ANTEC
LEYDEN

ALEXYS LC 100
Pump

user manual

Edition 5, 2007 182.0010

CE

DECLARATION OF CONFORMITY

The manufacturer hereby declares that the product

ALEXYS LC 100 pump	type 182

to which this declaration relates, is in conformity with the following directives:

Safety (73/23/EEC)

Safety requirements for laboratory equipment (Class I, Installation cat. II, Pollution degree 2)	EN61010-1

EMC (89/336/EEC)

Electromagnetic compatibility, generic emission standard	EN50081-1/2
Electromagnetic compatibility, generic immunity standard	EN50082-2
Emission standard- Information Technology Equipment (ITE)	EN 55022,Class B (CISPR22)
Harmonic current emissions	EN 61000-3-2
Voltage fluctuations and flicker	EN 61000-3-3

Attention

Use manufacturer-supplied cable(s) only to connect all I/O's with other devices. Thoroughly connect the shielding to common. Manufacturer will not accept any liability for damage, direct or indirect, caused by connecting this instrument to devices, which do not meet relevant safety standards.

July 26, 2007

Intended use

For research purposes only. While clinical applications may be shown, this instrument is not tested by the manufacturer to comply with the In Vitro Diagnostics Directive.

WEEE directive

All equipment of Antec Leyden which are subjected to the WEEE directive shipped after August 13, 2005 are compliant with the WEEE marking requirements. Such products are labelled with the “crossed out wheelie”, depicted on the left site.

The symbol on the product indicates that the product must not be disposed as unsorted municipality waste.

Collection & recycling information

Please ship the instrument back to the manufacturer (Antec Leyden, the Netherlands) at the end-of-life time of the product. The manufacturer will take care of the proper disposal and recycling of the instrument at its facilities.

Shipping address for the end-of-life products:

Antec Leyden
Industrieweg 12
2382NV Zoeterwoude
The Netherlands

In case of questions, or if further information is required about the collection & recycling procedure, please contact your local distributor.

ROHS directive

Our instruments are currently exempt from the RoHS directive because they fall under WEEE Annex IA categories 8 and 9, which includes medical devices and monitoring and control instruments. Nevertheless, we have taken steps to eliminate all restricted substances from our products.

Antec Leyden is an ISO 9001:2000 certified company.

Symbols

The following symbols are used on the ALEXYS LC 100:

Consult the manual for further safety instructions

Frame or chassis ground terminal

The following pictograms are used in the ALEXYS LC 100 manual:

Caution

Caution, risk of electric shock or other electrical hazard (high voltage)

Safety practices

The following safety practices are intended to ensure safe operation of the instrument.

Electrical hazards

Removal of protective panels on the instrument can result in exposure to potentially dangerous voltages. Therefore, disconnect the instrument from all power sources before disassembly. Untrained personnel should not open the instrument.

WARNING - RISK OF FIRE REPLACE FUSE AS MARKED	WARNING - RISK OF ELECTRIC CHOCK DISCONNECT POWER BEFORE SERVICING
AVERTISSEMENT - RISQUE DE FEU REMPLACEZ LE FUSIBLE COMME INDIQUE	AVERTISSEMENT - RISQUE DE CHOC ELECTRIQUE COUPER L'ALIMENTATION AVANT LA MAINTENANCE

Replace blown fuses with fuses of proper type and rating as stipulated on the rear panel and specified in the installation section of this manual. The fuse holder is integrated in the mains connector. Ensure that the instrument is never put in operation with fuses of a different type. This could cause fire.

Connect the ALEXYS LC 100 to a grounded AC power source. Check the position of the mains selector (is set to 230 V on delivery - if necessary set

to 115 V). Check fuses, on delivery this is set to 0.25 A slow (180 - 250 V) or 0.50 A slow (90 - 130 V).
The instrument should be connected to a protective earth via a ground socket. The power source should exhibit minimal power transients and fluctuations. Replace faulty or frayed power cords.

General precautions

Perform periodic leak checks on LC tubing and connections. Do not close or block the drain. Do not allow flammable and/or toxic solvents to accumulate. Follow a regulated, approved waste disposal program. Never dispose of such products through the municipal sewage system.

Spare parts and service availability

Manufacturer provides operational spare parts of the instrument and current accessories for a period of five years after shipment of the final production run of the instrument. Spare parts will be available after this five years period on an ‘as available’ basis.
Manufacturer provides a variety of services to support her customers after warranty expiration. Repair service can be provided on a time and material basis. Contact your local supplier for servicing. Qualified chemists on both contractual and as-needed basis can provide technical support and training.

Table of contents

C H A P T E R 1

Introduction

Congratulations on your purchase of the ALEXYS LC 100 HPLC pump. The LC 100 with its semi-floating piston design is a vital component of the ALEXYS 100 LC-EC system. As part of the system pump contribution to baseline noise is practically zero. For µL flow rates a micro head is available and can be easily exchanged. High pressure gradient as well as flow rate programming is supported by the ALEXYS data system. Because the use of buffers and salts is indispensable in LC-EC the LC 100 is equipped with a piston back flush module to maximize the piston and seal life span.

The ALEXYS LC 100 can be used as a highest quality stand-alone pump to be incorporated in any LC system.

C H A P T E R 2

Installation guide

Unpacking

Inspect the *transport box* for possible damage as it arrives. Immediately inform the transport company in case of damage, otherwise she may not accept any responsibility. Keep the transport box as it is designed for optimum protection during transport and it may be needed again. Carefully unpack the instrument and inspect it for completeness and possible damage. Contact your supplier in case of damage or if not all marked items on the checklist are included.
Prior to shipment, your pump has been inspected and tested to ensure the best possible performance. A certificate of performance is included in the ship kit.

Installation

The installation procedure consists of following steps:
1. mounting of pump head
2. electrical connections and settings
3. tubing and fittings
4. priming system with water

To unpack the ALEXYS LC 100, lift it from its box by both hands. Install the pump in an area, which meets the environmental conditions listed in Table I. Place the LC 100 on a flat and smooth surface.

Table I. Environmental conditions.

Parameter	Requirement
Storage temperature	0 – 50 ^{o}C
Storage humidity	0 – 90%, non-condensing
Operating temperature	15 – 30 ^{o}C
Operating humidity	20 – 80%, non-condensing

Avoid precipitation of buffer salts in organic solvents. Always start with flushing system with water when switching solvents.

Never switch off the LC 100 with mobile phase in it as salts will precipitate an clog the system. First flush with water, subsequently with water/methanol.

Step 1. Mounting of pump head

1. Remove the black transport cover and mount the pump head. Install the head using the supplied four bolts. Tighten the bolts only with gentle force!

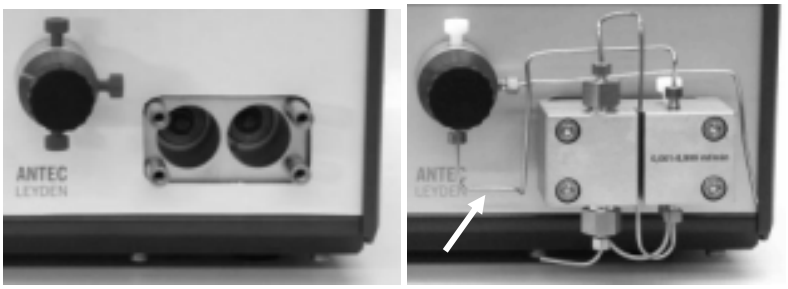

Fig. 1. Installation of pump head.

2. Connect outlet of the pump head to the **under-side** of the purge valve using the supplied s-shaped tubing (Fig. 1, arrow).

Step 2. Electrical connections and settings

3. Before connecting to mains, check the position of the voltage selector and switch to the correct position if necessary. The switch is set to 230 V as a standard, if necessary set to 115 V and change fuses.

When changing mains voltage setting also replace fuses according to rear panel fuse rating.

4. Connect LC 100 mains to OR 100 mains outlet using the power supply cord supplied in OR 100 kit (Fig. 2).

Fig. 2. Rear panel connectors AC and LC 100. D - A - P refers to detector , auto sampler and pump. 'PC' is connection with computer, 'mains' outlet is connected to OR 100.

Step 3. Tubing and fittings

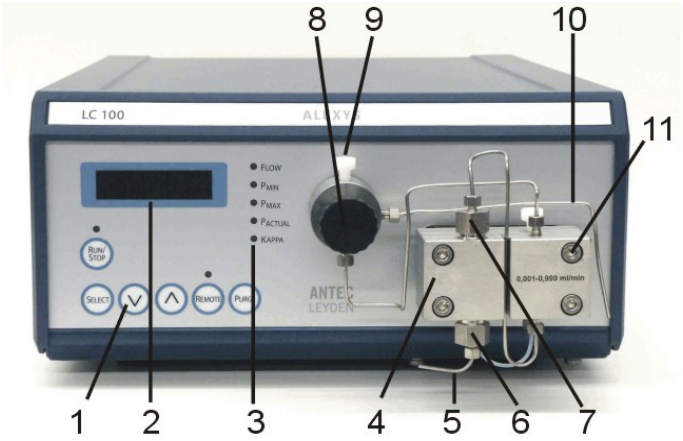

Fig. 3. LC 100 front panel. (1) control keys, (2) display, (3) mode indication LED's, (4) pump head assembly, (5) solvent inlet, (6) inlet check valve, (7) outlet check valve, (8) purge valve with built-in pressure transducer, (9) solvent outlet high pressure side, (10) vent outlet, (11) pump head mounting screws.

5. Switch on the pump, all LED's should light up for about two seconds.
6. Set all parameters to the desired value using Select and ▲or ▼ buttons.
7. Connect the pump inlet (Fig. 3 [5]) to the degasser unit using the supplied FEP tubing. Apply force to push the FEP tubing **all the way up** to the bend in the metal tubing (use sandpaper if necessary).
   In newer models this connection is made with the LC 100 inlet coupler (pn 250.1580, Fig. 4).

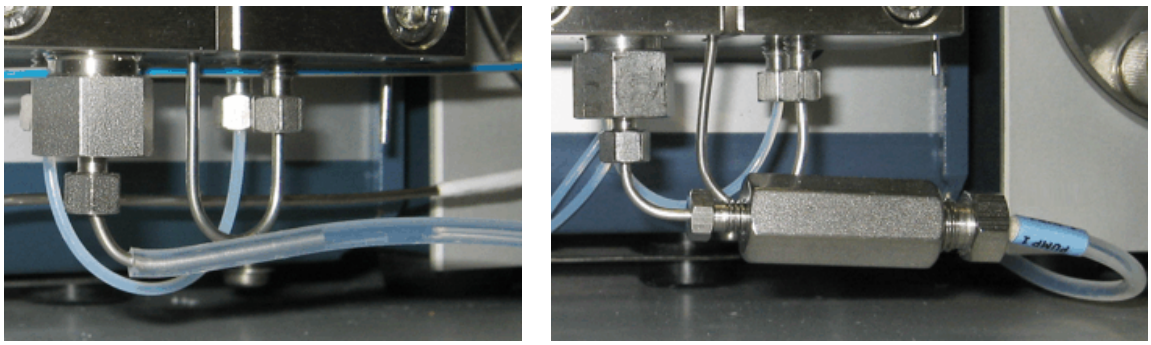

Fig. 4. Pump head inlet tubing connections. Left: tight fit Teflon tubing. Right: LC 100 inlet coupler.

8. The solvent filter should be placed between degasser and mobile phase reservoir (only water for first use). Never use a metal inlet filter in mobile phase bottle!

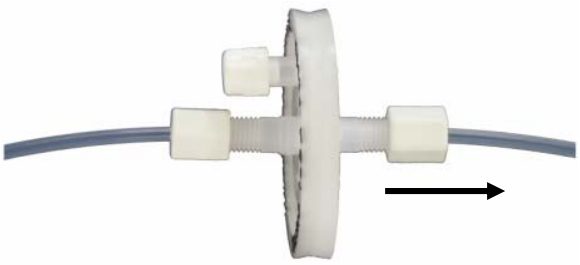

Fig. 5. In-line filter, arrow indicates flow direction. The white knob on top is an air vent which should be opened for filling the filter with mobile phase.

Read specifications on solvent filter. Aqueous type of filters are:
- NOT suitable for high percentages modifier and are
- NOT acid resistant.
Use another type of filter in these cases.

The LC 100 is standard equipped with aqueous type of piston seals made of UHMW Polyethylene. Do not use organic modifier (MeOH, Acetonitril) concentrations higher than 30%v/v in your mobile phase. In case of mobile phases with high organic modifier concentration use teflon-based seals.

9. Connect the pump outlet (Fig. 3 [9]) to the pulse dampener using supplied stainless steel capillary and stainless steel ferrules and nuts.
10. Depending on your configuration connect pulse dampener to a manual or electrically actuated injector or auto sampler. Consult the manual of auto sampler and detector for further installation instructions.

Fig. 6. Piston wash with syringe and wash liquid. Fitting 4 leads to wash liquid, 3 is connected to a syringe. Flush every day with 1 mL of wash liquid using the syringe.

11. Install the piston wash by connecting both pump heads using Teflon tubing as shown in Fig. 6. For aqueous mobile phases wash liquid is usually water. When modifier is used in the mobile phase also the wash liquid may contain modifier (10 – 25 % methanol).

<u>Always</u> connect the piston wash and make sure that the low pressure compartment of the LC 100 is filled with wash liquid. The LC 100 is standard equipped with <u>aqueous</u> type piston wash seals. The presence of wash liquid is necessary for sufficient lubration to prevent damage of the piston wash seals. Also in this case do not use organic modifier (MeOH, Acetonitril) concentrations higher than 30%v/v in your wash liquid.

12. Connect the piston wash syringe to secondary pump head (Fig. 6 [3]).
13. Connect the piston wash outlet tubing (Fig. 6 [4]). Lead Teflon tubing to wash reservoir and fill the wash with water by withdrawal of the syringe.
14. Make sure all connections are leak free. Check also connection 1-2 in Fig. 6.

Step 4. Priming system with water

15. Open purge valve (Fig. 3 [8]).
16. Connect the supplied syringe with supplied silicon tubing to vent outlet (Fig. 3 [10]). Fill the solvent supply line and the pump head with the solvent. Press the purge key to enable rapid self-priming.
17. When primed with liquid stop purging and shut the purge valve, **apply only gentle force**.
18. Click ‘Select’ button until ‘Flow’ LED is indicated. With up/down button set the required flow rate. Start pumping by pressing the Run/Stop key.
19. Click ‘Select’ button until ‘P actual’ LED is indicated. The pump pressure is shown in display.
20. For control from ALEXYS data system switch off the flow (Start/Stop) and press Remote on pump keyboard. Pump control window will show 2 question marks until desired flow rate is entered and ‘Set’.

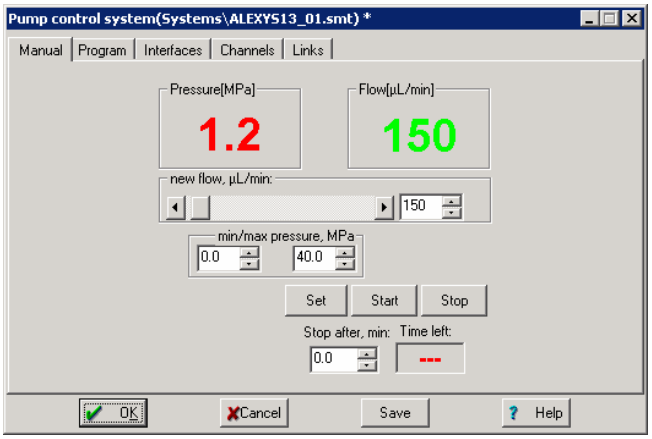

Fig. 7. Control of pump from ALEXYS data system. Use the slider to set the flow rate and click ‘Set’ button.

The pump is now ready for use. Continue with installation procedure of other parts of HPLC system, following instructions in corresponding manual. In combination with electrochemical detection a passivation step with 15% HNO_3 is recommended (see installation guide of detector). First remove all parts that are not acid-resistant such as nylon inlet filters, column and flow cell.

See installation guide of detector for passivation procedure with 15% HNO_3.

C H A P T E R 3

Description of instrument

Front panel

Fig. 8. Front panel LC 100.

Element	Description
Run/Stop	Run/stop key to start or stop pumping. A LED above this key indicates the status (run = on).
SELECT	Pressing this key selects one of five input channels, the active one being indicated by one of the five LED's. Repeated pressing steps through the five input channels cyclically.
▲or ▼	Input keys that increase (▲) or decrease (▼) the displayed value in the active channel.
Remote	Activates external control indicated by the corresponding LED. Values cannot be changed via the keypad when active.
PURGE	Maximises the flow rate determined by the pump head installed, and starts pumping. Can only be activated during pump stop.
Flow	If selected the flow rate is displayed. The flow is adjusted by pressing one of the keys ▲or ▼ .
P_{min}	If selected the minimum pressure limit for safety shutdown is displayed and can be adjusted. If set to 00.0, the minimum pressure shutdown function is inactive.
P_{max}	If selected the maximum pressure limit for safety shutdown is displayed and can be adjusted.
P_{actual}	Displays actual pressure.
Kappa	If selected compressibility is displayed and can be adjusted. For aqueous buffers the value should be zero.

Pump head

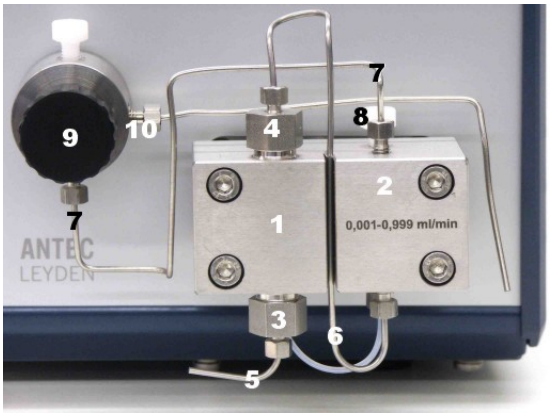

Fig. 9. Pump head.

Element	Description
1. Primary cylinder	
2. Secondary cylinder	
3. Inlet check valve	
4. Outlet check valve	
5. Solvent inlet	This capillary (1 mm ID) must be connected to the solvent reservoir by Teflon tubing with 1.6 mm I.D.
6. S-capillary	Connects primary with secondary pump head.
7. High-pressure solvent outlet	Is connected to the purge valve
8. Piston wash connection	
9. Purge valve	Has a built-in pressure transducer
10. Vent outlet	Low-pressure outlet if purge valve is open

Back panel

Fig. 10. Back panel.

Element	Description
Mains inlet	Connect mains power cable
Voltage selector	Select 115 or 230 V, change fuses as indicated
Fuse holder	Integrated in mains inlet, 5 x 20 mm fuses.
Auxiliary controls connector	For external control of pump, not used in combination with ALEXYS data system
Pressure output socket	High resolution pressure output (1 mV/MPa) enables pressure monitoring through analogue input of AC 100.
Time constant switch	Rise time for pressure monitor output: high (2 s) or low (20 ms).
Current loop / RS232 switch	Switch for external control. Always use ‘RS232’ in ALEXYS 100 LC-EC system.
RS 232 connector	For connection with AC 100 acquisition controller. Set pump to remote by ‘Remote’ key on front panel.
Current loop connector	Not applicable in combination with ALEXYS

C H A P T E R 4

Operation

Electrochemical detection is a sensitive detection technique characterised by extremely low detection limits. A detection limit of 100 pmole/L for catecholamines is no exception. Improving detection limits will always be limited by the weakest link in an LC-EC system. Therefore, in the ALEXYS 100 LC-EC system all hardware is carefully selected to warrant optimum performance.
In daily practice a couple of 'rules' must be obeyed to fully exploit the incredible linear dynamic range and low detection limits of an EC detector. These are not only hardware related, but also refer to mobile phase composition, degassing, temperature and pH stability and several other issues.

Mobile Phase

Mobile phase requirements

- Electrochemically clean, HPLC grade or better
- Ion strength 20 - 200 mmole/L
- Buffer pH near pKa
- In-line 0.2 µm filter & degassing of mobile phase
- EDTA for trapping of metal ions

Mobile phase must be clean as it is in direct contact with the working electrode in EC detection. High purity chemicals including water is a pre requisite. Adding EDTA to the buffer traps electrochemically metals such as Fe^{2+} by forming an inactive complex. However at higher working potentials (typically > 1.2 V vs. salt bridge AgCl ref) also EDTA can become electrochemically active and is not recommended. In that case only a passivation step with 15% HNO_3 is recommended (see installation guide).
Electrolytes assure contact between 3 electrodes in an electrochemical flow cell. Low ion strength destabilises an EC system and noise will increase. Extremely high buffer concentrations cause problems of salt formation. Therefore concentrations between 20 and 200 mmole/L are recommended.
Also constant pH is important for baseline stability and reproducible results. Stability of pH is best when close to pK_a of a buffer ion. Often used buffers are phosphate, carbonate, acetate and citrate. Modifiers such as methanol, propanol and acetonitril can be used without problems in DC amperometry, but not in pulsed amperometric detection as peaks are

strongly attenuated. In our experience the quality and expiration date of organic modifiers can be a problem resulting in increased noise levels. Metal inlet frits in mobile phase bottles are not advised as metal ions are introduced that increase baseline noise. Instead 0.2 µm nylon inlet filters are recommended.

Vacuum degassing

Considerable amounts of the gases N_2, O_2 and CO_2 may be dissolved in HPLC mobile phases. Whenever the temperature changes, solvents are mixed or a pressure reduction occurs, these gases may show up as very small air bubbles. To avoid noisy baselines an in-line vacuum degasser has been integrated in the ALEXYS 100 LC-EC system. It has a very low dead volume (less than 0.5 mL) and a high degassing efficiency. The advantage over helium degassing is that the ALEXYS degasser does not change the mobile phase composition.

Helium degassing

Degassing using helium is an effective and universally applicable method but only recommended when working in reductive electrochemical detection. All gases except helium are removed completely. Helium is not EC active and does not change the mobile phase properties significantly.
To prevent mobile phase contamination, only high-purity helium must be used.

Setting parameters

For computer control see ALEXYS data system manual how to change pump settings. Switch of stand-alone pumping (Start/Stop) and press remote to connect to ALEXYS data system.
When working stand-alone all operational parameters are set using the LC 100 keyboard. Use Select button to go up and down in menu to select Flow, Pmin, Pmax, Pactual or kappa. Corresponding LED lights up when selected. When using up or down button (▲or ▼) parameters are changed.

Pressure limit safety shutdown

Whenever a leakage in high-pressure tubing occurs, an automatic shutdown of the pump is important. Therefore, the ALEXYS LC 100 is provided with a low-pressure safety shutdown circuit. This circuit stops the pump

when the pressure drops below 'P_{min}'. This value may range from 0.1 MPa to the maximum pressure limit. To allow the pressure to build up in the whole LC system, after every pump start using the 'Run/Stop' key, there is a time interval of some minutes in which the circuit cannot be activated. If needed, the whole 'P_{min}' safety shutdown circuitry can be deactivated if 'P_{min}' is entered as 00.0 MPa. As a consequence, at zero pressure the pump will not stop.
Similarly, whenever blocking of a capillary or the column by the sample occurs, a high-pressure safety shutdown circuitry is needed. The shutdown function immediately reacts to prevent damage. The ALEXYS LC 100 shutdown circuit stops the pump in less than 20 ms whenever the actual pressure exceeds P_{max}. P_{max} values accepted range from 0.1 MPa to the pump head limit.
If a P_{max} or P_{min} limit has been attained, the pump stops, the actual pressure at the moment of shutdown is displayed, and the indicator LED 9 or 10, fig. 2, starts blinking, thus marking the kind of limit that has occurred.

Compressibility correction

The ALEXYS LC 100 is able to attain a pressure of 50 MPa (60 MPa for micro head). At such a pressure the density of liquids is substantially higher than at low-pressure conditions. The compressibility K of liquids is defined at constant temperature T as:

$$\kappa = -\frac{1}{V} \cdot \left(\frac{dV}{dP}\right)_T$$

where V is the volume of the liquid and P the pressure on it.

Non-zero compressibility implies that a solvent is compressed on pressurization. Hence, during the working stroke of the piston the mobile phase is compressed until the value attained behind the outlet valve. During this (pre)compression phase, no solvent is delivered. Since the compressibility varies with solvents, the compression phase depends on the mobile phase composition. It also depends on the system pressure. To prevent pulsations the second piston delivers in the priming and compression phase of the first.

Table II. Isothermal compressibility K of some solvents

Solvent	**K (10^{-11} Pa^{-1})**
water	45
acetone	128.9
acetonitril	97.4
dichloroethane	111.9
dimethylformamide	80
dioxane	60
ethanol	115
ethyl acetate	100
methanol	125
hexane	167.2
propanol	98
tetrahydrofuran	95

The ALEXYS LC 100 can correct for compressibility differences of solvents by means of a kappa adjustment. This adjustment reduces pulsations and makes the flow rate virtually independent from compressibility and pressure changes. In good approximation this is simply achieved by keying in the appropriate K-value from Table II or from data books. If lowest pulsation is desired, this can be reached by manually adjusting the K -value as follows:

1. Connect pressure monitor output with one of the input channels of AC 100 using pressure monitor cable.
2. Set filter switch on back panel to the ‘LOW’ position.
3. Install a backpressure capillary to pump outlet and run the pump at the desired flow rate. About 50 cm of 80 µm capillary should be enough to get about 100 bar at 1 mL/min.
4. Observe the pressure signal. Key-in stepwise increasing values for the compressibility in the ‘Kappa’ input mode using the keys ▲or ▼
5. Monitor the pressure signal simultaneously, which will change starting from low K -values to higher ones.

For aqueous buffers minimum pressure pulsation occurs at factory set K value of 0.

This value is dependent on the mobile phase composition and on the pump head as well. The numbers found in this way are rough estimates for the absolute compressibility.

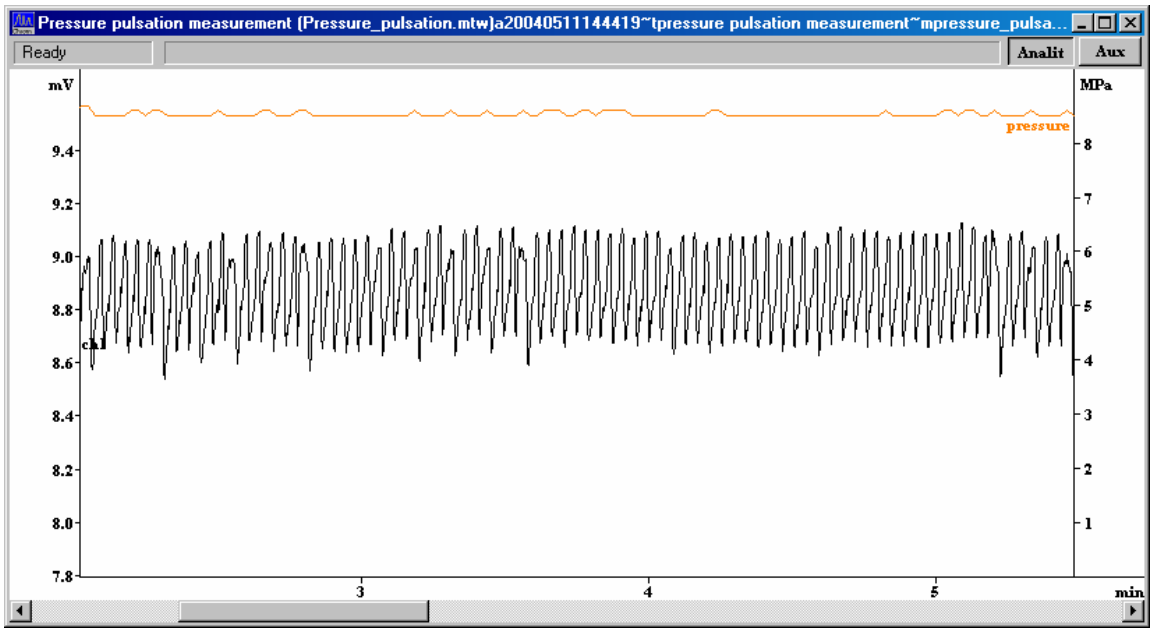

Fig. 11. Measurement of pressure: connecting pressure monitor to analogue input of ALEXYS data system. No pulse damper applied.

Flow correction

In manufacturing pump heads and components there are small tolerances that cannot be avoided. So the flow rate entered can be slightly different from the one selected. To compensate for this the flow can be corrected. The correction factor Nx = 1.00 ± 0.1 by steps of 0.01.has a range from 0.90 to 1.10, implying that the flow can be corrected by ± 10%. To have access to the correction factor N_x the SELECT key must be pressed longer than 2 seconds. Then a value for N appears and can be changed with the ▲or ▼key. If N is adjusted SELECT must be pressed again to return to standard control functions. The value will be stored. However, if the battery is empty or fails this value is lost.

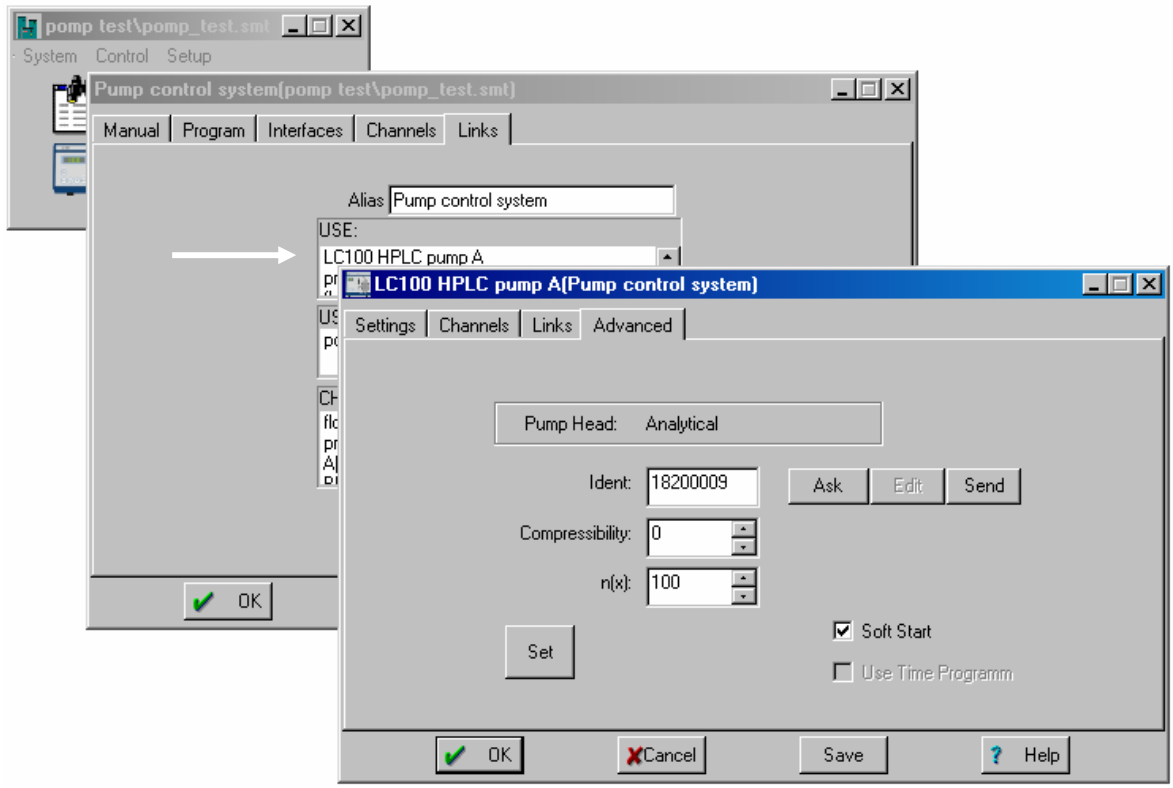

Fig. 12. Advanced pump settings in ALEXYS data system. Double clicking on LC100 HPLC pump A (arrow) opens a second window with advanced settings. Flow rate correction n(x) is entered as a percentage.

C H A P T E R 5

Maintenance

All moving parts are subject to normal wear and tear. It is therefore unavoidable that certain parts have to be replaced once in a while. The LC 100 is constructed in a way that worn out parts can easily be reached and replaced with a minimum of downtime.
In this section maintenance of most common parts is described:
- replacement of seals
- replacement of pistons
- maintenance of check valves

starting with disassembly of the pump head.

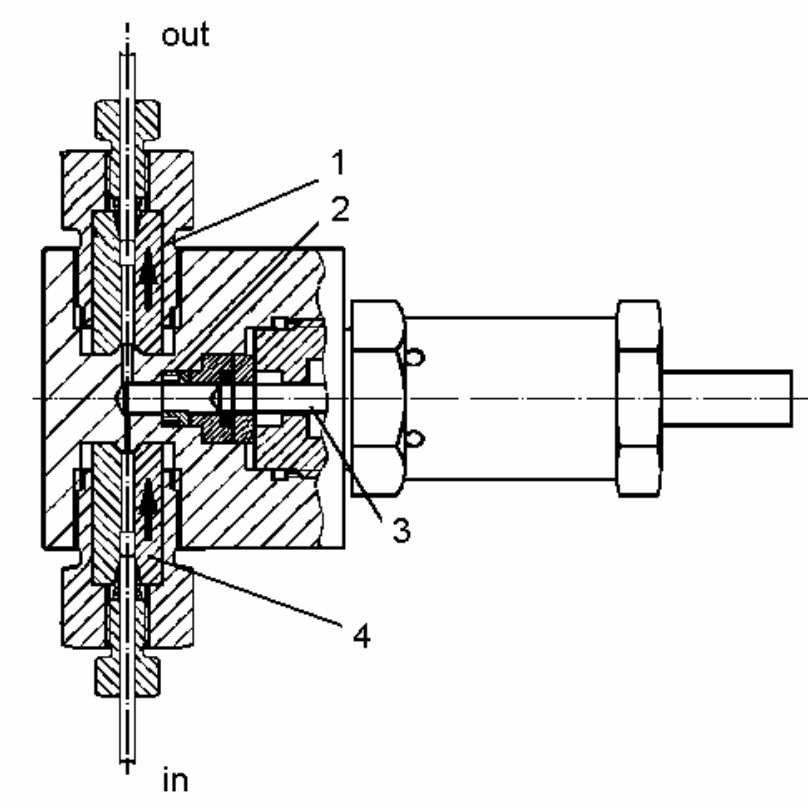

Fig. 13. Cross-section of primary pump head with check valves (1, 4), seal (2) and piston (3).

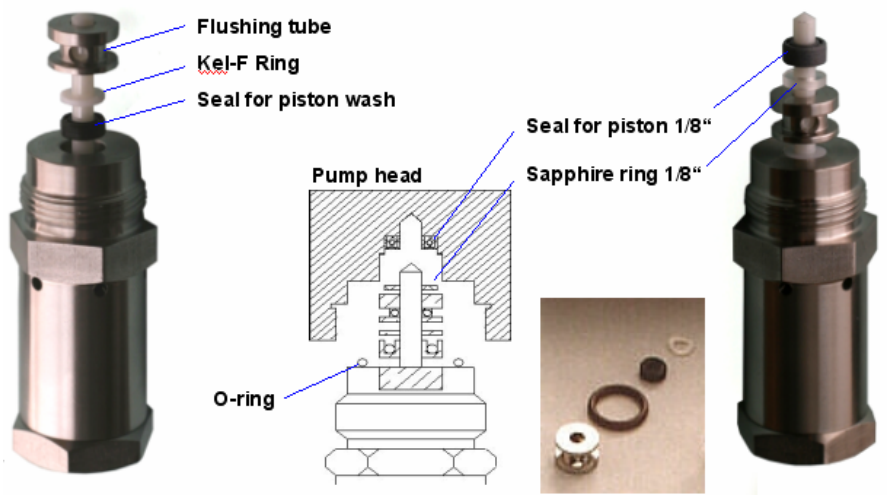

Fig. 14. Pump head with all parts.

Description	Part std.	Part micro
Pump head body	182.0336	182.0302
Seal for piston*	182.0416	182.0418
Sapphire ring*	182.0348	182.0322
Washing tube	182.0362	182.0334
Kel-F-ring	182.0360	182.0332
Piston wash seal	182.0422	182.0420
O-ring	182.0364	182.0364

* 1/8“ for standard, 1/16“ for micro.

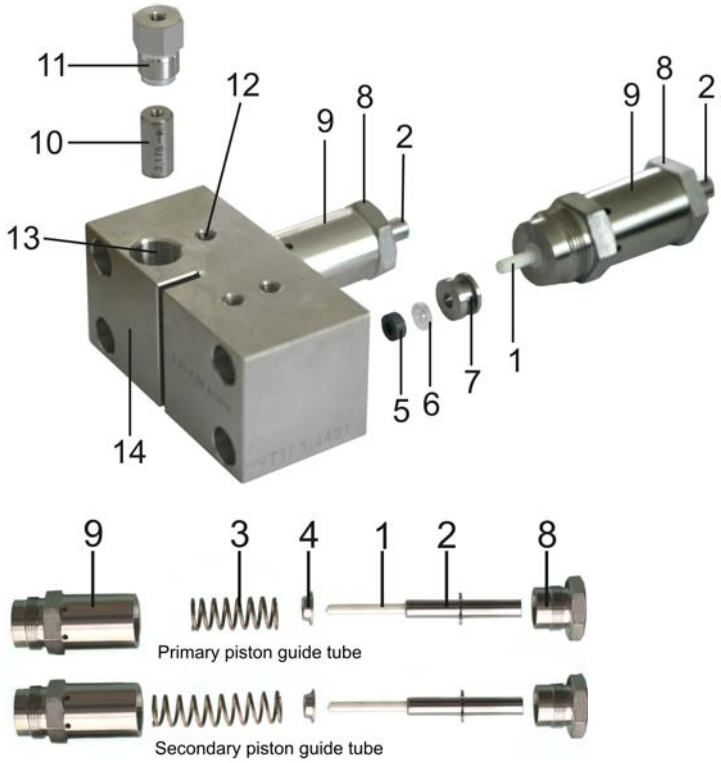

Fig. 15. Exploded view of piston and pump head(analytical version).

Nr	Description	Part std.	Part micro
1-9	Piston assembly complete primary	182.0338	182.0304
1-9	Piston assembly complete secondary	182.0344	182.0304
1, 2	Piston & shaft	182.0346	182.0320
3	Spring primary	182.0318	182.0318
3	Spring secondary	182.0312	182.0318
4	Teflon guide	182.0340	182.0306
5	Seal for piston	182.0416	182.0418
6	Sapphire ring	182.0348	182.0322
7	Washing tube	182.0362	182.0334
8	Piston cartridge screw	182.0308	182.0308
9	Piston cartridge	182.0314	182.0314
-	Check valve inlet	182.0352	182.0326
10	Check valve outlet	182.0354	182.0328
11	Check valve screw	182.0356	182.0356
14	Pump head body	182.0336	182.0302

Replacement of seals

Lifetime of seals is limited to 6 – 10 months due to normal wear and tear. Leakage of solvent around the pump head usually indicates damage to seals and replacement is necessary.

Avoid excessive wearing of parts: use aqueous seals with < 35 % organic modifier. Use solvent seals with > 25 % organic modifier.

Always use the correct seals for your application. Standard the LC 100 is equipped with aqueous type seals.
To replace seals, the pump head is removed from the pump, piston guide tubes are taken out (it is not necessary to open the pistons!). Seals should be replaced using a special piston replacement tool. This tool can be purchased by your local representive.

1. Remove the pump head from the pump by removing the outlet tubing and the four bolts at the front of the pump head. Put marks on the piston cylinders which one is right, and left!
2. Remove the piston cartridges from the head.

Note that both piston guide tubes are different for the analytical pump head, place marks and do not interchange them!

3. Take the O-ring, flushing unit and sapphire ring out of the pump head. If the sapphire ring does not come out, use a pipette tip to pick it up.

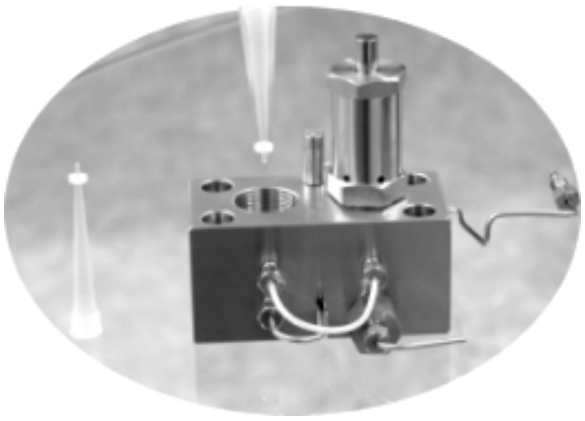

Fig. 16. Taking out the sapphire ring carefully, using a pipette tip.

4. Remove old seal using special tool as shown in Fig. 17. Place the shaft in the pump head; screw the tool in old seal and with the thread of the tool pull out gently the old seal.

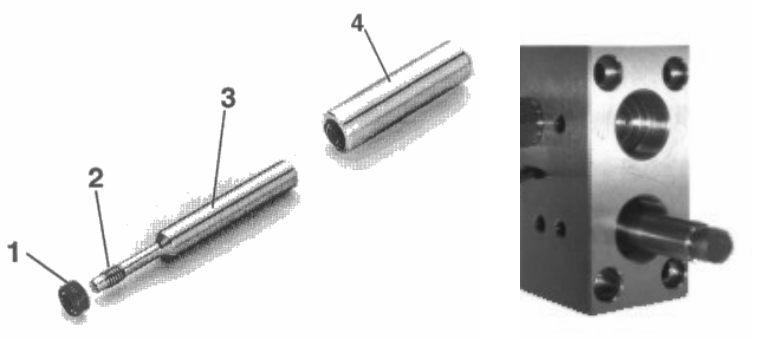

Fig. 17. Tool for taking out old seal. Piston seal (1), thread (2), shaft (3) and setting sleeve (4).

Removing the seal is destructive, therefore the seal must only be taken out when worn out or damaged by salt abrasion.

5. After removal of seals clean the pump head thoroughly with water and check carefully that no particles are left.
6. Press the new seal firmly but manually into the tool, the seal spring being in outside position (must be visible). Then put the tool into the pump head and press the seal into its seat inside the pump head using the other end of the piston pullout tool.

Fig. 18. New seal in replacement tool.

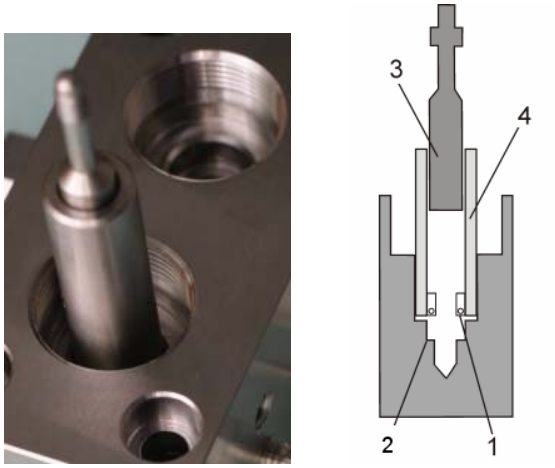

Fig. 19. New seal (1) pushed in place (2) using seal tool (3, 4).

7. When reinstalling the pistons in the pump head: **keep the pistons always in upward position**. Place piston seal, Kel-F-ring, washing tube, and sapphire support ring on the piston (Fig. 20).
8. Place the piston in the head and tighten the pump piston in its position. Apply only gentle force!

During assembly keep piston in upward position, to avoid parts falling of the piston.

Fig. 20. During assembly keep piston in upward position.

Note that all bolts must be finger tight only.

Never scratch over the sealing surface in the pump head. Avoid any hard tools touch this sealing surface, at all.

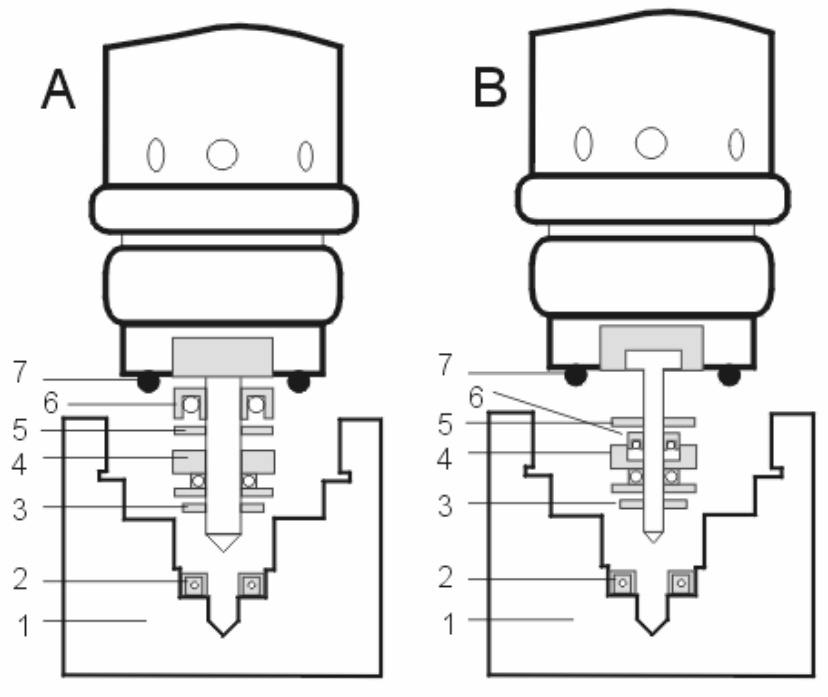

Fig. 21. Standard (A) and micro (B) pump head.

Pump head parts
1. Pump head
2. High pressure seal
3. Sapphire ring
4. Washing tube (or flushing tube)
5. Kel-F ring
6. Piston wash seal
7. O-ring

Micro head is different from standard head:

- washing seal (6) is integrated in the washing tube (4)
- mounting order Kel-F ring (5) is exchanged
- smaller piston diameter

Replacement of piston wash seals

Avoid excessive wearing of parts: use aqueous seals with < 35 % organic modifier. Use solvent seals with > 25 % organic modifier.

To replace the piston wash seals execute the following procedure:

Analytical pump head

1. Remove the white Kel-F ring covering the piston wash seal located inside the cavity in the piston cartridge (Fig. 21, 5).
2. Remove the piston wash seals (Fig. 21, 6) from its seat in the piston cartridge using the seal replacement tool.
3. After removal of seals clean the seats in the piston cartridge thoroughly with water and check carefully that no particles are left.
4. Install new piston wash seals manually. The seal springs should face towards the pump head. So the seals should be visible from the outside of the piston cartridge.
5. Re-assembly the piston guide tubes.

Micro pump head

1. For the micro pump head the piston wash seals (Fig. 21, 6) are integrated in the flushing tube. Remove the piston wash seals from its seat in the flushing tube using the seal replacement tool.
2. After removal of seals clean the seats in the flushing tube thoroughly with water and check carefully that no particles are left.
3. Install new piston wash seals manually. The seal springs should face towards the pump head. So the springs should not be visible from the outside of the flushing tube.

Cleaning check valves

When servicing check valves make sure that inlet and outlet check valves are not interchanged. Put marks on the check valves if necessary. See Fig. 13 for cross sectional view.

1. Remove the pump head from the pump by removing the outlet tubing and the four bolds at the front of the pump head. Put marks on the check valves!
2. Remove the check valves from the head, put them in a beaker with methanol and clean them by placing beaker in an ultrasonic bath for about 5 min.
3. After cleaning, take out valves and shake the valve in up and down ward direction. Listen for ticking of the ball in the check valves. This indicates that the check valves are freely moving, if not, repeat step 2.
4. After cleaning the check valves assemble the pump head and re-install.

Note that check valve assemblies 3 and 4 are different, do not interchange them!

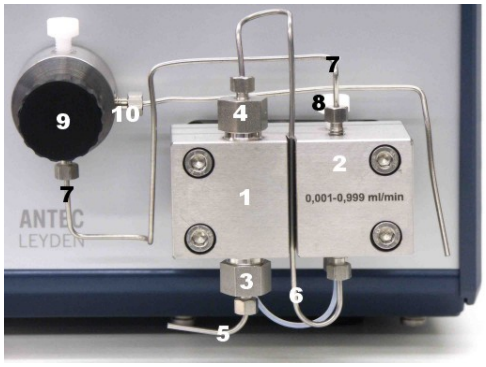

Fig. 22. Pump head with check valves (3 and 4). Outlet capillary (7) and four bolds must be removed before the head can be taken off.

Interchanging the inlet and outlet valves will destroy the piston seal since an extreme pressure is then built-up inside the working cylinder. In this case the pressure transducer will not sense the pressure inside the cylinder and hence automatic overpressure shutdown cannot work!

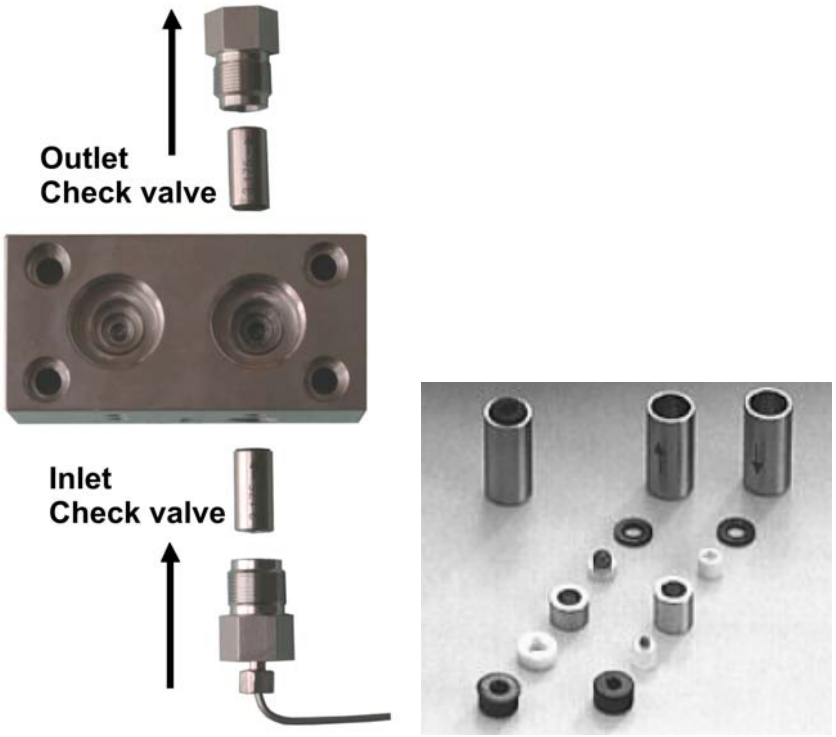

Fig. 23. Exploded view of pump head and check valves. Note the arrows on the check valves should point the direction as indicated.

Replacement of pistons

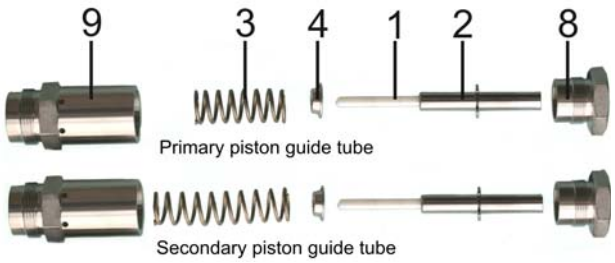

Fig. 24 Exploded view of piston guide tubes of analytical pump head: 1. piston, 2. piston shaft, 3. spring, 4. ring, 8. piston cartridge screw, 9. piston cartridge.

1. Remove the pump head from the pump by removing the outlet tubing and the four bolts at the front of the pump head. Put marks on the piston cylinders which one is right, and left!
2. Remove the piston cartridges from the head.

Note that both pistons guide tubes for the analytical pump head are different, do not interchange them!

3. Take the O-ring, flushing unit and sapphire ring out of the pump head. If the sapphire ring does not come out, use a pipette tip to pick it up (Fig. 16).
4. Open the piston guide tubes but be careful because the spring inside the cartridge is under pressure. Keep firm contact with shaft and bold while disassembling other wise parts will spring away or even hurt the operator.
5. After replacement assemble the pump head and re-install.

Precautions and system shutdown

The ALEXYS 100 LC-EC system has been developed for continuous operation. For maximum stability it is advised to leave the system ON continuously. If preferred, the flow cell may be switched OFF at night. However, precautions should be taken when:

- system is not in use for longer period of time (more than a few days)
- using mobile phase buffers at pH < 2 or pH > 9

- using corrosive mobile phase

1. Always switch off flow cell when changing to solvents without electrolytes.
2. First read column manufacturer recommendations. Take out column in case of solvent incompatibility.
3. Start always with flushing LC system with water when switching solvents.

Corrosive solvent precautions

When the system is not in use overnight or weekends switch off the cell and read column manufacturer recommendations. Flush with an appropriate solvent and only take out column in case of solvent incompatibility. Flush system with pure water and set the HPLC pump to a low flow rate. Next working day change water for mobile phase and continue operation. Before switching on the cell make sure mobile phase has been flushed throughout the system.
Corrosive solvents are: high concentrations of sodium hydroxide (carbohydrate analysis) or other buffers of pH>9 or pH < 2.

System shutdown

When not in use for a number of days flush the ALEXYS 100 LC-EC system first with water then with 75/25 water/methanol. Store column and flow cell according to manufacturer recommendations. Then turn off all ALEXYS system components.
Before using the LC 100 again, completely purge the system with the correct mobile phase before reconnecting the column and restarting the system.

Avoid precipitation of buffer salts in organic solvents. Start always with flushing system with water when switching solvents.

Replace blown fuses with fuses of proper type and rating stipulated on the side panel and specified in the installation section of this manual. The fuse holder is integrated in the mains inlet.

C H A P T E R 6

Trouble shooting

Power status LED off

Possible cause	Remedy
No power	Plug in power cord, if OR 100 is used check power on this device
Power off	Switch on device using the on/off button at the front panel

No RS232 communication

Possible cause	Remedy
No RS232 connection	Plug in RS232 cable(s)
Wrong COM port assigned	Reconfigure LC 100 in ALEXYS data system. See ALEXYS software manual.
AC 100 does not respond to commands	Reset AC 100 following the reset procedure described in maintenance section of AC 100 manual

Excessive pump pulsations

Possible cause	Remedy
Detector related problem	See detector trouble shooting guide
Back pressure too low for pulse damper	For optimal dampening of flow pulsations a backpressure of at least (7 MPa = 70 bar =1000 psi) is needed. Use backpressure regulator or restriction capillary *between pulse damper and injector* to increase pressure
Check valve	Clean or replace check valves.
Seal	Replace seal
Piston	Open pump head, clean or replace pistons

Leakage

Possible cause	Remedy
Connectors	Check connectors, replace if necessary
Leakage of seal	Replace seal

When trouble shooting is not successful and problems persist please contact your local supplier for service.

A P P E N D I X 1

Specifications ALEXYS LC 100 Pump

General specifications	Power	115 or 230 VAC ± 10 %, 50/ 60 Hz, 50 VA max
	Operating conditions	15 – 30°C, 20 – 80% RH, non-condensing
	Flow rate	0.01 - 4.99 mL/min, 0 - 50 MPa micro: 0.001- 0.999 mL/min, 0 - 60 MPa
	Flow rate accuracy	≤ 1.5 % at 20 - 80 % FS
	Displacement volume	primary piston 45 (11) µL secondary piston 14,5 (3.5) µL for analytical (micro) head
	Pressure protection shut down	max: 0 to FS, switch-off time ≤ 20 ms min: 0.1 to FS, switch-off time ~ 2 min
	Keyboard & display	flow rate, compressibility, pressure (actual, max, min)
	Auxiliary outputs	suction phase, pump stopped
	External control	full control, flow rate programming, high-pressure gradient programming (up to quaternary) with ALEXYS data system, automatic pump head identification
Physical specifications	Dimensions	40 (D) x 26 (W) x 13 (H) cm including pump head 15.8 “ (D) x 10.1” (W) x 5.1” (H)
	Weight	8.6 kg (18.9 lbs) including pump head

A P P E N D I X 2

Accessories

The following set of accessories is supplied with each ALEXYS LC 100

Part no	Description	Qty
182.0200	LC 100 ship kit	1
182.0524	Wrench 1/4” – 5/16”	1
182.0526	Allen key 4mm/DIN 911	1
182.0408	Syringe	1
182.0386	Hollow needle	1
182.0388	Silicon tubing	10 cm
182.0400	Teflon tubing	2 m
182.0402	Stainless steel capillary 1.6 / 0.25mm diameter	50 cm
182.0404	Fitting screws	2
182.0406	Ferrules	2
182.0528	Power cord EUR	1
182.0530	Power cable US (UL-certified)	1
182.0532	Replacement fuse 0.25 AT (EUR)	1
182.0534	Replacement fuse 0.5 AT (US)	1
182.0536	Allen screws mounting pump heads	4
250.7204	Ship kit list	1
182.0542	Instruction sheet line voltage selector	1
182.0544	Serial cable 9F-25F pins, 1 meter	1
	Inline solvent filter	1

Index