ALEXYS LC 100
Pump

PQ

Identification

The undersigned engineer certifies that he/she is trained and qualified to perform an PQ on the LC 100 pump.

Performer: ---------- ---------- ----------
Name Signature Initials

Company ----------

Title: ----------
(Anted Leyden representative trained and qualified to perform PQ procedures)

The undersigned reviewer/customer accepts that the above-mentioned engineer is trained and certified to do PQ/OQ on all Antec Equipment.

Reviewer/
Customer: ---------- ---------- ----------
Name Signature Initials

Company: ----------

Title: ----------
(Owner-designated authorized person)

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Instrument

☐ LC 100 p/n: ..... s/n: .....

☐ Pump head Analytical / Micro

Manufacturer Antec Leyden

Supplier .....

Date of delivery .....

Warranty until .....

Test equipment

AC 100	Part number	.....
	Serial number	.....
	Calibration date	.....
or		
Line recorder	Model	.....
	Serial number	.....
	Calibration date	.....
Flow meter	Model	.....
	Serial number	.....
	Calibration date	.....
ALEXYS data system	Revision	.....
		.....

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

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Verified by (customer): …….. Deviations (Y/N): ……

Comments:

C H A P T E R 1

Introduction

This document describes the Operational Qualification procedure as advised by the manufacturer. It is a result from our interpretation of many regulations and laboratory practises. In addition, feedback from users and representatives helped us to finalize this procedure.

A complete PQ for the LC 100 pump consists of two qualification checks. The following test procedures should be executed to check if the pump operates within specifications:

- Flow rate deviation measurement
- Residual pressure pulsation measurement

It may be necessary to re-adjust the pump in case the performance specifications are not met. For detailed information about pump readjustment consult the LC 100 service manual (p/n 182.0020).

All qualification checks have to be approved, or should be marked "n/a" if not applicable. Any deviation observed must be documented in the 'non-conformance' record. All relevant documents regarding this operational qualification must be filed together in one location.

As regulations and customer requirements may change, manufacturer reserves the right to introduces changes without prior notice. For details on functionality, operation and theory reference is made to the instrument users manual.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

C H A P T E R 2

PQ for LC 100 pump

Test configuration

Mobile phase	Degassed & filtered HPLC-grade water
Piston wash	HPLC-grade water
Capillary	1/16” PEEK restriction capillary (127 um I.D)
Flow meter	calibrated flowmeter or graduated analytical pipette & stopwatch
P	system pressure between 100 - 200 bar
P signal	Analog pressure output of the pump monitored using an AC 100 and ALEXYS data system software, sampling frequency 10 Hz, or line-recorder
Pmonitor	LC 100 time constant: high (2 sec)
kappa	45 (compressibility)

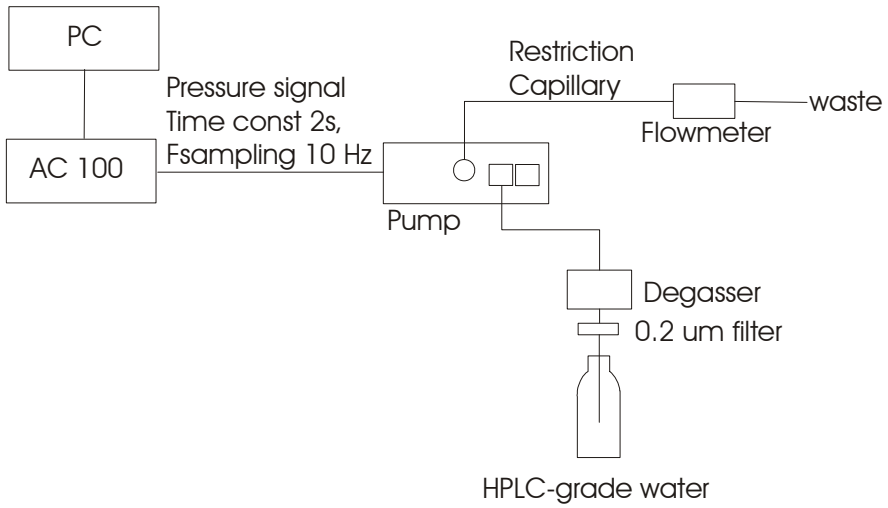


Fig. 1. Test set-up for measuring flow rate deviation & pressure pulsation.

Before running a PQ test the pump should be running for at least 5 minutes under the specified test conditions.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

All tests described in the PQ procedure are based on a LC 100 with analytical pump head. In case of a LC 100 with micro pump head all flow rates mentioned in the tests should be changed accordingly. The flow rate of the micro head should correspond to the same flow rate in % FS (Full Scale) as the analytical pump head. The PQ tests are performed at 20 and 40% FS which corresponds to the following flow rates:

	Analytical	Micro
20 % FS	1 mL/min	200 µL/min
40 % FS	2 mL/min	400 µL/min

Pressure pulsations test

Before starting the Pressure pulsation test download the latest version of the zip file "LC100_P-test_revxx.zip" from the Antec web site at http://www.antecleyden.com/. The zip file contains ALEXYS system and method files to perform the test, and an Excel worksheet "LC100_P-test_revxx.xls" were the residual pressure pulsation is calculated automatically. Store the ALEXYS system and method files in the corresponding directories in the ALEXYS program files directory.

1. Open the ALEXYS system file "LC100_P-test_xxxyy.smt".
2. Connect AC 100 (controlled by ALEXYS software) to the analogue pressure monitor (1mV/MPa) to monitor the pump pressure .
3. Set the compressibility Kappa to 45.
4. Set TIME CONST. to HIGH (2s).
5. Set pump to REMOTE.
6. Start run in Alexys. Subsequently a run will start of 15 minutes. The first 10 minutes the pressure signal is monitored at a flow rate 1 mL/min. After 10 minutes the flow is stopped automatically. The program will continue measuring for 5 minutes in order to determine the pressure at 0 mL/min. A typical pressure profile obtained with such test is shown in figure 2.

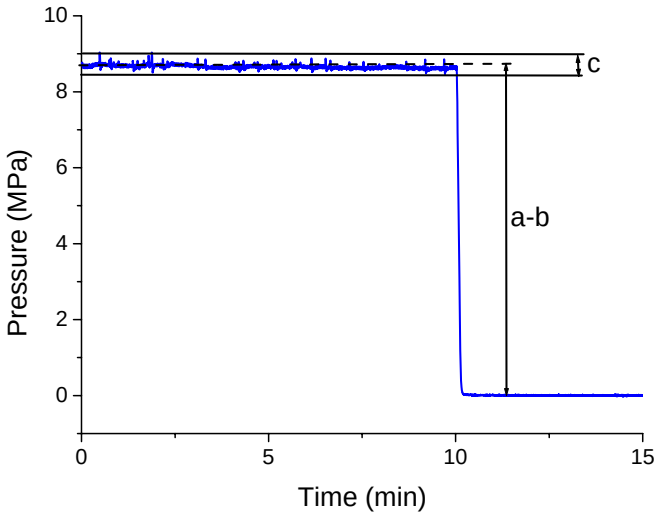


Fig 2. Calculation of pressure pulsations %P.

7. Export the Pressure data to ASCII in ALEXYS using the option "export raw data to txt" in the file menu. Do not include the retention time for export, only the pressure data.
8. Open the generated txt file and select all Pressure data. Subsequently, copy the data into the second column "pressure (MPa)" in the Excel worksheet "LC100_P-test_revxx.xls"

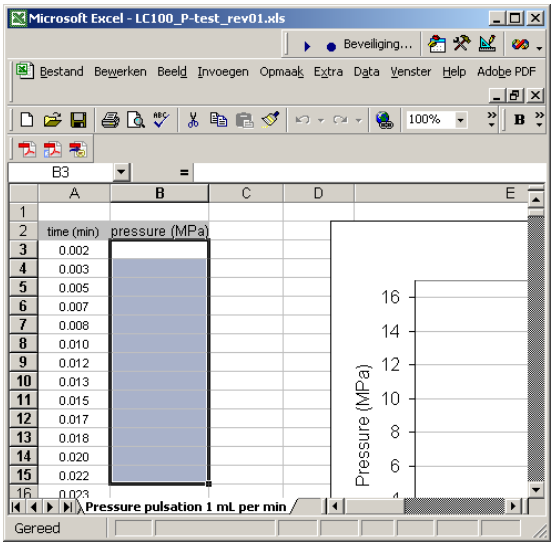


Fig 3. Spread sheet to calculate the Residual P pulsations (%P).

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

The Residual P pulsations (%P) will be automatically calculated in the Excel worksheet:

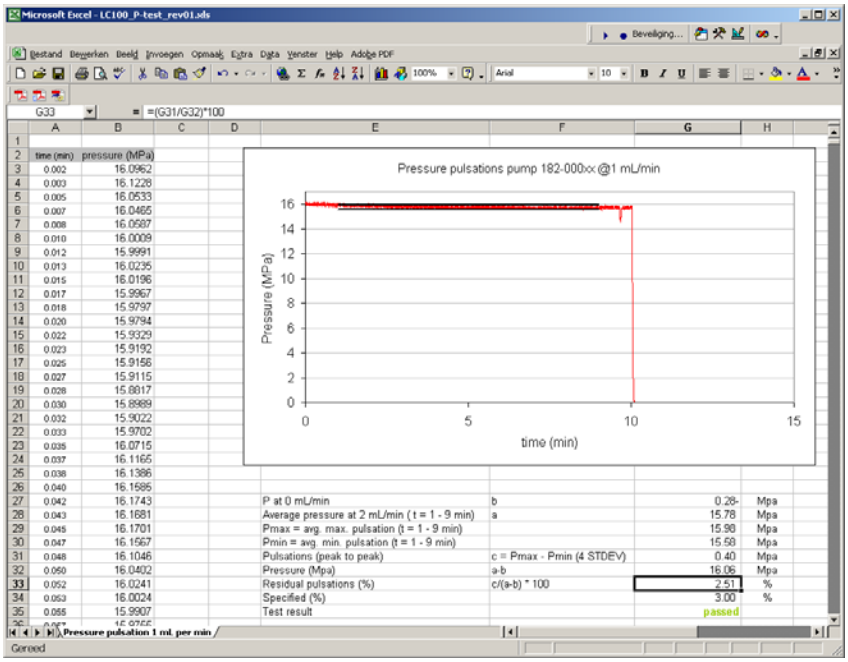


Fig 4. Example of results Residual P pulsations.

Pressure pulsations %P must be smaller than < 3%.

9. If the instrument fails test, first check for leakage or air in system, if pump head is assembled correctly and all critical parts (check valves, pistons etc.) are in tact . If the problem remains consult the test & readjustment section in the service manual.

When using a line-recorder to record the pressure profile, please follow the procedure below to determine the residual pressure pulsations:

1. Connect a line-recorder to the analogue pressure monitor (1mV/MPa) to monitor the pump pressure .
2. Set the compressibility Kappa to 45.
3. Set TIME CONST. to HIGH (2s).
4. Set the flow rate to 1 mL/min.
5. Press <R/S> to start the pump.
6. Record the pressure signal for 10 minutes via the pressure monitor output at 1 mL/min.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

7. After 10 minutes stop the flow and record the pressure signal for 5 more minutes to determine the pressure at 0 mL/min.

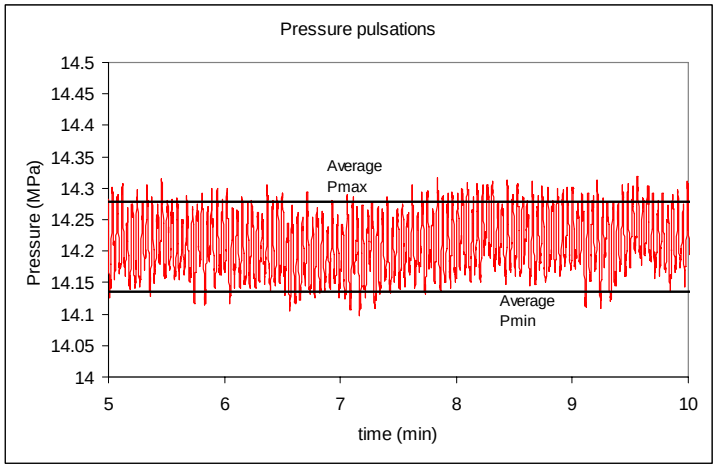


Fig. 5. Pressure signal with lines indicating Pmax and Pmin.

8. When using a line recorder determine the <u>average</u> maximum and minimum pressure values at 1 mL/min, as depicted in figure 5 . Draw straight parallel lines through minimum and maximum. Subsequently, determine c (c = Pmax-Pmin).

9. Calculate the residual pressure pulsations in the following way (see figure 2):

$$\%P = \frac{c}{(a-b)} * 100$$

Where:

c = Pmax - Pmin (MPa) *or* 4σ .

a-b = Actual system pressure (average pressure at 1 mL/min - average pressure at 0 mL/min)

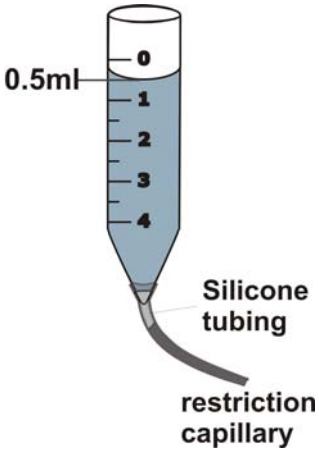
Pressure pulsations %P must be smaller than < 3%.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Flow rate deviation test

1. Set the flow rate to 1 mL/min and check if purge valve is closed.
2. Connect the inlet of the graduated analytical pipette to the outlet of the restriction capillary using a short piece of silicon tubing (see figure 6). Or connect the restriction capillary to a calibrated flow meter.
3. Press <Run/Stop> to start the pump.



Correct volume reading from the pipette is at the lowest point of the meniscus.

Fig. 6. Flow measurement with graduated 5 mL pipette.

4. Measure the flow rate with the pipette using the following procedure: start the stopwatch exactly at the moment that the liquid meniscus in the pipette passes the 4 mL line (for a good reading it is important that the pipette is kept perfectly vertical).
5. Stop the flow rate exactly at the moment that the liquid meniscus in the pipette passes the 0 mL line.
6. Calculate the actual flow rate. Flow rate = 4 mL/ stopwatch time (min). In case of using a calibrated flow meter complete 5 flow meter readings and calculate the average. Subsequently calculate the flow deviation:

$$\text{Flow deviation \%} = \frac{(\text{actual flowrate} - \text{set flow rate})}{\text{set flow rate}} * 100$$

7. Repeat the flow deviation measurement three times, the Relative Standard Deviation (RSD) of a triplicate

measurement should be < 1 %. If this is not the case please repeat all three measurements. The average flow deviation (n=3) should be smaller than +/- 3%.

8. Repeat this experiment at 2 mL/min. Measure the flow deviation three times, the Relative Standard Deviation (RSD) of a triplicate measurement should be < 1 %. If this is not the case please repeat all three measurements. The average flow deviation (n=3) should be smaller than +/- 3%.
9. Also at a flow rate of 2 mL/min perform a triplicate the flow deviation should be smaller than +/- 3%.
10. If the instrument fails test, first check for leakage or air in system, if pump head is assembled correctly and all critical parts (check valves, pistons etc.) are in tact . If the problem remains consult the test & readjustment section in the service manual.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

C H A P T E R 4

PQ certification

The Operational Qualification has been carried out in accordance to the PQ procedure and has been carried out to the satisfaction of both parties. All tests as described in this document have been successfully completed, and all results are within specifications.

Test results	Spec	Measured	Result
Pressure pulsations at 20% FS	< 3 %	%	☐ passed ☐ failed
Flow deviation at 20% FS	< 3 %	%	☐ passed ☐ failed
Flow deviation at 40% FS	< 3 %	%	☐ passed ☐ failed

Executing technician

Technician name & signature	…….. ……..
Company	
Date	

Customer (authorised to sign)

Name & signature	…….. ……..
Company/dept.	
Date	

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Comments

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

C H A P T E R 5

Non-conformance record

Any case of non-conformance found during the PQ procedure should be documented and signed for acceptance or corrective action taken.

Table I

Ref.	Non-conformance and action taken	Signature customer	Sign. executing technician
1		……	……
2		……	……
3		……	……
4		……	……
5		……	……
6		……	……

Verified by (customer): …….. Deviations (Y/N): ……

Comments: