of the valve block and lead it off to the waste bottle.

- Attach the desired 6.1825.XXX Sample Loop to both "loop" connections of the valve block.

Function check
Switch on all instruments.
Set the two switches "Remote" and "Pump" on the 708 Sampling Unit to "on".
Set the "Remote" switch on the two 683 Pump Units to "off".
Switch the 670 Titroprocessor to "manual control" (MC). Test the control lines:

1: Solvent addition
2: Aspiration
4: Switch on pump of 708 Sampling Unit
5: Switch valve of 708 Sampling Unit to "inject"
6: Switch valve of 708 Sampling Unit to "fill"
Activate stirrer.

The flow rate should be regulated by means of the contact pressure (see page 2) to ca. 5 - 7 mL/min (ca. 2 drops per second). In case of problems, see page 19.

4.3 Example of PAGE 3 of the 670 Titroprocessor

The following method includes commands for control of the 708 Sampling Unit and a sample changer. The latter are marked with "SC". If the sample is supplied without a sample changer, simply omit these commands.

```
 1  OMOVE1;                  SC                    18  MEAS 1;
 2  CTR 12;       5.0 s      Aspirate              18a
 3  OCTR12;       100 ms                           18b
 4  WPOS 1;                  SC                    18c
 5  CTR 16;       100 ms     708 "Fill"            19  DYNT 1;                    Titration
 6  OCTR16;       100 ms                           19a
 7  CTR 14;       100 ms     708 Pump on           19b
 8  NOP   ;       XX s 1)    Fill sample loop      19b
 9  CTR 11;       YY s 2)    Add solvent           20  TSTOP ;
10  OCTR11;       100 ms                           20a
11  CTR 15;       100 ms     708 "inject"          20b
12  OCTR15;       100 ms                           20c
13  STIR 1;                  Stirrer on            21  OSTIR1;                    Stirrer off
14  RPOS 1;                  SC                    22  CTR 12;          5.0 s     Aspirate
15  SHIFT1;                  SC                    23  OCTR12;CTR 11;   3.0 s     Rinse
16  NOP   ;       XX s 1)    Rinse sample loop     24  OCTR11;CTR 12;   5.0 s     Aspirate
17  OCTR14;       100 ms     708 Pump off          25  OCTR12;CTR 11;   3.0 s     Rinse



26  OCTR11;CTR 12;  5.0 s    Aspirate
27  OCTR12;CTR 11;  3.0 s    Rinse
28  OCTR11;
29  END   ;
```

Remarks

1): The waiting times depend on the size of the sample loop, the flow rate and the viscosity of the solutions. With a flow rate of ca. 5 – 7 mL/min and dilute aqueous solutions, the following waiting times result:

Sample loop	Waiting time
6.1825.060 5 ml	120 s
6.1825.050 1 ml	35 s
6.1825.040 0.5 ml	20 s

2): When the sample loop is being filled, transfer solution (solvent) is conveyed to the titration vessel. The waiting time for the addition of more solvent depends on this volume and the size of the titration vessel.

4.4 Operation

Switch the two switches "Pump" and "Remote" on the 708 Sampling Unit to "on".
Adjust the flow rate by means of the contact pressure (see page 2) to 5 – 7 mL/min (ca. 2 drops per second). In case of problems, see page 19.
Start method on 670 Titroprocessor.

Warning: The samples must not contain any solids! (danger of blockage)

Switching off
If the 708 Sampling Unit is switched off, lift up tubing cassette. This measure prolongs the life of the pump tubing!
If the 708 Sampling Unit is not used for some time, the wet system should be rinsed with solvent: Before switching off, siphon in solvent "as a sample" for ca. 3 min.

4.5 Calibration of the sample loop

The exact volume of the sample loop is determined with a titratable solution (e.g. NaOH).

- Pipette a known volume V_1 of your solution into the titration vessel. Perform, e.g. 5 determinations and calculate the mean value of the titrant consumption → t_1.
- Now add the same solution via the sample loop of the 708 Sampling Unit. Again perform 5 determinations and determine the mean value of the titrant consumption → t_2. Same conditions and same titration parameters as above!
- Calculate the volume of the sample loop V_{loop} using the following formula:

$$V_{loop} = \frac{V_1 * t_2}{t_1}$$

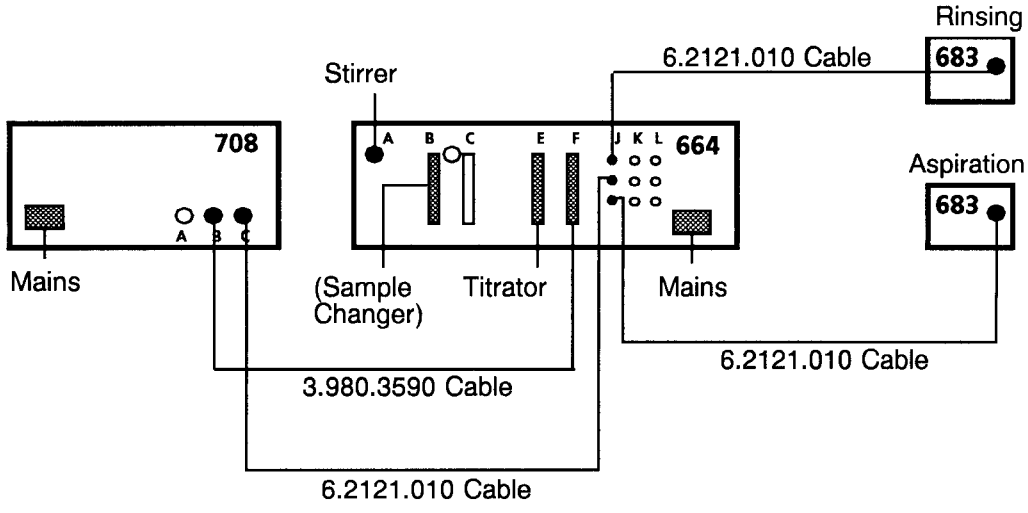
- Write the volume on the rough surface of the small plate of the sample loop.
- Conversion of the volume into the sample weight:
  Weigh an exactly known volume V_0 of your sample → m_0. The weight is calculated using

$$m_{sample} = \frac{m_0 * V_{loop}}{V_0}$$

5. Working with a titrator and the 664 Control Unit

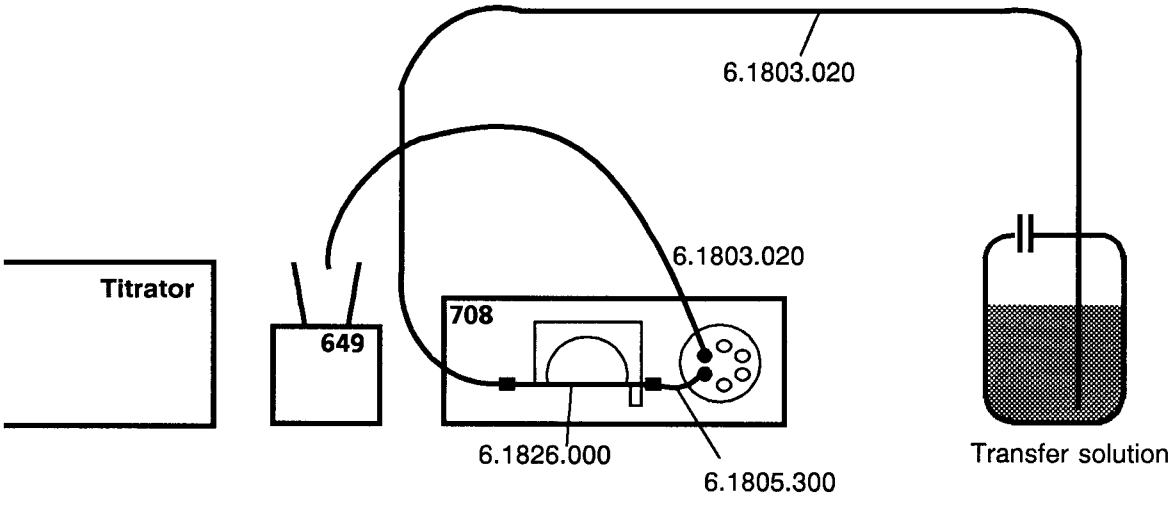
5.1 Cable connections

The instruments are interconnected as follows:



5.2 Tubing connections

- Fit 6.1808.020 Tubing Connectors to both 6.1826.000 pump tubings. Hold the tubing with a cloth to facilitate insertion of the tubing connectors. Keep a set of pump tubings on hand as a replacement.
- Remove both tubing cassettes from the holder of the 708 Sampling Unit.
- Setting up 1st pump circuit:

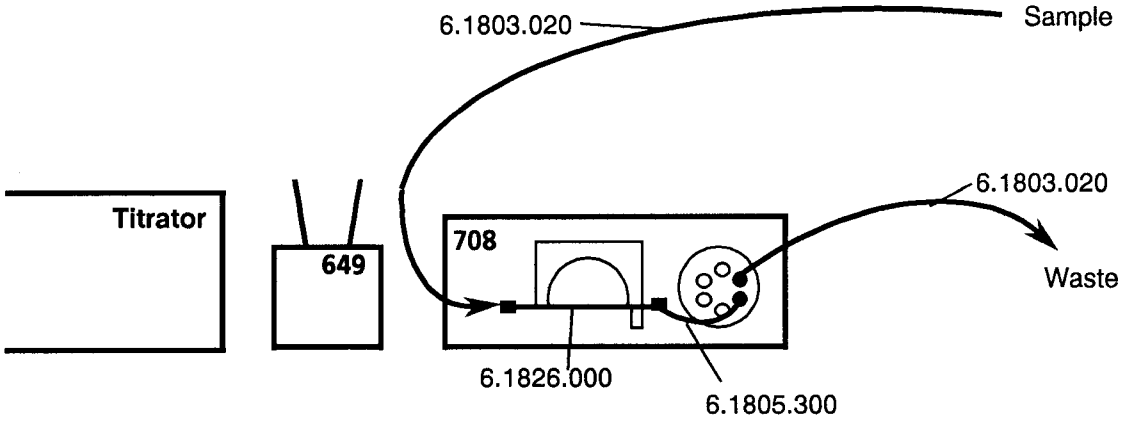


. Lay a pump tubing in the guide of a cassette so that the stopper is on the left (see page 2). Replace cassette in the holder ensuring the pump tubing is free from kinks.

. Connect right end of the pump tubing using 6.1805.300 tubing to "trans.sol" of the valve block.

. The left end of the pump tubing leads to the transfer solution.
Cut 6.1803.020 tubing using a sharp cutting tool (e.g. razor blade) to the correct length.
Attach 6.1811.020 Nipple to one end of tubing: Slide nipple with thread facing end of tubing over the tubing. Cut tubing diagonally to a length of ca. 1 cm. Slide seal with the white part facing the end of the tubing over the tubing. Hold tubing with flat-nose pliers and move seal. Finally, cut off tubing level with seal using a razor blade.
Connect nipple to left end of pump tubing and route to transfer solution.
. Fit 6.1811.020 Nipples to both ends of a ca. 60 cm length of 6.1803.020 tubing. Make connection between "to cell" of valve block and burette tip in the titration vessel and position burette tip in the titration vessel using 6.1446.030 Stopper.

- Setting up 2nd pump circuit:



. Insert second pump tubing in the tubing cassette and place the cassette in the holder of the 708 Sampling Unit.
. Use 6.1805.300 tubing to connect the right end of the pump tubing to "smpl" of the valve block.
. The left end of the pump tubing leads to the sample.
Attach a 6.1811.020 Nipple to another piece of 6.1803.020 tubing. Connect this to the left end of the pump tubing and route to sample.
If you supply samples using a sample changer, also fit a 6.1811.020 Nipple to the other end of the 6.1803.020 tubing. Attach a 6.1543.060 Burette Tip to this and insert it in the sample head of the sample changer.
. Use another piece of 6.1803.020 tubing fitted at one end with a 6.1811.020 Nipple for the waste tubing. Connect this tubing to "waste" of the valve block and lead off to the waste bottle.

- Attach the desired 6.1825.XXX Sample Loop to the two "loop" connections of the valve block.

Function check

Set the "Remote" switch on the two 683 Pump Units to "on".
Switch on all instruments.
Set the two switches "Remote" and "Pump" on the 708 Sampling Unit to "on".
Manually activate the different switches on the 664 Control Unit:
V1: 683 Rinsing Pump is activated.
V2: Pump of 708 Sampling Unit is switched on.
V3: 683 Aspiration Pump is activated.
Switch on stirrer.

Set the "Remote" switch on the 708 Sampling Unit to "off" and regulate the flow rate by means of the contact pressure (see page 2) to ca. 5 – 7 mL/min (ca. 2 drops per second). Then switch "remote" on again. In case of problems, see page 19.

5.3 Program of the 664 Control Unit and setting the waiting and valve opening times

The 6.9960.002 special EPROM of the 664 Control Unit contains 2 modes for control of the 708 Sampling Unit:

Mode 4: Control of the 708 Sampling Unit, a rinsing pump and an aspiration pump. If a sample changer is attached, no sample beaker should not be lifted to the working station (glass beaker detector must not respond).

Mode 5: As mode 4, but including control of the sample changer

Program sequence	1) Aspirating with pump via V1 ←←←←←←←←←← 2) Start 708 pump (valve V2 opens) ↑ 3) 708 to "Fill" position ↑ 4) Waiting time (Δt1) [1] ↑ 5) Addition of rinsing solution during waiting time (Δt3) [2] ↑ 6) 708 to "Inject" position ↑ 7) Stirrer on ↑ 8) Waiting time (Δt4) [1] for 9) Stop 708 pump (valve V2 closes) ↑ 10) Titration N-1 11) Stirrer off ↑ 12) Aspirating titrated sample with pump via valve V1 samples 13) Rinsing with pump (V3) ↑ 14) Aspirating with pump via V1 ↑ 15) Rinsing with pump (V3) ↑ 16) Aspirating with pump via V1 ↑ 17) Rinsing with pump (V3) →→→→→→→→→→ once N samples have been processed, the red "stop" lamp lights up.
Settings on the 664 Control Unit	V1: 0...15 s Aspiration with pump V2: 0...15 s 708 pump on as long as V2 is open V3: 0...15 s Rinsing with pump Δt1: 0...5 min Waiting time for filling of sample loop "Fill" Δt3: 0...15 s Addition of auxiliary solution (with the rinsing pump) Δt4: 0...5 min Waiting time for transfer of the sample "Inject"

Remarks

1): The waiting times Δt1 and Δt4 depend on the size of the sample loop, the flow rate and the viscosity of the solutions. With a flow rate of ca. 5 - 7 mL/min and dilute aqueous solutions, the following waiting times and settings of Δt1 and Δt4 result (set time = 1/20 of the effective waiting time):

Sample loop	Waiting time	Set time
6.1825.060 5 ml	120 s	**6 s**
6.1825.050 1 ml	40 s	**2 s**
6.1825.040 0.5 ml	20 s	**1 s**

2): When the sample loop is being filled, transfer solution (solvent) is added to the titration vessel. The waiting time Δt3 for the addition of more solvent depends on this volume and the size of the titration vessel.

5.4 Operation

Switch the two switches "Pump" and "Remote" on the 708 Sampling Unit to "on".
Adjust the flow rate by means of the contact pressure (see page 2) to 5 - 7 mL/min (ca. 2 drops per second). In case of problems, see page 19.
Start method on 664 Control Unit.

Warning: The samples must not contain any solids! (danger of blockage)

Switching off
If the 708 Sampling Unit is switched off, lift up tubing cassette. This measure prolongs the life of the pump tubing!
If the 708 Sampling Unit is not used for some time, the wet system should be rinsed with solvent: Before switching off, siphon in solvent "as a sample" for ca. 3 min.

5.5 Calibration of the sample loop

The exact volume of the sample loop is determined with a titratable solution (e.g. NaOH).

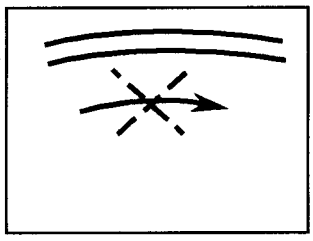
- Pipette a known volume V_1 of your solution into the titration vessel. Perform, e.g. 5 determinations and calculate the mean value of the titrant consumption → t_1.
- Now add the same solution via the sample loop of the 708 Sampling Unit. Again perform 5 determinations and determine the mean value of the titrant consumption → t_2. Same conditions and same titration parameters as above!
- Calculate the volume of the sample loop V_{loop} using the following formula:

$$V_{loop} = \frac{V_1 * t_2}{t_1}$$

- Write the volume on the rough surface of the small plate of the sample loop.
- Conversion of the volume into the sample weight:
  Weigh an exactly known volume V_0 of your sample → m_0. The weight is calculated using

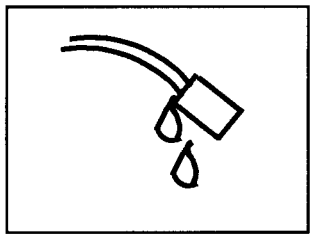
$$m_{sample} = \frac{m_0 * V_{loop}}{V_0}$$

6. Troubleshooting, rectification of malfunctions



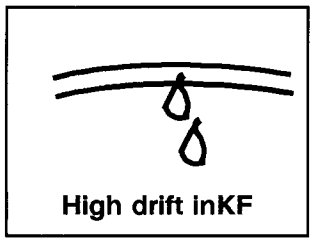
Insufficient or non-existent flow rate

- Increase contact pressure of pump tubing, see page 2.
- Check all connections for tightness.
- Use new pump tubing.
- Undo all connections of the pump circuit. First undo the first connection in the flow direction after siphoning on of the liquid. If liquid still flows here, check the next connection, etc.
  If liquid transport stops at the valve block:
  . Valve block obstructed: Unscrew valve block. Clean with solvent, compressed air and a needle, then screw it on again (tighten evenly and without much pressure).
  If liquid transport stops "at intervals":
  Tubing ends squeezed: Cut off seal.
  . Cut tubing diagonally to a length of ca. 1 cm. Slide seal with white part facing end of tubing over tubing. Cut off tubing level with seal using a razor blade.



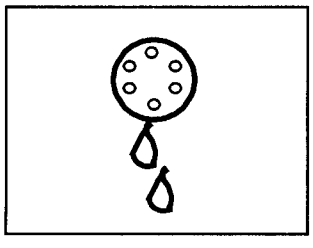
Leaking of tubing nipples

- Cut off seal. Cut tubing diagonally to a length of ca. 1 cm. Slide seal with white part facing end of the tubing over the tubing. Cut tubing off level with seal using a razor blade.
- Use new 6.1811.020 Nipple.



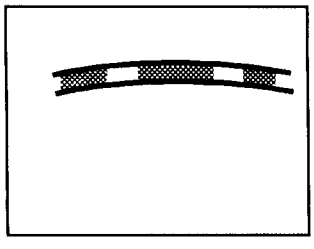
Tubing leaking or high drift in KF titrations

- Use new tubing or new 6.1826.000 pump tubing.



Leaks at valve block

- Tighten 3 Phillips screws of valve block more.
  Tighten screws evenly.



Air bubbles in pump circuit

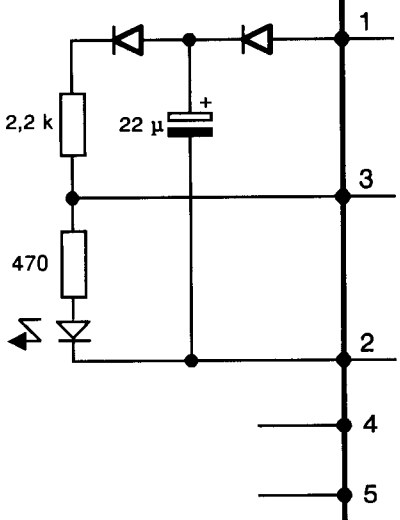
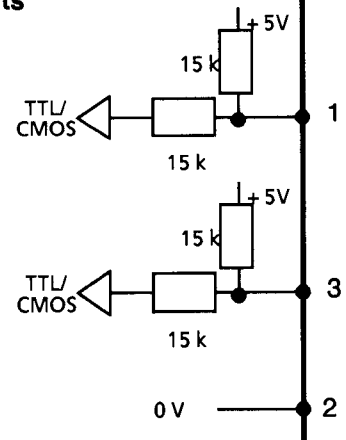
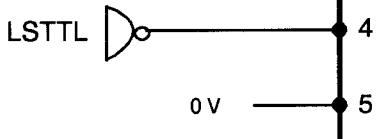
- Immerse tubing which siphons the liquid on completely.
- Check connections for tightness.
- When connected to 698 Autosampler and on use of a "light" solvent (e.g. methanol), use 6.1822.010 waste tubing.

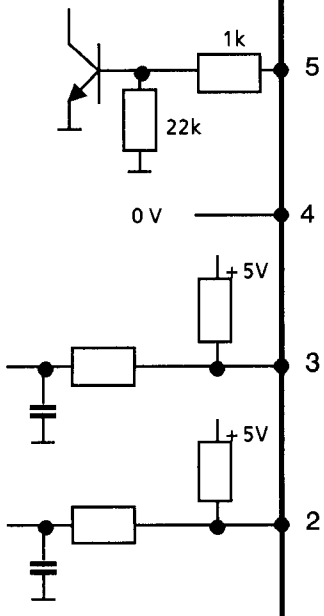
7. Appendix

7.1 Technical specifications

Pump type	2-channel peristaltic pump
Pump capacity	
with 6.1826.000 pump tubing, internal diameter 2.54 mm	5 ...7 ml/min (depending on contact pressure)
Pressure	max. 4 bar
Sample requirements	
Nature	Clear liquids
Sample loops, volume	50 µl...5000 µl
Viscosity (with standard tubing)	max. ca. 200 cp
Materials	
Tubing connections	PTFE
Pump tubing	FPM (Viton)
Valve block	PTFE, PCTFE
Housing	Light-metal injection moulding, multicoat stove enamel
Remote control	
via control lines	Pump: on/off, Valve: fill/inject
Safety requirements	Designed and tested in accordance to IEC publication 348, safety class I. For indoor use only. These instructions for use contain information and warnings which must be observed by the user to ensure safe operation and to retain the apparatus in safe condition.
Ambient temperature	
Nominal operating range	5 ... 40 °C
Storage transport	– 20 ... 70 °C
Mains connection	
Mains voltage	100...125 V and 200...250 V, adjustable
Mains frequency	50...60 Hz
Greatest power consumption	ca. 50 VA (when valve switched)
Fusing	Slow-blow fusible cutout (1 A), 5 x 20 mm
Dimensions	
Width	250 mm
Height	127 mm
Depth	ca. 350 mm
Weight	ca. 5.7 kg

7.2 Pin assignment

	external	Function
"Pump" socket 		ON : 24 V AC OFF: 0 V AC ON: + 5 V DC OFF: 0 V DC not used
"Valve" socket **Inputs** 		Inject = low (active). Additional condition for "Inject", see socket "Titrino", page 22. Fill = low (active)
Outputs 		External start: Pulses at a frequency of 100... 200 Hz, as long as line 2 of the "Titrino" socket is active (if the socket is activated at all) and sets the 708 to "Inject".

	external	Function
"Titrino" socket **Inputs**  **Outputs** 	5 4 3 2 1	Socket is activated (+ 5 V) Gives external start (on "Valve" socket, pins 4 and 5). If this line is "low" (active) and Inject (on "Valve" socket, pin 1) is "low" too, then the 708 is set to "Inject" and a start pulse is outputted (start pulse of ca. 150 ms duration).

No liability whatsoever will be accepted for damage caused by improper interconnection of instruments.

7.3 Warranty

The warranty regarding our products is limited to rectification free of charge in our workshops of defects that can be proved to be due to material, design or manufacturing faults which appear within 12 months from the day of delivery. Transport costs are chargeable to the orderer.
For day and night operation, the warranty is valid for 6 months.

Glass breakage in the case of electrodes or other glass parts is not covered by the warranty. Checks which are not a result of material or manufacturing faults are also charged during the warranty period. For parts of outside manufacture insofar as these constitute an appreciable part of our instrument, the warranty stipulations of the manufacturer in question apply.

With regard to the guarantee of accuracy, the technical specifications in the Instructions for Use are authoritative.

Concerning defects in material, construction or design as well as the absence of guaranteed features, the orderer has no rights or claims except those mentioned above.

If damage of the packaging is evident on receipt of a consignment or if the goods show signs of transport damage after unpacking, the carrier must be informed immediately and a written damage report demanded. Lack of an official damage report releases METROHM from any liability to pay compensation.

If any instruments and parts have to be returned, the original packaging should be used if at all possible. This applies above all to instruments, electrodes, burette cylinders and PTFE pistons. Before embedment in wood shavings or similar material, the parts must be packed in a dustproof package (for instruments, use of a plastic bag is imperative). If open assemblies are enclosed in the scope of delivery that are sensitive to electromagnetic voltages (e.g. data interfaces etc.) these must be returned in the associated original protective packaging (e.g. conductive protective bag). (Exception: assemblies with built-in voltage source belong in a non-conductive protective packaging). For damage which arises as a result of non-compliance with these instructions, no warranty responsibility whatsoever will be accepted by METROHM.

7.4 Scope of delivery and ordering designations

708 Sampling Unit	**2.708.0010**
including the following accessories:	
1 PTFE tubing with M6 thread, length 15 cm	6.1805.300
4 Tubing connectors	6.1808.020
1 Nipple with M6 thread	6.1811.020
2 Capillary tubings with M6 thread, length 1 m	6.1822.010
1 Sample loop, volume ca. 100 µl	6.1825.020
1 Sample loop, volume ca. 250 µl	6.1825.030
2 Pump tubings, FPM (Viton)	6.1826.000
1 Mains cable with cable socket, type CEE(22),V	
Cable plug to customer's specifications:	
type SEV 12 (Switzerland...)	6.2122.020
type CEE(7),VII (Germany...)	6.2122.040
type NEMA/ASA (USA...)	6.2122.070
1 Instructions for Use for 708 Sampling Unit	8.708.1003

For instrument combinations ready for use the following additional instruments and accessories are needed:

684 KF Coulometer, 698 Autosampler and 703 Ti Stand

1 KF Coulometer	2.684.0140
with printer and corresponding cable	
1 Autosampler	2.698.0010
1 Ti Stand	2.703.0010
(Thread adaptor for Riedel de-Haën and Fluka reagents	6.1618.000)
1 Cable 698 Autosampler – 708 Sampling Unit	6.2128.040
1 Cable 698 Autosampler – 684 KF-Coulometer	6.2128.050
1 Twin burette tip	6.1824.010
1 Stopper for twin burette tip	6.1446.060
1 Stopper with nipple and O ring	6.2730.030
1 Electrode holder	6.2021.020
1 Holding tube	6.1228.000

684 KF Coulometer, 698 Autosampler und 649 Magnetic Swing-out Stirrer

1 KF Coulometer	2.684.0140
with printer and corresponding cable	
1 Autosampler	2.698.0010
1 Magnetic Swing-out Stirrer	2.649.0040
1 Cable 698 Autosampler – 708 Sampling Unit	6.2128.040
1 Cable 698 Autosampler – 684 KF Coulometer	6.2128.050
1 Connector for glass tube with M6 thread	6.1811.000
2 PTFE tubings with M6 thread, length 40 cm	6.1805.310

701 KF Titrino and 698 Autosampler

1 Autosampler	2.698.0010
1 KF Titrino	2.701.0010
with printer and corresponding cable and exchange unit	
1 Ti Stand	2.703.0010
1 Cable 698 Autosampler – 708 Sampling Unit	6.2128.060
1 Cable 701 KF Titrino – 708 Sampling Unit	6.2128.070
1 Twin burette tip	6.1824.010
1 Stopper for twin burette tip	6.2730.050

Instead of the 703 Ti Stand, the 649 Magentic Swing-out Stirrer can be used (ordering number 2.649.0040).

670 Titroprocessor, 677 Drive Unit and 2 x 683 Pump Unit

Instruments, exchange unit, electrode	
1 Cable 670 Titroprocessor – 677 Drive Unit and 708 Sampling Unit	3.980.3580
1 Titration vessel	6.1415.220
1 Titration vessel lid	6.1414.010
1 Rotor nozzle	6.2740.000
1 Guide sleeve for rotor nozzle	6.1823.000
1 SGJ sleeve for guide sleeve	6.1236.040
1 Burette tip (sample transport into the titration vessel)	6.1543.060
1 Aspiration tip	6.1543.120
2 Movable ball stoppers	6.1446.030
1 Aspiration tube	6.1805.200
1 Screw connector for aspiration tube on 683 Pump Unit	6.1820.030
evtl. 1 sample loop, see "General accessories", page 26	6.1825.XXX
1 PTFE tubing for various connections, length 5 m	6.1803.020
4 Nipples with M6 thread	6.1811.020
1 PTFE tubing with M6 thread, length 15 cm	6.1805.300

If the samples are transported with a sample changer, the following additional accessories are needed:

1 Burette tip (for siphoning on the sample)	6.1543.060
1 Nipple with M6 thread	6.1811.020
evtl. PTFE tubing for various connections, length 5 m	6.1803.020

Titrator, 664 Control Unit and 2 x 683 Pump Unit

Instruments, exchange unit, electrode and cable Titrator – 664 Control Unit	
Special EPROM for 664 Control Unit	6.9960.002
1 Cable 664 Control Unit – 708 Sampling Unit (Valve)	3.980.3590
3 Cables 664 Control Unit – Pumps (708 and 683)	6.2121.010
1 Titration vessel	6.1415.220
1 Titration vessel lid	6.1414.010
1 Rotor nozzle	6.2740.000
1 Guide sleeve for rotor nozzle	6.1823.000
1 SGJ sleeve for guide sleeve	6.1236.040
1 Burette tip (sample transport into the titration vessel)	6.1543.060
1 Aspiration tip	6.1543.120
2 Movable ball stoppers	6.1446.030
1 Aspiration tube	6.1805.200
1 Screw connector for aspiration tubing on 683 Pump Unit	6.1820.030
evtl. 1 sample loop, see "General accessories", page 26	6.1825.XXX
1 PTFE tubing for various connections, length 5 m	6.1803.020
4 Nipples with M6 thread	6.1811.020
1 PTFE tubing with M6 thread, length 15 cm	6.1805.300

If the samples are transported with a sample changer, the following additional accessories are needed:

1 Burette tip (for siphoning on the sample)	6.1543.060
1 Nipple with M6 thread	6.1811.020
evtl. PTFE tubing for various connections, length 5 m	6.1803.020

General accessories for 708 Sampling Unit

Sample loop, volume ca. 50 µl	6.1825.010
Sample loop, volume ca. 100 µl	6.1825.020
Sample loop, volume ca. 250 µl	6.1825.030
Sample loop, volume ca. 500 µl	6.1825.040
Sample loop, volume ca. 1 ml	6.1825.050
Sample loop, volume ca. 5 ml	6.1825.060
Pump tubing	6.1826.000
Tubing connectors for pump tubing	6.1808.020
PTFE tubing for various connections, 5 m	6.1803.020
Nipple with M6 thread for 6.1803.020 Tubing	6.1811.020
PTFE tubing with M6 thread, length 15 cm	6.1805.300
PTFE tubing with M6 thread, length 40 cm	6.1805.310
Capillary tubing with M6 thread, length 1 m	6.1822.010
Twin burette tip	6.1824.010

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