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Filtration Systems, Inc Report for WSRC SpinTek Rotary Microfilter Testing

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Westinghouse Savannah River Company (WSRC) contracted with Filtration Systems, Inc to conduct a microfiltration test using their SpinTek Centrifugal filter and 5.6 M sodium, average salt solution simulated Savannah River Site (SRS) High Level Waste. The purpose of the test was to evaluate the centrifugal filter as a possible replacement for the Mott crossflow filters in the current design of the alpha removal flowsheet.

Filtration Systems conducted the test using the SpinTek STII rotary microfilter. The insoluble solids loading varied between 0.6 g/L and 60 g/L. The testing was conducted with several deviations from the original scope of work.

- Because of their unavailability, aluminum nitrate and cesium nitrate were not added to the feed solution (this change was approved by WSRC).
- The tests were conducted with a 0.1 m ceramic filter rather than a 0.5 m porous metal filter. This change was not approved by WSRC, and Filtration Systems will conduct additional tests with a 0.5 m porous metal filter.
- Filtration Systems added nitric acid to the feed solution to reduce the pH from 14 to 12.5. They have since confirmed that the filter has been successfully tested on solutions with pH as high as 14.

Filtration Systems, Inc provided WSRC with a report describing the test results (see Attachment 1). The significant results are the following:

- At an insoluble solids loading of 0.6 g/L, the filter flux was 0.21 gpm/ft^2.
- At an insoluble solids loading of 2.8 g/L, the filter flux was 0.19 gpm/ft^2.
- At an insoluble solids loading of 12.9 g/L, the filter flux was 0.15 gpm/ft^2.

- At an insoluble solids loading of 60 g/L, the filter flux was 0.13 gpm/ft^2.
- The measured filter flux is significantly higher than the current baseline.

Attachment 1
Task Description

The Savannah River Site (SRS) is developing a process to decontaminate High-Level Waste that entails removing the liquid waste from the tank and processing this waste for disposal. Once the liquid waste is removed from the tank, monosodium titanate is added to precipitate the strontium and selected actinides from the liquid stream. Also, the process stream may have entrained metal hydroxide particles commonly called sludge.

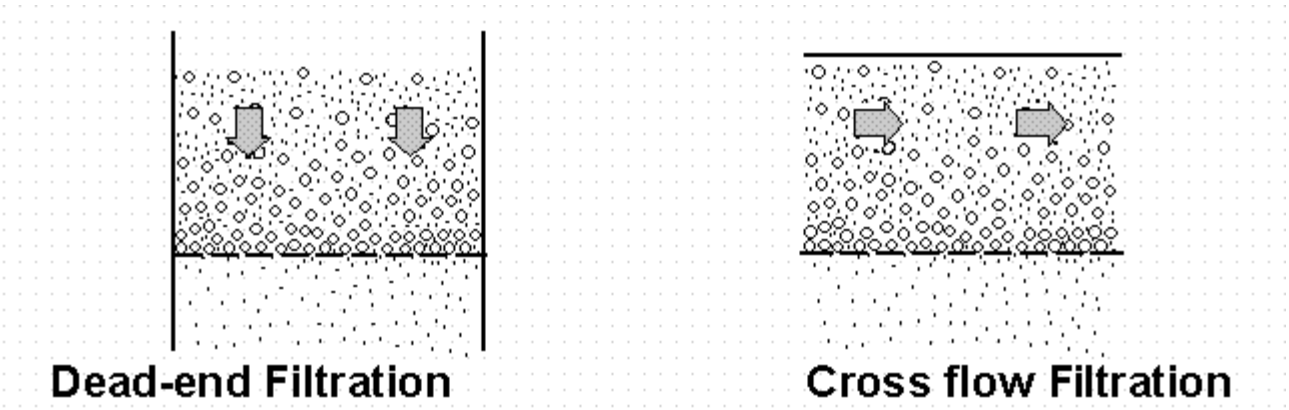
A separation process is sought that will filter the solids from this treated waste stream, produce a clean filtrate for subsequent processing, and concentrate the solids for vitrification. The SpinTek STII rotary microfilter is a candidate for this separation process and is being evaluated under this test program for filtrate quality, ability to operate with a high filtrate throughput when operating on a 5.6M Sodium salt solution with a high concentration of suspended solids.

To properly evaluate the SpinTek process the feed solution to the system is based upon a surrogate recipe provided by Westinghouse Savannah River Site (WSRC), the operating contractor on the site. Suspended solid contaminants will be added in four steps and the system's performance will be measured at these four concentration points.

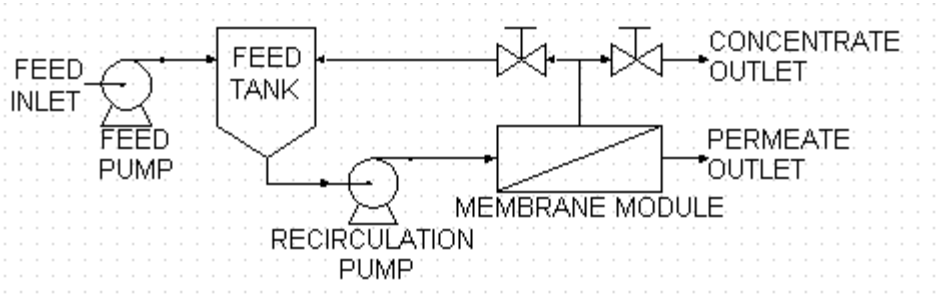
Samples of the feed and filtrate will be drawn at each of the contaminant solids levels for subsequent analysis.

Membrane Technologies

It is helpful to distinguish between conventional dead end filter and cross flow filtration systems like the SpinTek STII rotary microfilter.



Most industrial filtration process involves a feed flow moving straight through a filter and the solids accumulate on the surface of the membrane. When the solids build up and blind the filter the process is stopped, the filter replaced, and the system put back into surface. All of the SpinTek systems are "cross flow filtration" where the fluid flow is tangential to the surface of the membrane creating a sweeping action that helps prevent the membrane fouling.

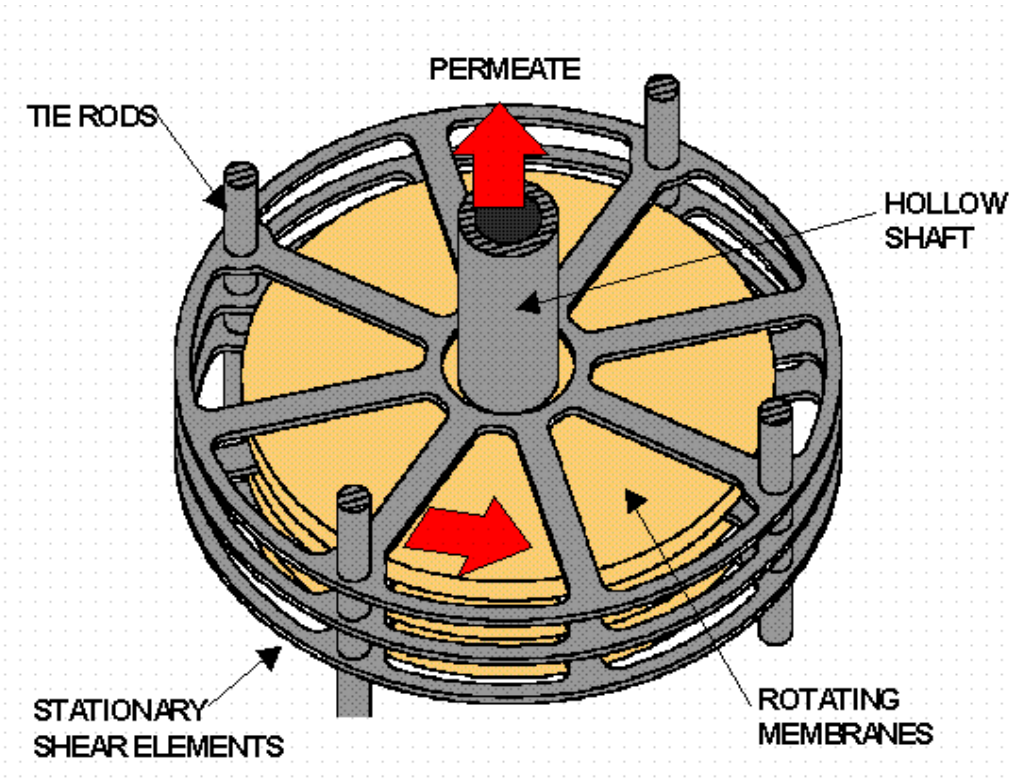


Feed is normally drawn from a tank, passed through a membrane module where some fluid will pass through the membrane as clean filtrate. The vast majority of the feed fluid, that creates shear across the membrane surface, returns to the feed tank. Cross flow filtration is hence a concentration process which under proper conditions can operate for long periods of time.

Description of the SpinTek Rotary Microfilter

The ST-II is a very rugged, compact and durable filtration system that uses membrane filters mounted on rotating disks that can produce high filtration flow rates, with stable performance, even when operating on slurries with high solids content. The membranes rotate at a tip speed velocity of 60 ft/sec that effectively cleans and sweeps solids off of the membrane surface providing reliable and stable filtrate throughput. An ST-II using 11" diameter disks has a rotational speed of 1,170 rpm.

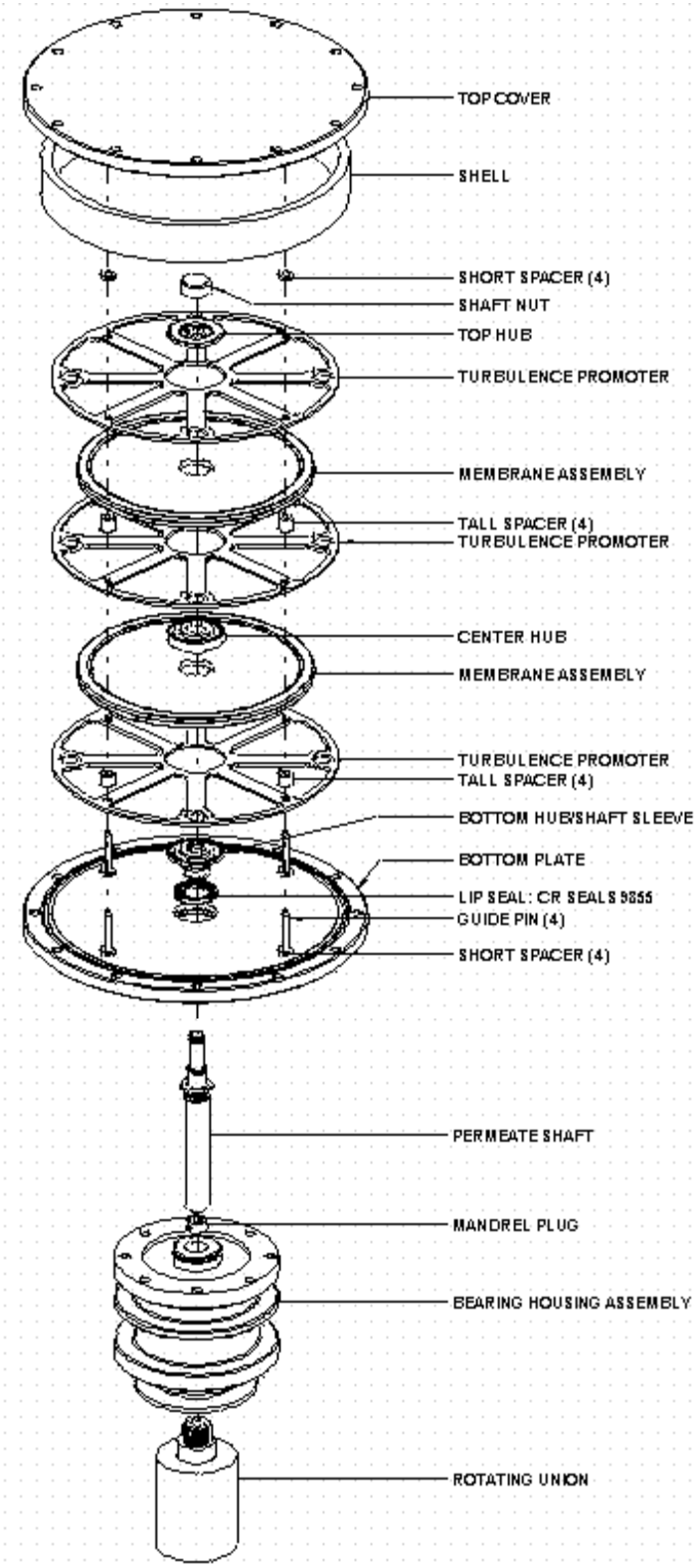
The ST-II is a compact, rugged system that can be designed to meet the DOD Mil-Std-810 specifications for durability and ruggedness.



The SpinTek ST-II High Shear Rotary Filter has rotating hollow disks covered with either commercially available or specialty flat sheet membranes. The disks are physically mounted and are hydraulically connected to a common hollow rotating shaft. The entire stack of membrane disks is enclosed within a pressure vessel. The feed fluid enters the vessel, flows between disks and across the membrane surface, where permeate flows through the membrane and exits through the hollow shaft. Concentrate exits the pressure vessel at the opposite end.

Stationary surfaces, or turbulent promoters, oppose the rotating membrane disks, generating large fluid shear rates across the membrane surface. This effectively eliminates the boundary layer that forms in conventional systems yielding very high fluid flux rates through the membranes. In addition, the stationary disks may have vane-like protrusions that resemble "wagon wheel" spokes to enhance fluid flow in and out of the channel between disks. The vanes can be placed at a distance of between 0.06" to 0.12" (1.5 mm to 3.0 mm) away from the membrane surface.

Comparison of High Shear SpinTek Rotary Filter to Conventional Cross Flow Filtration



The high shear SpinTek membrane process is similar to that of conventional membrane systems with the exception of

the rate at which the process fluid is recirculated. Since the cross flow velocity is controlled by rotating the membranes, the recirculation flow rate is set based on the amount of permeate removed. Pressure is decoupled from the feed flow rate, allowing more control over the driving force pressure and independent control of cross flow velocity. This allows optimization of performance for a very broad range of feed characteristics.

Cross flow velocity is then controlled directly and its magnitude is significantly greater than that of conventional membrane systems. A conventional membrane system may have a cross flow velocity of 8 ft/sec. while the SpinTek ST-II high shear membrane typically operates at 60 ft/sec. This has a dramatic influence on concentration polarization, enhances performance, and allows selectivity to be determined by the membrane pore size instead of by the boundary layer that forms on top of the membrane.

Because of its unique design, the ST-II has open channels uninhibited by typical mesh type turbulence promoters. Unlike conventional spiral wound membranes or hollow fibers, this wide channel allows the system to process a wide variety of viscous solutions containing moderate to high solids. In addition, unlike conventional membrane systems that rely upon adhesives and plastics for spacers and binders, SpinTek Systems has developed an innovative ceramic coated metallic membrane disk that not only uses mechanical sealing methods but can be decontaminated. Frequently, a conventional membrane device, even though utilizing a sophisticated membrane, will be limited in its use for military and DOE applications due to incompatible adhesives or materials of construction.

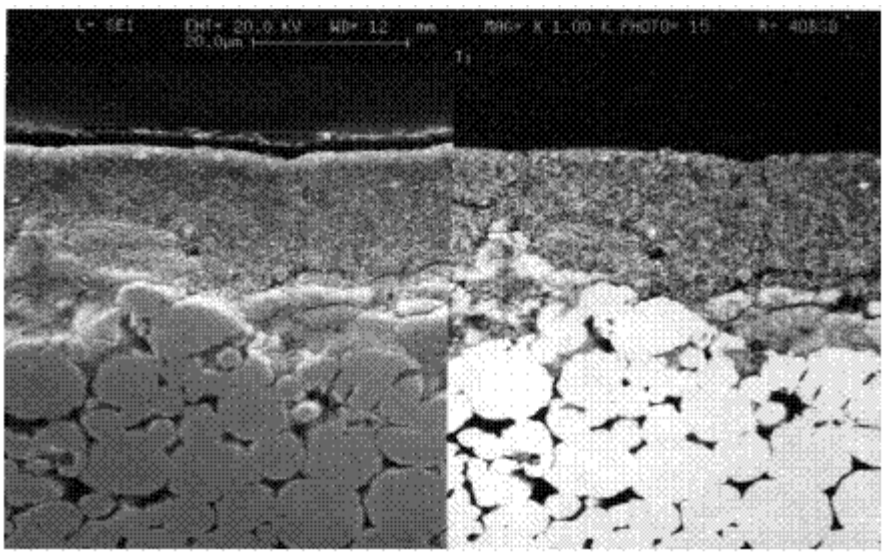
Ceramic Membranes

In early versions of the ST-II systems polymeric and early generations of ceramic membranes were used. A new ceramic membrane was developed with a pore size of 0.1 microns for use in the STII. The membrane has a thin titanium dioxide/alumina/silica dioxide layer on top of a stainless steel substrate. The top membrane coating is approximately 15 microns thick while the lower substrate is 250 microns.

The ceramic material being deposited is nanometer in size and is made of a combination of titanium dioxide, alumina and silica dioxide. The very small size of the nanopowder ceramic produces a very smooth to the touch surface that resists trapping of contaminants within the pore of the membrane.

The membrane is not brittle but ductile enough to rolled into a tube ½" (12mm) without cracking either the stainless support or the ceramic coating.

Shown below is a scanning electron microscope view of a 0.1 micron membrane showing the 3 micron size stainless steel particles coated by the nanopowder ceramic layer.



0.1 micron membrane

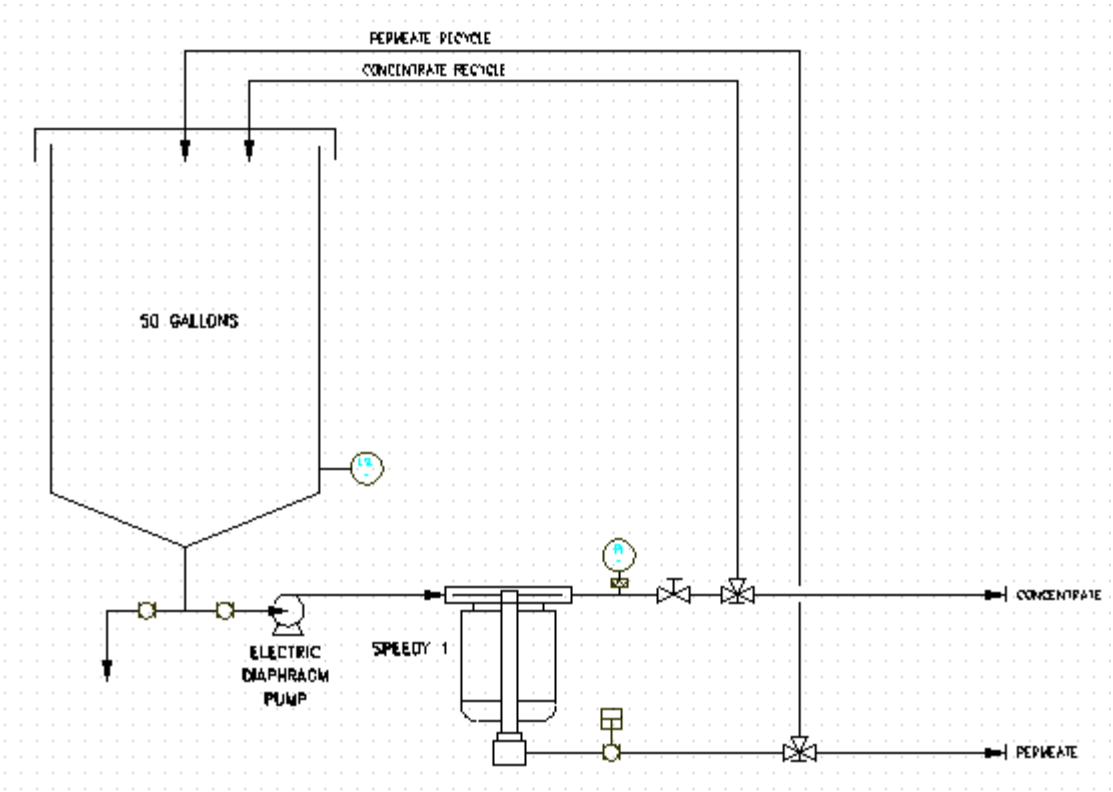
This membrane has been successfully tested in previous work on solutions with a pH up to 14 with no damage

occurring to the membrane ceramic coating or the stainless steel substrate.

Testing Apparatus

The SpinTek STII rotary microfilter uses a system similar to that shown below. A feed tank contains the feed surrogate solution and is continuously agitated by an electric mixer. Feed is drawn from the bottom of the tank by a multi-stage centrifugal pump and under pressure sent to the STII.

Valving is provided so that the both the concentrate and filtrate can be returned to the feed tank in a complete recycle mode. Alternatively, either concentrate or filtrate can be separately removed from the system.



In addition, the STII pilot system has variable controls for both feed pressure and rotor speed.

Technical Objectives

The primary objective is to determine if the SpinTek STII, using a 0.1 micron ceramic filter, can provide clean filtrate and have stable filtrate throughput while operating on a SRS precipitant feed solution.

Specific objectives:

- Operate the STII with clean deionized water to determine clean water filtrate throughput.
- Prepare a surrogate brine solution using the WSRC provided recipe.
- Operate the STII on this "clean" brine to determine filtrate throughput.
- Add MST and Sludge, provided by WSRC, as the precipitated suspended solids in four steps of increasing concentrate.
- Determine filtrate quality and system throughput with the four different concentrations of suspended solids.
- Send the filtrate and feed samples collected during the four concentration runs and send to SRS for subsequent analysis.
- Write a final report documenting the work performed during this entire test.

Test Plan

- The STII pilot system uses a single Ryton™ disk with ceramic membrane applied to both sides of the disk. The effective usable membrane for a single disk is one square foot. The system uses a new ceramic membrane disk with a nominal pore size of 0.1 microns and is operated at standard operating conditions of 40 psig feed pressure and 1,170 Rpm rotor speed. Flux will be stabilized and measured.
- A brine solution will be prepared using the WSRC provided recipe and the same membrane disk will again be operated at standard conditions, flux stabilized, and measured.
- MST and Sludge contaminants will be added to the brine solution in four steps with the first step having the least amount of contaminants and the subsequent steps having increasing concentration.
- After the "clean" brine is stabilized for filtrate throughput the system will be shut down, the membrane rinsed under fresh water, and reinstalled in the system. This shut down/rinse step will be performed before each addition of MST and Sludge.
- The final test will use the brine solution at the highest MST and Sludge concentration and the rotor speed will be reduced from 1,170 Rpm to 148 Rpm as a preliminary indication of rotor speed impact on filtrate throughput.
- Samples will be drawn of filtrate and feed during for each test for subsequent analysis by WSRC.

Test Work

A recipe has been provided by WSRC for both the brine make-up and the solids to be added. The first and second column lists the constituents and their concentration. The third and fourth column shows the linearly scaled constituent concentration of our 100 liter batch in kilograms and grams.

The aluminum nitrate and the cesium nitrate were eliminated from this test due to lack of availability. The WSRC personnel approved this deviation in the recipe. The pH of the resultant solution was approximately 14 and this was lowered to 12.5 by 7.5 liters of concentrated nitric acid.

Table #1 Surrogate Recipe

Constituents	Tanks		Scaled		Scaled	
	Recipe		Recipe		Recipe	
Deionized Water	600	liters	38.8	liters	38.8	liters
NaOH	119.23	kg	7.712	kg	7712	grams
Al(NO3)3*9H2O (*)	110.03	kg	0	kg	0	grams
MIX FOR 1 HOUR						
Na2CO3	16.05	kg	1.038	kg	1038.2	grams
Na2SO4	20.16	kg	1.304	kg	1304.0	grams
Na3PO4*12 H20	3.48	kg	0.225	kg	225.1	grams
NaC2O4	0.51	kg	0.033	kg	33.0	grams
Na2SiO3	0.462	kg	0.030	kg	29.9	grams
Na2MoO4	0.0458	kg	0.003	kg	3.0	grams
NaCl	1.38	kg	0.089	kg	89.3	grams
NaF	1.27	kg	0.082	kg	82.1	grams
KNO3	1.43	kg	0.092	kg	92.5	grams
CsNO3 (**)	25.8	g	0.000	g	0.0	grams
NaNO3	96.1	kg	6.216	kg	6216.0	grams
NaNO2	33.95	kg	2.196	kg	2196.0	grams
HNO3 (***)	0	liters	7.5	liters	7.5	liters
ADD DEIONIZED WATER	946	liters	61.2	liters	61.2	liters
TOTAL DEIONIZED WATER	1546	liters	100	liters	100	liters

(*): Aluminum nitrate was not available and was eliminated from recipe per Michael Poirer.						
(**): Cesium nitrate was not available and was eliminated from recipe per Michael Poirer.						
(***): Without the aluminum nitrate the pH of the solution was 13.5-14. The nitric acid was added						
to lower the pH to 12.5						

The first set of tests used four different concentrations of MST and Sludge. The amounts added are show in the table below.

Table #2 Sludge and MST Concentrations

Test #	Suspended Solids	% Solids in Original	Conc of Sludge		100% Sludge or		Diluted Sludge	
	Consitutent	Sample Containers	or MST		MST Required		Required	
Test #1	MST	11%	0.287	grams/liter	2.7	grams	265	grams
	Sludge	13%	0.315	grams/liter	2.5	grams	252	grams
	Total of Sludge/MST		0.602	grams/liter	5.2	grams	517	grams
Test #2	MST	11%	1.34	grams/liter	12.4	grams	1238	grams
	Sludge	13%	1.46	grams/liter	11.7	grams	1168	grams
	Total of Sludge/MST		2.8	grams/liter	24.1	grams	2406	grams
Test #3	MST	11%	6.17	grams/liter	57.0	grams	5702	grams
	Sludge	13%	6.73	grams/liter	53.8	grams	5384	grams
	Total of Sludge/MST		12.9	grams/liter	110.9	grams	11086	grams
Test #4 (*)	MST	11%	28.7	grams/liter	265.3	grams	2652	grams
	Sludge	13%	31.3	grams/liter	250.4	grams	25040	grams
	Total of Sludge/MST		60	grams/liter	515.7	grams	27692	grams
% Solids in Original Sample Containers: % solids of the raw MST or Sludge provided by WSRC.								
Concentration of Sludge or MST: Solids concentration to be added to the clean brine solution.								
100% Sludge or MST Required: The amount of 100% Sludge or MST required for the 100 liter batch of brine.								
Diluted Sludge or MST Required: The amount of 13% Sludge and 11% MST required for 100 liter brine batch.								

(*) The original feed volume of 100 liters had to be reduced to 22 liters due to the lack of enough MST and Sludge for this concentration level.

The new ceramic 0.1 micron ceramic membrane was installed and tested for clean water throughput. The brine solution was mixed and the membrane again tested for filtrate throughput and stability.

The system was shut down, the membrane removed and rinsed under fresh water. The first level of contaminants was added and the system operated and stable throughput measured and feed and filtrate samples drawn. This procedure was repeated for Tests #2, and #3.

Table #3 Filtration Run at Test 1,2 and 3 Solids Concentration

Feed Tank	Filtrate		Time	Feed	Press	Rotor	Temp	Comments
Volume, l	mls/min	gpm/sqft	hour/min	mls/min	PSIG	Rpm	F	
103	1125	0.297	0:00	293	40	1170	66	Clean DI water, initial flux
103	1115	0.295	0:30	295	40	1170	68	Clean DI water, final flux
103	1050	0.277	0:00	510	40	1170	70	Clean Brine solution, initial flux
103	1025	0.271	0:30	509	40	1170	72	Clean Brine solution, final flux

103	877	0.232	0:00	450	40	1170	74	**Test #1** initial flux
103	775	0.205	2:00	505	40	1170	80	**Test #1**, final flux
103	795	0.210	0:00	499	40	1170	73	**Test #2**, initial flux
103	730	0.193	2:00	505	40	1170	80	**Test #2**, final flux
103	610	0.161	0:00	505	40	1170	84	**Test #3**, initial flux
103	570	0.151	2:00	512	40	1170	93	**Test #3**, final flux

There was not enough MST and Sludge provided to operate Test #4 with the proper solids levels and use a 100 liter feed solution volume. Based upon the STII's minimum operating volume of 22 liters it was decided to add the proper level of suspended solids and then concentrate the 100 liter feed solution down from 100 liters to 22 liters. The calculation for this volume reduction is shown below and determined that a 3.28x concentration was the target level.

Table #4 Concentration Calculation to Meet Test 4 Solids Concentration

Conc	Volume		MST/Sludge	
1x	103	liters	11,086	grams
2x	51.5	liters	22,172	grams
3x	25.8	liters	44,344	grams
3.28x	22.2	liters	51,565	grams
4x	12.9	liters	88,688	grams

The following table shows that the concentration to 3.28x required 2 hours, 45 minutes of operation. Filtrate throughput stabilized after an additional 1 hour, 15 minutes of operation.

Table #5 Concentration Run for Test #4

Feed Tank	Filtrate		Time	Feed	Press	Rotor	Temp	Comments
Volume, l	mls/min	gpm/sqft	hour/min	Mls/min	PSIG	Rpm	F	
103	510	0.135	0:00	463	40	1170	79	1x concentration factor
93	497	0.131	0:15	466	40	1170	83	
84	498	0.132	0:30	448	39	1170	87	
74	502	0.133	0:45	434	40	1170	89	
65	500	0.132	1:00	433	39	1170	93	
57	498	0.132	1:15	412	40	1170	92	
51	488	0.129	1:30	448	40	1170	96	2x concentration factor
43	492	0.130	1:45	453	40	1170	98	
37	504	0.133	2:00	470	39	1170	102	
31	505	0.133	2:15	451	40	1170	101	
25	500	0.132	2:30	448	40	1170	101	3x concentration factor
22	496	0.131	2:45	456	40	1170	102	3.28x Concentration stopped
22	508	0.134	3:00	446	40	1170	102	3.28x
22	501	0.132	3:15	432	39	1170	102	3.28x
22	500	0.132	3:30	448	40	1170	102	3.28x

After the completion of Test #4 it was decided to add the remainder of the MST and Sludge and a portion of the previously removed filtrate to demonstrate operation of the STII at the maximum possible solids loading.

The following table indicates the concentration run from 48 liters down to 22 liters and the results of this test.

Table #6 Balance of Sludge and MST Added to Test #4 Feed Solution

Feed Tank	Filtrate		Time	Feed	Press	Rotor	Temp	Comments
Volume, l	mls/min	gpm/sqft	hour/min	Mls/min	PSIG	Rpm	F	
48	270	0.072	0:00	261	40	1170	83	
40	290	0.076	0:30	481	40	1170	87	
22	316	0.083	1:30	296	41	1170	101	3.28x

Lowering the rotor speed from 1170 to 148 to determine filtrate throughput decline performed a final test.

Table #7 Rotor Speed Reduced on Test #4 Feed Solution

Feed Tank	Filtrate		Time	Feed	Press	Rotor	Temp	Comments
Volume, l	mls/min	gpm/sqft	hour/min	Mls/min	PSIG	Rpm	F	
22	270	0.072	0:00	261	40	**148**	83	Ending concentration of Test #5
22	148	0.039	0:10	619	40	**148**	96	used as feed for Test #6.
22	140	0.037	0:50	600	41	**148**	97	

The test was stopped at the end of 50 minutes of operation though the flux decline had not stopped.

Analysis of the Data

The filtrate samples from all of the tests were "water" clear and there didn't appear to be any suspended solids in the samples. The samples were allowed to settle for 24 hours and there still did not appear any residue at the bottom of the containers.

Flux decline, based upon increasing feed concentrations, was expected and did occur. The stabilized filtrate throughput from Test #1 was 295 gfd, Test #2 was 278 gfd, and Test #3 was 217 gfd. This equates to the filtrate flow rate, per square foot of membrane, of 0.20 gpm, 0.19 gpm, and 0.15 gpm, respectively. All of these were significantly higher than the SRS target of 0.02 gpm per square foot of membrane.

Test #4, at the end of the 3.28x concentration run was still 190 gfd (0.13 gpm/sqft), with clean filtrate and a stable throughput.

Test #5 showed a lower initial flux than Test #4 though the initial solids concentration in the Test #5 feed solution was actually lower than Test #4 and this was unexpected. The flux was stable and even increased slightly from 103 gfd to 120 gfd, probably due to either temperature variations or the spinning action of the disk slightly cleaning the membrane surface.

Test #6, with lower rotor speed, showed an almost immediate filtrate throughput drop from 103 gfd to 53 gfd or a reduction of 55.8%. The flux had not stabilized and was continuing to decline at the completion of this test run.

Feed water temperature varied from a low of 79 F and 102 F and the slight variation in filtrate throughput due to temperature variation was not adjusted for.

Summary

- Filtrate during the entire series of tests were "water" clear with no suspended solids apparent in the samples.
- After settling of the collected feed samples it was easily apparent that the solids loading had significantly increased, as expected, during the series of Tests #1-5. This should be confirmed by subsequent analytical testing.

- The filtrate throughput was higher than the WSRC target of 0.02 gpm/square foot of membrane even during Test #4 where it had dropped to a low of 0.13 gpm/sqft.
- When the membrane was rinsed under water before the start of every test, except Test #6, the small amount of solids on the membrane surface quickly washed off. It is fully expected that a simple in-situ cleaning of the membrane with surfactant would produce the same results.
- We believe that this series of tests confirmed that the SpinTek STII should continue as a viable candidate for this application.