



ClinRep[®]

Instruction Manual
for the
amperometric
Detector

L-3500A

Table of Contents	Page
1. INSTALLATION	1
1.1 Unpacking the Unit	1
1.2 Set up Location	1
1.3 Main supply	1
1.4 Checking the delivered parts	2
1.5 Merck Recipe Amperometric Detector LC 3500A packing list	2
2. FUNCTION	3
2.1 Elements of the front of the unit	3
2.1.1 Potential adjustment	4
2.1.2 Switch for Cell mode	4
2.1.3 Switch for output filter	4
2.1.4 Switch for filter order	4
2.1.5 Switch panel for Sensitivity and Polarity of the output	5
2.1.6 Key to display the potential between the auxiliary electrode and the working electrode	5
2.1.7 Key for the display of the total current	5
2.1.8 Key to display the current compensated by Autozero	6
2.1.9 Function key for manual Autozero	6
2.2 Elements of the rear of unit	7
2.2.1 Setting the Autozero levels	7
3. OPERATION	8
3.1 Electronic cabling for a system with recorder	8
3.2 Electronic cabling for a system with Integrator	8
3.3 Electronic cabling for a system with the Merck HPLC-Manager	9
3.2 Checking the electronics of the detector	9
3.2.1 Checking the potential setting and the current	9
3.2.1 Checking the Out nA display and the Autozero	10
3.3 Passivation of the HPLC Unit with half concentrated nitric acid	10
3.3.1 Reason for Passivation	10
3.3.2 Performing the Passivation	10
3.3.3 Passivation of the injector system	11
3.4 „Starting“ and set up of the measurement conditions	11
3.4.1 Potential set up	11
3.3.2 Switching the cell mode from „Standby“ to „Work“	11
3.3.3 Measuring the sample	11
COMPONENTS	<i>Fehler! Textmarke nicht definiert.</i>
TECHNICAL INFORMATION	13

1. INSTALLATION

1.1 Unpacking the Unit

Unpack the transport box, by removing the detector and the standard accessories and placing them on a level, sturdy table.

1.2 Set up Location

Note: Do not expose the detector or the cell to a direct draft, due to the fact that the zero-line stability and the repetition ability may be adversely effected.

Choose a location where:

- 1) The temperature does not vary too much during the procedure and where the temperature remains between 10 and 30° Celsius.
- 2) The relative humidity is between 45 and 85%.
- 3) The unit is not exposed to direct sunlight or a draft. Avoid the placement close to a window.
- 4) the unit is well aired.
- 5) No open flame is used.
- 6) No aggressive gases are present.
- 7) No strong vibrations or for the unit detectable shaking is present.
- 8) No strong magnetic or electric field is present.

1.3 Main supply

- 1) Main voltage: 115 or 230 V
Possible fluctuations have to be within +/- 10% of the specified values.
- 2) Frequency: 50 or 60 Hz
Possible fluctuations have to be within +/- 4% of the specified frequencies.
- 3) Installation of the Power Cable
Make sure that the main power switch is on „OFF“, then plug the main power cable into the socket on the back of the unit

Attach the other end of the cable to the alternating current supply (AC).

The used main cable has three poles. The unit is grounded according to the international safety norms.

Make sure that the alternating current is interference free and has a stable current level. A strong and fast current change disturbs the function of the electronics and of the microprocessor and can cause a malfunction.

Before moving the unit to a new location please pull the main power supply cable due to safety reasons.

1.4 Checking the delivered parts

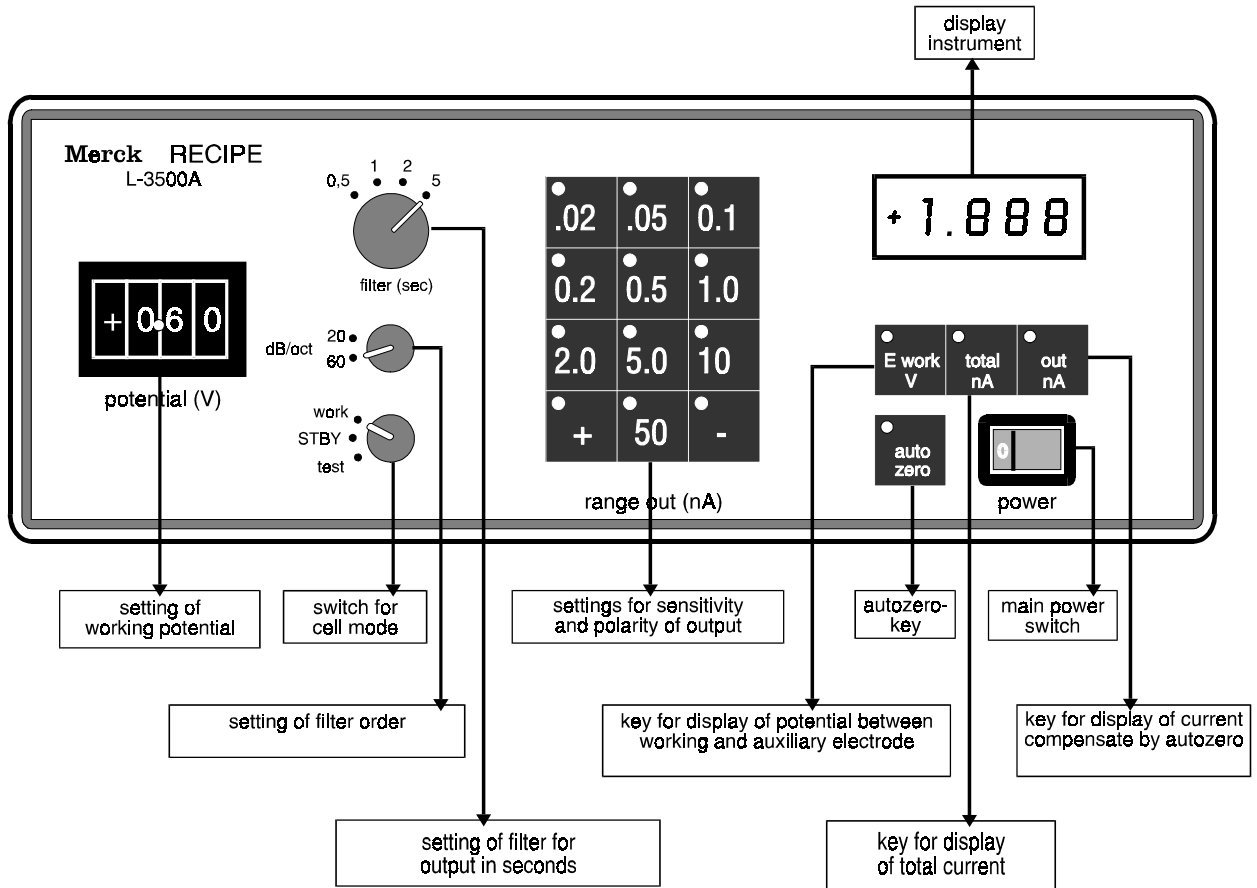
After unpacking, make sure that the delivered parts match with those on the packing list. If a part should be missing or needs to be replaced, please contact Merck.

1.5 Merck Recipe Amperometric Detector LC 3500A packing list

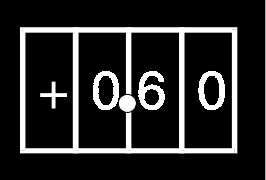
1. Electronic unit 1x
2. Cable:
 - a) Main power cable
 - b) Cell connection cable
 - c) Connection cable ECD exit to the integrator (cable shoe)
 - d) Connection cable ECD exit to the PC-data system (green Merck plug)
 - e) Connection cable Autozero for the integrator (cable shoe)
 - f) Connection cable Autozero - Start out (autosampler, detector etc.) (green Merck plug)
3. Cell housing with mounting rod and cell (without reference electrode)
4. Cell accessories
 - a) Reference electrodes 2x
 - b) Cell gasket 30 μ m 2x
 - c) Cell gasket 50 μ m 3x
 - d) Mounting rod
 - e) Fitting screws 2x
 - f) Ferrules 4x
 - g) Washers for cell connection 8x
 - h) Mounting mechanism for fitting 1x

2. FUNCTION

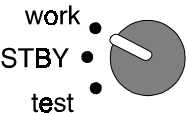
2.1 Elements of the front of the unit



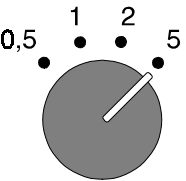
2.1.1 Potential adjustment

 <p>potential (V)</p>	<p>Adjustment for the oxidation or reduction potential in the range of +1.999 to -1.999</p>
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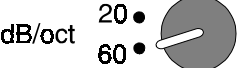
2.1.2 Switch for Cell mode

	<ol style="list-style-type: none"> 1. In the switch position „Standby“ the electronic connection with the cell is turned off. 2. In the position „Work“ the cell is connected electronically i.e. the potential is present and the current running through the cell is measured. 3. In the position „Test“ an electric dummy cell (100 MOhm resistance) is connected. With this it is possible to check the function of the detector electronics (see pg. ...)
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2.1.3 Switch for output filter

 <p>filter (sec)</p>	<p>For most measurements the position 2 or 5 are advisable. Especially with very fast separations the optimum set up has to be determined experimentally.</p>
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2.1.4 Switch for filter order

	<p>In the position 60 dB/oct high frequency disturbance signals are filtered out most effective. Generally this position should be chosen. Only with extremely fast chromatography a change to 20dB/oct can become necessary.</p>
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2.1.5 Switch panel for Sensitivity and Polarity of the output

● .02	● .05	● 0.1	<p>Is used for the amplification or reduction of the output. Usually a setting of 10 nA per 1 Volt (i.e. 10mV) as an output signal, are used when using the Integrator or the PC evaluation system. The recording sensitivity can be set at the Integrator or PC.</p> <p>The + or - keys are used to change the polarity of 1 V or 10 mV of the outputs.</p>
● 0.2	● 0.5	● 1.0	
● 2.0	● 5.0	● 10	
● +	● 50	● -	
range out (nA)			


2.1.6 Key to display the potential between the auxiliary electrode and the working electrode

● E work V	<p>Shows the value of the potential between the auxiliary and working electrode. Since surface effects can play a role here, the value can fluctuate significantly from the set potential, without having any influence on the detection or the actually active potential between the mobile phase and the working electrode. With great fluctuations of the shown value from the set potential in addition to detection problems (e.g. low sensitivity) the cell, especially the reference electrode should be checked. Short term fluctuations with a well used system are also a practically sure sign for a malfunction of the cell or the chromatographic system.</p>
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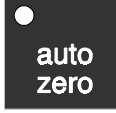
2.1.7 Key for the display of the total current

● total nA	<p>Shows the value of the total current generated in the cell. It is important for the determination of the quality of the mobile phase. Degassing the mobile phase can result in a quality increase.</p>
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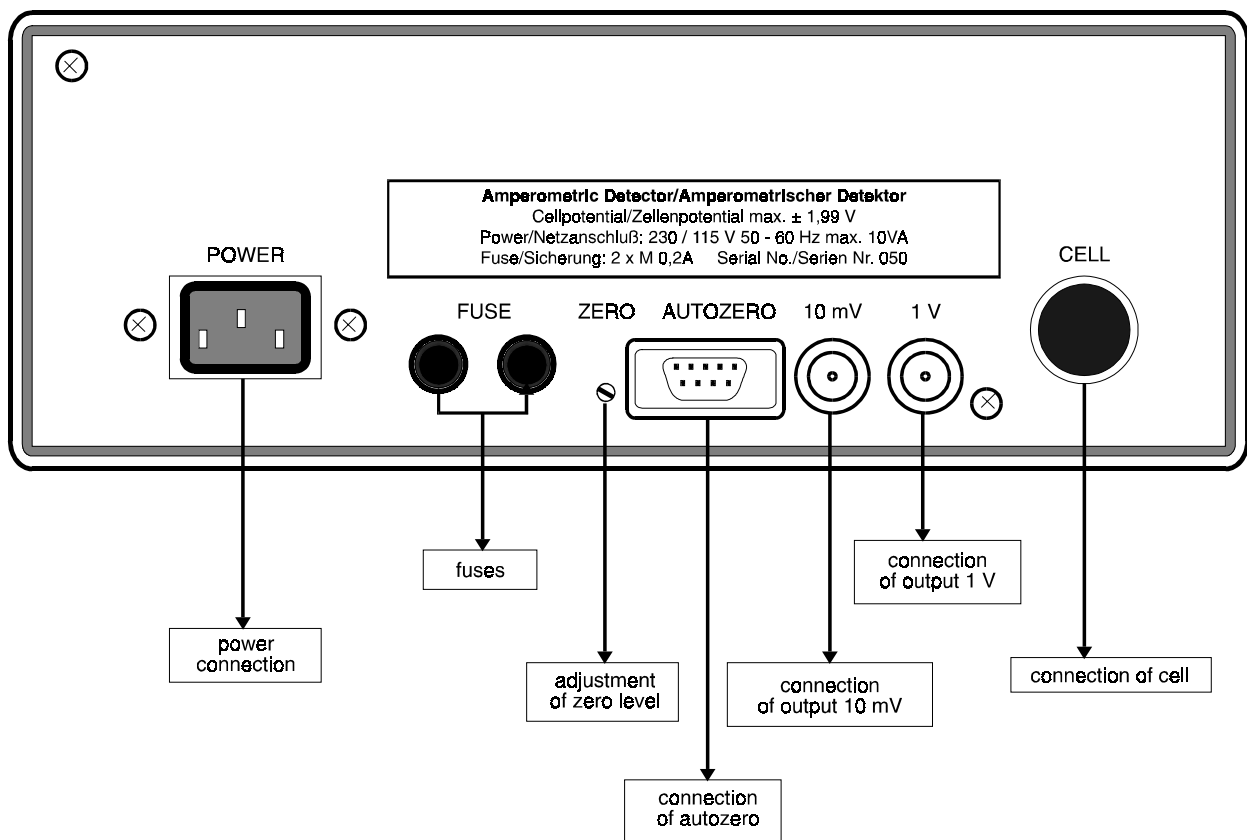
2.1.8 Key to display the current compensated by Autozero

	Shows the current minus the offset compensation by Autozero. At the 1V or 1mV exit an according current is present.
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2.1.9 Function key for manual Autozero

	Manual Autozero key for the compensation of the basic current. (Basic signal of the mobile phase)
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2.2 Elements of the rear of unit

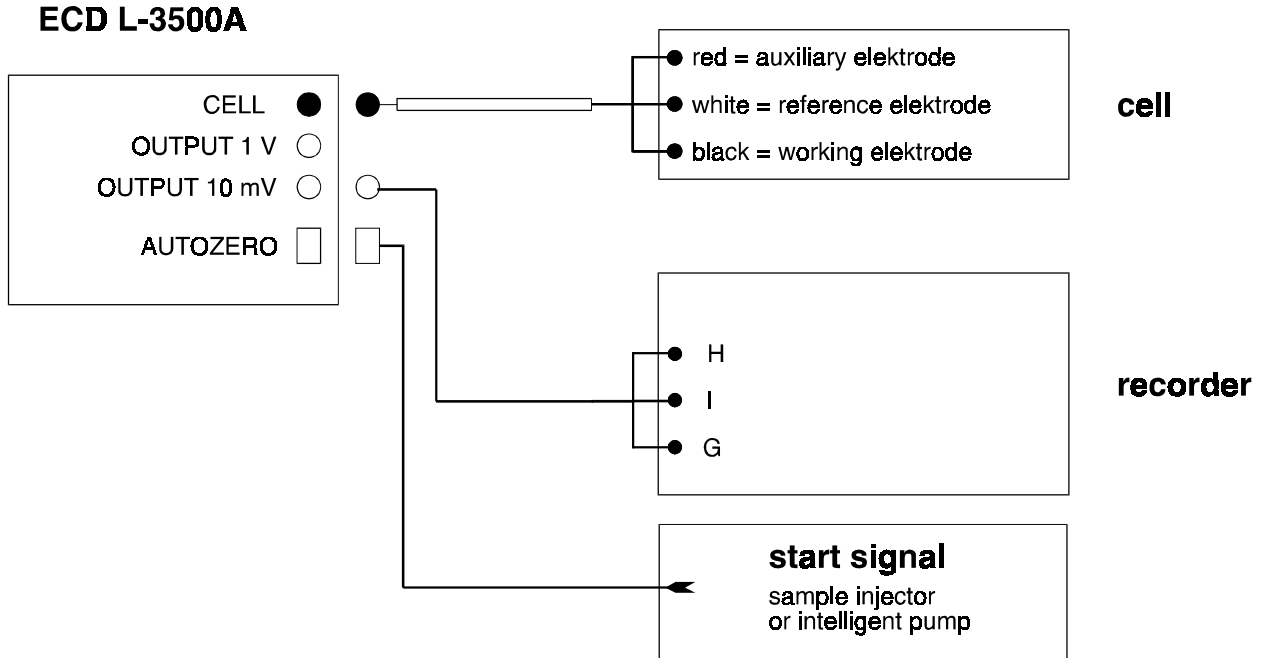


2.2.1 Setting the Autozero levels

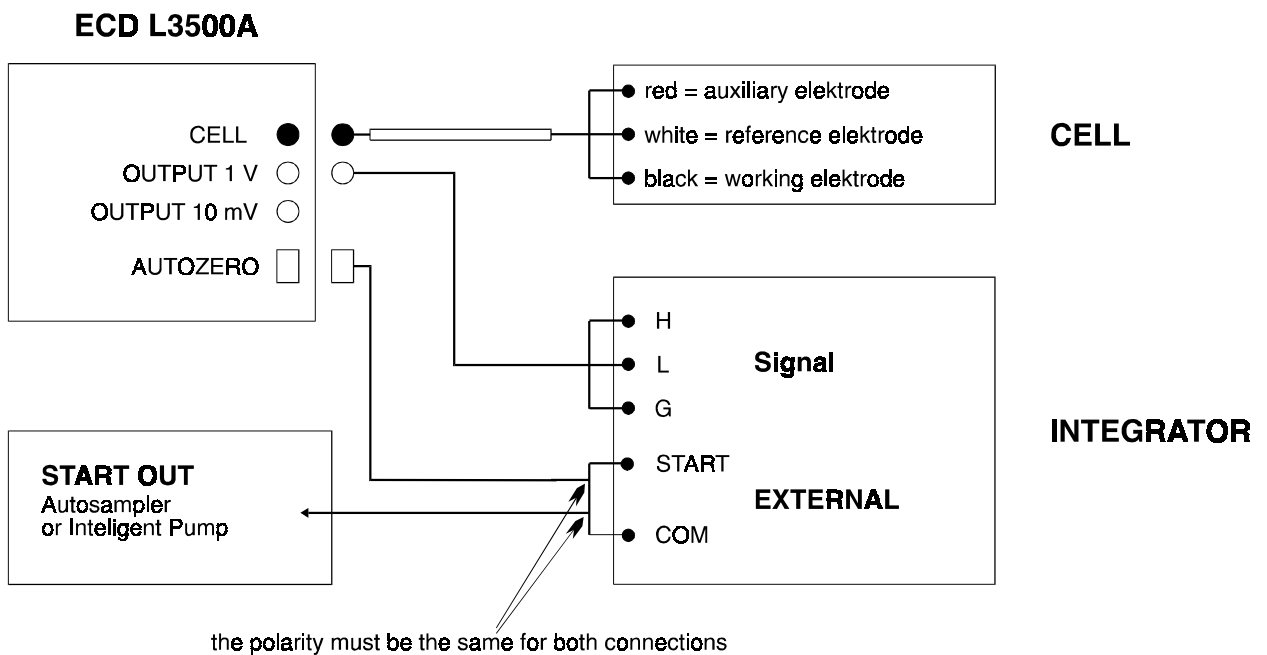
The Autozero level (exit current after using Autozero) can be adjusted individually by turning the setscrew. This can be used to change the zero point by ± 0.25 nA related to the range 10 nA/1V or 10 mV (e.g. to adapt an Integrator).

3. OPERATION

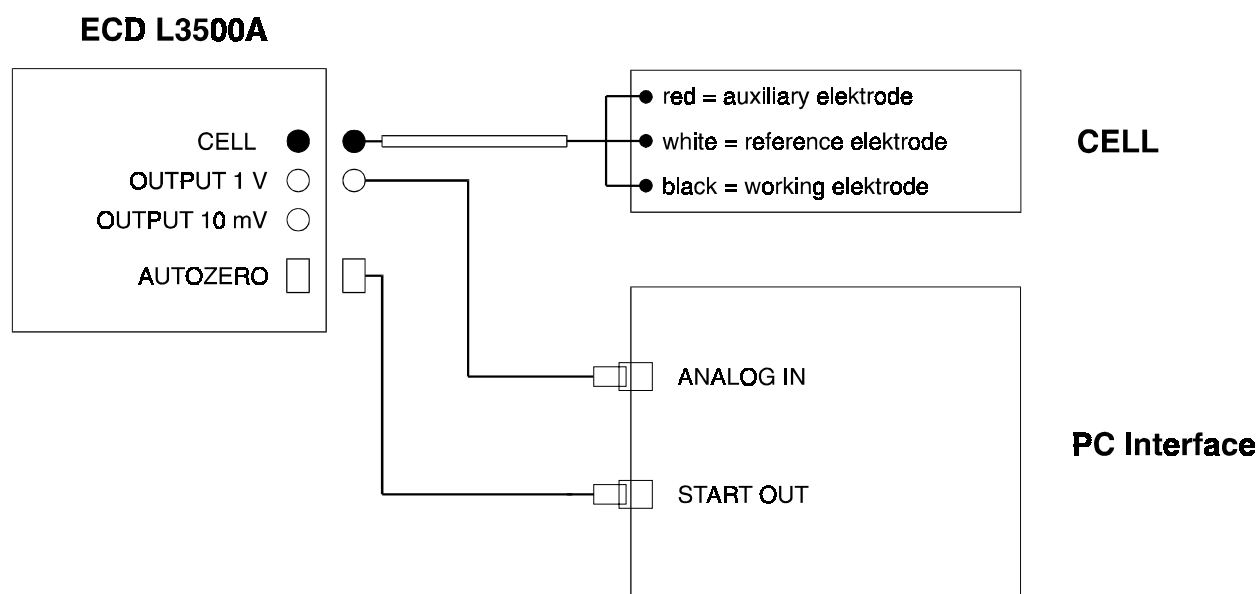
3.1 Electronic cabling for a system with recorder



3.2 Electronic cabling for a system with Integrator



3.3 Electronic cabling for a system with the Merck HPLC-Manager



3.2 Checking the electronics of the detector

3.2.1 Checking the potential setting and the current

Set the switch for the cell mode to the position „TEST“

potential-setting	display-key	display
0 V	E _{work}	0 V ± 0.002 V (digital display 0.0±1)
0 V	total	0 nA ± 0.1 nA
+0.5 V	E _{work}	+0.5 V ± 0.002 V
	total	+5 nA ± 0.1 nA
+1.0 V	E _{work}	+1.0 V ± 0.002 mV
	total nA	+10 nA ± 0.2 nA

Of course, this check can also be done with a negative potential setting. **Since this is a digital display, the last digit can fluctuate by ± 1.** If these fluctuations are significantly greater, a defect in the electronics is very probable. Please contact the customer service.

3.2.1 Checking the Out nA display and the Autozero

The polarity change + or - in the „range out“ field is indicated in the position „out nA“. The switch „range out“ from 0.02 to 50 nA is done after the measurements for the display „total“ and „out“. Thus, the position does not change the display.

3.3 Passivation of the HPLC Unit with half concentrated nitric acid

3.3.1 Reason for Passivation

A new HPLC unit has to be passivated before using it with the electrochemical detector. Metal ions in the solution e.g. Fe^{2+} are substances, which can be reduced or oxidized. Thus, large disturbances can be possible (e.g. high base current, uneven base line, ghost peaks etc.).

It is also advisable, to repeat this passivation of the HPLC unit from time to time (dependent on usage every 2 to 3 month), especially when malfunctions occur, which can not be traced to single components of the unit.

3.3.2 Performing the Passivation

All components of the unit, except the column and the detector cell have to be passivated (preoxidized).

This is done with the following steps:

- a) Connect the pump, the sampler, the column oven and all capillaries with the exception of the column and the detector cell.
- b) Place the exit capillary into a safe waste container.
- c) Flush the system with HPLC water for 15 min. with a flow of 1-2 ml per min.
- d) Then flush for approx. 10 min. with isopropanol.
- e) Then flush for approx. 15 min. with water.
- f) Now flush the system with nitric acid for 30 min. with a flow of 1 ml/min. (65% nitric acid solution 1:1 diluted with water).
- g) Now flush the system again with HPLC water with a flow of 1-2 ml/min. until the pH of the exiting solution is again neutral. Refresh the water in the solvent container several times to make sure that the nitric acid is flushed out of the frit in the mobile phase.
- h) Then flush the system for approx. 15 min. with the intended mobile phase.
- i) Connect the column and the detector cell.

3.3.3 Passivation of the injector system

The injector system also has to be passivated completely.

3.3.3.1 Hand injection system

Inject half concentrated nitric acid several times during the passivation phase. During the following irrigation with water the equal amounts of water have to be injected.

3.3.3.2 Autosampler

The half concentrated nitric acid also has to be used as the washing solution during the passivation phase. Repeat the wash steps several times and inject half concentrated nitric acid several times with the largest possible volumes.

According to this, the irrigation has to be done with water.

3.4 Start and set up of the measurement conditions

3.4.1 Potential set up

Set the wanted potential in the „Standby“ cell mode. To choose the potential it is best to follow literature results or figures given from the HPLC-Kit manufacturer. Are both not available a set of samples with increasing potentials have to be performed.

3.3.2 Switching the cell mode from „Standby“ to „Work“

When the HPLC-System is running continuously with a constant flow rate, switch the cell mode to „work“. Watch the basic current on the display (Key „total nA“). It is possible that the display begins to blink in the beginning, if the basic current is high in the starting phase. Dependent on the potential and the mobile phase the display should continually decrease and stabilize at a constant value. The unit and the detector are now ready to analyze.

3.3.3 Measuring the sample

Choose 10 nA for emaciation. Press the „Autozero“ key and inject a sample. According to the chromatogram i.e. the responses of the substances an amplification or emaciation can be set on the Integrator or PC-system first. If this should not be enough, change the sensitivity with the detector keys (0.02 to 50 nA), With especially high signal flows the 1 mV exit can be used as an emaciation.

4. SPARE PARTS LIST

(For the cell see instruction part of the cell)

Order No.	Description	Quantity
EC281	Main power supply	1
EC282	Cell connection cable	1
EC283	Exit cable universal with cable shoe	1
EC284	Exit cable Merck with green flat plug	1
EC285	Autozero cable universal with cable shoe	1
EC286	Autozero cable Merck with green flat plug	1

5. TECHNICAL INFORMATION

Principal:

Amperometric Detector with 3 Electrode-System

Cell:

Working electrode:	Glassy Carbon in Borosilicate glass; optional: Ag, Au, Cu, Ni, PT in Kel-F
Auxiliary electrode:	V4A-stainless steel
Reference electrode:	Silver/Silverchloride refill;
Diaphragm:	Glass diaphragm; optional base stable zirconium oxid diaphragma
Cell volume:	1,5 μl with 30 μm cell gaskets optional 0,75 μl /15 μm or 2,5 μl /50 μm
Materials:	Teflon, stainless steel

Electronics:

Working potential:	0 to $\pm 1,999\text{V}$
Input current range:	0 to $\pm 200\text{nA}$
Measurement range:	0,02 to 50nA adjustable
Offset compensation:	to $\pm 200\text{nA}$, accuracy better than 0,05%
Manual offset range	$\pm 2,5\%$ the range
Autozero connection:	switching voltage 5V max. 20nA, impulse time min: 100 msec
Active filter:	20/60 dB adjustable
Time constants:	0,5, 1, 2, 5 sec
Noise level:	0,3 pA at 100M Ω at time constant 5 sec and 60 dB filter
Outputs:	0-10mV / 0-1V (max. 10V)
Main supply:	115V, 230V, 50-60 Hz
Weight:	4,0 kg; Cell approx.. 0,2 kg
Dimensions:	Electronic unit: 255 (w) x 310 (d) x 105 (h) mm Cell in Faraday's cage: 120 (w) x 110 (t) x 155 (h) mm