

CHIRALYSER - MP

Instruction Manual

Version CHI 7.2x (LED 426 nm)

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1.0 Notes and Warnings

This apparatus has been designed and manufactured in accordance with EN 60335/1 resp. IEC / CE and has left the factory in perfect condition as regards the relevant safety and CE regulations.

In order to maintain this perfect condition and to ensure safe operation, the user should observe the notes and warnings contained herein.

Prior to switching this apparatus on, please make sure that the mains voltage corresponds to the voltage stated on the apparatus and that the mains supply is correctly grounded. The protective effect must not be reduced by any unearthed extension lead.

Any interruption of the earth wire inside or outside the apparatus or loosening of the earth wire connection can make an apparatus dangerous. Intentional interruption is not permissible.

Opening of covers or removing of parts may expose live parts. Connections points may also be live !

Before making any adjustments, servicing, overhauls or before replacing any parts, the apparatus must be disconnected from the mains supply, if it needs to be opened.

If it becomes necessary to work on the apparatus whilst it is open, this must only be carried out by a skilled person, who is familiar with the dangers involved.

Care has to be taken to ensure that only fuses of the indicated type and nominal amperage are used. It is neither permissible to use mended fuses nor to short-circuit the fuse holder. The apparatus should not be operated, if there are any doubts about its safety, and it should then be taken out of service and secured against unintentional use.

Reasons for suspecting the apparatus to be unsafe, include the following:

- visible damage,**
- no function,**
- prolonged storage under unfavourable conditions,**
- severe stresses due to unsuited transport.**

1.1 Function Principle

From the LED (1) light passes through the lens system (2) and gets linear polarized by the polarization filter P1 (3).

After that, the light passes the flow cell (4) and the Faraday coil (5) with the Faraday rod (6) inside.

The Faraday rod consists of a special glass material which rotates the plane of the polarized light synchronous to the frequency f_1 of the electrical field applied to the coil. (Faraday modulation).

After passing the second polarization filter P2 (7), which is cross arranged to the first polarization filter, the light is modulated twice the frequency f_1 .

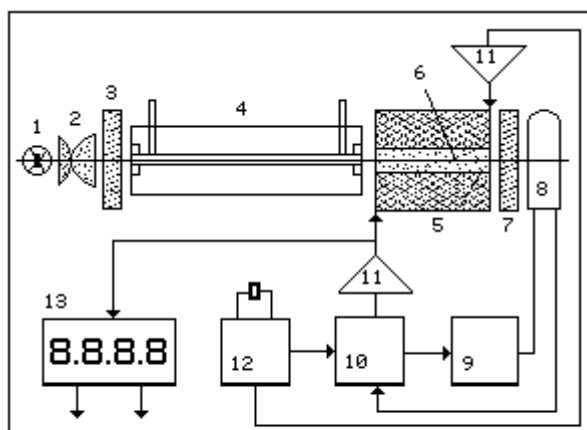
The voltage controlled photomultiplier (8) converts the modulated light into a photocurrent.

In the electronic synchronisation stage (10) the phase of the photocurrent is compared with the phase of the modulator frequency f_1 .

An optical active compound in the flow cell rotates the plane of the polarized light and causes a phase-offset in the synchronisation stage.

This phase-offset generates via the power amplifier (11) a compensation current into the Faraday coil which causes an electrical field which compensates the rotation of the optical active compound. (Faraday compensation).

The compensation current is proportional to the optical rotation of the optical active compound. Positive (d) - or negative (l) rotations are displayed (12) by positive or negative currents.



- 1 LED
- 2 lens system
- 3 polarisation filter P1
- 4 flow cell
- 5 Faraday coil
- 6 Faraday rod
- 7 polarisation filter P2
- 8 photomultiplier
- 9 intensity control
- 10 synchronisation
- 11 power amplifier
- 12 time base
- 13 display processor

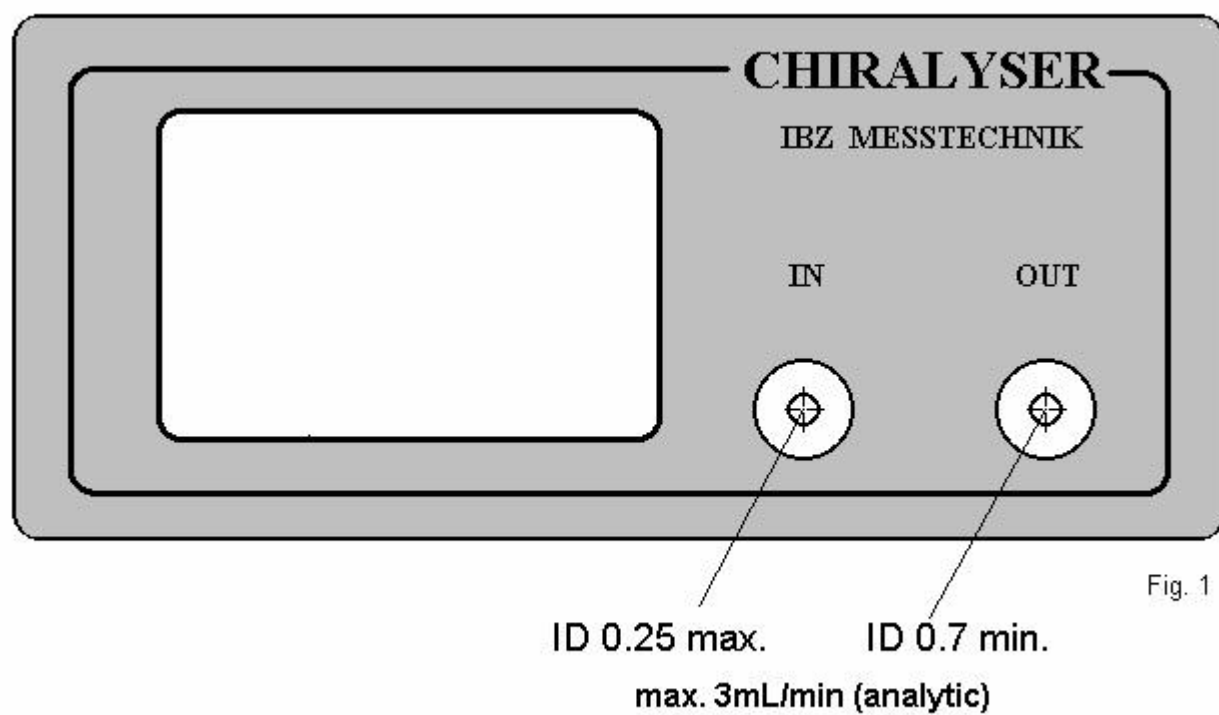


Fig. 1



2. Installation

Place the CHIRALYSER nearby to the outlet of the column or to the outlet of any detector on a solid and vibration-free location. There must be no other objects placed upon the instrument.

Connect the **IN** port with a 1/16" capillar with an inner diameter of **0.25 mm** (or less) and suitable fitting to the outlet of the column or the outlet of a previous detector.

Connect the **OUT** port with a 1/16" capillar with an inner diameter of **0.7 mm** (or more) and suitable fitting to the system waste. The CHIRALYSER has to be always the last detector connected to the HPLC system because of it's sensitivity to back pressure caused by other detectors. Disregard will have high risc for braking the glass windows of the flow cell.

ATTENTION:

NEVER use tubings less than **0.7mm** inner diameter at the **OUT** port.
Serious damage will occure !

There must be no back pressure at the OUT port.
No else detectors must be connected to the OUT port.

Only use degassed mobile phase and do not exceed maximum flow rate (3 ml/ min for analytical cell).

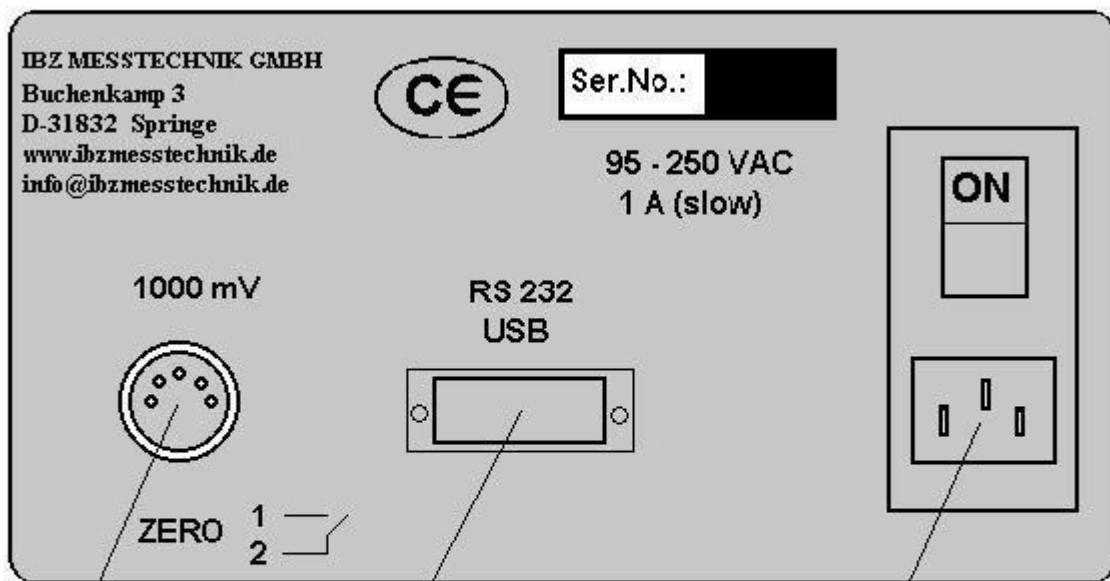


Fig. 2

Analog Interface

Serial Interface RS232
USB (optional with adapter)

Mains
95 - 250 VAC



2.1 Electrical connections

Mains

Make sure that your ac mains voltage is identical to the value printed on the identification label at the back side of the instrument. Operation without proper ground connection is dangerous and will cause malfunction of the instrument.

Analog Interface and AUTOZERO

Connect the analog cable to your system's A/D converter input port or to an Integrator or Chart Recorder. Notice the polarity marked on the cable. The output voltage range of the CHIRALYSER is 1000 mV related to the selected baseline offset and measuring range. Default setting is 500 mV.

The analog cable also carries the signal for the AUTOZERO contact. Contact has to be closed for minimum 0.5 sec to create ZERO in the display. There must be no external potential (voltage) at the contact.

IMPORTANT : Use only original analog cable attached to the consignment. See section 8.0

Serial Interface

Connect the serial cable to your system's RS232c input port. Configure your system's serial input port to:

9800 baud, 8 data, 1 stop, 1 start, no parity.

The actual measuring value is obtained by sending "?" (ASCII 63) to the CHIRALYSER.

IMPORTANT : Use only original serial cable attached to the consignment. For full remote control via serial interface see section 8.1

USB Interface (optional)

Connect the USB adapter to the serial interface connector and to your system's USB port. Start the driver CD and follow the instructions given by the installation program.

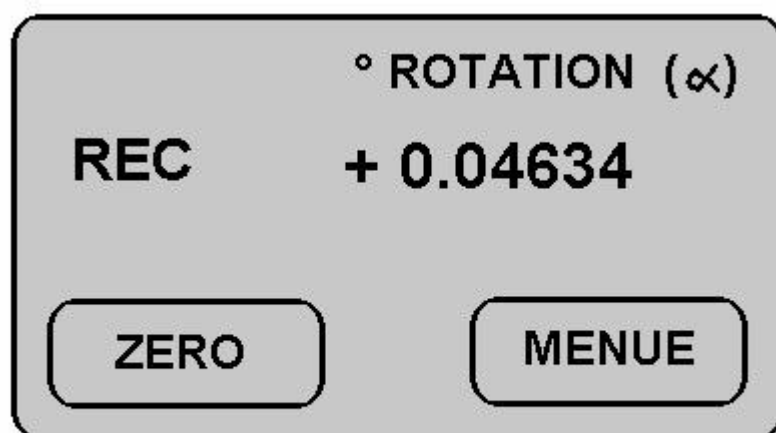


Fig. 5

Analog interface overrange. Touch ZERO

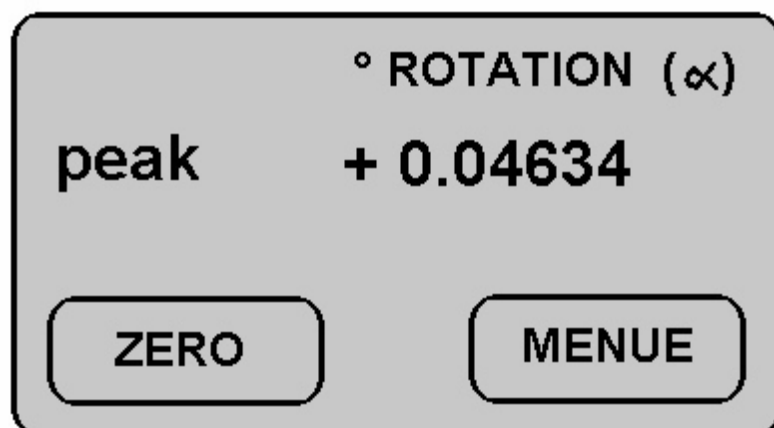


Fig. 6

Peak detection, see section 3.1

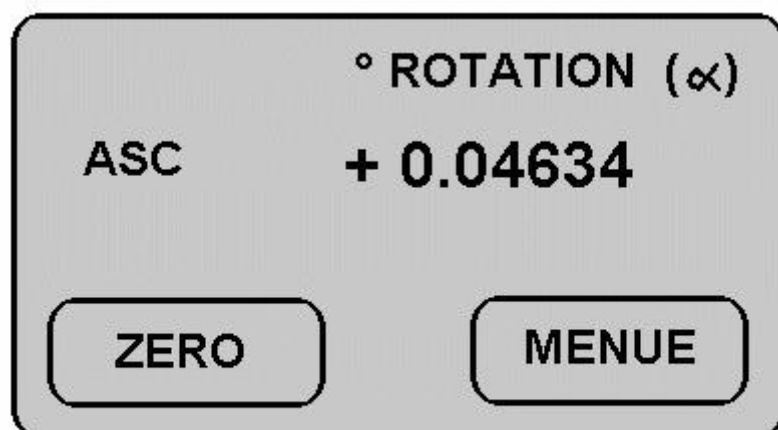


Fig. 7

Automatic Symmetry Calibration, see section 4.5

3.0 Start of measurement

After switching POWER on, the CHIRALYSER displays



Fig. 3

Wait until flow and pressure is balanced and stable and the touch **START**.

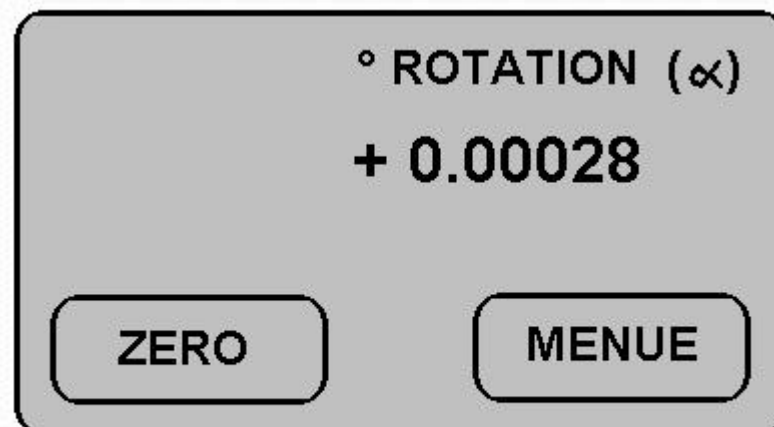


Fig. 4

Flashing α indicates correct data sampling.

CHIRALYSER is ready for measuring.

Touch **ZERO** to start operation in the default mode.

Display might show some additional information

(see Fig. 5 – 7, left side).

3.1 Peak detection

An optically active compound is indicated by **peak** in the display. Baseline drift and noise, specially during warm up (approx.30 min.), will also be indicated by **peak**.

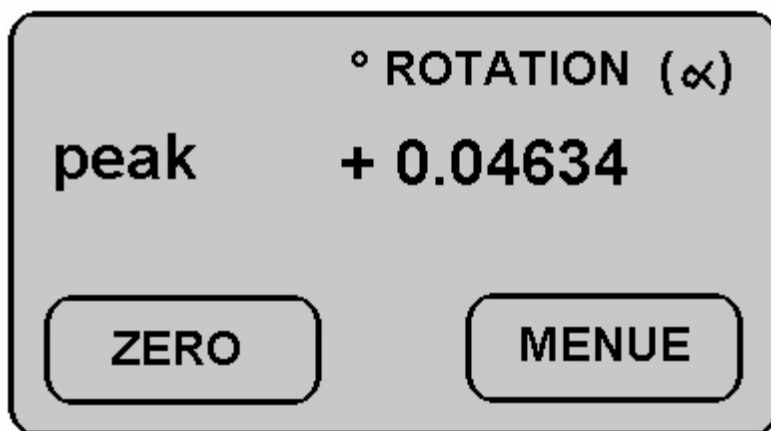


Fig. 6

Increase time constant (AVR) if baseline noise and drift
- under balanced conditions - will indicate **peak** in the display.

See section 4.0

4.0 Change instrument settings

Touch **MENUE** for changing the instrument settings

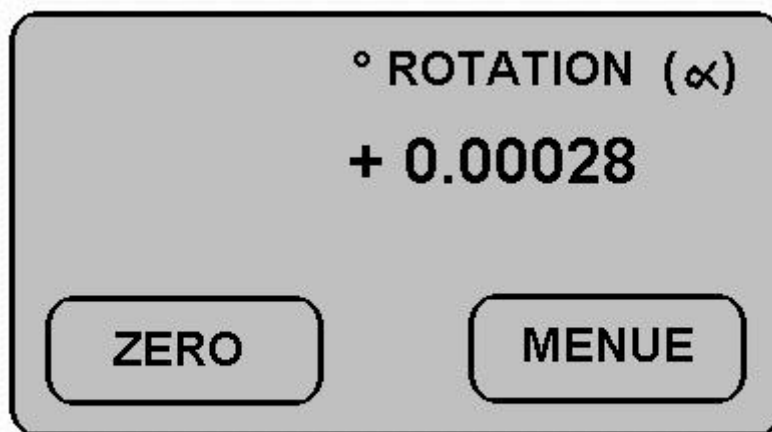


Fig. 4

CHIRALYSER will display

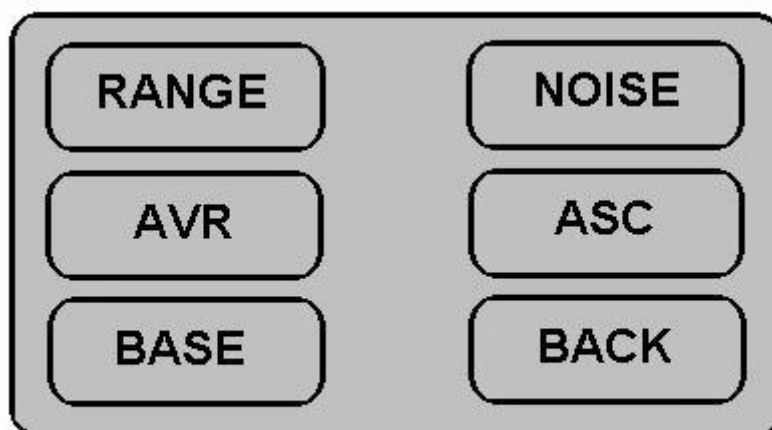


Fig. 8

Select **parameters** to change settings.
Touch **BACK** to go back to the measuring mode.

4.1 RANGE

Default setting for the measuring range is 8.0 mdeg. / 1000 mV full scale.
Range limits are 4.0 mdeg. / 1000 mV for highest sensitivity and
512.0 mdeg. / 1000 mV for lowest sensitivity.

Select appropriate RANGE by touching the arrow keys.
Touch OK to go back to the measuring mode.



Fig.9

4.2 AVR (time constant)

Default setting for the AVR time constant is “OFF” (no time constant). In this setting baseline noise is visible, but lowest limit of detection is attainable.

AVR settings can be changed within 0.2 sec (fast response) to 3.6 sec (slow response).

The AVR settings may affect baseline noise and peak geometry.

Select appropriate AVR by touching the arrow keys.

Touch **OK** to go back to the measuring mode.

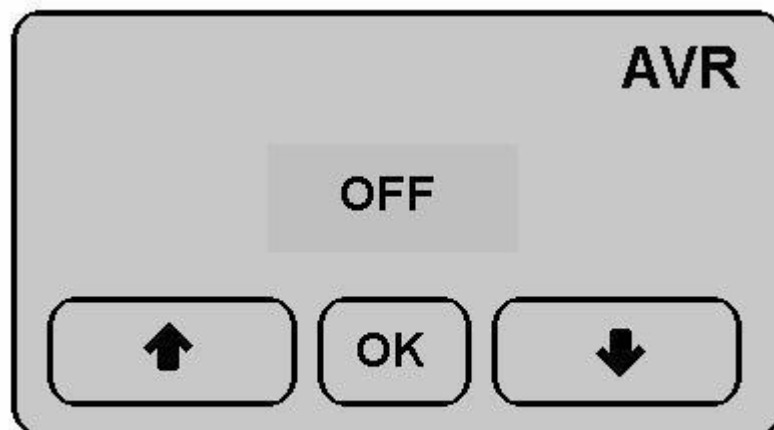


Fig. 10

4.3 BASE

Default setting for the baseline offset is 500.0 mV.
Baseline offset limits are 20.0 and 980.0 mV.
Use default setting for symmetrically measuring range.

Select appropriate BASE by touching the arrow keys.
Touch **OK** to go back to the measuring mode.

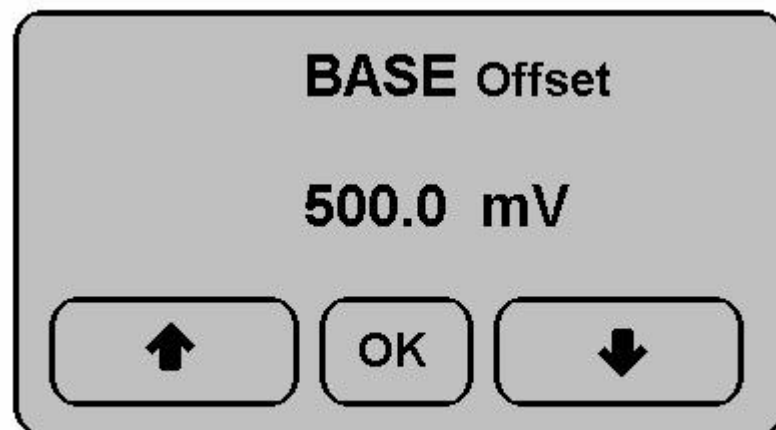


Fig. 11

4.4 NOISE

The displayed noise value effects the dynamic discrimination level of the AVR settings. Readings are only for diagnostic purposes and cannot be changed by user.

Touch **OK** to go back to the measuring mode.



Fig. 12

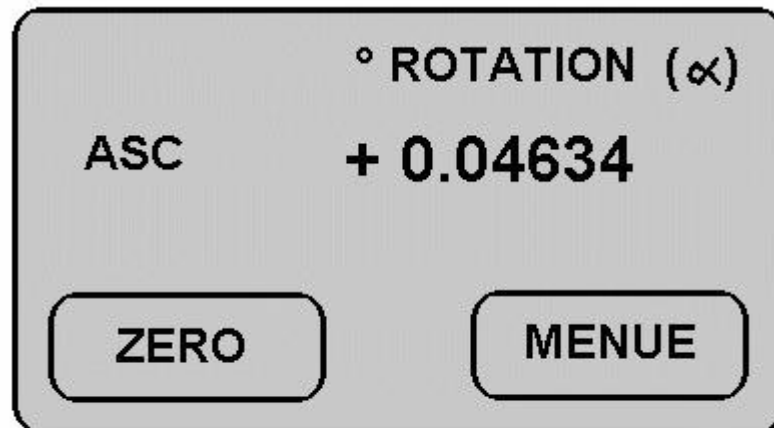


Fig. 7

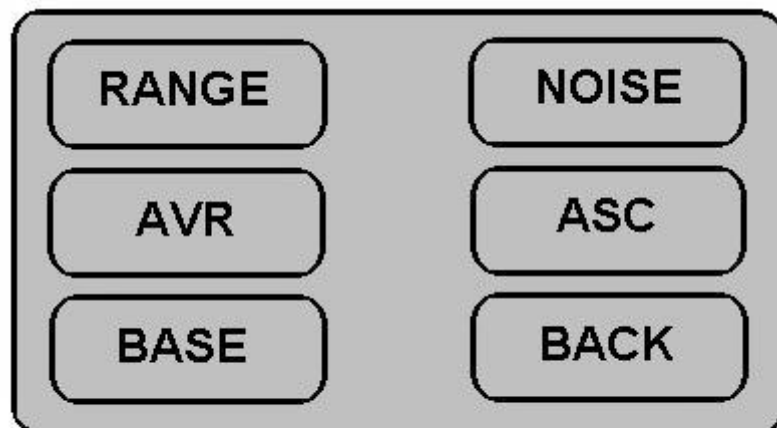


Fig. 8

4.5 ASC (Automatic Symmetry Calibration)

Internal optical offset, caused by high refractive index of the mobile phase or mechanical stress to the cell windows will display flashing ASC in the operational mode (Fig. 7).

Touch **MENUE** (Fig. 8) and then **ASC** to start the Automatic Symmetry Calibration.

CHIRALYSER will display



Fig. 13

When terminated, CHIRALYSER will display



Fig 14

Touch **OK** to go back to the measuring mode.

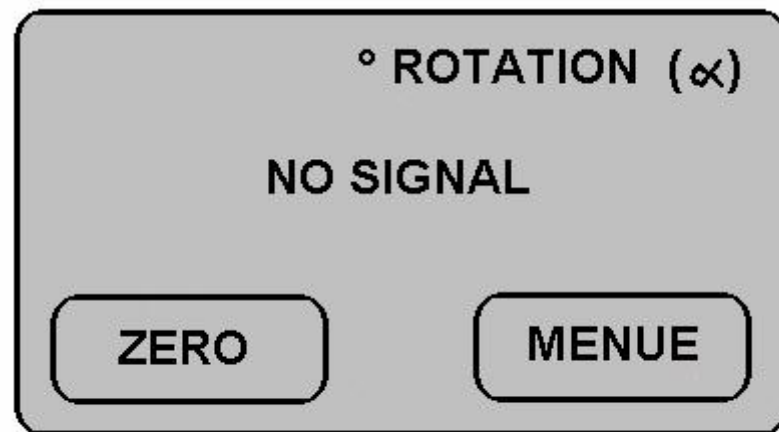


Fig. 15



Fig. 16

5.0 Troubleshooting

	Error message	NO SIGNAL
	Meaning	Air bubbles inside flow cell. Windows unclean or broken.
	Remedy	Use only degassed mobile phase. Raise flow temporary to max. 3 ml/min. Purge with isopropanol to remove bubbles. Clean or replace windows. (see section 6.1)
	Error message	ASC overrange
	Meaning	Internal optical offset too high, Automatic Symmetry Calibration cannot cancel optical offset.
	Remedy	Adjust optical balance (see section 6.1)
operation. water).	Error message	Display indicates “peak” at baseline
	Meaning	Baseline is noisy. Small air bubbles inside flow cell. Instrument not yet thermically balanced. Incompatible mobile phase (e.g.: organic /
	Remedy	Wait until instrument is balanced. Inject isopropanol to remove bubbles. Use compatible degassed mobile phase.
	Error message	Baseline drift
	Meaning	Instrument not yet thermically balanced
	Remedy	Wait until instrument is balanced. Keep temperature constant.

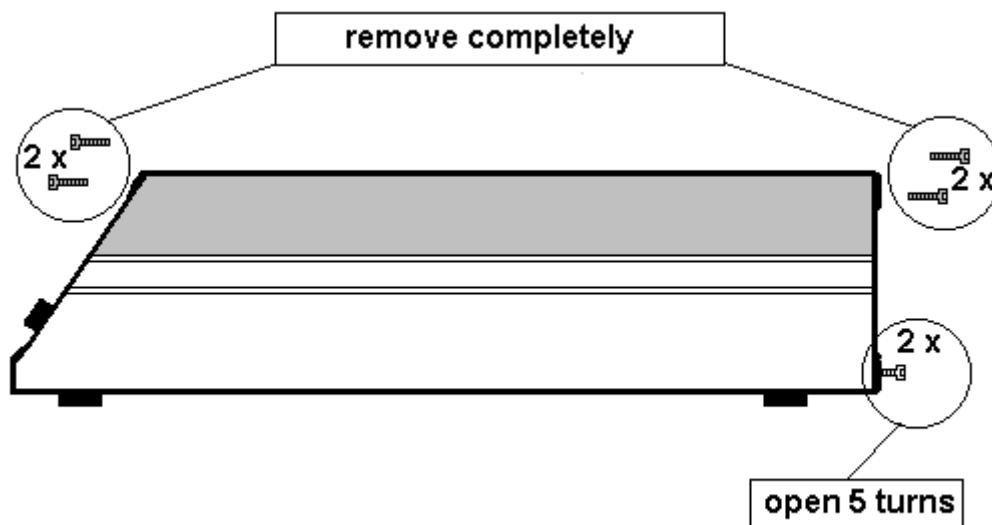
6.0 Service instructions

This chapter contains some easy to do service instructions.
The CHIRALYSER supports the user with diagnostic help.

Opening of enclosures or removing of parts may expose live parts.
Connection points may also be live !
If it becomes necessary to work on the apparatus whilst it is open, this must only be carried out by a skilled person, who is familiar with the dangers involved.

See: Notes and warnings at the beginning of this manual.

For the following service instructions, the enclosure has to be open.
Switch off power and open / remove the screws according this drawing.
Bend backplane slightly and lift off the upper part of the enclosure.



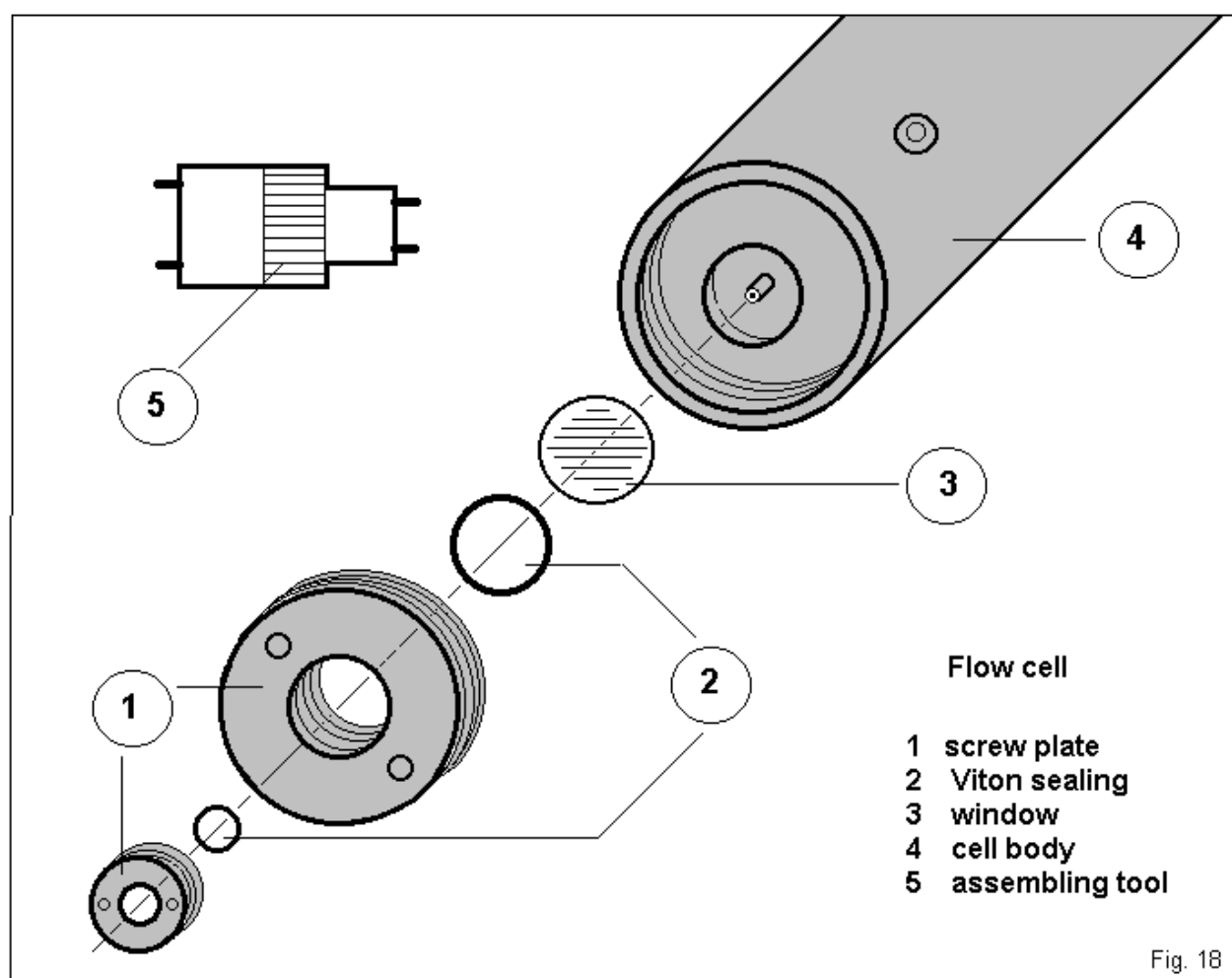


Fig. 18



6.1 Disassembling the flow cell

The flow cell must be disassembled if the windows are broken or require cleaning.

For do so, follow these instructions:

- Switch off power and open the enclosure according the drawing in section 6.0
- Disconnect the 1/16" fittings at the flow cell (4).
- Remove the cell holder (clamp); take the flow cell to a clean place for disassembly.
- Open the flow cell by unscrewing the screw plate (1) with the assembly tool (5).
- Replace the defective or unclean window together with its VITON seal (2) The area around and under the window must be absolutely clean; wipe away any particle very carefully.
- Carefully screw the flow cell closed with the assembly tool with very light torque.
- Repeat the same procedure with the other side of the flow cell.
- Very carefully, tighten both sides – slowly with little torque.
If a screw plate is tightened too quickly, or with too much torque, stress may be induced to the windows, causing baseline drift.
- Fix the cell with the cell holder (clamp) and reconnect the 1/16" fittings to the flow cell.
- close the enclosure and fix screws.

Switch the instrument on and touch START.

Expect some drift for some time, until stress is distributed in the mechanical / optical system and the instrument gets balanced.



Fig. 16

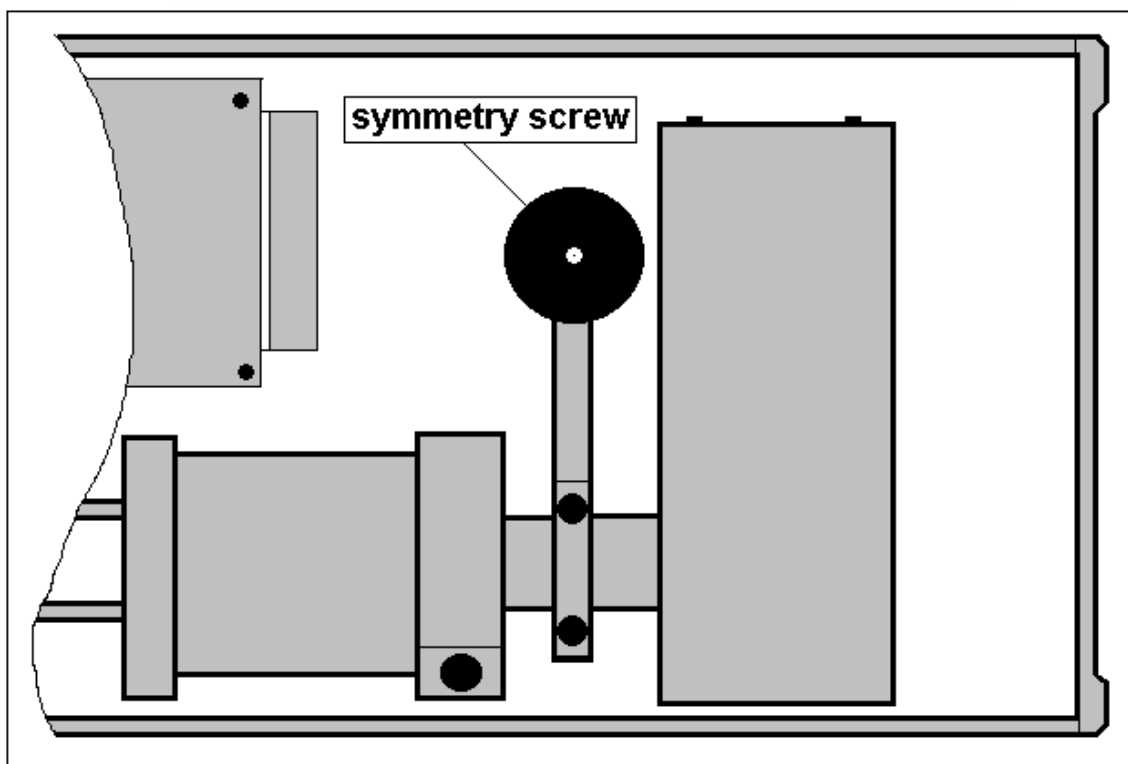


Fig. 17



Fig. 13

6.2 Mechanical Adjustment of the optical balance

The CHIRALYSER is factory set to an symmetrically measuring range with excellent optical balance.

This balance may be upset through flow cell disassembly or mechanical shock by transport and may cause peak cut off and internal optical offset. This internal optical offset is indicated by displaying a flashing ASC in the operational mode.

Starting the ASC routine (see section 4.5), will cancel the optical offset.

In case of excessive optical offset, indicated by ASC overrange (see Fig. 16), the Automatic Symmetry Calibration cannot cancel the optical offset completely and needs mechanical adjustment of the optical balance.

During the mechanical adjustment, power is on and flow rate should be 1 ml/min.

Do not touch any electronic device nor the printboard inside the instrument. There is a high risk of electric shock.

Open the enclosure according the drawing in section 6.0

Mark the position of the symmetry screw with a soft pencil.

Slowly rotate the symmetry screw (Fig. 17) clockwise until display indicates "please wait" (Fig. 13).

Do not rotate more than 5 turns clockwise. If there is no result, rotate anti-clockwise for maximum 10 turns.

Close enclosure and continue operation.

7.0 Preparative Cell

This CHIRALYSER is optionally provided with a preparative measuring cell with an optical length of 100 mm, volume 300 μ l.

Recommended flow rate is 5 to 100 ml/minute.

Do not exceed 30 bar (450 psi) pressure at the outlet (OUT) port and use only degassed mobile phase specially when flow rate is low.

The detector may be much too sensitive in its default settings. To reduce sensitivity, first make an ASC (Automatic Symmetry Calibration) with suitable flow according section 4.5 in this manual and then select appropriate RANGE.

If sensitivity is too high even in the highest RANGE setting (512.0 mdeg.), a shorter measuring cell is recommended.

Installation

The 100 mm cell is placed towards the light source and the "IN" tubings must be changed to 1/16" with inner diameter 1.0 mm to minimize backpressure to the column. Fix clamp midside of the cell.

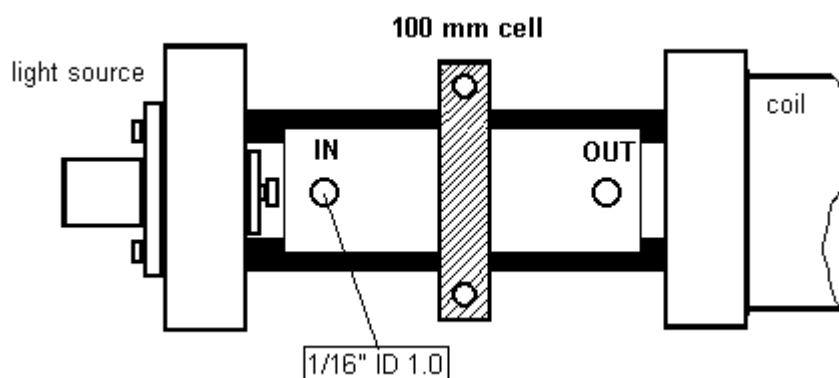


Fig. 19



8.0 Pin connections

The Analog cable carries the signal (1000 mV) for the external A/D converter and the connection for the AUTOZERO contact.

Analog cable (5 pole connector male / open)

<u>Pin</u>	<u>meaning</u>	<u>color</u>
1	Signal Ground	red
2	Auozero - close contact to pin 1 -	green
3		
4	analog signal negative potential	white
5	analog signal positive potential	yellow

Serial interface cable (9 pole D-sub connector male / female)

<u>Pin</u>	<u>CHIRALYSER (female)</u>
1	DCD
2	TxD (transmit data)
3	RxD (receive data)
4	DTR
5	GND (signal ground)
6	DSR
7	RTS
8	CTS
9	n.c.



8.1 Remote control via serial interface

Remote control is given by sending ASCII code to CHIRALYSER according schedule

FORMAT 9600 / 8 / 1 / 1

ASCII		mode	action
65	(A)	START	start CHIRALYSER
66	(B)	ZERO	set display ZERO
87	(W)	DISPLAY	display off
75	(K)	DISPLAY	display on
63	(?)	REQUEST DATA	send measuring data to host
67	(C)	MENUE	enable MENUE for all settings
73	(I)	MENUE	back to measuring mode
68	(D)	RANGE	enable RANGE settings
76	(L)	RANGE	increase RANGE
77	(M)	RANGE	decrease RANGE
78	(N)	RANGE	back to measuring mode
70	(F)	AVR	enable AVR settings
82	(R)	AVR	increase AVR
83	(S)	AVR	decrease AVR
84	(T)	AVR	back to measuring mode
72	(H)	BASE	enable BASE settings
79	(O)	BASE	increase BASE
80	(P)	BASE	decrease BASE
81	(Q)	BASE	back to measuring mode
69	(E)	HV	enable HV
85	(U)	HV	back to measuring mode
71	(G)	ASC	enable ASC
86	(V)	ASC	back to measuring mode