

1 CHANGE HISTORY:

Version	Description of Change
00	Initial Release

2 PROJECT INFORMATION:

Project Name	Project Manager/Lead	REL RFT
Luna	Jim Usowicz (x3580)	721014774

3 DEVICE UNDER TEST / ANALYSIS:

Qty	Description	Project Phase	PN	Rev
4	1/16" Zr Ball, Al Seat Check Valve	Feasibility	289013004	C
4	Optimize Ceramic CV Cartridge, 1/16"	Feasibility	289002430	A
4	Assy, Cartridge, Check Valve	Feasibility	289000144	F

Note: Units tested are assumed to be representative of future production units unless otherwise noted

4 SYNOPSIS:

4.1 Purpose: The purpose of this test is to evaluate the robustness of a prototype ceramic ball-and-seat check valve (PN 289013004) that is intended to address "Loss of Prime" issues observed by customers running buffered solvents with Alliance Separations Modules. The performance of this prototype design will be compared to that of the check valve options already offered by Waters under the same user conditions. Findings will be communicated to engineering to enable them to make a decision about whether or not further testing will be required for this design.

4.2 History: A common complaint to be addressed during the Luna Project is loss of prime. Customers claim this occurs when the standard ruby sapphire Alliance check valve (PN 289000144) is installed, but not when the third-party "Optimize" ceramic check valve (PN 2890002430) is installed. The test condition for which the loss of prime error is reported to occur is when phosphate buffer/ACN gradients are used. Customers have been rejecting the other two ceramic ball-and-seat check valves offered by Waters (PN 289001463 and 289001551). The Optimize check valve design is rated to ~3000psi, while Alliance can operate up to 5000psi. The goal is to provide a ceramic ball-and-seat check valve that will not throw this loss of prime error and will allow customers to operate successfully within the Alliance pressure range. A prototype design (PN 289013004) is being considered and the reliability of this design must be tested. One customer, Aurobindo provided details for a method they use (which is USP-certified), which reportedly causes precipitate to form during the gradient. In tests run at Waters, this method has been used to represent a typical customer method.

5 ASSIGNMENTS:

Assigned REL Engineer	Assigned REL Support	RDE or Technical Contact
David Seigal (x2643)	Kurt Banker (x3547)	Austin Allen (x2780)

6 PROPOSED SCHEDULE:

6.1 Total Test Time: ~250 Days

Activity	Est. Time to Completion
Set Up	5 Days
Check Valve Cycling	245 Days
Total	250 Days

Note: Schedule assumes no issues arise during testing

7 PROPOSED SETUP:

7.1 Check valves will be cycled in two different ways, depending on the solvents being used

Test Solvent	Setup Used For Cycling	# of Test Setups
100% Acetonitrile	Alliance Pump Tray Assembly	3
Aurobindo Gradient	Alliance Pump Tray Assembly	3

7.2 The Aurobindo gradient takes the following form: Solvent A is 20% of (50 mM potassium phosphate buffer in Milli-Q H₂O with a pH of 3 ± 0.05 and 80% of (100% MeOH). Solvent B is 100% (HPLC-grade ACN).

The gradient table for one injection runs as follows,

Time	% Solvent A	% Solvent B	Gradient Curve
0-19 minutes	60	40	Linear (Curve 6)
20-27 minutes	30	70	Linear (Curve 6)
28-35 minutes	60	40	Linear (Curve 6)

7.3 There are 3 unique check valves being evaluated (Section 3) and there will be 1 test setup of each test solvent running 2 of the same type of check valves continuously.

7.4 Both setups will use 70% Milli-Q H₂O/30% MeOH for seal wash.

7.5 Acetonitrile setups will be recycling both mobile phase and seal wash, while Aurobindo setups will not be recycling seal wash or mobile phase.

7.5.1 Seal wash and mobile phase on acetonitrile setups will be replaced with fresh solvents every other week.

7.5.2 Aurobindo setups will be replaced with solvent as needed. Bottles will be cleaned every other week to prevent buffer precipitation.

7.6 Both setups will be run with 3000psi of backpressure.

7.6.1 Test setups will be run at 1.5mL/min.

7.7 Number of pump cycles will be calculated based on total solvent pumped (in liters) and pump stroke volume (in µL).

7.8 The check valves will be cycled until one PM duration's (1year) worth of usage is exceeded, or until the tested check valve fails.

Note: Deviations from proposed setup will be captured in electronic log, which can be found in, fileshare\WetLabBackup\Projects\Alliance\Luna_Alliance+\Prototype Ceramic Check Valve

8 MEASUREMENT EQUIPMENT:

Description	Calibration ID	Calibration Due
Accumet AB150 pH Meter	AB92343602	31-DEC-2018

9 QUALIFICATION OF TEST EQUIPMENT:

Description	Requirements	Reference
Alliance Pump Trays (6)	Fit for Purpose Tests Req'd	9.2

9.1 Verify the Alliance pump tray can flow and maintain pressure.

10 TEST PROCEDURE:

10.1 Periodic Testing:

- After check valve installation, prime seal wash pumps and set all system flows to 1.5mL/min.
- Record max pressure and pressure deltas in the corresponding electronic log for each test stand. Record pressure delta and flow rate at each periodic test, as well.
- Through the onboard "Direct Function" menu, run a "Static Leak Test."
 - Follow onscreen instructions to conduct test.

- Record all results and observations in electronic log.
- Failures: If a static leak test fails, verify the cause of the failure (seals, fittings, check valves, tubing).
 - Loss of prime errors can also be caused by the GPV outlet tube becoming clogged.
 - If cause of leak test failure is any component other than the check valves, replace that part and make note of replacement in electronic log.
 - If cause of leak test failure is the check valve, make note of observation before resuming cycling.
 - If static leak test fails because of a check valve 3 consecutive times, replace the failed check valve and make note of cycle count at time of replacement in electronic log.
- After successful leak test, or appropriate diagnosis of a failed test, resume cycling.
- Periodically run a static leak test 2x per week.
- Back up electronic logs after every periodic test.

10.2 Instructions:

- Cycling: (6) test stands will be tested in parallel.
- Test stands are to be run 7 days/week.
- Test stand status, periodic test data and observations will be captured in electronic log files.

10.3 Bi-Weekly Tasks

- Empty solvent reservoirs of acetonitrile and seal wash and clean bottles by rinsing with Milli-Q H₂O.
- Replace with fresh solvent in a dry, clean bottle.
- Mark the date of solvent replacement on the chemical identification tag.

10.4 Daily Tasks

- Verify that all test stands are running at 1.5mL/min and at 3000 ± 250psi. Record this information and pressure delta in the electronic log.
- Ensure solvent and seal wash bottles are full and that the seal wash pumps are operating.
- Verify that solvent has been replaced according to schedule.
- Record any issues and/or troubleshooting in the electronic log.
- Additional tasks may be added at the discretion of REL engineer and noted in the electronic log file.

11 ACCEPTANCE CRITERIA:

- 11.1 Findings and data collected during this evaluation will be given to engineering so that they are able to make a decision about whether or not this design will be a viable option for customers moving forward. Check valves will be considered passing if they pass onboard static leak test and do not cause "Loss of Prime" or other errors that alter performance. If seen consistently, these issues will constitute a potential check valve failure.
- 11.2 Inspect all failed check valves at teardown for signs of wear or contamination. Potential failure modes include, but are not limited to, ball being stuck to seat, ball not sealing in seat, and/or housing gasket(s) creeping/leaking.
- 11.3 No observations or issues that could negatively impact customer satisfaction, including safety.

12 DISPOSITION OF PARTS AND DATA:

- 12.1 REL will store data for two years or the life of the product, whichever is longer.
- 12.2 REL will document disposition of parts in electronic log.

13 APPENDICES AND REFERENCES:

- 13.1 FSD SeparationsModuleAndColumnModules: DIR721002483
- 13.2 REL RFT Prototype Luna Ceramic Check Valve: Survey Test: DIR721014774
- 13.3 Information and Data Location: \\fileshare\\WetLabBackup\\Projects\\Alliance\\Luna_Alliance+\\Prototype Ceramic Check Valve

13.4 Electronic Logs: \\fileshare\WetLabBackup\Projects\Alliance\Luna_Alliance+\Prototype Ceramic Check Valve\Sys#_DDMMYYYYY.xlsx

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