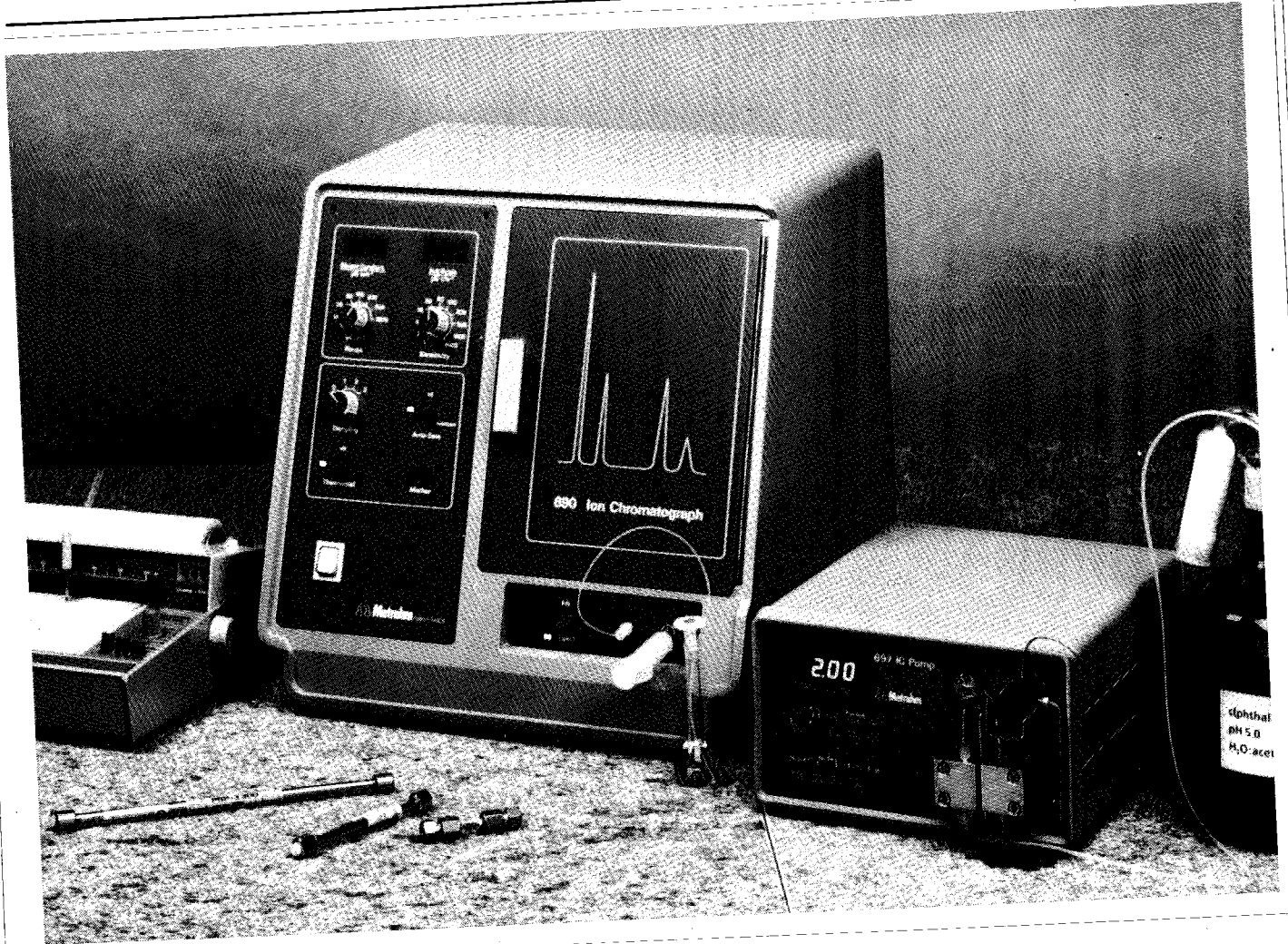


Metrohm Instructions for use

690 Ion Chromatograph Series 07...



 **Metrohm**
Measurement in Chemistry

METROHM Ltd.
CH-9101 Herisau
Switzerland
Phone 071/53 11 33
Telefax 071/52 11 14
Telex 88 27 12 metr ch

8.690.1033



Ionenchromatograph 690

Serie 7 ...

Netzanschluss:

Netzspannung	$U = 100, 117, 220, 240 \text{ V} \pm 10\%$
Netzfrequenz	$f = 50 \dots 60 \text{ Hz}$
Leistungsaufnahme	$S = 50 \text{ VA}$

Gebrauchsanweisung 8.690.1031

8.690.1033 Instructions for Use

690 Ion Chromatograph

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690 Ion Chromatograph

Instructions for use

1. Introduction

The **690 Ion Chromatograph** is a measuring instrument for the chromatographic analysis of ionic components. As *Fig. 1* shows, it comprises an injector, an insulated column compartment, a thermostatted conductivity detector and the electronics necessary for detection of the measuring signal and control of the heating. The following **three versions** are available:

- 2.690.0010 Ion Chromatograph with 1 manually operated injector
- 2.690.0020 Ion Chromatograph with 1 electrically operated injector
- 2.690.0030 Ion Chromatograph with 2 electrically operated injectors and manually operated valve for column selection

The 690 Ion Chromatograph is the main component of an **ion chromatography system** that can be expanded by the user personally to suit his own requirements. In the minimum configuration (see *Fig. 1*), this system consists of the central 690 instrument together with a column, the 697 IC Pump and a data recording device (recorder and/or integrator). The 690 Ion Chromatograph is, however, designed in a manner such that practically all HPLC peripheral units and accessories such as precolumns, additional separation columns, additional detectors, other injection systems, autosamplers and laboratory data systems available on the market can be integrated in the system without any problem.

The present **Instructions for Use** are essentially confined to the description of the 690 Ion Chromatograph. The explanations have been deliberately designed to provide even inexperienced HPLC users with an introduction to the mode of operation of the instrument and the special features of this analytical method.

As a supplement to these Instructions for Use, the **8.690.2013 Application File** containing detailed examples from diverse application fields is also supplied with every 690 Ion Chromatograph. In addition, *Section 1* of the file contains a short introduction to the fundamental theoretical principles of ion chromatography, principally for the benefit of newcomers to this field.

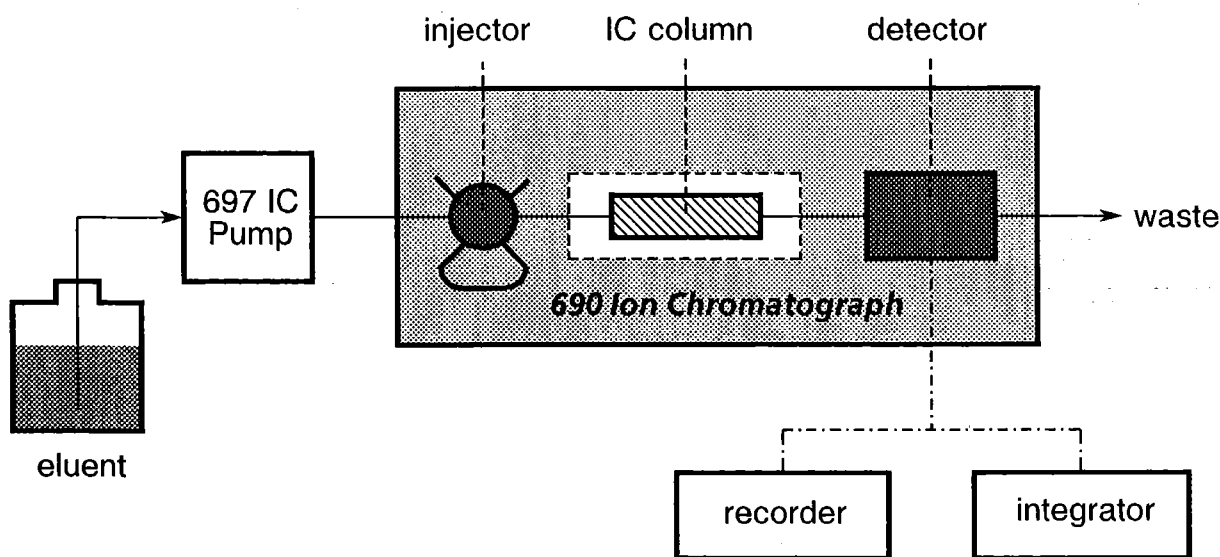


Fig. 1: Block diagram of the ion chromatography system

2. Control Elements

2.1. 690.0010 Ion Chromatograph (with manually operated injector)

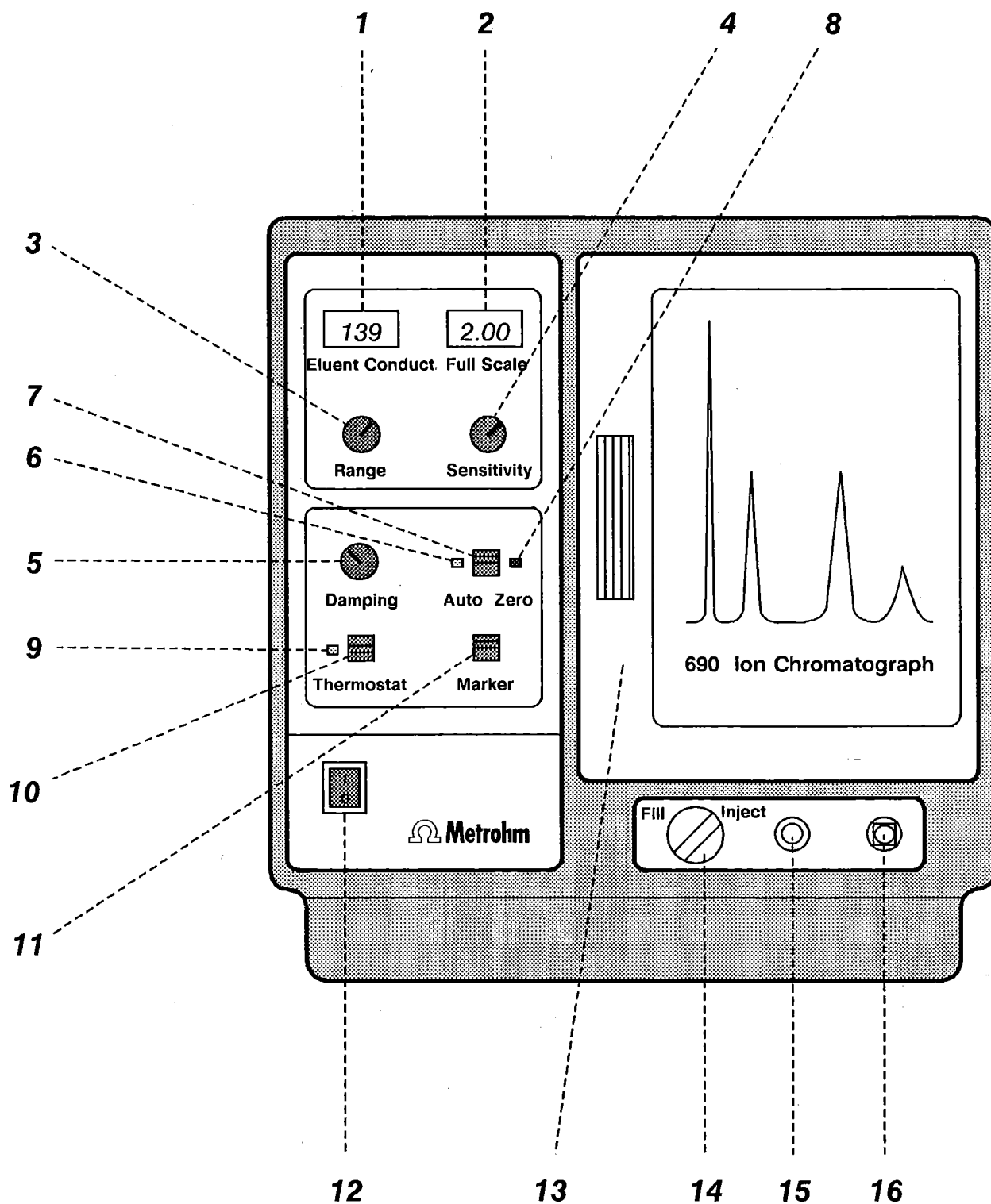


Fig. 2: Front of the 690.0010 Ion Chromatograph

- 1 Eluent Conduct.**
Display of the eluent conductivity in $\mu\text{S}/\text{cm}$
Range: 0...1000 $\mu\text{S}/\text{cm}$
If the measured conductivity falls outside the measuring range selected, " / " (digit 1 at the extreme left with no following digits) appears in the display (\rightarrow reset range)
- 2 Full Scale**
Display of the full scale operating range in $\mu\text{S}/\text{cm}$
The operating range is defined by the settings of **Range** and **Sensitivity**:
Full Scale = Range/Sensitivity
Range: 0.1...1000 $\mu\text{S}/\text{cm}$
If conductivity falls below the lower limit of this range, " / " (digit 1 at the extreme left with no following digits) appears in the display (\rightarrow reset range and/or sensitivity)
- 3 Range**
Setting of the measuring range in $\mu\text{S}/\text{cm}$
Range: 10...1000 $\mu\text{S}/\text{cm}$
- 4 Sensitivity**
Setting of the sensitivity
Range: 1...2000
- 5 Damping**
Damping (Butterworth filter, 4th order) of the measuring signal
stage 0: $\tau = 0.125 \text{ s}$
stage 1: $\tau = 0.5 \text{ s}$
stage 2: $\tau = 1 \text{ s}$
stage 3: $\tau = 2 \text{ s}$
- 6 Auto Zero display**
When the electronic background compensation is switched on, the green lamp lights up
- 7 Auto Zero**
Automatic zeroing of the measuring signal (electronic background compensation)
Triggering the zeroing (repeatable):
press switch downwards briefly (green lamp lights up)
Switching off:
push switch upwards briefly (green lamp goes out)
- 8 Overload display**
Illumination of the red lamp shows that the measuring signal lies outside $\pm 150\%$ of the full scale range selected
- 9 Thermostat display**
The green lamp lights up when the heating is switched on
- 10 Thermostat**
On/off switch for the heating of the conductivity detector
- 11 Marker**
Toggle switch for marking (ca. 10% of the full scale range)
Triggering: press switch downwards briefly
- 12 ON/OFF**
Switching the instrument on and off (mains switch)
I: On 0: Off
When in operational readiness, the red lamp in the switch lights up
- 13 Door for wet system/interior**
- 14 Injection valve switch**
Position "Fill":
filling of the sample loop
Position "Inject":
injection of the loop contents
- 15 Connection for 6.1822.000 extractor tubing**
- 16 Connection for 6.2816.020 syringe**

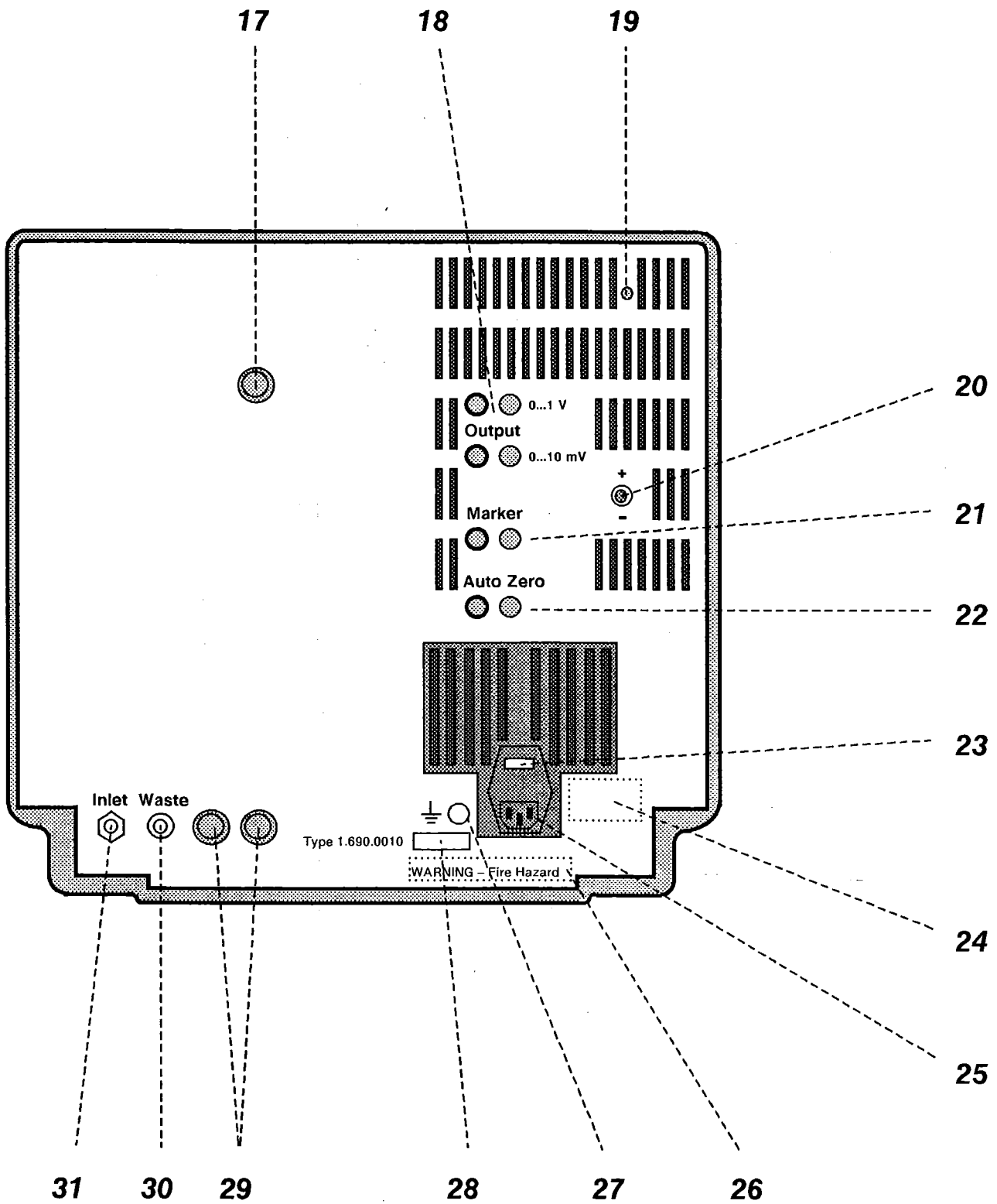


Fig. 3: Rear of the 690.0010 Ion Chromatograph

- 17 Rear panel opening**
(sealed with plastic stopper)
for additional inlet and outlet lines to the column compartment
- 18 Output**
Connection sockets for line recorder/integrator
(Analogue signal output
0...10 mV and 0...1 V)
- 19 Adjustment screw**
To regulate the heating block temperature
between 25...45 °C
This temperature is set at 35 ± 1 °C in the factory and should not be altered without good reason!
- 20 Polarity selector switch + / -**
- 21 Marker connection**
Connection sockets for external control of the marker function (see Section 7.2)
- 22 Auto Zero connection**
Connection sockets for external control of the Auto Zero function (see Section 7.2)
- 23 Display of the set mains voltage**
- 24 Data for the fuse and power consumption**
- 25 Mains connection**
Cold appliance input socket type CEE(22), VI
- 26 Important notice**
"WARNING: Fire hazard!
For continued protection replace only with the same type and rating of fuse!"
- 27 Earth socket**
- 28 Model plate**
Details of the type, serial and instrument number.
All these numbers must be quoted in full in any inquiries to METROHM or the national agency!
- 29 Rear panel openings**
(sealed with plastic stoppers)
for additional inlet and outlet lines (e.g. compressed air inlet, additional detector) to the column compartment
- 30 Waste**
Connection for 6.1822.010 PTFE microcapillary tubing (outlet to waste container)
- 31 Inlet**
Connection for 6.2620.030 steel capillary (inlet of the eluent from the pump)

2.2. 690.0020 Ion Chromatograph (with electrically operated injector)

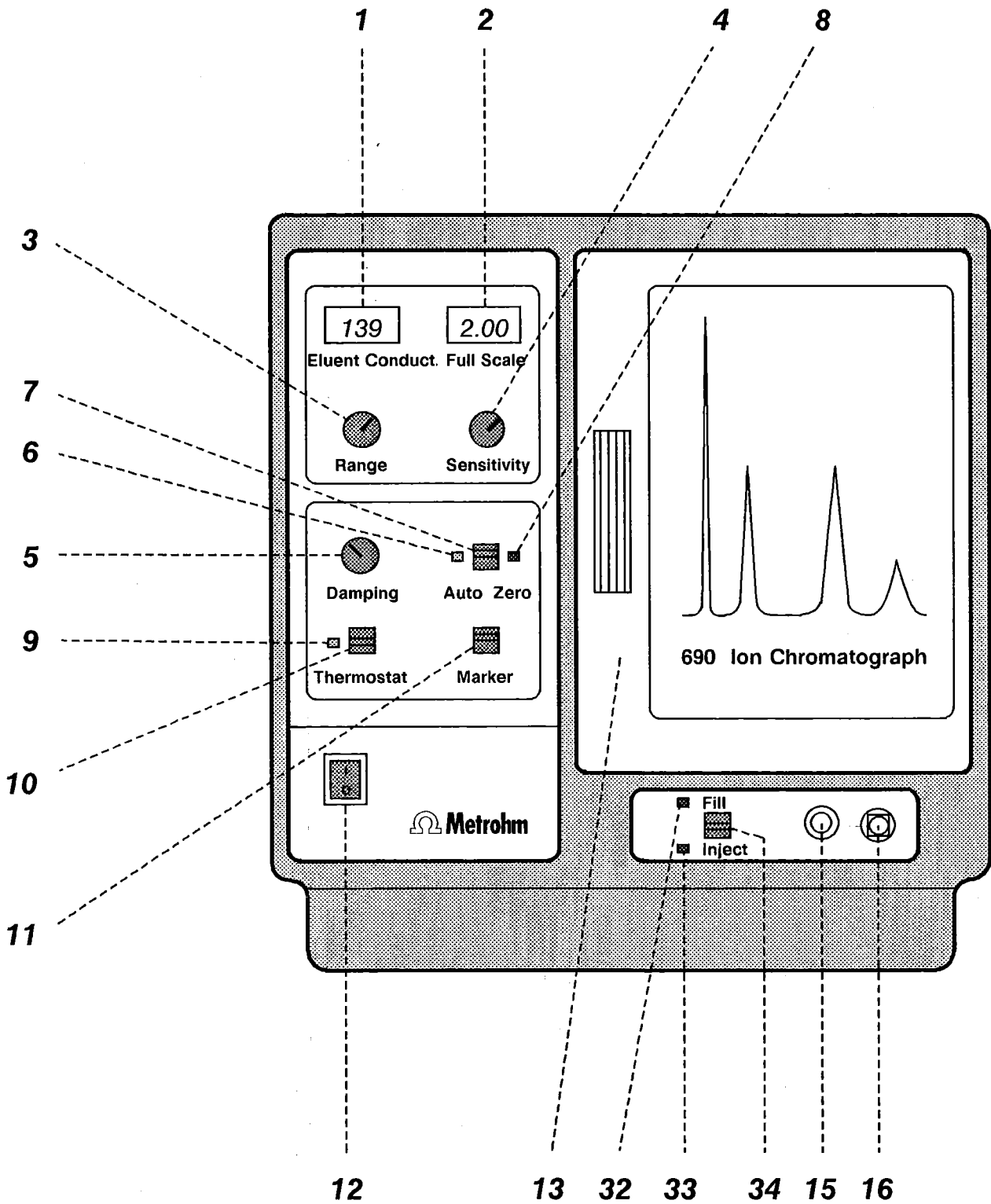


Fig. 4: Front of the 690.0020 Ion Chromatograph

- 1 Eluent Conduct.**
Display of the eluent conductivity in $\mu\text{S}/\text{cm}$
Range: 0...1000 $\mu\text{S}/\text{cm}$
If the measured conductivity falls outside the measuring range selected, "1" (digit 1 at the extreme left with no following digits) appears in the display (\rightarrow reset range)
- 2 Full Scale**
Display of the full scale operating range in $\mu\text{S}/\text{cm}$
The operating range is defined by the settings of **Range** and **Sensitivity**:
Full Scale = Range/Sensitivity
Range: 0.1...1000 $\mu\text{S}/\text{cm}$
If conductivity falls below the lower limit of this range, "1" (digit 1 at the extreme left with no following digits) appears in the display (\rightarrow reset range and/or sensitivity)
- 3 Range**
Setting of the measuring range in $\mu\text{S}/\text{cm}$
Range: 10...1000 $\mu\text{S}/\text{cm}$
- 4 Sensitivity**
Setting of the sensitivity
Range: 1...2000
- 5 Damping**
Damping (Butterworth filter, 4th order) of the measuring signal
stage 0: $\tau = 0.125 \text{ s}$
stage 1: $\tau = 0.5 \text{ s}$
stage 2: $\tau = 1 \text{ s}$
stage 3: $\tau = 2 \text{ s}$
- 6 Auto Zero display**
When the electronic background compensation is switched on, the green lamp lights up
- 7 Auto Zero**
Automatic zeroing of the measuring signal (electronic background compensation)
Triggering the zeroing (repeatable):
press switch downwards briefly (green lamp lights up)
Switching off:
push switch upwards briefly (green lamp goes out)
- 8 Overload display**
Illumination of the red lamp shows that the measuring signal lies outside $\pm 150\%$ of the full scale range selected
- 9 Thermostat display**
The green lamp lights up when the heating is switched on
- 10 Thermostat**
On/off switch for the heating of the conductivity detector
- 11 Marker**
Toggle switch for marking (ca. 10% of the full scale range)
Triggering: press switch downwards briefly
- 12 ON/OFF**
Switching the instrument on and off (mains switch)
1: On 0: Off
When in operational readiness, the red lamp in the switch lights up
- 13 Door for wet system/interior**
- 15 Connection for 6.1822.000 extractor tubing**
- 16 Connection for 6.2816.020 syringe**
- 32 Indicator lamp "Fill"**
If the red lamp lights up, the injection valve is in position "Fill" (the sample loop is filled).
- 33 Indicator lamp "Inject"**
If the red lamp lights up, the injection valve is in position "Inject" (the loop contents are injected).
- 34 Injection valve switch**
Position "Fill":
filling of the sample loop
Position "Inject":
injection of the loop contents

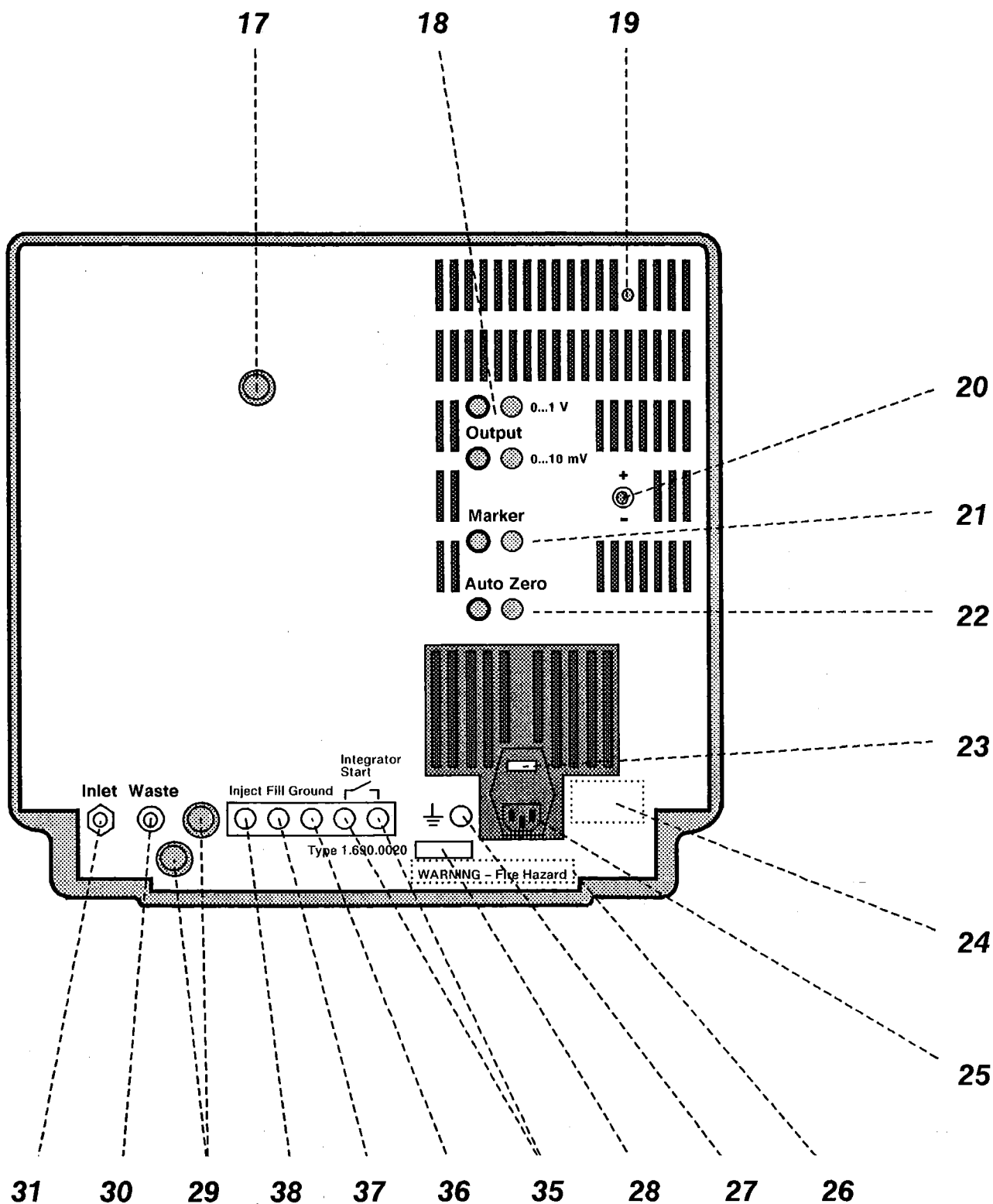


Fig. 5: Rear of the 690.0020 Ion Chromatograph

- 17 Rear panel opening**
(sealed with plastic stopper)
for additional inlet and outlet lines to the column compartment
- 18 Output**
Connection sockets for line recorder/integrator
(Analogue signal output
0...10 mV and 0...1 V)
- 19 Adjustment screw**
To regulate the heating block temperature
between 25...45 °C
This temperature is set at 35 ± 1 °C in the factory and should not be altered without good reason!
- 20 Polarity selector switch + / -**
- 21 Marker connection**
Connection sockets for external control of the marker function (see Section 7.2)
- 22 Auto Zero connection**
Connection sockets for external control of the Auto Zero function (see Section 7.2)
- 23 Display of the set mains voltage**
- 24 Data for the fuse and power consumption**
- 25 Mains connection**
Cold appliance input socket type CEE(22), VI
- 26 Important notice**
"WARNING: Fire hazard!
For continued protection replace only with the same type and rating of fuse!"
- 27 Earth socket**
- 28 Model plate**
Details of the type, serial and instrument number.
All these numbers must be quoted in full in any inquiries to METROHM or the national agency!
- 29 Rear panel openings**
(sealed with plastic stoppers)
for additional inlet and outlet lines (e.g. compressed air inlet, additional detector) to the column compartment
- 30 Waste**
Connection for 6.1822.010 PTFE microcapillary tubing (outlet to waste container)
- 31 Inlet**
Connection for 6.2620.030 steel capillary (inlet of the eluent from the pump)
- 35 Connection socket "Integrator Start"**
- 36 Connection socket "Ground" for the 698 Autosampler**
- 37 Connection socket "Fill" for the 698 Autosampler**
Connection for external switching the injection valve into the "Fill" position
- 38 Connection socket "Inject" for the 698 Autosampler**
Connection for external switching the injection valve into the "Inject" position

2.3. 690.0030 Ion Chromatograph (with 2 electrically operated injectors)

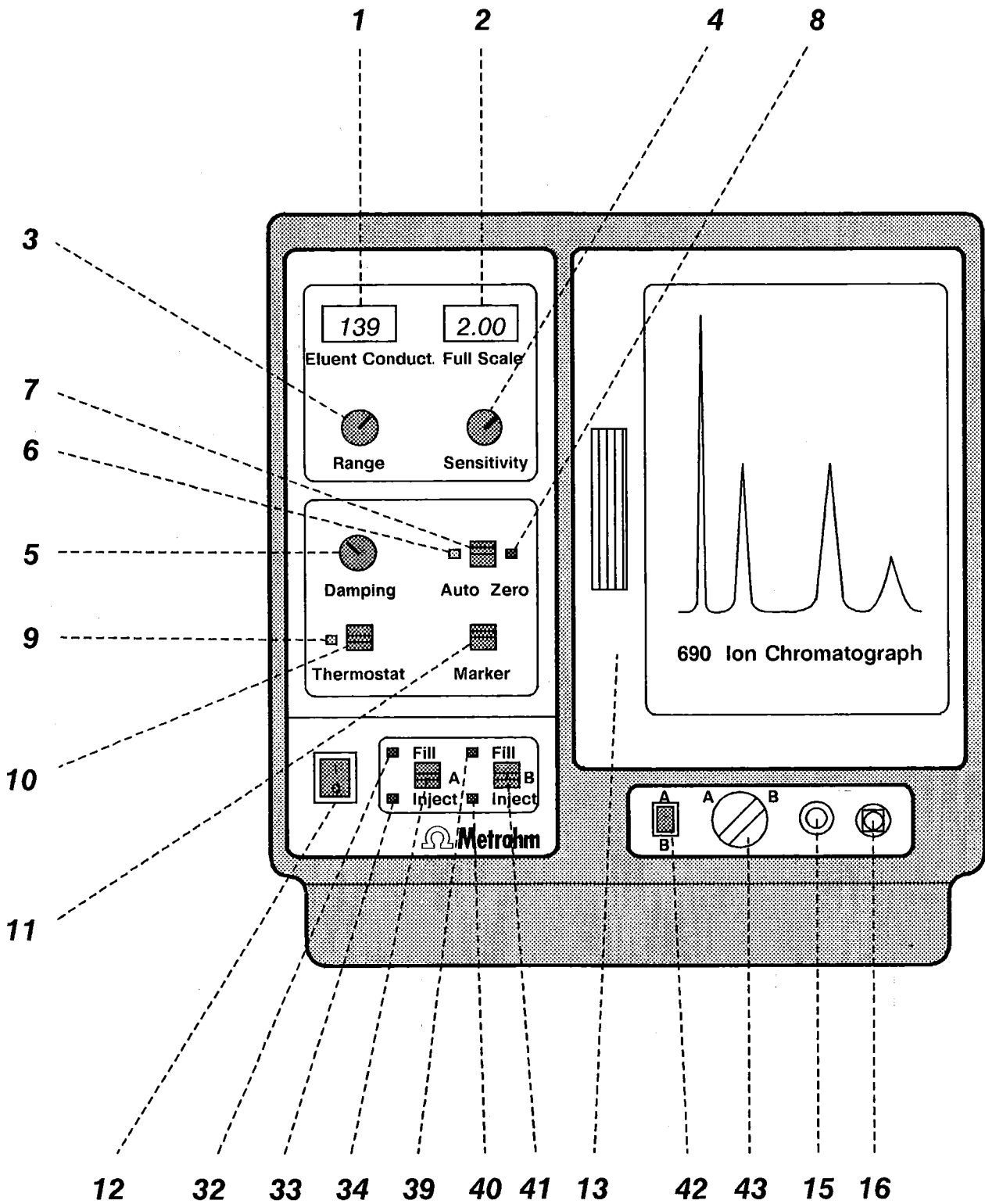


Fig. 6: Front of the 690.0030 Ion Chromatograph

- 1 Eluent Conduct.**
Display of the eluent conductivity in $\mu\text{S}/\text{cm}$
Range: 0...1000 $\mu\text{S}/\text{cm}$
If the measured conductivity falls outside the measuring range selected, "1" (digit 1 at the extreme left with no following digits) appears in the display (\rightarrow reset range)
- 2 Full Scale**
Display of the full scale operating range in $\mu\text{S}/\text{cm}$
The operating range is defined by the settings of **Range** and **Sensitivity**:
Full Scale = Range/Sensitivity
Range: 0.1...1000 $\mu\text{S}/\text{cm}$
If conductivity falls below the lower limit of this range, "1" (digit 1 at the extreme left with no following digits) appears in the display (\rightarrow reset range and/or sensitivity)
- 3 Range**
Setting of the measuring range in $\mu\text{S}/\text{cm}$
Range: 10...1000 $\mu\text{S}/\text{cm}$
- 4 Sensitivity**
Setting of the sensitivity
Range: 1...2000
- 5 Damping**
Damping (Butterworth filter, 4th order) of the measuring signal
stage 0: $\tau = 0.125$ s
stage 1: $\tau = 0.5$ s
stage 2: $\tau = 1$ s
stage 3: $\tau = 2$ s
- 6 Auto Zero display**
When the electronic background compensation is switched on, the green lamp lights up
- 7 Auto Zero**
Automatic zeroing of the measuring signal (electronic background compensation)
Triggering the zeroing (repeatable):
press switch downwards briefly (green lamp lights up)
Switching off:
push switch upwards briefly (green lamp goes out)
- 8 Overload display**
Illumination of the red lamp shows that the measuring signal lies outside $\pm 150\%$ of the full scale range selected
- 9 Thermostat display**
The green lamp lights up when the heating is switched on
- 10 Thermostat**
On/off switch for the heating of the conductivity detector
- 11 Marker**
Toggle switch for marking (ca. 10% of the full scale range)
Triggering: press switch downwards briefly
- 12 ON/OFF**
Switching the instrument on and off (mains switch)
1: On 0: Off
When in operational readiness, the red lamp in the switch lights up
- 13 Door for wet system/interior**
- 15 Connection for 6.1822.000 extractor tubing**
- 16 Connection for 6.2816.020 syringe**
- 32 Indicator lamp "Fill" for column A**
If the red lamp lights up, the injection valve **A** is in position "Fill" (the sample loop is filled).
- 33 Indicator lamp "Inject" for column A**
If the red lamp lights up, the injection valve **A** is in position "Inject" (the loop contents are injected).
- 34 Injection valve switch for column A**
Position "Fill":
filling of the sample loop
Position "Inject":
injection of the loop contents
- 39 Indicator lamp "Fill" for column B**
If the red lamp lights up, the injection valve **B** is in position "Fill" (the sample loop is filled).
- 40 Indicator lamp "Inject" for column B**
If the red lamp lights up, the injection valve **B** is in position "Inject" (the loop contents are injected).
- 41 Injection valve switch for column B**
Position "Fill":
filling of the sample loop
Position "Inject":
injection of the loop contents
- 42 Switch for injector selection of the connector block (50) A/B**
Position A:
The connection sockets (35...38) refer to injector/column **A**
Position B:
The connection sockets (35...38) refer to injector/column **B**
- 43 Valve switch for selection of column A or B**

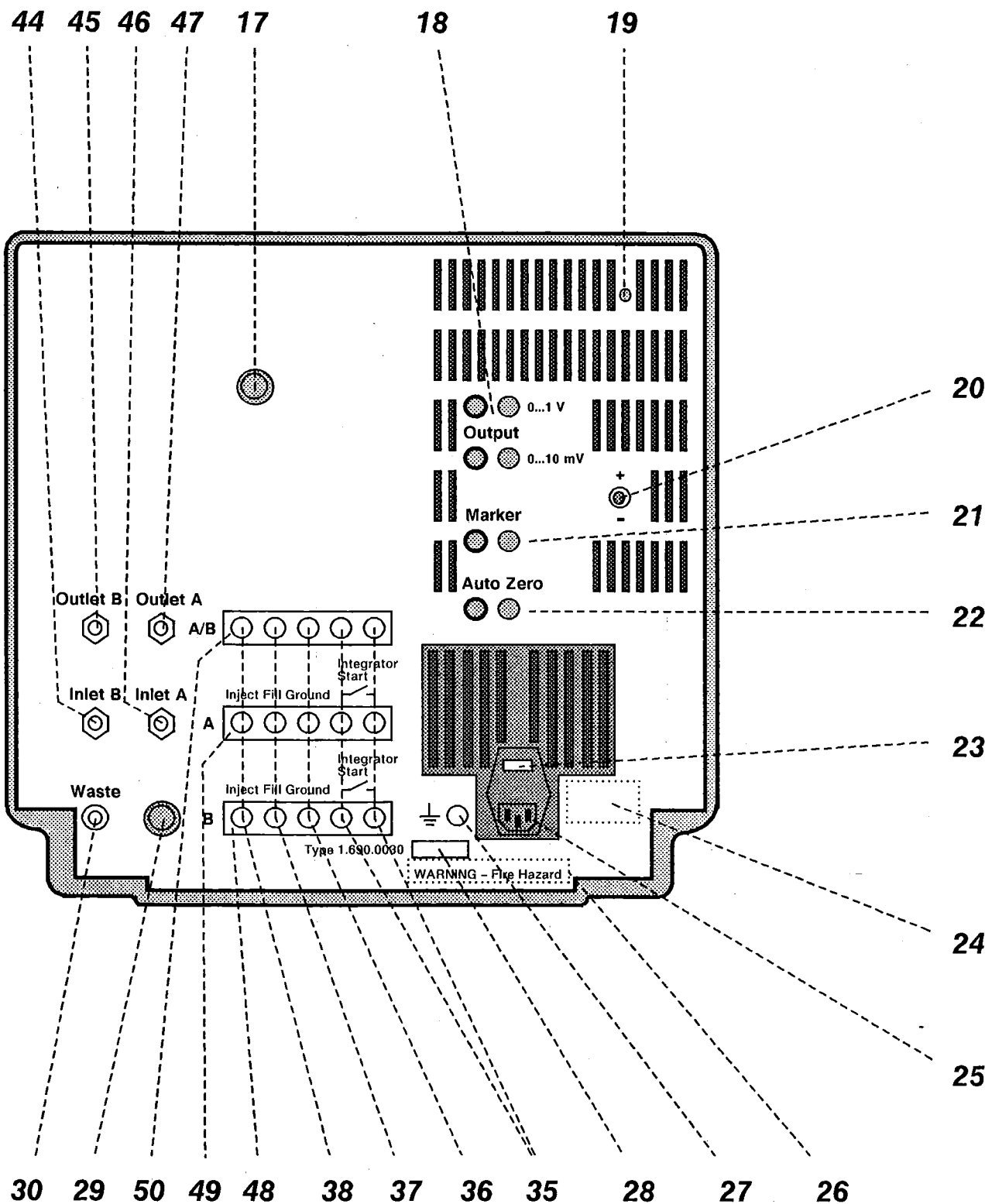


Fig. 7: Rear of the 690.0030 Ion Chromatograph

- 17 Rear panel opening**
(sealed with plastic stopper)
for additional inlet and outlet lines to the column compartment
- 18 Output**
Connection sockets for line recorder/integrator
(Analogue signal output
0...10 mV and 0...1 V)
- 19 Adjustment screw**
To regulate the heating block temperature
between 25...45 °C
This temperature is set at 35 ± 1 °C in the factory and should not be altered without good reason!
- 20 Polarity selector switch + / -**
- 21 Marker connection**
Connection sockets for external control of the marker function (see Section 7.2)
- 22 Auto Zero connection**
Connection sockets for external control of the Auto Zero function (see Section 7.2)
- 23 Display of the set mains voltage**
- 24 Data for the fuse and power consumption**
- 25 Mains connection**
Cold appliance input socket type CEE(22), VI
- 26 Important notice**
"WARNING: Fire hazard!
For continued protection replace only with the same type and rating of fuse!"
- 27 Earth socket**
- 28 Model plate**
Details of the type, serial and instrument number.
All these numbers must be quoted in full in any inquiries to METROHM or the national agency!
- 29 Rear panel openings**
(sealed with plastic stoppers)
for additional inlet and outlet lines (e.g. compressed air inlet, additional detector) to the column compartment
- 30 Waste**
Connection for 6.1822.010 PTFE microcapillary tubing (outlet to waste container)
- 35 Connection socket "Integrator Start"**
- 36 Connection socket "Ground" for the 698 Autosampler**
- 37 Connection socket "Fill" for the 698 Autosampler**
Connection for external switching the injection valve into the "Fill" position
- 38 Connection socket "Inject" for the 698 Autosampler**
Connection for external switching the injection valve into the "Inject" position
- 44 Inlet B**
Eluent inlet to column B
Connection for 6.2620.020 steel capillary (inlet of the eluent from the pump)
- 45 Outlet B**
Eluent outlet from column B
Connection for 6.1822.010 PTFE microcapillary tubing (outlet to waste container)
- 46 Inlet A**
Eluent inlet to column A
Connection for 6.2620.020 steel capillary (inlet of the eluent from the pump)
- 47 Outlet A**
Eluent outlet from column A
Connection for 6.1822.010 PTFE microcapillary tubing (outlet to waste container)
- 48 Connector block B for injector B**
- 49 Connector block A for injector A**
- 50 Connector block A/B for injector A or B**
Selection A ↔ B by switch (43)

3. Installation

Note: Normally, the installation procedures described for the "690 Ion Chromatograph" are guilty for all three instrument versions. Different procedures are specified for each version separately.

3.1. Setting up the instrument

3.1.1. Packaging

The 690 Ion Chromatograph is supplied together with the separately packed accessories in a special package designed to afford extremely good protection. This contains two shock-absorbent foam liners foamed to the individual shape and wrapped in double plastic blue sheeting. The instrument itself is packed in an evacuated polyethylene bag to exclude dust. It is advisable to keep the special packaging since only this ensures that no transport damage is incurred if the instrument has to be returned.

3.1.2. Transport protection

For protection during the transport, the detector block in the interior is screwed onto the rear of the instrument. The adjusting of the detector block is described in *Section 3.7.2*. If the instrument has to be returned, the detector block must be rescrewed onto the rear.

3.1.3. Control

Immediately after receipt of the shipment, a check must be made to ensure that it is complete and has arrived without damage (compare with delivery note and accessories list in *Section 8*). If transport damage has occurred, see instructions in *Section 9 "Warranty"*.

3.1.4. Set up

To ensure smooth operation, the 690 Ion Chromatograph should be set up in the laboratory at a place free from vibration and protected from any corrosive atmosphere and contamination by chemicals. This also applies to all other components of the ion chromatography system. To avoid disturbing influences of temperature on the insulated column compartment, the entire system (incl. pump and eluent reservoir) must be protected from direct sunlight.

3.2. Mains connection and switching on the instrument

3.2.1. Altering the voltage

Before switching on the 690 Ion Chromatograph for the first time, check to ensure that the mains voltage set on the instrument (readable in display **(23)** above the mains connection **(25)**) conforms with the local mains voltage. If this is not the case, alter the voltage as follows:

- 1▶ Pull out mains cable.
- 2▶ Open plastic cover above the mains connection **(25)** with the aid of a screwdriver.
- 3▶ Take out voltage selector drum and place in the correct position. (Never turn the drum when in place!)
- 4▶ Remove fuse below the voltage selector drum and compare its specifications with the data **(24)** on the rear panel. If necessary, replace fuse.
- 5▶ Press on plastic cover until it snaps in.

3.2.2. Thermal fuse

The 690 Ion Chromatograph is provided with additional protection by a thermal fuse; METROHM service must be called in if it blows.

3.2.3. Mains cable

The instrument is supplied with one of three mains cables:

- 6.2122.020 with plug SEV 12 (Switzerland ...)
- 6.2122.040 with plug CEE(7), VII (Federal Republic of Germany ...)
- 6.2122.070 with plug NEMA/ASA (USA ...)

which are three-cored and fitted with a plug with earthing pin. If a different plug has to be fitted, the yellow/green lead (IEC standard) must be connected to the protective earth. If no socket with earth is available, the instrument must be connected to a perfect earthing conductor via its earthing socket (27) (→).

3.2.4. Mains connection

The mains cable is plugged into connection socket (25).

3.2.5. Switching on the instrument

The instrument is switched on using mains switch (12) at the front: I = On 0 = Off
Operational readiness is indicated by illumination of the red lamp in the mains switch.

3.3. Capillary connections

3.3.1. Steel connectors

All high pressure tubing between the feed pump and the detector of the 690 Ion Chromatograph consists of steel capillaries (i.d. = 0.25 mm, e.d. = 1/16") screwed onto the appropriate connections by means of the two steel connector pieces 6.2620.010 ferrule and 6.2620.000 compression fitting. To mount the connectors onto a capillary, proceed as follows:

- 1▶ Cut off a piece of appropriate length from the 6.2620.020 steel capillary (i.d. = 0.25 mm, e.d. = 1/16", length L = 3 m).
To achieve a perfect cut surface, it is best to use the 6.2621.040 capillary tubing cutter available as an option.
- 2▶ Slid a 6.2620.000 compression fitting and a 6.2620.010 ferrule over the end of the capillary as shown in Fig. 8.
- 3▶ To avoid dead volumes, push the end of the capillary completely into the appropriate connection.
- 4▶ Tighten the compression fitting with the 6.2621.010 open-end spanner 1/4".

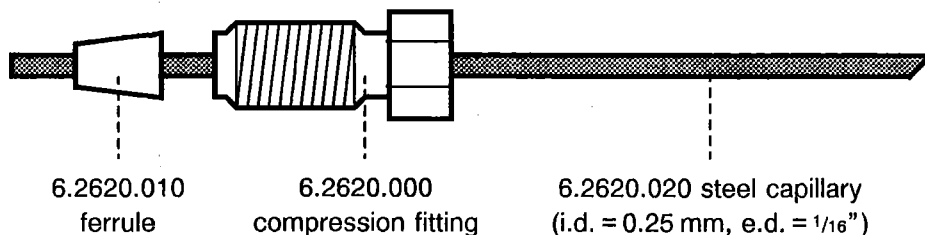


Fig. 8: Steel connectors

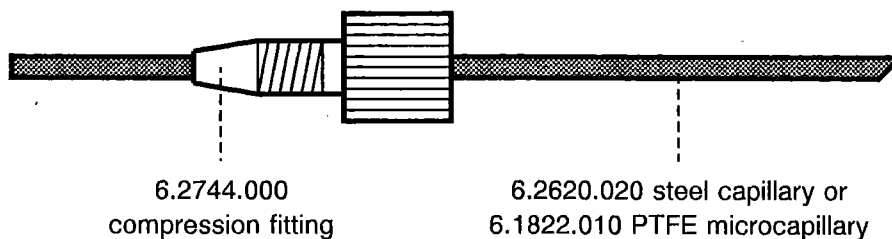


Fig. 9: PVDF connectors

3.3.2. PVDF connectors

Instead of steel connectors (see *Section 3.3.1*) you can use the 6.2744.000 PVDF connectors for steel capillary connections up to a pressure of 200 bar. These connectors are especially suited for connecting the 6.1822.010 PTFE microcapillaries (i.d. = 0.3 mm). To mount the connectors onto a capillary, proceed as follows:

- 1▶ Cut off a piece of appropriate length from the steel or PTFE capillary.
To achieve a perfect cut surface with steel capillaries, it is best to use the 6.2621.040 capillary tubing cutter available as an option.
- 2▶ Slid a 6.2744.000 compression fitting over the end of the capillary as shown in *Fig. 9*.
- 3▶ To avoid dead volumes, push the end of the capillary completely into the appropriate connection.
- 4▶ Tighten the PVDF compression fitting by hand (use no tools!).

3.4. Connection of the 697 IC Pump

3.4.1. General

Any commercial HPLC pump can be employed for operation of the 690 Ion Chromatograph. Since the attainable sensitivity depends to a large extent on the quality of the pump, METROHM recommends use of the **697 IC Pump** with minimal residual pulsation and excellent flowrate stability.

For the **startup procedure and operation of the 697 IC Pump**, please refer to the 8.697.1003 Instructions for use. The eluent, which must be degassed and filtered (cf. *Section 6.3*), is selected on the basis of the column installed in the 690 Ion Chromatograph and the separation problem at hand (see *Section 1-02* of the 8.690.2013 Application File). It must be ensured that the eluent employed is miscible with the solvents contained in the pump head and in the column (check manufacturers' specifications).

3.4.2. Pulsation dampener

To protect the column material from pressure drops caused by the injector the use of a pulsation dampener connected between the pump and the 690 Ion Chromatograph is recommended. The optional **6.2620.050 Portmann pulsation dampener** is very well suited to this purpose (see *Section 8.4.2*).

The 6.2620.050 pulsation dampener has two connections for steel capillaries with arbitrary flow direction.

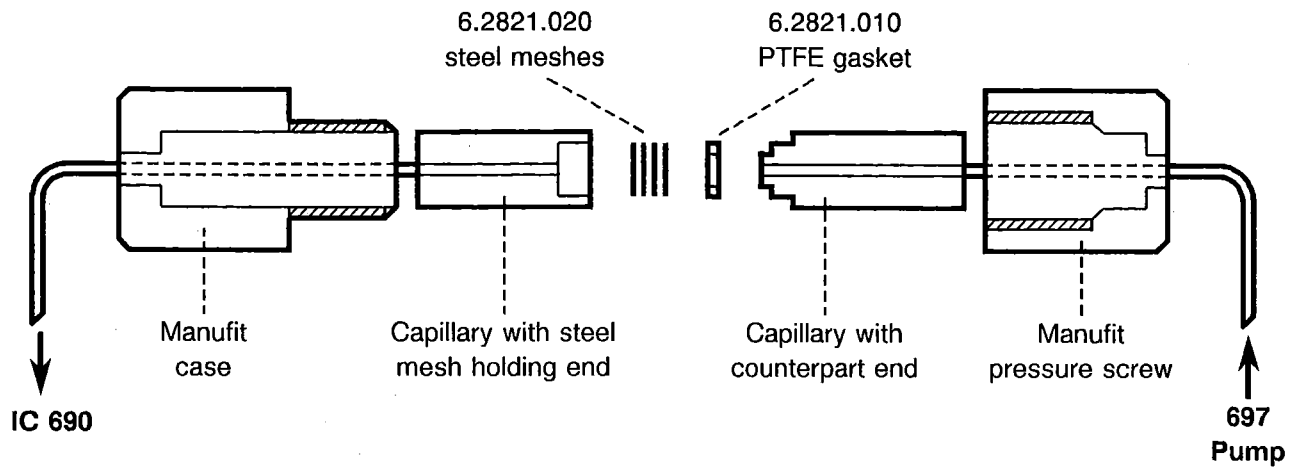


Fig. 10: Installing the 6.2821.000 Filter Unit Manufit

3.4.3. Installing the Filter Unit Manufit

To avoid contamination of the piston seals of the 697 IC Pump by abrasive particles, it is advantageous to fit an in-line filter between the pump and the 690 Ion Chromatograph. The optional **6.2821.000 Manufit filter unit** (see Section 8.4.8) is extremely well suited to this purpose. For mounting this filter, proceed as follows (see Fig. 10):

- 1▶ Introduce steel capillary with steel mesh holding end into Manufit case.
- 2▶ Insert 4 steel meshes (6.2821.020) into the steel mesh holding end.
- 3▶ Press the 6.2821.010 PTFE gasket into the steel mesh holding end.
- 4▶ Introduce steel capillary with counterpart end into Manufit pressure screw.
- 5▶ Stick together the two capillary end pieces.
- 6▶ Screw together tightly by hand the Manufit case and the Manufit pressure screw.

To exchange dirty steel meshes proceed in opposite order.

3.4.4. Connection 690 Ion Chromatograph - 697 IC Pump

- **690.0010 and 690.0020 Ion Chromatograph:**

Connect the 697 IC Pump to the 690 Ion Chromatograph as shown in Fig. 11. All steel capillaries are mounted as described in Section 3.3.1:

- 1▶ 690 Ion Chromatograph: connection (31) (inlet)
- 2▶ 697 IC Pump: connection (4)
(see 8.697.1003 Instructions for use)

- **690.0030 Ion Chromatograph:**

For operation of the 690.0030 Ion Chromatograph two independent 697 IC pumps are needed. Connect the two pumps **A** and **B** to the 690 Ion Chromatograph as shown in Fig. 11. All steel capillaries are mounted as described in Section 3.3.1:

- 1▶ 690 Ion Chromatograph: connection (46) (inlet **A** for pump **A**)
connection (44) (inlet **B** for pump **B**)
- 2▶ 697 IC Pump: connection (4)
(see 8.697.1003 Instructions for use)

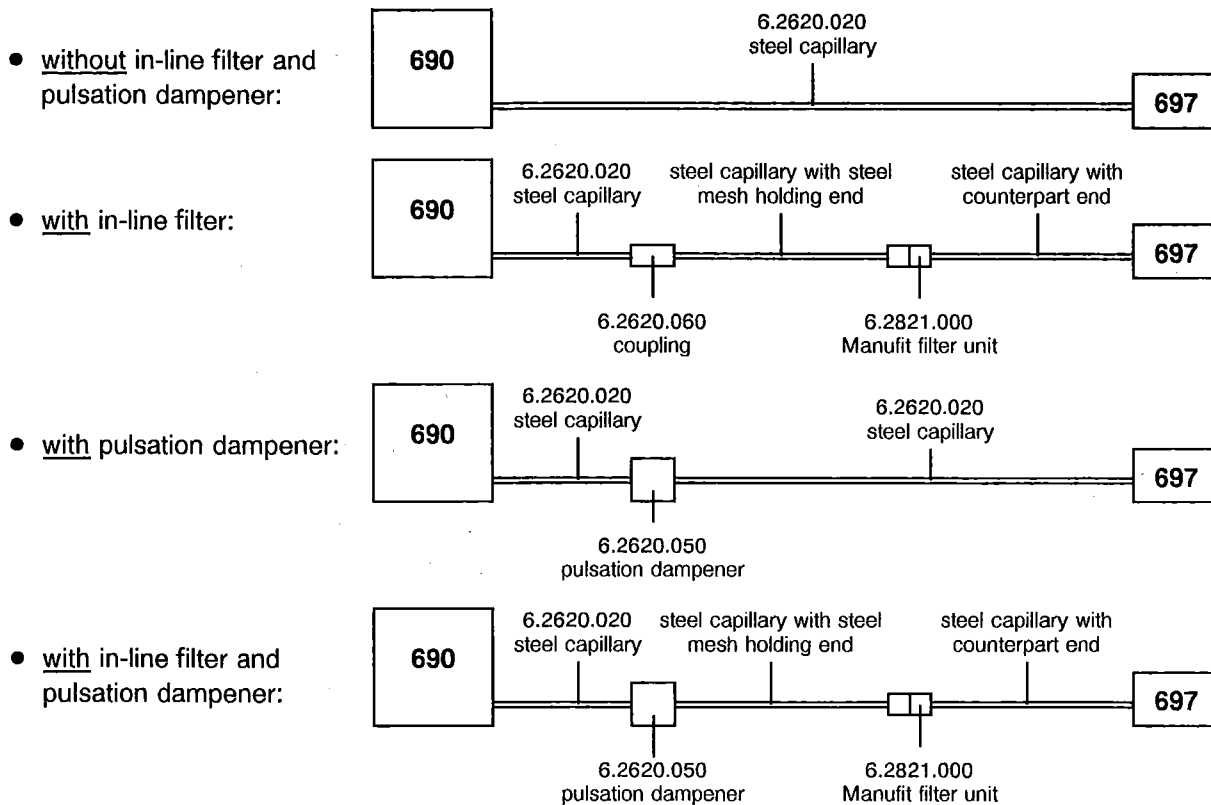


Fig. 11: Connection 690 Ion Chromatograph – 697 IC Pump

3.4.5. Filling the capillary up to the column connection

• **690.0010 Ion Chromatograph:**

Before insertion of the column, the connection capillary between the 697 IC Pump and the 690 Ion Chromatograph has to be filled up to the end of the capillary (65) (see Fig. 13) with eluent and be rinsed:

- 1▶ Turn the injection valve switch (14) into the "Inject" position.
- 2▶ Switch on the pump and deliver eluent until it emerges at the end of capillary (65).
- 3▶ Put a beaker under the end of capillary (65).
- 4▶ Rinse the connection 697 IC Pump – 690 Ion Chromatograph with eluent for about 10 min.
- 5▶ Switch off the pump.

• **690.0020 Ion Chromatograph:**

Before insertion of the column, the connection capillary between the 697 IC Pump and the 690 Ion Chromatograph has to be filled up to the end of the capillary (65) (see Fig. 13) with eluent and be rinsed:

- 1▶ Press downwards briefly the injection valve switch (34). The red lamp (33) indicates that the injection valve is in the "Inject" position.
- 2▶ Switch on the pump and deliver eluent until it emerges at the end of capillary (65).
- 3▶ Put a beaker under the end of capillary (65).
- 4▶ Rinse the connection 690 Ion Chromatograph – 697 IC Pump with eluent for about 10 min.
- 5▶ Switch off the pump.

- **690.0030 Ion Chromatograph:**

Before insertion of the columns, both connections capillaries between the 697 IC Pump and the 690 Ion Chromatograph have to be filled up to the end of the capillaries (71) and (76) (see Fig. 14) with eluent and be rinsed:

- **Connection for column A:**

- 1▶ Press downwards briefly the injection valve switch (34). The red lamp (33) indicates that the injection valve **A** is in the "Inject" position.
- 2▶ Switch on pump **A** and deliver eluent until it emerges at the end of capillary (71).
- 3▶ Put a beaker under the end of capillary (71).
- 4▶ Rinse the connection pump **A** – 690 Ion Chromatograph with eluent for about 10 min.
- 5▶ Switch off pump **A**.

- **Connection for column B:**

- 1▶ Press downwards briefly the injection valve switch (41). The red lamp (40) indicates that the injection valve **B** is in the "Inject" position.
- 2▶ Switch on pump **B** and deliver eluent until it emerges at the end of capillary (76).
- 3▶ Put a beaker under the end of capillary (76).
- 4▶ Rinse the connection pump **B** – 690 Ion Chromatograph with eluent for about 10 min.
- 5▶ Switch off pump **B**.

3.5. Connection of the waste container

The 6.1822.010 PTFE microcapillary tubing (i.d. = 0.3 mm, e.d. = 1.5 mm, length $L = 1$ m) is used for the connection 690 Ion Chromatograph – waste container:

- 1▶ Connect the nipple with flange attached to the tubing to connection (30) (Waste) at the rear of the 690 Ion Chromatograph. Tighten the plastic nipple securely by hand.
- 2▶ Introduce the open end of the tubing into a waste container of sufficient capacity and fix it in place.

3.6. Installing the precolumn

3.6.1. General

The use of easily exchangeable precolumns protects the separation columns and prolongs their life time. The precolumns available from METROHM (see Section 8.4.7) are either real precolumns or precolumn cartridges, which are used together with a 6.2821.050 twin cartridge holder or a 6.2821.040 cartridge head.

New IC precolumns are normally filled with solution and sealed at both ends. Before the precolumn is installed in the system, it must be ensured that this solution is freely miscible with the eluent used (check manufacturer's specifications).

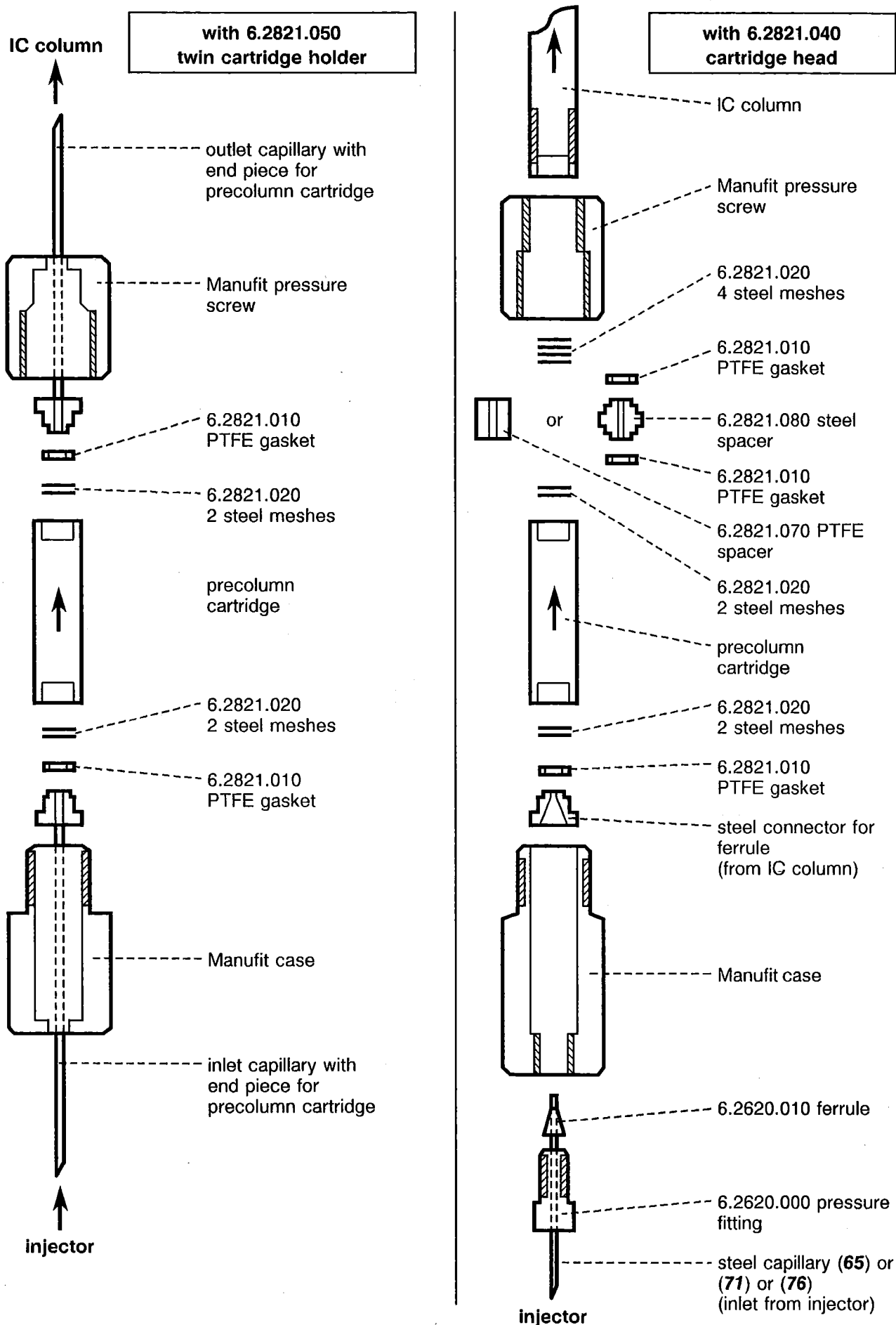


Fig. 12: Installing the precolumn cartridges

3.6.2. Precolumn cartridges with 6.2821.050 twin cartridge holder

For installing the precolumn cartridges proceed as follows (see Fig. 12):

- 1▶ Introduce inlet capillary with end piece for precolumn cartridge into Manufit case.
- 2▶ Introduce outlet capillary with end piece for precolumn cartridge into Manufit pressure screw.
- 3▶ Remove end caps from the precolumn cartridge.
- 4▶ Stick the two capillary end pieces onto the precolumn cartridge (pay attention to the flow direction if specified on the precolumn).
- 5▶ Screw tightly together Manufit case and Manufit pressure screw by hand.
- 6▶ Shorten the outlet capillary of the precolumn to about 5 cm and mount the connectors (cf. Section 3.3.1).
- 7▶ Mount the connectors to the inlet capillary of the precolumn (cf. Section 3.3.1).
- 8▶ Screw inlet capillary to connection (64) or (71) or (76) (inlet from injector) by means of the 6.2620.060 coupling.
- 9▶ Put a beaker under the end of the outlet capillary.
- 10▶ Switch on 697 IC Pump and rinse the precolumn with eluent for about 10 min.
- 11▶ Switch off the pump.

3.6.3. Precolumn cartridges with 6.2821.040 cartridge head

For installing the precolumn cartridges proceed as follows (see Fig. 12):

- 1▶ Remove end caps from the column.
- 2▶ Remove the fastening screw from the inlet of the IC column.
- 3▶ Take out steel connector for ferrule from the fastening screw.
- 4▶ Remove end caps from the precolumn cartridge.

with 6.2821.070 PTFE spacer:

- 5▶ Remove 6.2821.010 PTFE gasket from the inlet side of the column
- 6▶ Remove 6.2821.010 PTFE gasket from the outlet side of the precolumn cartridge
- 7▶ Put the 6.2821.070 PTFE spacer on the column (refrigerate first if necessary)
- 8▶ Put the precolumn cartridge on the 6.2821.070 PTFE spacer (pay attention to the flow direction specified on the precolumn)

with 6.2821.080 steel spacer (option):

- 5▶ -
- 6▶ -
- 7▶ Put the 6.2821.080 steel spacer on the column
- 8▶ Put the precolumn cartridge on the 6.2821.080 steel spacer (pay attention to the flow direction specified on the precolumn)

- 9▶ Screw Manufit pressure screw onto the column.
- 10▶ Put the steel connector for ferrule on the precolumn cartridge.
- 11▶ Screw tightly together Manufit case and Manufit pressure screw by hand.

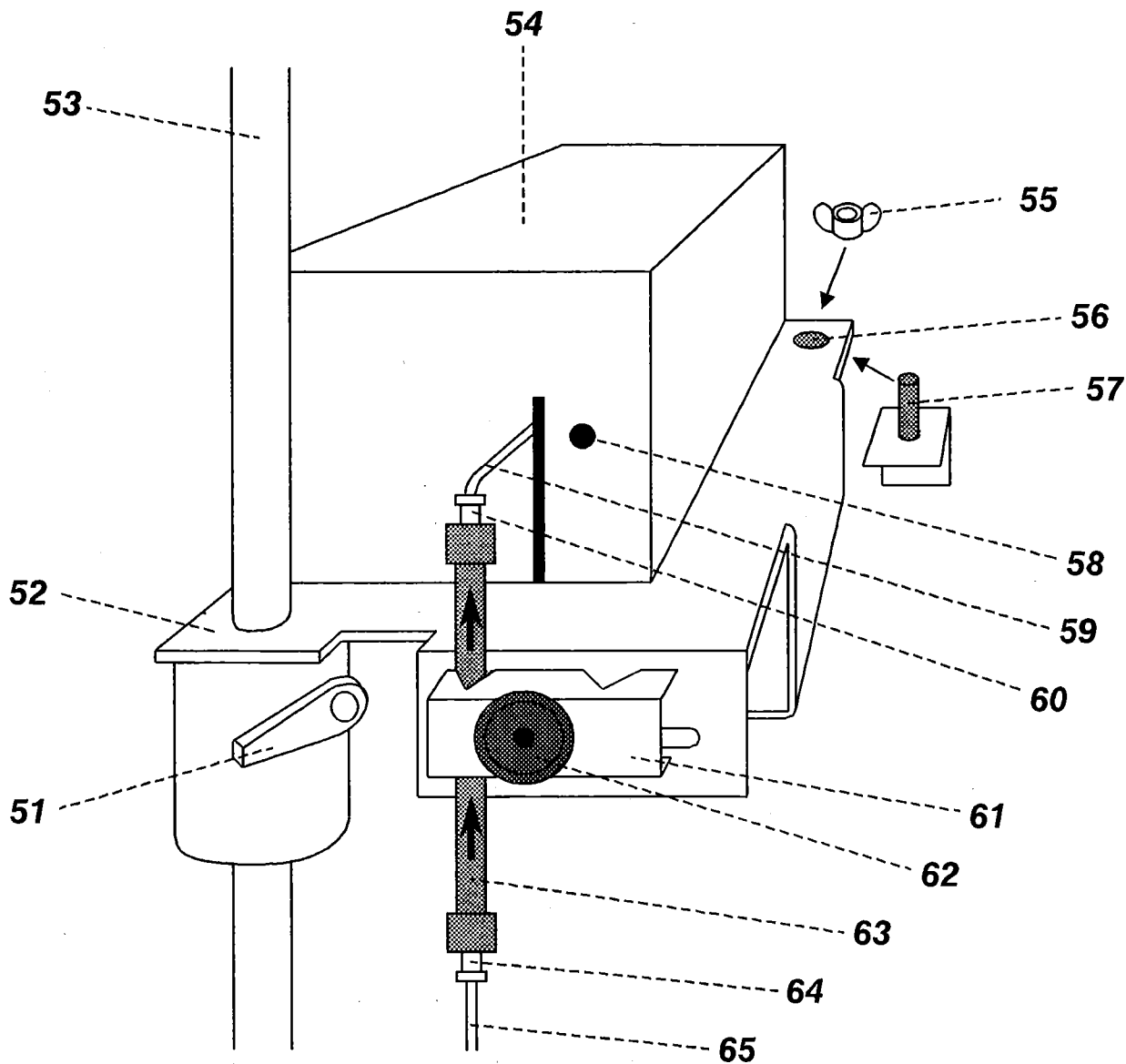


Fig. 13: Detector and column at the 690.0010 and 690.0020 Ion Chromatograph

- | | |
|--|---|
| 51 <i>locking lever to lock the platform</i> | 60 <i>upper column connection</i>
(6.2620.000 pressure fitting and
6.2620.010 ferrule) |
| 52 <i>platform</i> | 61 <i>column support</i> |
| 53 <i>stand rod</i> | 62 <i>turning knob for locking the column
support</i> |
| 54 <i>detector block</i> | 63 <i>column</i>
(pay attention to the flow direction when
installing) |
| 55 <i>fly nut for transport protection screw</i> | 64 <i>lower column connection</i>
(6.2620.000 pressure fitting and
6.2620.010 ferrule) |
| 56 <i>opening for transport protection screw</i> | 65 <i>capillary connection to injector</i> |
| 57 <i>transport protection screw</i>
(mounted on the rear of the instrument) | |
| 58 <i>opening for external temperature sensor</i> | |
| 59 <i>capillary connection to detector block</i> | |

3.6.4. 6.1009.010 IC anion precolumn SUPER-SEP

This precolumn has two connections for steel capillaries and is installed as follows:

- 1▶ Remove end caps from the precolumn.
- 2▶ Screw precolumn to connection (64) [(70) or (75) resp.] of the capillary (65) [(71) or (76) resp.] (inlet from injector) (see Fig. 13 and Fig. 14 resp.).
- 3▶ Cut off a short piece of the 6.2620.020 steel capillary (i.d. = 0.25 mm, e.d. = 1/16", length L = 3 m) and mount the connectors (cf. Section 3.3.1)
- 4▶ Screw the prepared steel capillary to the other end of the precolumn (cf. Section 3.3.1).
- 5▶ Put a beaker under the outlet of the precolumn.
- 6▶ Switch on 697 IC Pump and rinse the precolumn with eluent for about 10 min.
- 7▶ Switch off the pump.

3.7. Installing the column

3.7.1. General

New IC columns are normally filled with solution and sealed at both ends. Before the column is installed in the system, it must be ensured that this solution is freely miscible with the eluent used (check manufacturer's specifications).

The IC columns and precolumns available from METROHM are listed in Section 8.4.7. With each column a test chromatogram and an information leaflet is supplied. Additional information concerning these columns can be found in Section 1-02 of the 8.690.2013 Application File.

3.7.2. Adjusting the detector block position

To protect the detector block (54) fixed on the platform (52) during transport, it is screwed onto the rear of the instrument (see Fig. 13). The position of the detector block is adjusted as follows:

- 1▶ Unscrew fly nut (55) (store up for another transport).
- 2▶ Loose locking lever (51) by turning anticlockwise.
- 3▶ Slid the platform (52) together with the permanently mounted detector block (54) to the desired height on the stand rod (53) and lock it in place again by retightening the locking lever (51).

3.7.3. Installing the column

- 690.0010 and 690.0020 Ion Chromatograph:

The IC column is installed as follows (see Fig. 13):

- 1▶ Remove the end caps from the column.
- 2▶ Screw the inlet end of column (63) onto the lower column connection (64) mounted on the capillary (65) or onto the outlet capillary of the precolumn mounted on the column (see Section 3.6).

Take care to ensure that the column is inserted correctly in accordance with the flow direction marked (arrow must point upwards).

In the case of columns with 1/4" thread, the 6.2620.040 accessory (adaptor 1/16"-1/4") must be inserted between the column and connection (64).

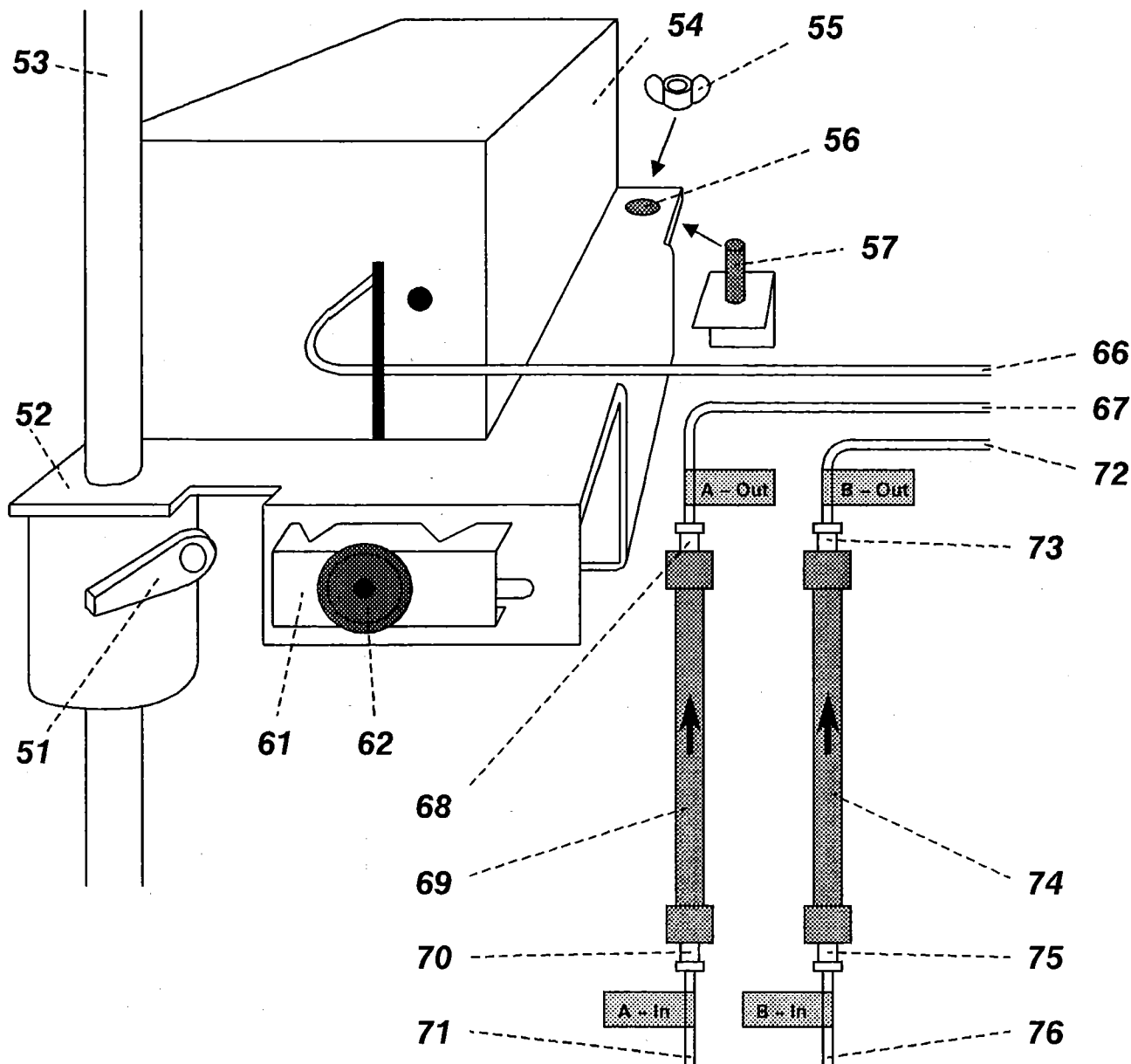


Fig. 14: Detector and column at the 690.0030 Ion Chromatograph

- | | | | | | |
|----|---|----|--|----|--|
| 51 | locking lever to lock the platform | 66 | capillary connection to detector block
(connection to column selection valve) | 71 | capillary connection to injector A |
| 52 | platform | 67 | capillary connection between column A and column selection valve
(outlet from column A) | 72 | capillary connection between column A and column selection valve
(outlet from column B) |
| 53 | stand rod | 68 | upper column connection for column A
(6.2620.000 pressure fitting and 6.2620.010 ferrule) | 73 | upper column connection for column B
(6.2620.000 pressure fitting and 6.2620.010 ferrule) |
| 54 | detector block | 69 | column A
(pay attention to the flow direction when installing) | 74 | column B
(pay attention to the flow direction when installing) |
| 55 | fly nut for transport protection screw | 70 | lower column connection for column A
(6.2620.000 pressure fitting and 6.2620.010 ferrule) | 75 | lower column connection for column B
(6.2620.000 pressure fitting and 6.2620.010 ferrule) |
| 56 | opening for transport protection screw | 71 | capillary connection to injector A | 76 | capillary connection to injector B |
| 57 | transport protection screw
(mounted on the rear of the instrument) | | | | |
| 61 | column support | | | | |
| 62 | turning knob for locking the column support | | | | |

- 3▶ Put a beaker under the outlet end of the column.
- 4▶ Switch on 697 IC Pump and rinse the column with eluent for about 10 min.
- 5▶ Switch off the pump.
- 6▶ Adjust the position of the detector block (54) to accommodate the length of the installed column:
Slid the platform (52) together with the permanently mounted detector block (54) to the desired height on the stand rod (53) and lock it in place again by retightening the locking lever (51).
- 7▶ Screw tightly the upper end of column (63) onto the upper column connection (60) mounted on the capillary (59). For columns with 1/4" thread, a 6.2620.040 adaptor is again inserted (connection 1/16"-1/4").
- 8▶ The connected column (63) can be additionally fixed using the column support (61):
Loosen the turning knob (62), slid column support (61) over column (63) and retighten turning knob (62).

• 690.0030 Ion Chromatograph:

The column A [column B] is installed as follows (see Fig. 14):

- 1▶ Remove the end caps from column A [column B].
- 2▶ Screw the inlet end of column A (69) [column B (74)] onto the lower column connection (70) [(75)] mounted on the capillary (71) [(76)] or onto the outlet capillary of the precolumn mounted on the column (see Section 3.6).

Take care to ensure that the column is inserted correctly in accordance with the flow direction marked (arrow must point upwards).

In the case of columns with 1/4" thread, the 6.2620.040 accessory (adaptor 1/16"-1/4") must be inserted between the column and connection (70) [(75)].

- 3▶ Put a beaker under the outlet end of the column.
- 4▶ Switch on 697 IC Pump and rinse the column with eluent for about 10 min.
- 5▶ Switch off the pump.
- 6▶ Screw tightly the upper end of column A (69) [column B (74)] onto the upper column connection (68) [(73)] mounted on the capillary (67) [(72)]. For columns with 1/4" thread, a 6.2620.040 adaptor is again inserted (connection 1/16"-1/4").
- 7▶ The connected columns can be additionally fixed using the column support (61):
First adjust the position of the detector block (54) if necessary. Then loosen the turning knob (62), slid column support (61) over column (63) and retighten turning knob (62).

3.7.4 Testing for leaks

The entire IC system must be tested for leaks before normal operation:

- 1▶ Set the desired flow rate (normally 1...3 mL/min) and the maximum upper pressure (20...30 bar above the pressure observed with the installed column) on the 697 IC Pump (see 8.697.1003 Instructions for use).
- 2▶ Switch on the 697 IC Pump and check all connections for leaks. If eluent flows out somewhere, tighten the pressure fitting concerned.
- 3▶ If the entire system is tight, condition the IC system (see Section 3.13).

3.8. Connection of syringe and extractor tubing

The 6.2810.020 syringe and the 6.1822.000 PTFE extractor tubing (i.d. = 0.9 mm, e.d. = 1.5 mm, length $L = 40$ cm) are needed to fill the injector loop valve. They are installed as follows:

- 1▶ Insert 6.2816.020 syringe (without needle) into connection socket (16) at the front of the instrument.
- 2▶ Screw 6.1822.000 extractor tubing into connection (15).

3.9. Connection of a recorder

The recorder is connected to the connection sockets (18) at the rear of the instrument, either to output 0...10 mV or 0...1 V. The polarity of the output signal at connection sockets (18) can be changed with the polarity selector switch (20) at any time.

If the METROHM 586 Labograph is used as recorder (see Section 8.4.4), the connection is made with the 6.2115.010 cable according to Fig. 15.

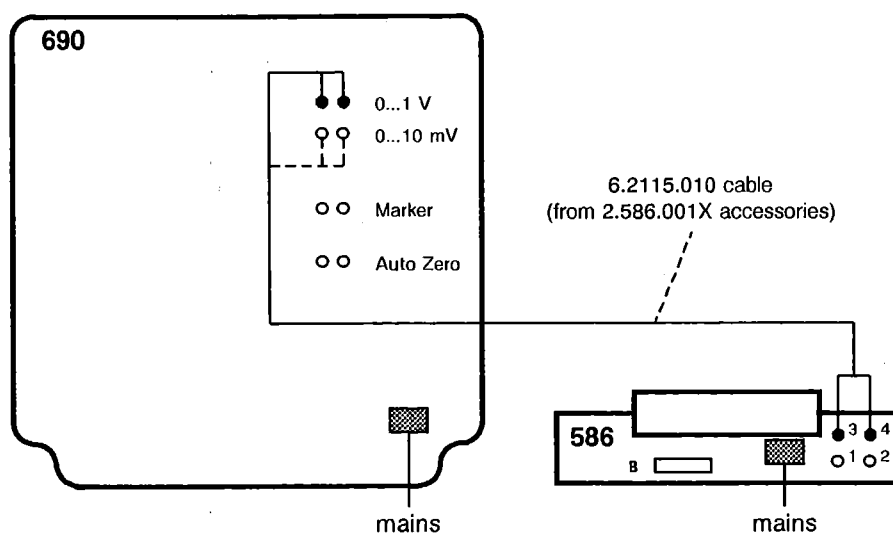


Fig.15: Connection of 586 Labograph at 690 Ion Chromatograph

3.10. Connection of integrator/PC

3.10.1. Measuring signal

An integrator or PC is connected to the connection sockets (18) at the rear of the instrument (see Fig. 16), either to output 0...10 mV or 0...1 V with a special integrator cable (normally part of the integrator accessories). You can also use a 6.2115.030 cable (option, see Section 8.2).

The polarity of the output signal at connection sockets (18) can be changed with the polarity selector switch (20) at any time.

3.10.2. Automatic integrator start

With the 690.0020 and 690.0030 versions the integrator can be started automatically when the electrical injector valve is put into position "Inject". For that the integrator must be connected to the connection sockets (35) "Integrator Start" by means of the 6.2115.030 cable (see Fig. 16, for technical specifications see Section 7.2).

With the 690.0030 Ion Chromatograph with two injectors there are three possibilities for connection on an integrator/PC (see Fig. 7):

- connection sockets (35) on connector block A (49) for injector A
- connection sockets (35) on connector block B (48) for injector B
- connection sockets (35) on connector block A/B (50) for injector A or B (selection A ↔ B by switch (43))

3.10.3. External control of injectors

With the 690.0020 and 690.0030 versions the electrical injectors can be triggered externally (e.g. Autosampler, PC). Connect the external apparatus to the connection sockets Ground (36), Fill (37) and Inject (38) (see Fig. 5 or Fig. 7, for technical specifications see Section 7.2).

With the 690.0030 Ion Chromatograph with two injectors there are three possibilities for connection on an integrator/PC (see Fig. 7):

- connection sockets (35) on connector block A (49) for injector A
- connection sockets (35) on connector block B (48) for injector B
- connection sockets (35) on connector block A/B (50) for injector A or B (selection A ↔ B by switch (43))

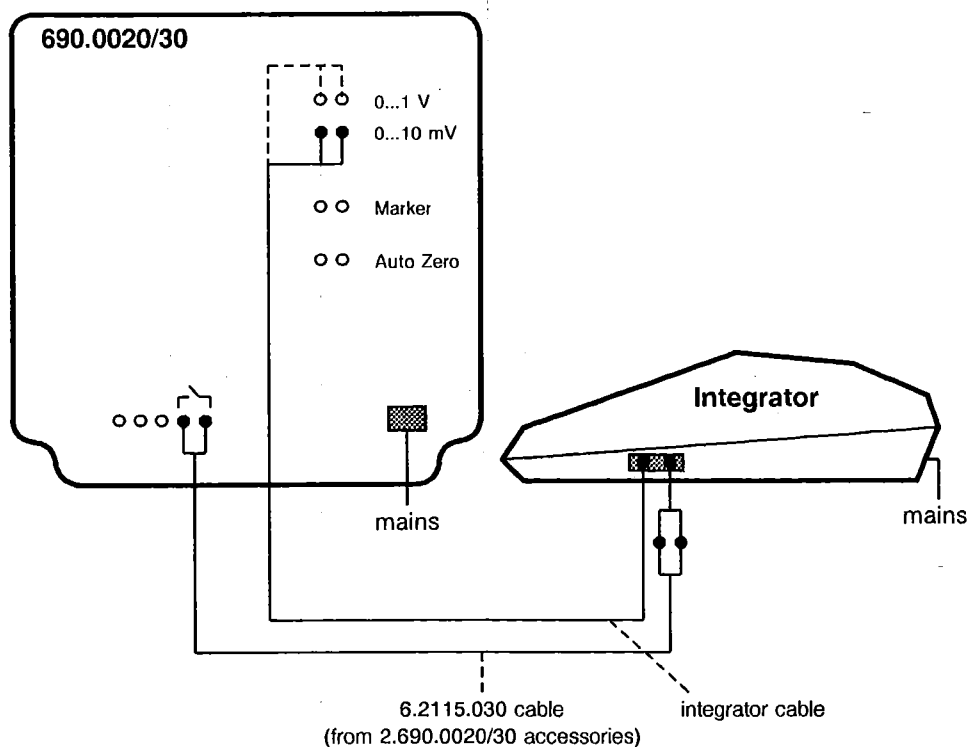


Fig.16: Connection of an integrator at the 690 Ion Chromatograph

3.11. Connection of 698 Autosampler

The 690.0020 and 690.0030 Ion Chromatographs with electrically operated injectors are ready for the connection of the 698 Autosampler (the 690.0010 Ion Chromatograph with manually operated injector must be converted first to version 690.0020 by means of the 6.5848.000 conversion kit).

For the installation and connection of the 698 Autosampler refer to 8.698.1013 Instructions for use.

3.12. Connection of additional detectors

If connection of an additional detector (e.g. 656 Electrochemical Detector) after the conductivity detector of the 690 Ion Chromatograph is desired, it must be taken into account that the coupling at output (30) "Waste" has a large dead volume. The PTFE microcapillary tubing attached to the conductivity detector must therefore be unscrewed from this coupling in the interior, shortened if necessary and connected directly with the inlet of the auxiliary detector. If the auxiliary detector is positioned in the interior, any electrical connecting cables can be led to the outside through openings (17) or (29).

3.13. Conditioning the system

Before any sample solutions can be injected, the whole system with column and detector must be conditioned until a stable baseline is obtained. First, the following adjustments are made with the pump switched on:

- 1▶ **Range:** The measuring range (0...10 to 0...1000 $\mu\text{S}/\text{cm}$) is set with control knob (3) such that the displayed eluent conductivity value (Eluent Conduct. (1)) falls within the selected range.

If the conductivity of the eluent is well above the upper value of the range, "/ " (digit 1 with no following digits) appears in display (1) (Eluent Conduct.). The next highest range must be set.

- 2▶ **Sensitivity:** The sensitivity (1...2000) is selected with control knob (4) such that the displayed full scale range (2) ($\mu\text{S}/\text{cm}$) corresponds with the desired working range.

To begin with, it is advisable to select a full scale range which is not too small as the conductivity of the eluent can still change drastically in the conditioning phase before the temperature has stabilized.

- 3▶ **Thermostat:** The heating of the detector block is switched on with toggle switch (10), and this is indicated by illumination of green lamp (9).

- 4▶ **Damping:** The damping is set to the desired stage with control knob (5).
Work can normally be carried out using setting 0. If the pump pulsations are excessive, the signal damping can be increased.

- 5► Auto Zero:**
- Briefly press toggle switch (7) downwards: the current measuring signal is set automatically at analogue output (18) to 0 V (electronic background compensation). When the background compensation is switched on, green lamp (6) is lit up.
 - Set the zero point on the recorder and/or integrator to the desired position (Offset).
 - To switch the Auto Zero function off, push toggle switch (7) upwards briefly; green lamp (6) is then extinguished.

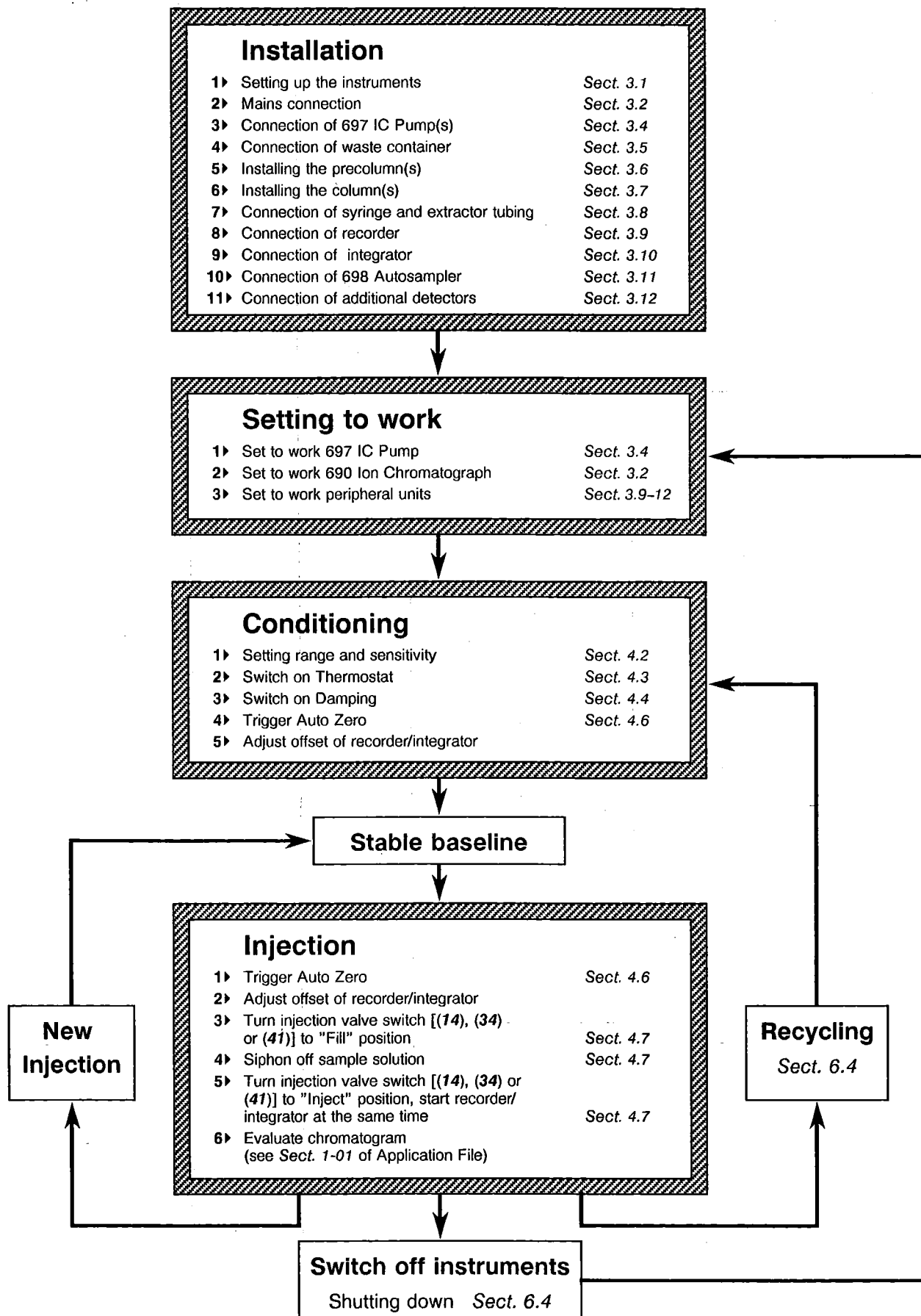
If red lamp (8) (overload display) lights up, this signifies that the measuring signal lies outside $\pm 150\%$ of the selected full scale range. In this case, either the Auto Zero switch (7) must again be pressed downwards or the sensitivity reduced using control knob (4).

Note: The Auto Zero function works only if the measured value remains relatively stable during the zeroing (e.g. not during the injection peak).

Depending on the sensitivity selected, it takes 30...60 min for the desired stability to be reached and hence before the IC system is ready to perform analyses. (If the eluent is changed, establishment of the ion exchange equilibrium on the column can also take a longer time.)

4. Operation

4.1. Sequence of operations



4.2. Setting range and sensitivity

4.2.1. Range

The measuring range is set using control knob (3) (**Range**). Seven positions from 0...10 $\mu\text{S}/\text{cm}$ up to 0...1000 $\mu\text{S}/\text{cm}$ are available. The measuring range must be chosen such that the conductivity value of the eluent employed falls within the selected range. The measured value of the eluent conductivity (**Eluent Conduct.**) then appears in display (1).

If the measured conductivity value clearly exceeds the upper range value, " / " (digit 1 at the extreme left with no following digits) appears in display (1). The next highest range must be set.

4.2.2. Sensitivity

The sensitivity can be set using control knob (4) (**Sensitivity**) in 11 positions from 1...2000. It is selected such that the full scale range ($\mu\text{S}/\text{cm}$) which appears in display (2) corresponds to the desired working range.

4.2.3. Full Scale

The full scale range (or working range), which is shown in display (2), is defined by the settings of **Range** and **Sensitivity**:

$$\text{Full Scale} = \frac{\text{Range}}{\text{Sensitivity}}$$

Fig. 17 shows a pictorial representation of these quantities. In the example plotted with a measuring range of 500 $\mu\text{S}/\text{cm}$ and a sensitivity of 2000, the full scale range is $500/2000 = 0.25 \mu\text{S}/\text{cm}$.

The maximum possible full scale range is limited to 1000 $\mu\text{S}/\text{cm}$; the minimum range is 0.1 $\mu\text{S}/\text{cm}$. If values below this lower limit are found, (which would theoretically be possible with an appropriate choice of **Range** and **Sensitivity**), " / " (digit 1 at the extreme left with no following digits) appears in display (2). **Range** and/or **Sensitivity** must then be reset.

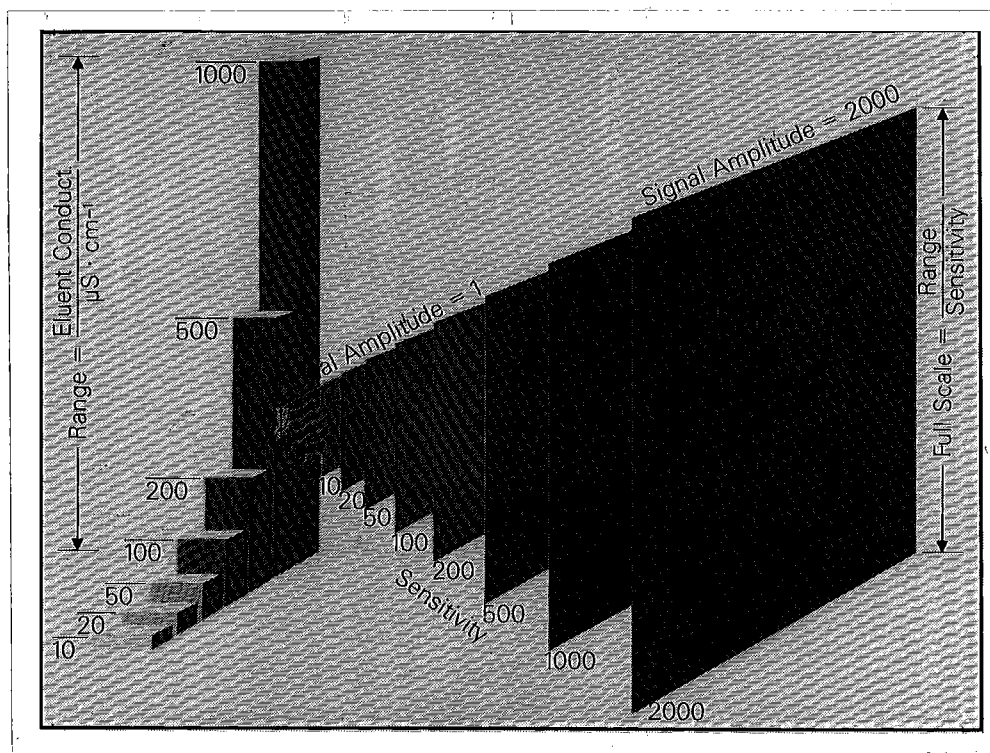


Fig. 17: Measuring range and sensitivity

4.3. Thermostatting

4.3.1. Switching on

The thermostatting of the conductivity measuring cell is switched on by pressing switch (10) downwards. The green indicator lamp (9) is lit when the thermostatting is operative.

4.3.2. Operating temperature

The operating temperature is set in the factory to 35 ± 1 °C and should not be changed without good reason. When the ambient temperature is constant, the heating built into the 690 Ion Chromatograph controls this measuring cell temperature to an accuracy of ± 0.01 °C thereby furnishing the prerequisite for highly sensitive determinations. Normally, 30...60 min must elapse after switching on before this temperature stability is reached.

To **change the operating temperature** of the Ion Chromatograph, the adjustment screw (19) at the rear of the instrument must be turned in a clockwise (temperature raising) or anticlockwise (temperature lowering) direction using a screwdriver. The range within which the temperature can be varied is 25...45 °C. If it is necessary to check the set temperature, this must be performed with a suitable temperature sensor, which can be inserted in opening (58) of the detector block (54) (see Fig. 13).

The measured conductivity value is always corrected to 20 °C with a fixed **correction factor** of 2.3%/°C independent of the set operating temperature. If the ambient temperature is constant, it is therefore also possible to work without thermostatting.

4.3.3. Switching off

The thermostatting of the conductivity measuring cell is switched off by pushing switch (10) upwards; green indicator lamp (9) goes out. It takes a relatively long time for the measuring cell to reach room temperature again.

4.4. Damping

A Butterworth filter (4th order) is built into the 690 Ion Chromatograph for electronic damping of the measuring signal. The damping can be set at four levels with control knob (5) (τ = time constant):

stage 0:	$\tau = 0.125$ s
stage 1:	$\tau = 0.5$ s
stage 2:	$\tau = 1$ s
stage 3:	$\tau = 2$ s

With pulse-free pumps with a constant flow rate, work is normally carried out at **stage 0**. If, during sensitive measurements, disturbing pulses are found or severe noise appears, an attempt can be made to diminish such disturbances by increasing the damping in stages. Standard solutions and samples must always be recorded with the same damping stage since the damping can influence the peak heights.

4.5. Marker

The Marker function serves to set marking spikes of 10% of the full scale range within the chromatogram. To trigger the Marker function, switch (11) is pressed downwards briefly. For external control of the Marker function via connection sockets (21) see Section 7.2.

4.6. Auto Zero

4.6.1. Function

"Auto Zero" is the term used to describe the **automatic electronic background compensation of the measuring signal**, i.e. the current measured conductivity value becomes the new zero point of the selected full scale range. Fig. 18 shows a pictorial representation of the mode of operation of the Auto Zero function. Before first-time actuation or after switching off the Auto Zero function, the zero point of the full scale range (FS) is set to 0 $\mu\text{S}/\text{cm}$; by pressing the Auto Zero switch (7), it is set automatically to the current measured value. The Auto Zero function can also be triggered externally via connection sockets (22) (see Section 7.2).

4.6.2. Triggering

To trigger the Auto Zero function, press switch (7) downwards briefly. Green lamp (6) indicates that the electronic background compensation is in operation. (Red overload lamp (8) may light up briefly during the zeroing.)

Note: The Auto Zero function works properly only if the measured value remains relatively stable during the zeroing (e.g. not during the injection peak).

4.6.3. Switching off

Push the Auto Zero switch (7) upwards briefly. Green lamp (6) goes out. The zero point of the full scale range is again set to 0 $\mu\text{S}/\text{cm}$.

4.6.4. Overload

Illumination of the red overload lamp (8) shows that the measured value lies outside $\pm 150\%$ of the full scale range selected (see Fig. 18).

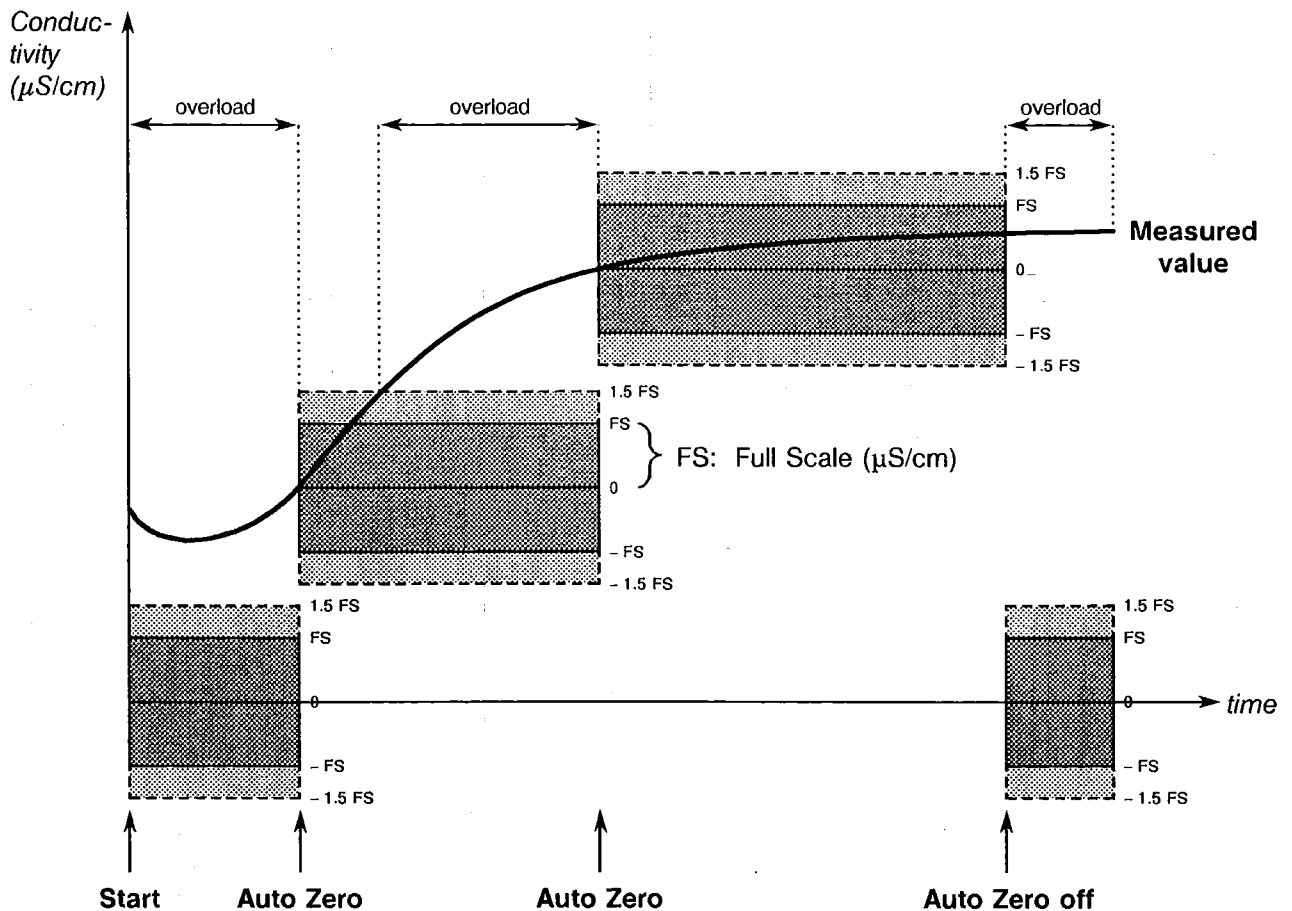


Fig. 18: Auto Zero

4.7. Injection of samples

4.7.1. Operating principle of the injector

- 690.0010 Ion Chromatograph:

A **manually** operated VALCO loop injection valve with a **100 µL loop** is built into the 690.0010 Ion Chromatograph for the injection of samples. If other sample volumes need to be injected, this loop must be replaced by a commercially available loop with the desired volume (e.g. 6.2620.080 sample loop 10 µL). Fig. 19 shows a schematic representation of the operating principle of the injector in the positions "Fill" and "Inject".

- 690.0020 Ion Chromatograph:

A VALCO loop injection valve – triggered **electrically** either by pressing switch (34) or externally by the 698 Autosampler – with a **100 µL loop** is built into the 690.0020 Ion Chromatograph for the injection of samples. If other sample volumes need to be injected, this loop must be replaced by a commercially available loop with the desired volume (e.g. 6.2620.080 sample loop 10 µL). Fig. 19 shows a schematic representation of the operating principle of the injector in the positions "Fill" and "Inject".

- 690.0030 Ion Chromatograph:

Two VALCO loop injection valves **A** and **B** – triggered electrically either by pressing switch (34) or (41) or externally by the 698 Autosampler – are built into the 690.0030 Ion Chromatograph for the injection of samples. Injector **A** has a **100 µL loop**, injector **B** a **10 µL loop**. If other sample volumes need to be injected, this loop must be replaced by a commercially available loop with the desired volume (e.g. 6.2620.080 sample loop 10 µL or 6.2620.090 sample loop 100 µL). Fig. 20 shows a schematic representation of the operating principle of the injectors in the positions "Fill" and "Inject".

4.7.2. Triggering the injector

- 690.0010 Ion Chromatograph:

- 1▶ Turn the injection valve switch (14) at the front of the 690.0010 Ion Chromatograph to the "Fill" position.
Control knob (14) must be turned quickly otherwise the pressure increases rapidly within a short space of time and this increase in pressure in the system causes the upper pressure limit sensor to signal automatic shutdown of the pump.
- 2▶ Immerse the extractor tubing attached to connector (15) in the sample solution.

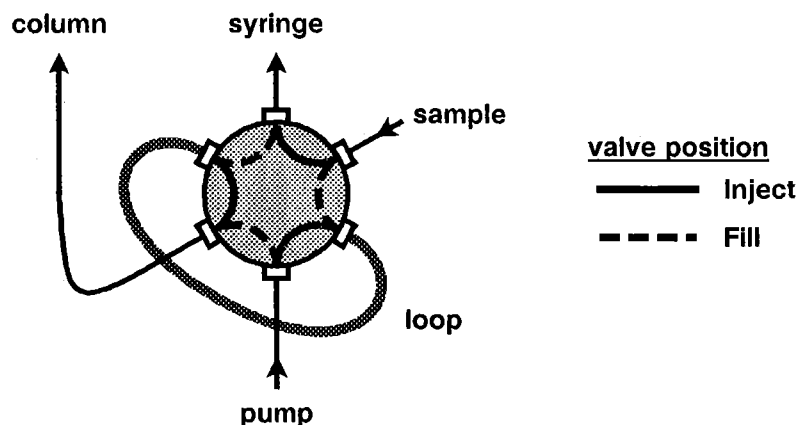


Fig. 19: Operating principle of the injector at the 690.0010/690.0020 Ion Chromatographs

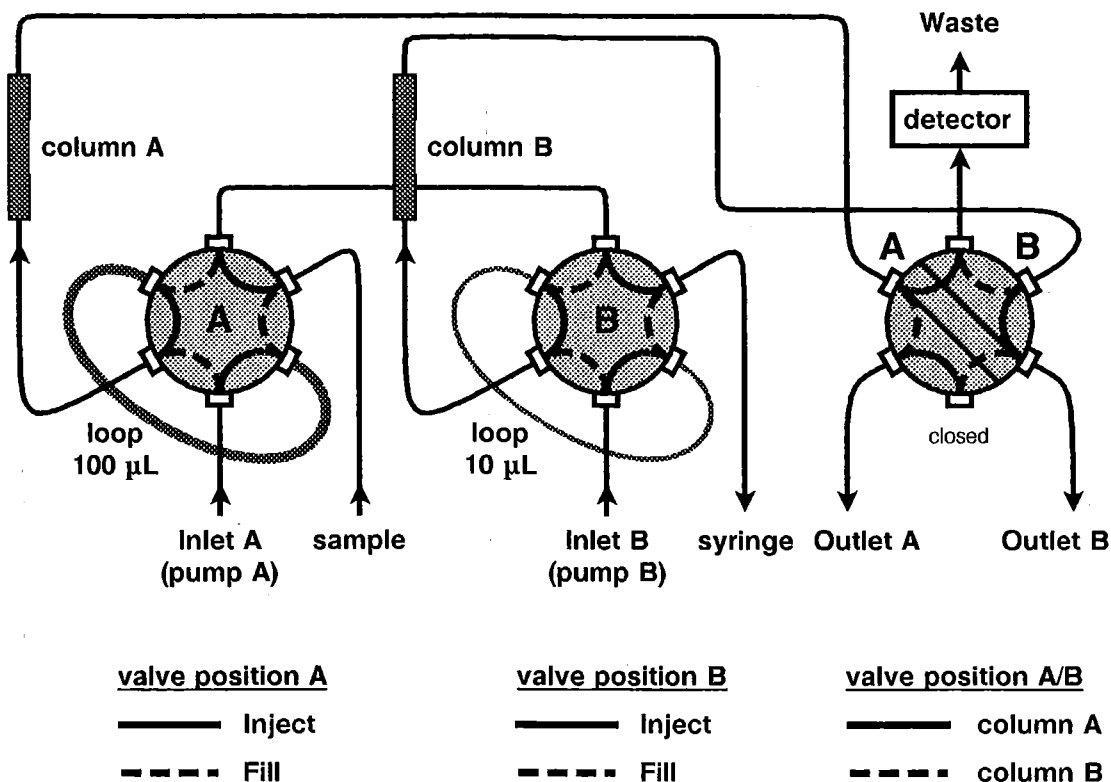


Fig. 20: Operating principle of the injector at the 690.0030 Ion Chromatograph (view from the front side of the instrument)

- 3▶ Siphon off the sample with the syringe attached to connection (16).
To avoid carryover of previous samples, it is advisable to siphon off a sufficiently large quantity of sample. When large concentration differences exist between samples, it is best to flush out the extractor tubing with dist. water between injections.
- 4▶ Turn the injection valve switch (14) to the "Inject" position (with sufficient rapidity, see above).
- 5▶ Start recorder and integrator manually at the same time.

● 690.0020 Ion Chromatograph:

- 1▶ Press injection valve switch (34) at the front of the 690.0020 Ion Chromatograph upwards briefly.
The red lamp (32) indicates that the injection valve is in position "Fill".
The injection valve can also be triggered externally by the 698 Autosampler (see Sect. 3.10.3 and 8.698.1013 Instructions for use).
- 2▶ Immerse the extractor tubing attached to connector (15) in the sample solution.
- 3▶ Siphon off the sample with the syringe attached to connection (16).
To avoid carryover of previous samples, it is advisable to siphon off a sufficiently large quantity of sample. When large concentration differences exist between samples, it is best to flush out the extractor tubing with dist. water between injections.

- 4▶ Press injection valve switch (34) at the front of the 690.0020 Ion Chromatograph downwards briefly.
The red lamp (33) indicates that the injection valve is in position "inject".
The injection valve can also be triggered externally by the 698 Autosampler (see Sect. 3.10.3 and 8.698.1013 Instructions for use).
 - 5▶ Start recorder and integrator manually at the same time.
An integrator connected to the sockets (35) is started automatically by triggering the injection valve (see Section 3.10.2).
- 690.0030 Ion Chromatograph (injector/column A or [B])
- 1▶ Put column selection valve (43) to position A [B].
 - 2▶ If peripheral units are connected to connector block A/B (50), put the injector selection switch (42) to position A [B].
 - 3▶ Press injection valve switch (34) [(41)] at the front of the 690.0030 Ion Chromatograph upwards briefly.
The red lamp (32) [(39)] indicates that the injection valve A [B] is in position "Fill".
The injection valve can also be triggered externally by the 698 Autosampler (see Sect. 3.10.3 and 8.698.1013 Instructions for use).
 - 4▶ Immerse the extractor tubing attached to connector (15) in the sample solution.
 - 5▶ Siphon off the sample with the syringe attached to connection (16).
If both injectors are in position "Fill", both sample loops are filled at the same time.
To avoid carryover of previous samples, it is advisable to siphon off a sufficiently large quantity of sample. When large concentration differences exist between samples, it is best to flush out the extractor tubing with dist. water between injections.
 - 6▶ Press injection valve switch (34) [(41)] at the front of the 690.0030 Ion Chromatograph downwards briefly.
The red lamp (33) [(40)] indicates that the injection valve A [B] is in position "Inject".
The injection valve can also be triggered externally by the 698 Autosampler (see Sect. 3.10.3 and 8.698.1013 Instructions for use).
 - 7▶ Start recorder and integrator manually at the same time.
An integrator connected to the sockets (35) is started automatically by triggering the injection valve (see Section 3.10.2).

5. Example: Determination of chloride, nitrate and sulphate in drinking water

A detailed description of the analysis of a drinking water sample with a 6.1005.000 anion column PRP-X100, which can also be found in *Application Bulletin No. 150 (IC 3-01 in the 8.690.2013 Application File)*, is given in what follows as an example of the recording and evaluation of an ion chromatogram.

5.1. Preliminaries

The ion chromatography system is set up as described in *Section 3* with the following apparatus being used in addition to the 690 Ion Chromatograph:

- Column: 6.1005.000 IC anion column PRP-X100 (see *Section 8.4.7*)
- Pump: 2.697.0010 IC Pump (see *Section 8.4.1*)
- Recorder: 2.586.001X Labograph (see *Section 8.4.4*)
- Integrator

The most important installation steps are once again described briefly below (details see sections mentioned).

5.1.1. Installing the 690 Ion Chromatograph

- | | |
|---|----------------------|
| 1▶ Setting up the instrument | <i>Section 3.1</i> |
| 2▶ Cheking the mains voltage | <i>Section 3.2.1</i> |
| 3▶ Mains connection | <i>Section 3.2.4</i> |
| 4▶ Switching on the instrument | <i>Section 3.2.5</i> |
| 5▶ Connection of the waste container | <i>Section 3.5</i> |
| 6▶ Connection of syringe and extractor tubing | <i>Section 3.8</i> |

5.1.2. Eluent preparation

- | | |
|--|--------------------|
| 1▶ Eluent preparation:
phthalic acid $c = 0.002 \text{ mol/L}$ in dist. water/acetone 90:10
(pH value of the solution adjusted to 5.0 with NaOH) | |
| 2▶ Microfiltration and degassing of the eluent | <i>Section 6.3</i> |
| 3▶ Stirring the eluent in the storage vessel | <i>Section 6.3</i> |

5.1.3. Installing the 697 IC Pump (*see 8.697.1003 Instructions for Use)

- | | |
|--|---------------------|
| 1▶ Setting up the pump | <i>Section 3.1*</i> |
| 2▶ Mounting the pump head | <i>Section 3.2*</i> |
| 3▶ Connecting the capillaries | <i>Section 3.3*</i> |
| 4▶ Mains connection | <i>Section 3.4*</i> |
| 5▶ Switching on the pump | <i>Section 3.4*</i> |
| 6▶ Deaerating the pump | <i>Section 3.5*</i> |
| 7▶ Setting the flow rate:
FLOW SET: 2.00 mL/min | <i>Section 4.2*</i> |
| 8▶ Setting the pressure limit values:
PRESSURE MIN: 0.5 MPa (= 5 bar)
PRESSURE MAX: 10.0 MPa (= 100 bar) | <i>Section 4.3*</i> |

5.1.4. Connection 697 IC Pump – 690 Ion Chromatograph

- 1▶ Mounting the pulsation dampener (option) Section 3.4.2
- 2▶ Installing the filter unit Manufit (option) Section 3.4.3
- 3▶ Capillary connection 697 IC Pump – 690 Ion Chromatograph (Fig. 11) Section 3.4.4
- 4▶ Rinse the capillary up to the lower column connection (60) or (68) for about 10 min Section 3.4.5

5.1.5. Installing the column

- 1▶ Screw the 6.1005.000 IC Anion Column to the lower column connection (60) or (68) Section 3.7.3
- 2▶ Rinse column with eluent for about 10 min Section 3.7.3
- 3▶ Screw the column to the upper column connection (64) or (70) Section 3.7.3
- 4▶ Switch on 697 IC Pump and check all connections for leaks Section 3.7.4

5.1.6. Connection of the recorder

- 1▶ Connect the 586 Labograph with the 6.2115.010 cable at the 690 Ion Chromatograph (Fig. 15) Section 3.9
- 2▶ Recorder settings (see 586 Instructions for use):
 - Voltage measuring range ΔU : 1000 mV
 - Chart speed: 5 mm/min
 - Zero setting U_0 : set to the desired position after pressing the Auto Zero switch
- 3▶ Setting the polarity of the output signal at the 690 Ion Chromatograph Section 3.9

5.1.7. Connection of the integrator

- 1▶ Connect the integrator with the supplied integrator cable at the 690 Ion chromatograph (Fig. 16) Section 3.10
- 2▶ Programming the integrator
- 3▶ Setting the polarity of the output signal at the 690 Ion Chromatograph Section 3.9

5.1.8. Settings at the 690 Ion Chromatograph

- 1▶ Setting range Section 4.2
 - Range: 500 $\mu\text{S/cm}$
- 2▶ Setting sensitivity Section 4.2
 - Sensitivity: 100 (\Rightarrow Full Scale = 5 $\mu\text{S/cm}$)
- 3▶ Switch on thermostat Section 4.3
 - Thermostat: On
- 4▶ Setting damping Section 4.4
 - Damping: 0

5.1.9. Conditioning

The entire system is now conditioned until the baseline has achieved the desired stability. With the selected flow rate and with the 6.1005.000 column, the pressure will be around 70 bar (7.0 MPa).

5.2. Calibration

5.2.1. Standard

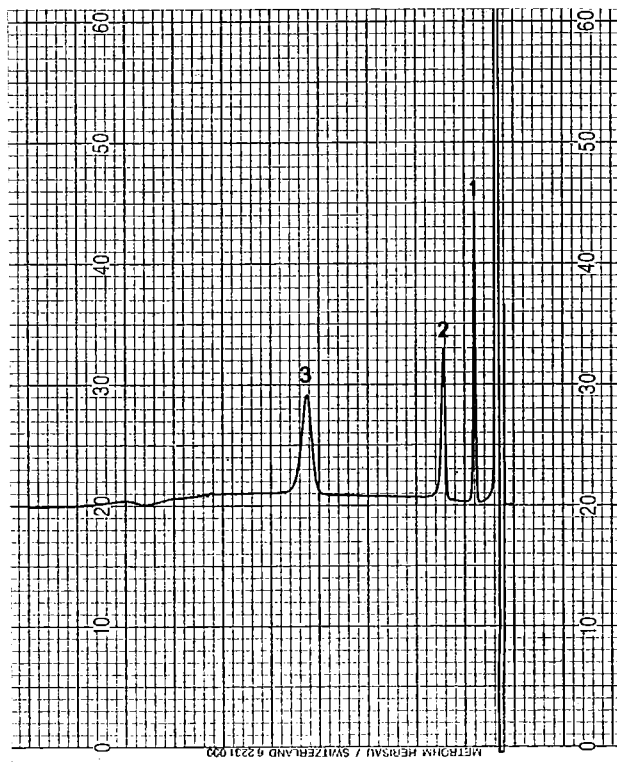
The following standard solution is used for calibration:

5 ppm Cl^- , 10 ppm NO_3^- , 10 ppm SO_4^{2-}
(as sodium or potassium salts in dist. water)

5.2.2. Injection

- 1▶ Press the "Auto Zero" switch on the 690 Ion Chromatograph. Reset the offset on the recorder and integrator if need be.
- 2▶ Turn injection valve switch (14) or (34) to position "Fill".
- 3▶ Immerse the extractor tubing attached to connection (15) in the standard solution.
- 4▶ With the aid of the syringe at connection (16), siphon off ca. 1 mL standard solution.
- 5▶ Turn injection valve switch (14) or (34) to position "Inject". At the same time, start the recorder and integrator.

Fig. 21 shows the chromatogram plotted with the 586 Labograph, whereas the chromatogram recorded and evaluated with the integrator is illustrated in Fig. 22.



- 1 5 ppm chloride
- 2 10 ppm nitrate
- 3 10 ppm sulphate

Fig. 21: Chromatogram of the calibration

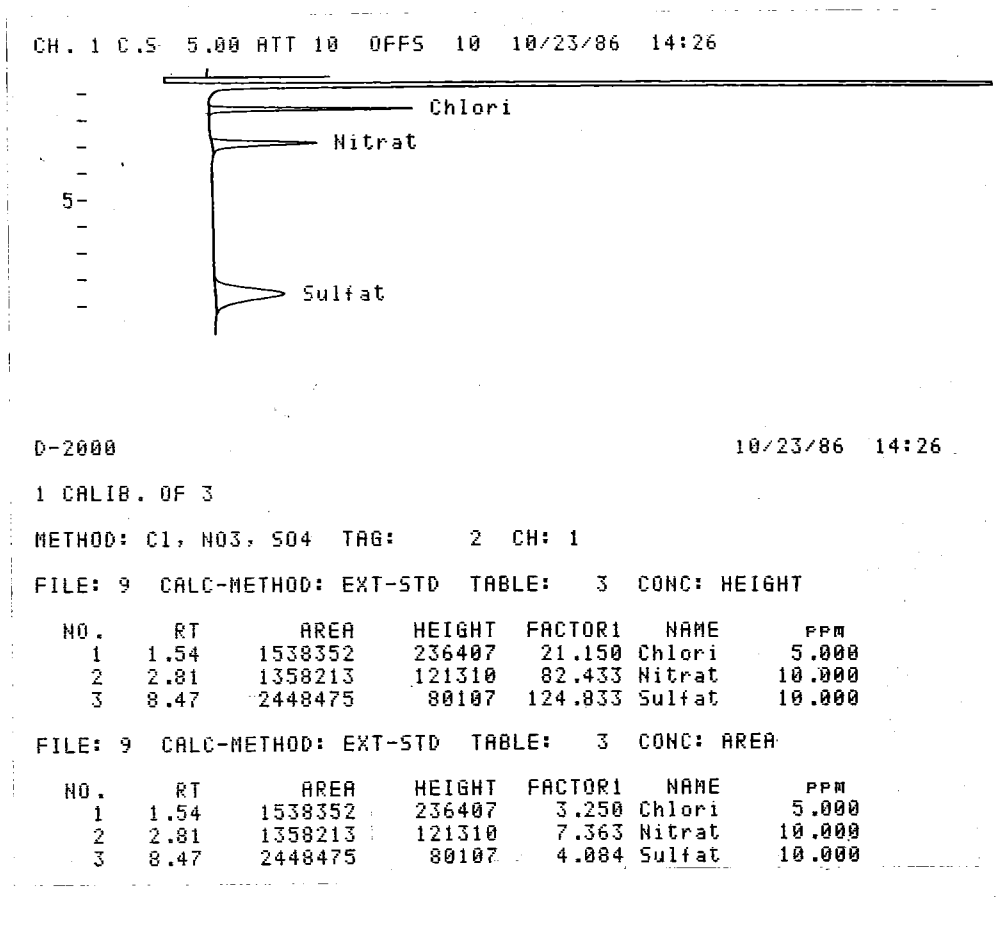


Fig. 22: Integrator evaluation of the calibration

5.3. Determination and evaluation

5.3.1. Injection of the drinking water sample

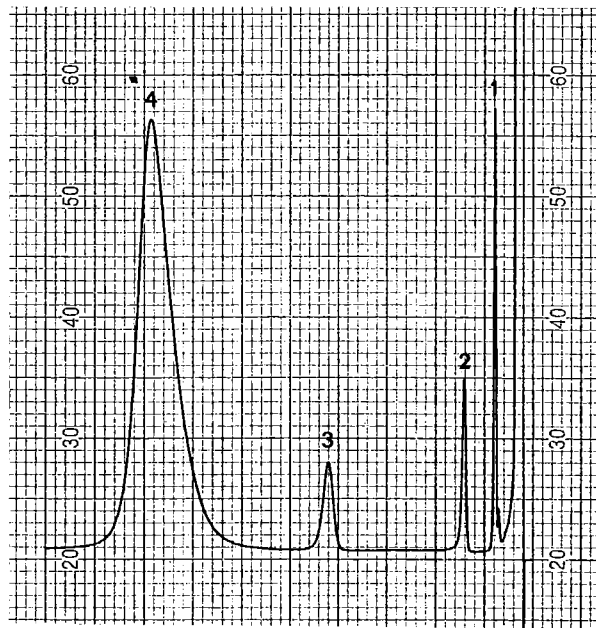
The drinking water sample is microfiltered (0.45 µm) before the injection. The injection is carried out in a manner analogous to the calibration (points 1▶...5▶, see Sect. 5.2.2). Fig. 23 shows the chromatogram plotted using the 586 Labograph, Fig. 24 the corresponding integrator evaluation.

5.3.2. Evaluation

The evaluation by the integrator is shown in Fig. 24. Evaluations of the areas and heights give very similar results.

The manual evaluation of the recorder chromatogram can be performed using the measured peak heights:

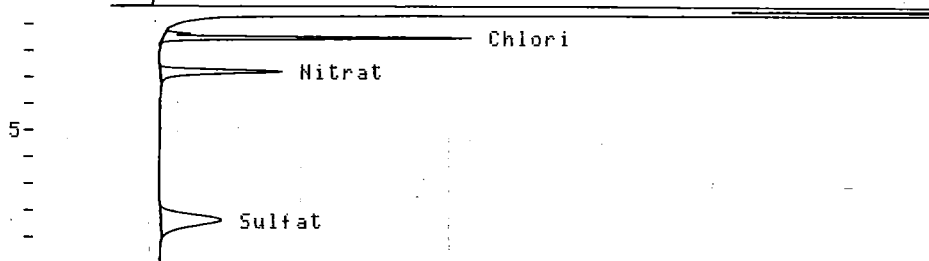
Ions	Standard		Drinking water sample	
	concentration	height	height	concentration
chloride	5 ppm	60 mm	90.5 mm	7.5 ppm
nitrate	10 ppm	31 mm	36 mm	11.9 ppm
sulphate	10 ppm	20.5 mm	18 mm	8.8 ppm



- 1 chloride
- 2 nitrate
- 3 sulphate
- 4 system peak

Fig. 23: Chromatogram of the drinking water analysis

CH. 1 C.S 5.00 ATT 10 OFFS 10 10/23/86 15:46



D-2000

10/23/86 15:46

METHOD: Cl, NO3, SO4 TAG: 6 CH: 1

FILE: 9 CALC-METHOD: EXT-STD TABLE: 3 CONC: HEIGHT

NO.	RT	AREA	HEIGHT	FACTOR1	NAME	PPM
2	1.52	2240860	356896	21.150	Chlori	7.548
3	2.77	1599556	140958	82.433	Nitrat	11.619
4	8.34	2080270	70077	124.833	Sulfat	8.747

FILE: 9 CALC-METHOD: EXT-STD TABLE: 3 CONC: AREA

NO.	RT	AREA	HEIGHT	FACTOR1	NAME	PPM
2	1.52	2240860	356896	3.250	Chlori	7.283
3	2.77	1599556	140958	7.363	Nitrat	11.776
4	8.34	2080270	70077	4.084	Sulfat	8.496

Fig. 24: Integrator evaluation of the drinking water analysis

6. Practical tips for ion chromatography

6.1. Columns

6.1.1. Separation efficiency

The analysis quality attainable with the 690 Ion Chromatograph depends to a large extent on the separation efficiency of the column used. The purchaser of an IC column should satisfy himself that the separation efficiency is adequate for the analysis problems at hand. The **characteristics of an IC column** such as capacity factors, selectivity, theoretical plate number and resolution (cf. Section "Theory of Ion Chromatography" in the 8.690.2013 Application File) should be specified on the standard chromatogram enclosed with every column and checked with one's own measurements. If difficulties arise, in each case the quality of the column must first be checked by recording a **standard chromatogram**.

The IC columns available from METROHM are listed in Section 8.4.7. For additional information concerning the columns refer to Section 1-02 of the 8.690.2013 Application File.

6.1.2. Protection

To protect the column from foreign bodies, which could adversely affect the separation efficiency, it is advisable to subject the eluent as well as all samples to **microfiltration** (filter 0.45 µm) and to siphon off the eluent through the **6.2821.060 suction filter**.

To avoid contamination of the piston seals by abrasive particles, it is advantageous to fit an **In-line filter** between the 697 IC Pump and the 690 Ion Chromatograph. The optional **6.2821.000 Manufit filter unit** (see Section 8.4.8) is extremely well suited to this purpose.

The use of easily exchangeable **precolumns** protects the separation columns and prolongs their life time. The precolumns available from METROHM (see Section 8.4.7) are either real precolumns or precolumn cartridges, which are used together with a 6.2821.050 twin cartridge holder or a 6.2821.040 cartridge head.

6.1.3. Storage

When not in use, ion chromatography columns should always be sealed and stored filled according to the manufacturer's instructions. Columns based on polymers can usually be stored filled with the eluent employed.

6.1.4. Dead volume

Dead volume at the ends of a column can be the cause of extreme peak broadenings or splitting (appearance of double peaks). Filling the column with glass beads (Ø 100 µm) can frequently help restore the separation properties.

6.1.5. Regeneration

If the separation properties of the column have deteriorated, it can be regenerated according to the column manufacturer's directions. The columns available from METROHM (see Section 8.4.7) all are supplied with a instruction sheet containing the regeneration procedures.

Caution: *With silica based columns don't use **alkaline eluents** for regeneration, otherwise the columns could be damaged!*

6.2. Pumps

6.2.1. Pulsation dampener

For sensitive measurements with the 690 Ion Chromatograph, high pressure pumps with a high flow rate stability and as free from pulsation as possible are needed. If the pulses found are too large, a pulsation dampener connected between the pump and the 690 Ion Chromatograph may possibly remedy the situation (e.g. the optional **6.2620.050 Portmann pulsation dampener**, see *Section 8.4.2*). In addition, the pulsation dampener protects the column material from pressure drops caused by the injector.

6.2.2. Maintenance

To protect the pump from foreign bodies, it is advisable to subject the eluent as well as all samples to **microfiltration** (filter 0.45 µm) and to siphon off the eluent through the **6.2821.060 suction filter**.

An unstable baseline (pulsation, flow rate variations) can be traced in many cases to dirty valves or defective, leaking piston seals.

Dirty valves are cleaned by flushing with water, RBS solution or organic solvents. The flushing treatment is reinforced by brief treatment in an ultrasonic bath. When the cleaned valves are reinstalled, it must be ensured that the flow direction is correct.

Pump seals are exchanged in accordance with the pump manufacturer's directions. For the 697 IC Pump, refer to *Section 5.2 "Maintenance work"* of the 8.697.1003 Instructions for use.

Salt crystals between piston and seal create abrasive particles which can get into the eluent. These lead to dirty valves, a rise in pressure and, in extreme cases, to scratched pistons. It is therefore absolutely essential to ensure that **no deposits** can form (see also *Section 6.3*).

6.3. Eluents

6.3.1. Treatment

Fresh eluents should always be subjected to **microfiltration** (filter 0.45 µm) and **degassed** (with N₂, He or vacuum). The eluent should be **stirred** continuously with a magnetic stirrer particularly when working with a closed loop (recycling) or when alkaline eluents are used.

The storage vessel containing the eluent must be sealed as tightly as possible to avoid excessive evaporation. This is especially important for eluents containing organic solvents (e.g. acetone), the evaporation of which can lead to long-term drift. If work is carried out in a very sensitive range, even a single drop of condensate falling back into the eluent can cause a perceptible change in the background conductivity.

6.3.2. Influence of various parameters on anion columns

- **concentration:** An increase in the concentration leads as a rule to shorter retention times and a more rapid separation but also to higher background conductivity.
- **pH:** A higher eluent pH shortens the retention times.
- **organic modifier:** Addition of an organic solvent (e.g. methanol, acetone, acetonitrile) to aqueous eluents generally lengthens the retention times. Divalent anions are usually more strongly influenced than monovalent anions.

6.3.3. Changing the eluent

When changing the eluent, it must be ensured that **no deposits** can be formed. Successive solutions must therefore be miscible. If it is necessary to flush the system with an organic reagent, several solvents with increasing or decreasing lipophilic character must therefore be used if possible (e.g. water ↔ acetone ↔ chloroform).

6.4. 690 Ion Chromatograph

6.4.1. Passivation

Passivation of the entire IC system (without column) by flushing with 20 ... 50 mL 0.2 mol/L HNO₃ is appropriate in the following cases:

- before switching to cation chromatography
- after working with ethylenediamine eluents
- when exceptional changes in the measuring properties of the cell are observed

The column is removed from the 690 Ion Chromatograph for passivation. The two connections (60) and (64) [or (68)/(70) and (73)/(75) resp. at the 690.0030 Ion Chromatograph] are connected directly with each other using the 6.2620.060 coupling supplied with the accessories.

6.4.2. Connections

All connections between injector, column and detector must be as short as possible, have minimum dead volumes and be absolutely tight. The PTFE capillary after the detector block must have no constrictions (the measuring cell has been tested to 50 bar back pressure).

6.4.3. Auto Zero

The Auto Zero switch functions properly only if the measured value remains relatively stable during zeroing (e.g. not during an injection peak). The output is set to 0 V during zeroing.

6.4.4. Recycling (closed circuit)

To keep the eluent consumption as low as possible during the idle state between injections (e.g. overnight), the so-called "recycling" procedure can be used. In recycling, the eluent flowing out of connection (30) [or (45) and (47) at the 690.0030 Ion Chromatograph] is led back directly into the eluent storage vessel. In this manner, the IC system is quickly ready for new injections without a long conditioning period.

Caution: Do **not** use the recycling procedure

- with alkaline eluents
- with the 6.1009.020 IC cation column SUPER-SEP

6.4.5. Shutting down

If the 690 Ion Chromatograph is shut down for a lengthy period of time, the entire IC system (with-out column) must be flushed with dist. water **free from salts**.

To flush the IC system remove the column; connect the two column connectors (60) and (64) [or (68)/(70) and (73)/(75) resp. at the 690.0030 Ion Chromatograph] by means of the 6.2620.060 Coupling. Flush with dist. water until the conductivity drops below 10 µS/cm.

At the end the system is flushed with methanol/water (1:4).

6.5. Malfunctions and their rectification

If difficulties appear in analyses with the ion chromatography system, their causes are best investigated in the order **column** → **pump** → **eluent** → **690 Ion Chromatograph**. Some of the malfunctions that can appear are listed in the following Table with details concerning possible causes and countermeasures.

Malfunction	Cause	Rectification
very noisy baseline, pulsation	<ul style="list-style-type: none"> • dirty pump valves • defective pump seals • quality of pump insufficient for selected sensitivity 	<ul style="list-style-type: none"> • clean valves (see <i>Section 6.2</i>) • replace pump seals (see <i>Section 6.2</i>) • use pulsation dampener, use more efficient pump or lower sensitivity
baseline drift	<ul style="list-style-type: none"> • thermal equilibrium not yet attained • leak in system • evaporation of organic solvent in eluent 	<ul style="list-style-type: none"> • condition system with heating switched on • check connections and make leakproof • seal eluent storage vessel better
marked pressure drop	<ul style="list-style-type: none"> • leak in system 	<ul style="list-style-type: none"> • check connections and make leakproof
marked pressure rise	<ul style="list-style-type: none"> • contamination of column inlet filter • contamination of filter in the 6.2821.000 Manufit Filter Unit • change in column packing due to injection of contaminated samples 	<ul style="list-style-type: none"> • clean or replace 6.2821.020 steel mesh(es) • clean or replace 6.2821.020 steel mesh(es) • regenerate column (see <i>Section 6.1</i>) or replace it <i>Note: samples must always be subjected to microfiltration</i>
chromatograms with poor resolution, change in the retention times	<ul style="list-style-type: none"> • deteriorated separation efficiency of IC column 	<ul style="list-style-type: none"> • regenerate column (see <i>Section 6.1</i>) or replace it
extreme peak broadening, splitting (double peaks)	<ul style="list-style-type: none"> • dead volume at column ends 	<ul style="list-style-type: none"> • fill column with glass beads ($\varnothing \leq 100\mu\text{m}$) or replace it

7. Technical Data

7.1. Technical Data of the 690 Ion Chromatograph

- **Measuring range (Range)** 0...10, 20, 50, 100, 200, 500, 1000 $\mu\text{S}/\text{cm}$

- **Sensitivity** 1 \times , 2 \times , 5 \times , 10 \times , 20 \times , 50 \times , 100 \times , 200 \times , 500 \times , 1000 \times , 2000 \times

- **Displays**
 - Eluent Conduct. Conductivity of the eluent in $\mu\text{S}/\text{cm}$
 - Full Scale Full scale deflection in $\mu\text{S}/\text{cm}$
(Full Scale = Range/Sensitivity)
Minimum value = 0.1 $\mu\text{S}/\text{cm}$

- **Detector**
 - Principle Thermostatted conductivity detector with 2 annular steel electrodes, alternating current measurement (measurement frequency = 1 kHz)
 - Cell volume 1.5 μL
 - Cell constant 17 cm^{-1}
 - Max. operating pressure 50 bar
 - Linearity up to $\pm 150\%$ of each full scale range deviations < 0.5% of full scale range
 - Drift (electronic) < 0.0013% of the selected measuring range/ $^{\circ}\text{C}$
 - Noise (electronic) typically: < 0.0003% of the set measuring range

- **Block heating**
 - Power up manual, operation indicated by green LED lamp
 - Block temperature 35 ± 1 $^{\circ}\text{C}$ (can be set to 25...45 $^{\circ}\text{C}$)
 - Temperature stability at constant ambient temperature $\leq \pm 0.01$ $^{\circ}\text{C}$

- **Temperature correction** Automatic correction of the measured conductivity to 20 $^{\circ}\text{C}$ with a temperature coefficient of 2.3%/ $^{\circ}\text{C}$

- **Output signal**
 - Analogue signal 0...1 V/full scale and 0...10 mV/full scale with 50% range reserve
 - Polarity switchable manually

- **Marker** ca. 10% of full scale range, triggered manually or externally

- **Auto Zero** Automatic zeroing (electronic background compensation) over entire selectable measuring range, triggered manually or externally
max. error: $\pm 0.6\%$ of full scale range

- **Damping**

Low-pass filter (Butterworth, 4th order)
stage 0: $\tau = 0.125$ s
stage 1: $\tau = 0.5$ s
stage 2: $\tau = 1$ s
stage 3: $\tau = 2$ s

- **Housing**

Polyurethane (PUR) rigid foam, thermally insulated interior for injector, column and detector

- **Injector**

690.0010 version: manually operated VALCO loop injection valve with 100 μ L loop (other loop volumes can be fitted)
690.0020 version: electrically operated VALCO loop injection valve with 100 μ L loop
690.0030 version: 2 electrically operated VALCO loop injection valves
injector A: 100 μ L loop
injector B: 10 μ L loop
(other loop volumes can be fitted)

- **Safety specifications**

In compliance with IEC publication 348

- **Mains connection**

Mains voltage $U_{\sim} = 100, 120, 220, 240$ V ± 10 %, switchable
Mains frequency $f = 50 \dots 60$ Hz
Power consumption
690.0010 version: max. continuous power consumption ca. 50 VA
690.0020/30 versions: max. continuous power consumption ca. 50 VA
(when the actuator switches, peak loads of max. 0.5 A mean value and 1.2 A maximum value can appear during max. 180 ms)
Fuse 5 mm \varnothing , 20 mm long
100 and 120 V: 0.63 A (slow-blow)
220 and 240 V: 0.315 A (slow-blow)

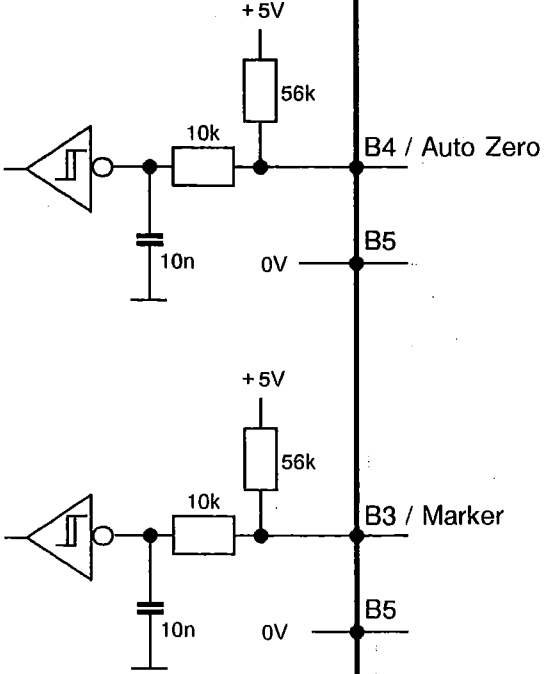
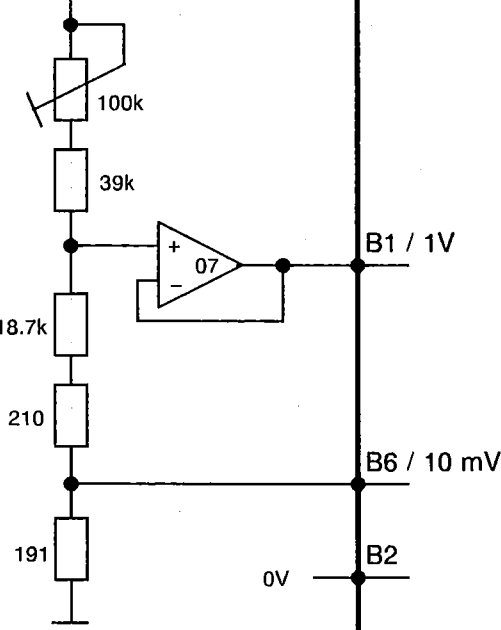

- **Dimensions**

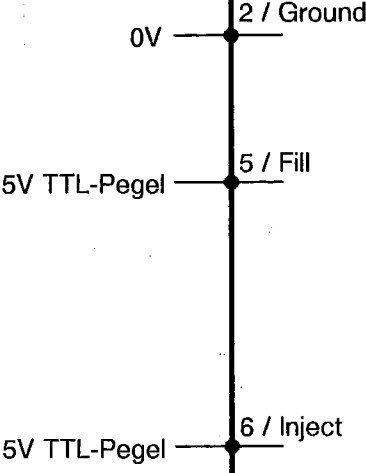
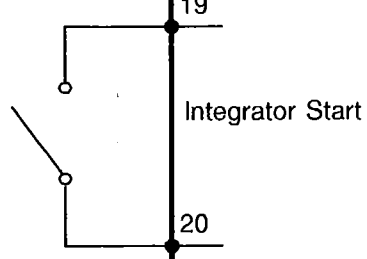

Width 435 mm
Height 430 mm
Depth 370 mm

- **Weight**

15 kg

7.2. Control inputs and outputs

3.690.0030	externally	Function
<p>Inputs</p> 		<p>L = Auto Zero on</p> <p>L = Marker</p>
<p>Analogue output</p> 		<p>Analogue output 0...1 V</p> <p>Analogue output 0...10 mV</p>
<p>Date 14.5.87/pa</p> 	<p>Control inputs and outputs</p> <p>690 Ion Chromatograph</p> <p style="text-align: right;">3.690.0030/690 4 E 1</p>	

A.601.0010 Actuator	externally	Function
<p>Inputs</p>		<p>The injection valve is switched to the "Fill" position.</p> <p>The injection valve is switched to the "Inject" position.</p>
<p>Ausgänge</p>		<p>When the injection valve has reached the "Inject" position, the switch is closed for 100 ms.</p>
<p>Date 27.4.89/um</p> 	<p>Control inputs and outputs</p> <p>6.5848.000 Autoinjector for 690.0020/30 Ion Chromatograph</p> <p style="text-align: right;">A.601.0010/690 4 E 2</p>	

8. Scope of Delivery and Ordering Designations

8.1. 690.0010 Ion Chromatograph

Ion Chromatograph with manually operated injector	2.690.0010
instrument including following accessories:	
1 × PTFE extractor tubing with connector, i.d. = 0.9 mm, e.d. = 1.5 mm, length $L = 40$ cm	6.1822.000
1 × PTFE microcapillary tubing (outlet tubing) with nipple and flange, i.d. = 0.3 mm, e.d. = 1.5 mm, length $L = 1$ m	6.1822.010
1 × Compression fitting for $1/16$ " steel capillary, set of 5	6.2620.000
1 × Ferrule for $1/16$ " steel capillary, set of 5	6.2620.010
1 × Steel capillary, i.d. = 0.25 mm, e.d. = $1/16$ ", length $L = 3$ m	6.2620.020
1 × Coupling for $1/16$ " steel capillary	6.2620.060
1 × Adjustable spanner, max. width 20 mm	6.2621.000
1 × Open-end spanner $1/4$ " × $5/16$ " for 6.2620.000 compression fitting	6.2621.010
1 × Spanner for plastic nipple for extractor and outlet tubing	6.2739.000
1 × Syringe, volumen $V = 10$ mL	6.2816.020
1 × Needle for 6.2816.020 Syringe	6.2816.030
1 × Mains cable cable socket type CEE (22), V; cable plug to order:	
type SEV 12 (Switzerland ...)	6.2122.020
type CEE (7), VII (Germany FR ...)	6.2122.040
type NEMA/ASA (USA ...)	6.2122.070
1 × Instructions for use (english)	8.690.1033
1 × Application File (english)	8.690.2013

8.2. 690.0020 Ion Chromatograph

Ion Chromatograph with electrically operated injector	2.690.0020
instrument including following accessories:	
1 × PTFE extractor tubing with connector, i.d. = 0.9 mm, e.d. = 1.5 mm, length $L = 40$ cm	6.1822.000
1 × PTFE microcapillary tubing (outlet tubing) with nipple and flange, i.d. = 0.3 mm, e.d. = 1.5 mm, length $L = 1$ m	6.1822.010
1 × Compression fitting for $1/16$ " steel capillary, set of 5	6.2620.000
1 × Ferrule for $1/16$ " steel capillary, set of 5	6.2620.010
1 × Steel capillary, i.d. = 0.25 mm, e.d. = $1/16$ ", length $L = 3$ m	6.2620.020
1 × Coupling for $1/16$ " steel capillary	6.2620.060
1 × Adjustable spanner, max. width 20 mm	6.2621.000
1 × Open-end spanner $1/4$ " × $5/16$ " for 6.2620.000 compression fitting	6.2621.010
1 × Spanner for plastic nipple for extractor and outlet tubing	6.2739.000
1 × Syringe, volumen $V = 10$ mL	6.2816.020
1 × Needle for 6.2816.020 Syringe	6.2816.030
1 × Connection cable 690 Ion Chromatograph – Integrator (for automatic integrator start)	6.2115.030
1 × Mains cable cable socket type CEE (22), V; cable plug to order:	
type SEV 12 (Switzerland ...)	6.2122.020
type CEE (7), VII (Germany FR ...)	6.2122.040
type NEMA/ASA (USA ...)	6.2122.070
1 × Instructions for use (english)	8.690.1033
1 × Application File (english)	8.690.2013

8.3. 690.0030 Ion Chromatograph

**Ion Chromatograph with 2 electrically operated injector
and manually operated column selection valve 2.690.0030**

instrument including following accessories:

1 ×	PTFE extractor tubing with connector, i.d. = 0.9 mm, e.d. = 1.5 mm, length L = 40 cm	6.1822.000
3 ×	PTFE microcapillary tubing (outlet tubing) with nipple and flange, i.d. = 0.3 mm, e.d. = 1.5 mm, length L = 1 m	6.1822.010
1 ×	Compression fitting for 1/16" steel capillary, set of 5	6.2620.000
1 ×	Ferrule for 1/16" steel capillary, set of 5	6.2620.010
1 ×	Steel capillary, i.d. = 0.25 mm, e.d. = 1/16", length L = 3 m	6.2620.020
2 ×	Coupling for 1/16" steel capillary	6.2620.060
1 ×	Adjustable spanner, max. width 20 mm	6.2621.000
1 ×	Open-end spanner 1/4" × 5/16" for 6.2620.000 compression fitting	6.2621.010
1 ×	Spanner for plastic nipple for extractor and outlet tubing	6.2739.000
1 ×	PVDF Compression fitting for 1/16" capillary, set of 5	6.2744.000
1 ×	Syringe, volumen V = 10 mL	6.2816.020
1 ×	Needle for 6.2816.020 Syringe	6.2816.030
2 ×	Connection cable 690 Ion Chromatograph – Integrator (for automatic integrator start)	6.2115.030
1 ×	Mains cable cable socket type CEE (22), V; cable plug to order: type SEV 12 (Switzerland ...)	6.2122.020
	type CEE (7), VII (Germany FR ...)	6.2122.040
	type NEMA/ASA (USA ...)	6.2122.070
1 ×	Instructions for use (english)	8.690.1033
1 ×	Application File (english)	8.690.2013

Subject to alterations!

8.4. Options

Additional equipment available on payment of extra charge:

8.4.1. 697 IC Pump	2.697.0010
IC Pump with minimal residual pulsation and excellent flowrate stability; incl. accessories	
Important spare parts:	
Special tool for installing the 6.2741.000 piston seal	6.2617.010
Piston seal	6.2741.000
Sapphire piston	6.2824.000
Outlet valve	6.2824.010
Inlet valve	6.2824.020
Sapphire ring	6.2824.030
Pumping head, complet	6.2824.040
Spring for main piston	6.2824.050
Spring for priming piston	6.2824.060
8.4.2. Portmann pulsation dampener	6.2620.050
Pulsation dampener for extra column protection	
8.4.3. 698 Autosampler	2.698.0010
For the automation of sample introduction. Capacity: 64 sample vials each with an effective volume of ca. 700 µL; incl. accessories	
Important spare parts:	
PP sample vials for 698 Autosampler, 1000 pcs	6.2743.000
Glass sample vials for 698 Autosampler, 1000 pcs	6.2413.000
Polyethylene caps (plasticizer-free), 1000 pcs	6.2743.010
8.4.4. 586 Labograph	
Single channel precision pen recorder for the recording of ion chromatograms for manual evaluation	
Recorder, incl. accessories, for mains frequency $f = 50$ Hz	2.586.0012
Recorder, incl. accessories, for mains frequency $f = 60$ Hz	2.586.0013
8.4.5. Elektrochemical detector (ELCD)	
As second or alternative detector, consisting of:	
641 VA Detector for signal detection	2.641.0010
656 Electrochemical detector	2.656.0020
(can be installed in the interior of the 690 Ion Chromatograph)	
Carbon paste electrode for 656 Electrochemical detector	6.0807.000
Carbon paste for 6.0807.000 Electrode	6.2801.020
8.4.6. Conversion kit	6.5848.000
To convert version 2.690.0010 of the Ion Chromatograph into version 2.690.0020 with electrically operated injector	

8.4.7. Columns and precolumns

For the most common anions (halides, nitrate, phosphate, sulphate, ...):	
IC anion column PRP-X100	6.1005.000
IC precolumn cartridge PRP-X100	6.1005.020
(installation by means of 6.2821.040 cartridge head or 6.2821.050 twin cartridge holder)	
For common anions and special applications:	
IC anion column SUPER-SEP	6.1009.000
IC anion precolumn SUPER-SEP	6.1009.010
IC precolumn cartridge PRP-1	6.1005.050
(installation by means of 6.2821.040 cartridge head or 6.2821.050 twin cartridge holder)	
For carboxylic and hydroxycarboxylic acids:	
IC ion exclusion column PRP-X300	6.1005.030
IC precolumn cartridge PRP-X300	6.1005.040
(installation by means of 6.2821.040 cartridge head or 6.2821.050 twin cartridge holder)	
For alkaline earth ions and certain other divalent cations:	
IC cation column Nucleosil 5SA	6.1007.000
IC precolumn cartridge Nucleosil 5SA	6.1007.010
(installation by means of 6.2821.040 cartridge head or 6.2821.050 twin cartridge holder)	
For alkali and ammonium ions:	
IC cation column Vydac 400	6.1008.000
For alkali and alkaline earth ions and ammonium ions:	
IC cation column SUPER-SEP (acc. to Schomburg)	6.1009.020
IC cation precolumn cartridge SUPER-SEP	6.1009.030
(installation by means of 6.2821.040 cartridge head or 6.2821.050 twin cartridge holder)	

8.4.8. Additional accessories

Connection 1/16" capillary / 1/4" screw thread	6.2620.040
(for connection of columns with 1/4" thread)	
Sample loop 10 µL	6.2620.080
Sample loop 100 µL	6.2620.090
Capillary tubing cutter	6.2621.040
Manufit filter unit; complet	6.2821.000
Spare parts:	
PTFE gasket for Manufit filter unit and 6.1005.000, 6.1005.030, 6.1007.000 and 6.1009.020 IC columns, set of 4	6.2821.010
Steel mesh for Manufit filter unit and 6.1005.000, 6.1005.030, 6.1007.000 and 6.1009.020 IC columns, set of 10	6.2821.020
Cartridge head; complet	6.2821.040
(for mounting precolumn cartridges directly onto columns)	
Spare parts:	
PTFE spacer, set of 5	6.2821.070
Steel spacer	6.2821.080
Twin cartridge holder	6.2821.050
(for mounting precolumn cartridges into the input capillary of the separation column)	
Suction filter with 1/16" capillary connector (supplied with the 697 IC Pump)	6.2821.060

9. 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.

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