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PERFORMANCE SPECIFICATION

TYPE OF SPECIFICATION (check one):

☐

SYSTEM (*SIGNATURE REQUIRED)

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SUB ASSEMBLY (✓ SIGNATURE REQUIRED)

PRODUCT: 2690

DOCUMENT NUMBER: 270694

ASSEMBLY NAME: INJECTOR / SEAL PACK
ASSEMBLY

REVISION: A

PART NUMBER AFFECTED: 270576

WRITTEN BY: STAN WHARTON

DATE: 3/22/96

APPROVED BY/DATE:

ACKNOWLEDGMENT/DATE:

* | PROJECT/FUNCTIONAL MANAGER

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REVISION HISTORY

REV.
A

DWO/ECO NUMBER
DWO-1632

DATE
4/22/96

ORIGINATOR
STAN WHARTON

1.0 SCOPE

This specification defines the performance requirements for the High Pressure Injector/Seal Pack Assembly, a sub-assembly of the 2690. A prerequisite to this specification is the understanding of the parts drawing which delineates the product. This also implies that all components conform to those documents.

2.0 RELEVANT SPECIFICATIONS

271037	Study, Needle Height, 2690 (in project folder)
271042	Spec, Software CAPS, 2690
270576	Assembly, Injector

3.0 GENERAL DESCRIPTION

The High Pressure Injector/Seal Pack sub-assembly in the 2690 is the mechanism used to collect samples and deliver them into the flowing mobile phase for transport downstream. This assembly consists of a stepper motor supported by two guide rods that are set into a casting which is anchored to the 2690 frame. The stepper motor is driven by a unipolar chopper drive circuit which turns a lead screw that drives a yoke along the guide rods. The yoke contains the injector needle which is moved through the seal pack that is mounted to the bottom of the casting. The seal pack is comprised of an upper and lower seal housing which sandwich a bevel body containing the two high pressure seals. These high pressure seals are designed to separate and seal the mobile phase from the outside environment. There are two optical interrupt switches that are mounted on a bracket that is anchored at the top and bottom to the motor support and the casting respectively. A flag mounted to the yoke passes through the interrupt switches when the needle is moved down or up.

The position of the needle in this assembly is determined by a combination of motor steps and the interrupt switches. The needle is required to move to the following positions during the injection cycle:

- down from the stream position to the seal position
- down from the seal position to the vial position
- up from the vial position to the seal position
- up from the seal position to the stream position

The upper most interrupt switch is used to define the stream position of the needle cross port. The seal position and the vial position are determined by motor steps. There is one other position called the "auto add" position. This position is used when the needle has been retracted from the vial in between "auto add" sample draws. The lower switch is used to determine the position of the needle for the "auto add" operations.

The seal wash housings (upper and lower) provide wash chambers to remove unwanted material that may be introduced into the high pressure fluid stream from salt deposits on the needle, or from sample contamination on the outside and/or point of the injector needle. In either case, the wash system is designed to void this material away from the high pressure seals and the needle compartment.

Two high pressure cup seals are installed into the bevel body of the subassembly. Between these two seals flows the high pressure mobile phase. During operation of the injector subassembly, the cross port hole leaves the cavity created by these two seals.

The first movement of the injector needle is into the "bubble check" (or seal) position. Bubble check (or seal) position is defined as the point at which the cross port is, in essence, shut off from both the atmosphere and the pressurized flow stream. At this position, certain high pressure valves switch states to equilibrated internal needle pressure with the atmosphere prior to leaving the seal pack.

The next motion of the needle is through the "Needle Wash" chamber. This needle wash chamber is constructed to "scrub" and "wipe" the needle during it's transition from the seal pack to the sample vial. The actual wash is accomplished by two methods: first; an interference fit between the porous polyethylene frits and the injector needle wipes the needle, second; the turbulence created by aspirating a much larger volume of air than fluid, dries the needle point of any droplets.

This agitation breaks away the residue from the frits and/or needle for easy evacuation downstream into the needle wash waste. Further description of actual air to fluid is defined under the physical performance section of this specification.

The performance of these high pressure seals are one of the most critical elements of the system. The minimum effective seal width is .050 inches. This allows for a .03 inch window for the "Bubble Check". This position is used during the injection sequence and compressibility sequence.

The seal matrix used in this position is UHMWPE. The seal matrix, a proprietary compound of the Fluorocarbon Company, is Flouroloy-G. The seal is pre-loaded with a set of elgiloy® split rings. This applies an initial load on the injector needle thus allowing the seal to take an initial directional set.

The following parameters describe the expected seal life.

Injectons:	10,000
Pump Pressure	1500 PSI
Cycle Note:	20 injections/hour
Needle Wash	on
Solvent	ACN/H ₂ O in 10 Molar Sodium Phosphate DiBasic

4.0 PHYSICAL PERFORMANCE

Pressure

Operating Pressure Range:	400 - 5000 PSIG
Maximum Dynamic Seal Pressure:	5000 PSIG
Maximum Static Seal Pressure:	6000 PSIG
Maximum Static High Pressure Leak Rate:	1.3 µl/min. @ 6000 PSIG
Maximum Static Low Pressure Leak Rate:	0.5 µl/min. @ 400 PSIG
Flow Rate Range:	0-25 ml/min.
Maximum Backpressure @ Max. Flow:	25 PSIG

Temperature

Operating	4° to 40° C
Shipping	-40° to 70° C (per spec 056529DS)

Chromatography

Band Spreading	50µl
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Needle Wash

Slurry Ratio (needle down)	5ml/minute Minimum H ₂ O 300cc/minute Minimum Air
Slurry Resistance	10 inch Hg
Injected Needle Wash Contamination	<0.1% from previous sample
Sample Carry-Over	<0.1% from previous injection
Precision	<0.5% RSD (20µl to 100µl)
Linearity	>0.999 coefficient of correlation (5µl to 100µl)

Stepping Motor

Step Angle:	1.8 Degrees per Step
Step Rate:	690 Full Steps per Second
Maximum Pull In Torque:	70 oz-in

Lead Screw

Thread Form:	5/16-8 (two starts)
Lead:	.250" per Revolution
Lead Accuracy:	.003" per Foot
Lead Screw Material:	303 Stainless Steel
Lead Nut Material:	Turcite X

Resolution:	.00125" per Step
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Excursion: stream position to vial position	2.571"
seal position to stream position	0.099"

Excursion Time: seal position to vial position	2.87 seconds
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Load Capacity:	22 Lbs. Nominal
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5.0 PERFORMANCE TESTING

Band Spreading

ALLOWABLE: Maximum 50 μ l step within 2690 seal pack.

List of Equipment

- Solvent Delivery System: M-590 (or equivalent) fitted with stainless steel 50 μ l heads.
- Detector: Low dispersion 2 cell conductivity detector (or equivalent).
- Data System: Waters 840, System 1 on IDEL1, SIM box (or equivalent).

Experimental Parameters

- Mobile Phase: 2mM HN03 in R.O. H₂O filtered (0.45 μ) and degassed.
- Flow Rate: 1.0ml/min (Select 2.0 on 590 with 50 μ l heads)
- Needle orientation relative to flow: Exit down stream.
- Backpressure: Approximately 200 PSIG.
- Sample: R.O. H₂O fresh.
- Data Rate: 100 points per second
- Data Reduction: "PEAKS" software package on Rev 6.21 (or later) of Waters 840 software (or equivalent).
- Data Acquisition Method: TUBES

Experimental Method

- Connect solvent reservoir to the pump inlet draw-off valve.
- Connect the blue line of the autosampler to the outlet of the pump.
- Connect the red line of the auto sampler to the sample cell inlet of the detector. From the outlet of the reference side, hook into line a seal pack with needle in the down position. From the opposite side of the seal pack, plumb to the inlet of the sample side of the cell. Total tubing volume to be 25 μ l.
- Connect the SAMPLE cell outlet to a waste bottle.
- Select System 1 on IDEL 1 (Waters 840) and run the TUBES method (6 - 1 μ l injections).
- Set pump flow rate to 2 ml/min.
- Program the auto sampler to run 1 - 1 μ l injection
- Wait for the 840 to display the INJECT WAIT message.
- Start the auto sampler.
- Wait for the 840 to display the INJECT WAIT message.
- Run the remaining 5 injections

The experiment is intended to examine the seal pack's interior design on band spread. The experiment places a slug of material into a stream for band width. Within the same stream is placed a "seal pack". The difference between the two is in essence the effect which the "sealpack" had on mixing and spreading.

Needle Wash

A) Injected Needle Wash Contamination (Cross Contamination)

ALLOWABLE: Less than 50 nanoliters

List of Equipment

- Solvent Delivery System: M-510 (or equivalent).
- Solvent Storage: 5 ltr. Kontes solvent reservoir with 7 PSIG helium blanket from Waters eluent stabilization module.
- Auto sampler: 2690 with Rev. 1 software or later.
- Column: 3.9mm x 15cm NovaPak C-18
- Detector: 440 Absorbance Detector (or equivalent).
- Data Collection and Storage: Waters 860, Idelvx version 1.2 (or equivalent)

Experimental Parameters

- Mobile Phase: 65/35 ACN/R.O. H₂O filtered (0.5μ) and degassed.
- Flow Rate: 1.0ml/min.
- Needle orientation relative to flow: Exit down stream.
- Backpressure: Approximately 2000 PSIG measured at pump transducer.
- Sample: Mobile phase.
- Needle wash solvent: 750 μgram/ml Ethylparaben dissolved in mobile phase.
- Run Time: 4.0 min.
- Data Rate: 5 points per second

Experimental Method

- Load Carousel. Vial 1 contains needle wash solvent (750mg/ml Methylparben) and vials 2 through 10 contain the sample (mobile phase).
- Prime the needle wash system.
- Run vial 1, which contains the same solvent as the needle wash, to quantify the area count. The injection sample size may be approximately .1μl. Quantify over six injections.
- Run 9 injections from vials 2 through 10 (81 injections) of approximately 10 μl. Quantify the results for the amount of needle wash solvent which has entered the flow streams. This process will show the amount of needle wash solution which has been; 1.) carried into the flow stream through the cross port needle and; 2) Dissolved or dropped into the vial because of poor aspiration of the needle and needle point itself (The needle point only gets cleaned by air).

B) Carry-Over Study

ALLOWABLE: Less than .1% of previous sample concentration

List of Equipment

- same as cross contamination test

Experimental Parameters

- Mobile Phase: same.
- Flow Rate: same.
- Needle orientation relative to flow: same.
- Backpressure: same
- Sample: 150 mg/ml Anthracene dissolved in 50/50 ACN/H₂O.
- Needle wash solvent: 10/90 ACN/ R.O. H₂O
- Run Time: same
- Data Rate: same

Experimental Method

- Load Carousel. Vial 1 contains the 750mg/ml Anthracene solution, and vial 2 through 7 contain the mobile phase.
- Replace the needle wash solvent with needle wash solvent (10/90 ACN/H₂O).
- Prime the needle wash system.
- Program the 715 to inject 6 - 2µl samples from a vial which contains 750 µgrams/ml of Anthracene. This is done to quantify the area count.
- Program the unit to now sample 5µl from vial 1, then 50 µl from vial 2. Repeat the sequence 5 additional times for a total of 12 injections from vial 1 and vial 2 combination. Repeat the test vial 1 to vial 3, vial 1 to vial 4, etc., through vial 1 to vial 7.
- Quantify the results of "carry over" of vial 1 which is spiked into vial 2 through 7. The amount of carry-over is the amount which was present on the needle or in the needle wash chamber during the movement of the needle through it's injection sequence. This will show; 1) the effectiveness of the sample removal from the shaft and the point of the needle; 2) the effectiveness of sample removal from the needle wash chamber in the seal pack; and 3) the effectiveness of sample removal from and between injections in general.

