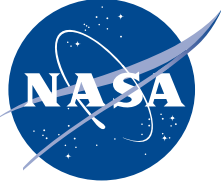


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ECLSS Sustaining Compatibility Testing on Urine Processor Assembly Nonmetallic Materials for Reformulation of Pretreated Urine Solution

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Marshall Space Flight Center, Huntsville, Alabama

September 2015

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LIST OF ACRONYMS AND SYMBOLS

CrO ₃	chromium trioxide
DA	Distillation Assembly
DMA	dynamic mechanical analysis/analyzer
ECLSS	Environmental Control and Life Support System
EPDM	ethylene propylene diene monomer
H ₂ SO ₄	sulfuric acid
H ₃ PO ₄	phosphoric acid
ISS	International Space Station
MIUL	Material Identification and Usage List
MSFC	Marshall Space Flight Center
M&P	Materials and Processes (Laboratory)
PEI	polyetherimide
PTFE	polytetrafluoroethylene
PVDF	polyvinylidene fluoride
TTS	time-temperature superposition
UPA	Urine Processor Assembly

NOMENCLATURE

E	stress relaxation modulus
ΔE	change in stress relaxation modulus
t_f	final time
t_o	initial time

TECHNICAL PUBLICATION

ECLSS SUSTAINING COMPATIBILITY TESTING ON URINE PROCESSOR ASSEMBLY NONMETALLIC MATERIALS FOR REFORMULATION OF PRETREATED URINE SOLUTION

1. INTRODUCTION

Environmental Control and Life Support System (ECLSS) Sustaining for the International Space Station (ISS) requested the Materials Test Branch/EM10 in the Materials and Processes (M&P) Laboratory at Marshall Space Flight Center (MSFC) perform compatibility testing on selected non-metallic materials used in the Urine Processor Assembly (UPA). On board ISS, urine and flush water are converted to potable water through the heart of the UPA: the Distillation Assembly (DA). The baseline urine solution is pretreated with 8.4 g/L of sulfuric acid (H_2SO_4) and 1.5 g/L of chromium trioxide (CrO_3) as an oxidizer. On several occasions in recent years, salt crystals have precipitated out of the pretreated urine (baseline) in both flight and development/ground units of the UPA. Such crystals are capable of plugging filters in UPA components. The crystals were analyzed in M&P and were determined to be mostly a mixture of calcium sulfate and calcium sulfite. ECLSS Sustaining proposed a new urine solution pretreated with phosphoric acid (H_3PO_4), mainly with the hope of eliminating or minimizing formation of the crystals. The concentration of CrO_3 in the new pretreat solution was kept the same, but the concentration of H_3PO_4 was increased by a factor of ≈ 2.3 compared to the H_2SO_4 pretreat solution to reach the desired solution pH of ≈ 2.0 . Regardless of the acid used in the pretreated urine formulation, the goals of the pretreatment remained to drive the pH low enough (≈ 2.0) to control microbial growth and the reaction of urea to ammonia.

1.1 History of Compatibility of Urine Processor Assembly Nonmetallic Materials With Pretreated Urine Solution

MSFC history to determine compatibility of UPA nonmetallic materials with pretreated urine is as follows:

- Laboratory testing was performed by EM10 in 2000 and 2002–2003 to determine compatibility of UPA nonmetallic materials with urine pretreated with H_2SO_4 for United States and Russian formulations, respectively. Compatibility was determined primarily by measuring material modulus (an indicator of stiffness) after immersion of samples in pretreated urine and concentrated brine solution for times up to six to eight months. In 2000, immersed samples were conditioned at 100 °F and 0.5 psia pressure to simulate UPA operating conditions. In 2002–2003, immersed samples were conditioned only at room temperature.¹ The Russian formulation containing the CrO_3 oxidizer is still used today.

- In April 2012, ECLSS Sustaining tasked MSFC M&P with determining manpower estimates for recertifying materials used on the UPA due to the proposed urine pretreatment with H_3PO_4 . The recertification effort would involve compatibility testing similar to that performed in 2000 and 2002–2003.
- In July 2012, MSFC M&P completed a white paper for ECLSS Sustaining on the proposed urine pretreatment with H_3PO_4 (C.D. Wingard, “ECLSS Sustaining White Paper for the Effect of a Proposed Acid Change in Pretreated Urine on the Compatibility of Non-Metallic Materials Used in the UPA,” NASA Marshall Space Flight Center, unpublished, 2012). Testing for compatibility of UPA nonmetallic materials with H_3PO_4 pretreat would need to be performed, and the white paper was written to provide an indicator for such material pretreat compatibility based on available technical literature.
 - Nine nonmetallic (polymeric) materials used on the UPA were evaluated for compatibility with H_3PO_4 pretreat based on available technical literature. The nine materials used in the UPA are directly wetted by pretreated urine, or indirectly wetted with ‘product steam’ containing urine vapors during UPA operation. The nine materials and their functions in the UPA are shown in table 1.
 - The highest approximate concentration of H_3PO_4 used in the proposed new pretreat would be 12.7% in the concentrated brine solution. Technical literature could not be found for compatibility of polymeric materials with urine pretreated with acid. While some data were found for compatibility of such materials with urine, the most meaningful data were for compatibility of such materials with aqueous solutions of H_3PO_4 , generally ranging from 1%–5% to 85% H_3PO_4 . In general, all nine polymeric materials in table 1 had excellent compatibility with aqueous solutions of 10% to 20%–30% H_3PO_4 .

Table 1. Nonmetallic materials on the UPA directly or indirectly wetted (listed in white paper for proposed urine pretreatment with H_3PO_4).

Material Name and Description	Function in the Urine Processor Assembly
Fluorocarbon elastomer (Parker V747-75)	O-ring seal on DA
Teflon polytetrafluoroethylene	O-ring seal, lip seal, and piston cup on DA
Polysulfone	Sensor body on DA
Norprene A-60-F thermoplastic elastomer	Pump tubing on Fluids Control Pump Assembly
Ultem polyetherimide 2300 with 30% glass reinforcement	Mount, drive pulley, demister plate, and frames on DA
Ethylene propylene diene monomer elastomer (Parker E751-65)	Drive belt O-ring on DA
Neoprene elastomer with Kevlar fibers	Drive belt on DA
Polypropylene	Demister screen on DA
VespeI SP-21 polyimide	Driven gear on DA

2. EXPERIMENTAL METHODS

2.1 Materials Used for Testing

2.1.1 Nonmetallic Materials Added to Original List

The nonmetallic materials listed in table 1 that were researched in the white paper were also used for the actual compatibility testing in EM10. A few materials were added to the list for testing and are shown in table 2. The ethylene propylene diene monomer (EPDM) rubber material listed in table 1 was found from the Material Identification and Usage List (MIUL) for the DA of the UPA to be used for two different functions with two different Parker Seals compounds of EPDM. The Scotch-Brite™ material was also found from the MIUL. The polyvinylidene fluoride (PVDF) transfer hose filter housing material was added based on e-mail communication between ECLSS engineers at MSFC and Johnson Space Center.

Table 2. Nonmetallic materials on the UPA directly or indirectly wetted that were later added to those in table 1 for compatibility testing.

Material Name and Description	Function in the Urine Processor Assembly
EPDM elastomer (Parker E515-80)	Fluid quick disconnect on DA
Kynar 740 PVDF	Filter housing for pretreated urine transfer hose assembly
Scotch-Brite aluminum oxide with nylon fibers and unidentified cured adhesive with calcium carbonate	Demister membrane on DA

2.1.2 Obtaining Materials and Sample Fabrication

Nine of the 12 nonmetallic materials in tables 1 and 2 were obtained in flat sheet geometry, as most had a nominal thickness of 0.125 in that was preferred for testing to determine material modulus (an indicator of stiffness) by dynamic mechanical analysis (DMA). Each DMA sample specimen was ≈ 2.5 in \times 0.5 in \times thickness. Most rigid or semirigid polymer flat sheets were machined in EM10 into the rectangular DMA samples. Several rigid or semirigid polymers such as Kynar®, Vespel®, and Ultem® were obtained from the distributors in molded or machined lengths of material in the 0.5-in width, and they were simply cut in EM10 into the desired 2.5-in lengths. Elastomeric/rubber sheet materials were not easily machinable and were cut into rectangular DMA samples with a sharp utility knife held against a stainless steel ruler. A variety of DMA rectangular samples that were machined or cut from rigid or semirigid polymers and softer elastomers are shown in figure 1. The neoprene rubber drive belt, polypropylene screen, and Scotch-Brite membrane were tested in the geometries in which they are actually used on the UPA. These materials were mostly cut with scissors into the correct DMA length-by-width dimensions, and sample thicknesses were used ‘as received.’ The materials for the drive belt, screen, and membrane that were used to produce samples are shown in figure 2.

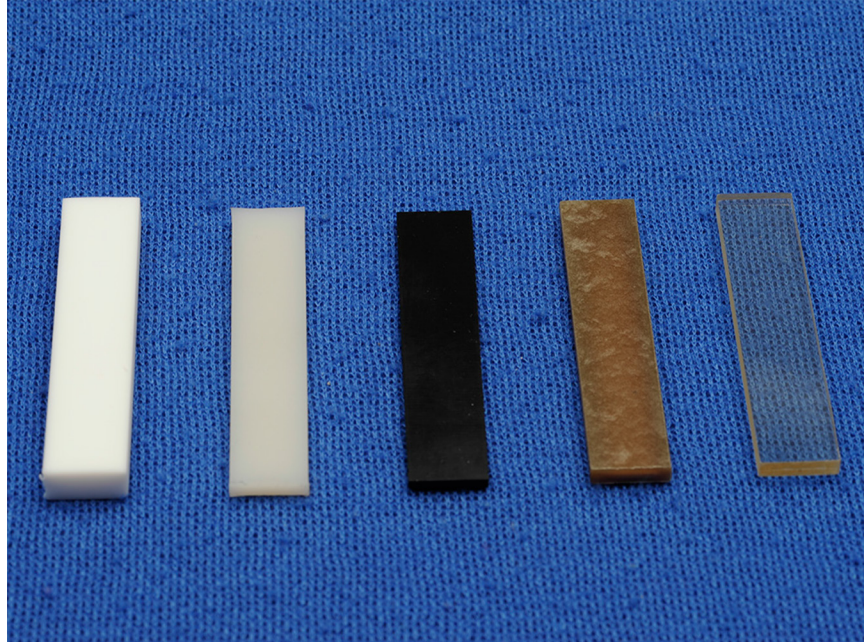


Figure 1. Several different molded virgin plastic or rubber sheet materials machined or cut into rectangular samples for DMA testing.

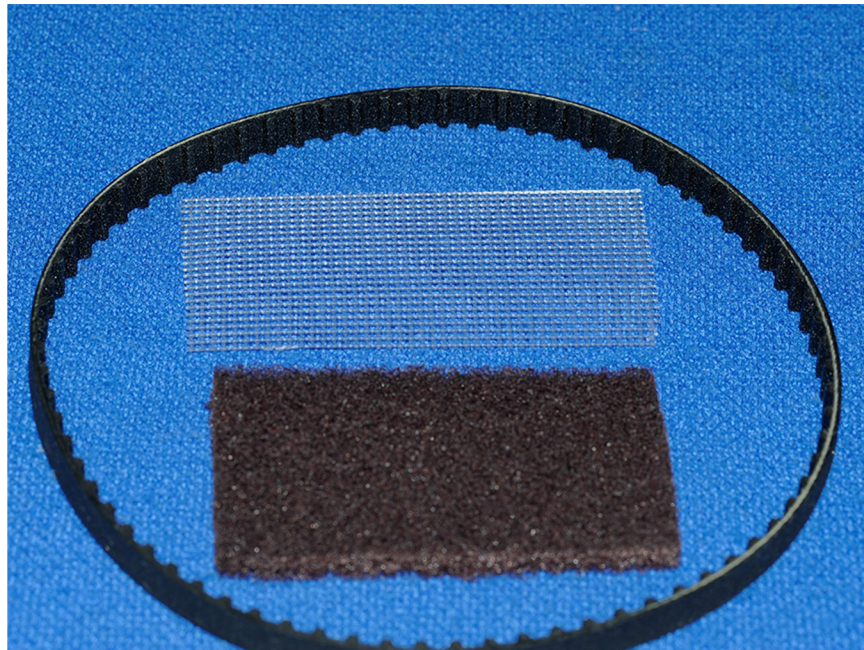


Figure 2. Several materials used to cut rectangular samples for DMA testing: neoprene drive belt, polypropylene screen, and Scotch-Brite.

2.2 Test Setup for Conditioning Samples Prior to Dynamic Mechanical Analysis Testing

2.2.1 Storage Jars

Several 1,000-mL Nalgene plastic jars made of polypropylene, each with a plastic screw cap, were used for immersing test samples in solutions of pretreated urine and concentrated brine.

2.2.2 Storage Racks

For immersing samples in each solution, samples were placed in a rack made of hardware cloth, a flexible wire mesh material with a grid size of 0.5 in. Up to 18 rectangular samples for one material type were placed in a rack.

2.2.3 Racks Loaded With Samples and Immersed in Solutions Until Compatibility Testing

The loaded rack was placed in the jar, liquid was poured completely over the samples, and the cap was screwed finger tight. Immersed samples in each capped jar were placed inside a fume hood at room temperature until compatibility testing was performed. For each type of solution, two loaded sample racks of two different types of materials were stored in solution in a single jar. Figure 3 shows one of the Nalgene storage jars and two storage racks, with each rack containing some test samples of a different material.



Figure 3. 1000-mL Nalgene plastic jar (left) used to contain flexible wire racks for containing up to 18 DMA samples per rack, and two wire racks containing samples of two different materials (right) were immersed in one solution (pretreated urine or brine solution) per jar. Each jar was hand-tightened and stored in a fume hood at room temperature until samples were removed for DMA testing.

2.3 Materials Testing

2.3.1 Appearance and Weight Gain of Rectangular Dynamic Mechanical Analysis Samples After Immersion in Solutions

After each rectangular DMA sample was removed from immersion in the solution (pretreat or brine), the sample was flushed with deionized water and patted dry with a lint-free cloth. Each cleaned sample—having been weighed on an analytical balance prior to immersion in solution—was quickly weighed again to determine weight gain due to immersion for a specified time.

2.3.2 Dynamic Mechanical Analyzer

The TA Instruments 2980 DMA was used in this work to measure storage modulus (indicator of material stiffness) of rectangular samples before and after immersion in pretreated urine and concentrated brine solutions for up to 180 days (\approx 6 months). Each rectangular sample was mounted and clamped horizontally in the DMA, as shown in figure 4. The cantilever clamp in the dual cantilever mode was used for all samples, and each sample was clamped at three points across the sample length. A torque wrench was used to tighten screws at the three points with a torque value appropriate for the material stiffness tested (table 3). The largest DMA cantilever clamp was used, with a fixed sample length of 35 mm for each sample tested. For most samples tested, the DMA sample length/thickness ratio was 10.8–10.9, which is satisfactory since TA Instruments prefers a ratio of ≥ 10 for obtaining quality modulus data.



Figure 4. A rectangular material sample (≈ 2.5 in \times 0.5 in \times thickness) mounted and clamped horizontally in the DMA with a cantilever clamp used in the dual cantilever mode. A torque wrench was used to tighten screws at all three points with a torque value appropriate for the material stiffness tested.

Table 3. Some test parameters used for DMA testing samples of nonmetallic materials listed in tables 1 and 2.

Material Name and Description	Sample Torque (\approx in-lb _f)	Sample Strain (%)	Sample Temperature Range ($^{\circ}$ C)
Fluorocarbon elastomer (Parker V747-75)	3	0.15	25–210
Teflon PTFE	6	0.07	25–265
Polysulfone	8	0.07	25–155
Norprene A-60-F thermoplastic elastomer	3	0.15	25–140
Ultem PEI with 30% glass reinforcement	8	0.07	25–175
EPDM elastomer (Parker E751-65)	3	0.15	25–130
EPDM elastomer (Parker E515-80)	3	0.15	25–130
Neoprene elastomer with Kevlar fibers	3	0.15	25–130
Polypropylene screen	5	0.15	25–175
Vespel SP-21 PI	8	0.07	25–290
Kynar 740 PVDF	6	0.07	25–115
Scotch-Brite	3	0.33	25–100

2.3.2.1 Dynamic Mechanical Analyzer Testing Mode and Test Parameters. Each material sample was tested in the DMA stress relaxation mode. In this mode, the strain was held constant at a fixed percentage depending on the material stiffness (table 3), and stress relaxation modulus E was measured as a function of time. For each test, no recovery time was measured following a time of displacement at a specified strain for each sample. For each material in table 3, the following test procedure for stress relaxation was used:

- (1) Equilibrate at 25 °C.
- (2) Displacement at specified strain percentage for time = 15 min.
- (3) Increase 5 °C.
- (4) Repeat step 2 until designated upper limit temperature for each material (table 3).

It should be noted that while each material in table 3 has an upper temperature for continuous use recommended by the manufacturer, none of these materials are typically exposed to higher than ≈ 85 °C during DA/UPA operation. The upper temperature limits for each material in table 3 were used for DMA testing to aid in data analysis and life prediction that is described in section 3.

2.3.2.2 Analysis of Dynamic Mechanical Analyzer Stress Relaxation Data by Time-Temperature Superposition. DMA data for each sample were analyzed with TA Instruments time-temperature superposition (TTS) software. TTS displays data as a log-log plot of stress relaxation modulus E versus time. With the choice of a reference temperature, relaxation segments at each isothermal temperature are shifted to short times (lower temperatures) and long times (higher temperatures) to produce a continuous ‘master curve’ over decades of time. To create the TTS master curve for each DMA sample, the reference temperature was chosen as 40 °C (104 °F), which is a reasonable average operating temperature of the DA of the UPA each day.

The plot in figure 5 illustrates the TTS principle and was taken from technical literature for stress relaxation data from tensile testing on a rubber material, with the reference temperature chosen as 25 °C.² The plot shows relaxation segments at different isothermal temperatures and creation of the master curve from the relaxation segments by use of a ‘shift factor.’

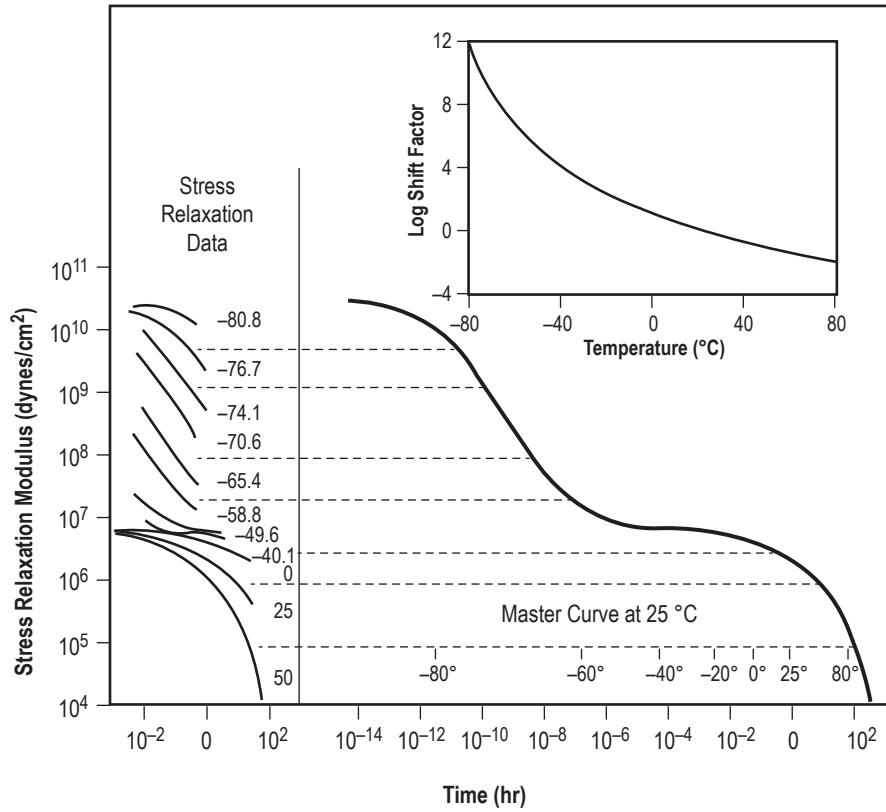


Figure 5. TTS for polyisobutylene rubber based on tensile stress relaxation data at various isothermal temperatures to create a 'master curve.'

3. RESULTS AND DISCUSSION

3.1 Appearance and Weight Gain of Rectangular Dynamic Mechanical Analysis Samples After Immersion in Solutions

For eight of the 12 nonmetallic materials tested, weight gain was no more than 1.5% after 180 days of immersion in both pretreated urine and brine solutions. The remaining four materials had considerably more weight gain in the two solutions after up to 180 days of immersion, as shown in table 4. The Scotch-Brite material absorbed considerable weight—26.5%–30% after 86–88 days of immersion in the two solutions—so no weight gain data were obtained for longer immersion times. Most DMA samples were somewhat discolored after immersion in the two solutions for immersion times up to 180 days. However, none of these immersed samples appeared to show any evidence of degradation from possible chemical attack by either solution.

Table 4. Nonmetallic materials with the highest weight gains after immersion times up to \approx 180 days in pretreated urine and concentrated brine solutions.

Material Name and Time Immersed in Solution	Weight Gain (%)	
	After Immersion in Pretreated Urine Solution	After Immersion in Concentrated Brine Solution
EPDM elastomers (Parker E751-65, E515-80) 180 days	3.5	7.0
Neoprene elastomer with Kevlar fibers 180 days	4.5	6.5
Scotch-Brite 86 days	26.5	–
88 days	–	30.0

3.2 Analysis of Dynamic Mechanical Analyzer Stress Relaxation Data by Time-Temperature Superposition

3.2.1 Method Chosen for Analysis of Dynamic Mechanical Analysis Data

For the TTS master curve of each DMA sample (virgin/control and immersed in solution for a specified time), a change in stress relaxation modulus ΔE was calculated for a short time t_o at a lower temperature, and a long(er) time t_f at a higher temperature. Some of the ‘ground rules’ for the DMA data analysis were as follows:

- ΔE for each immersed sample was compared to ΔE for a virgin sample, with the same values of t_o and t_f chosen for each immersed and virgin sample.
- If ΔE for each immersed sample was 100% of ΔE for the virgin sample, there was no change in ΔE due to effects of prolonged immersion in either solution.
- The goal of the TTS master curve for each DMA sample was to reach/predict a stress relaxation modulus E after 10 years of continuous use at a reference temperature of 40 °C.

3.2.2 Representative Time-Temperature Superposition Master Curves for Several Nonmetallic Materials

3.2.2.1 Elastomeric/Rubber Materials (Low Modulus). In general, elastomeric/rubber materials did not yield smooth, continuous TTS master curves. The isothermal relaxation segments for these materials tended to ‘bunch’ and did not spread out over decades of time. For that reason, it was not possible to obtain a 10-year prediction of stress relaxation modulus E . Figure 6 is a plot of $\log E$ versus \log time for a sample of virgin neoprene drive belt material and shows how the isothermal relaxation segments mostly bunched together. Figure 6 also illustrates how the change in stress relaxation modulus ΔE was determined based on values of E at an initial time (t_o) and final time (t_f). Figure 7 is a plot of $\log E$ versus \log time for a sample of EPDM rubber (Parker E515-80) after immersion in pretreated urine for 180 days. The isothermal relaxation segments are more ‘feathered’ than those in figure 6, but the segments did not spread out well over time.

Scotch-Brite material is also somewhat similar to elastomeric/rubber materials in the type of TTS master curve it produced. Figure 8 is a plot of $\log E$ versus \log time for a sample of Scotch-Brite after immersion in brine solution for 88 days.

One exception for elastomeric materials was the Norprene® A-60-F thermoplastic elastomer, which contains some polypropylene plastic dispersed in an EPDM rubber matrix. This material did produce a smooth, continuous TTS master curve that extended to a 10-year prediction at 87,600 hours (and beyond) for E . Figure 9 shows a plot of $\log E$ versus \log time for a sample of Norprene A-60-F after immersion in pretreated urine for 84 days.

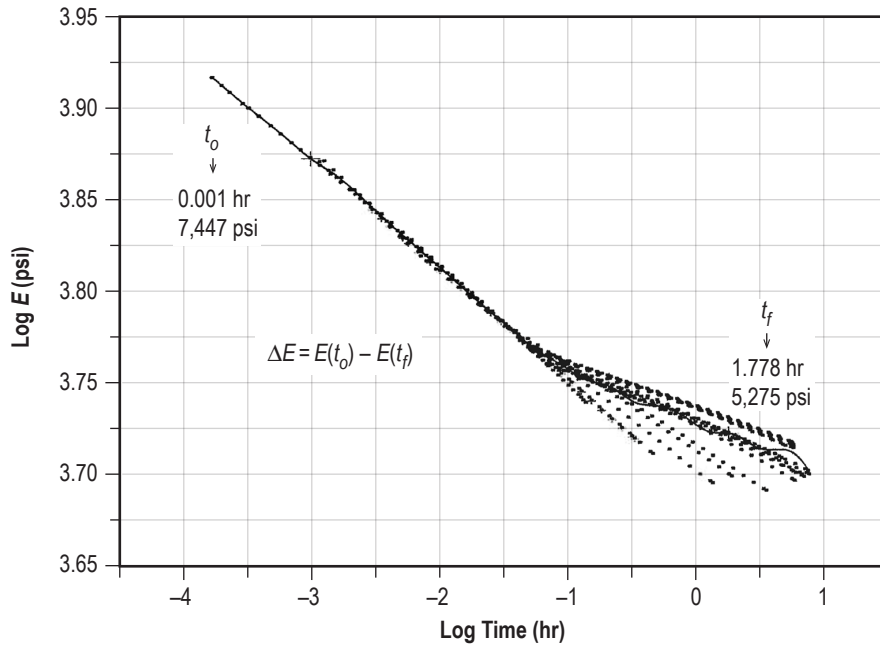


Figure 6. TTS master curve of $\log E$ versus \log time for a virgin sample of neoprene rubber drive belt. The change in DMA stress relaxation modulus E was calculated at short and long(er) times on the master curve: $\Delta E = E(t_o) - E(t_f)$.

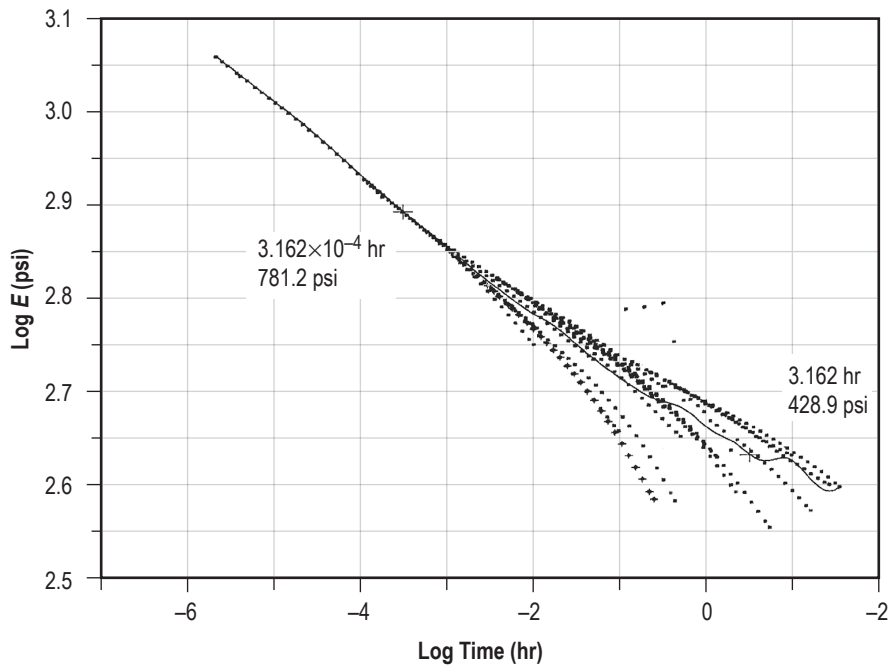


Figure 7. TTS master curve of $\log E$ versus \log time from DMA data on a sample of E515-80 EPDM rubber after immersion in pretreated urine for 180 days.

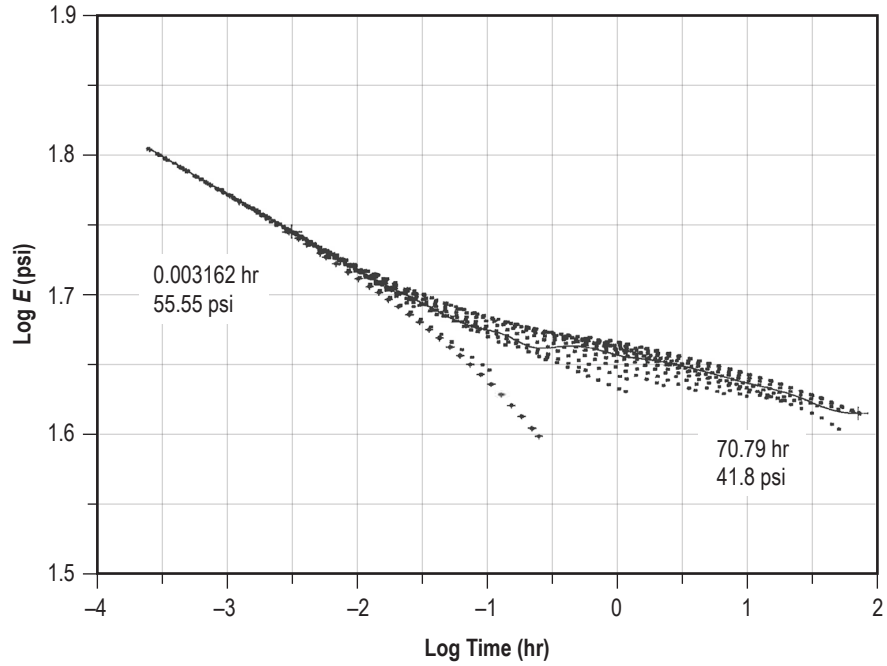


Figure 8. TTS master curve of $\log E$ versus \log time from DMA data on a sample of Scotch-Brite after immersion in brine solution for 88 days.

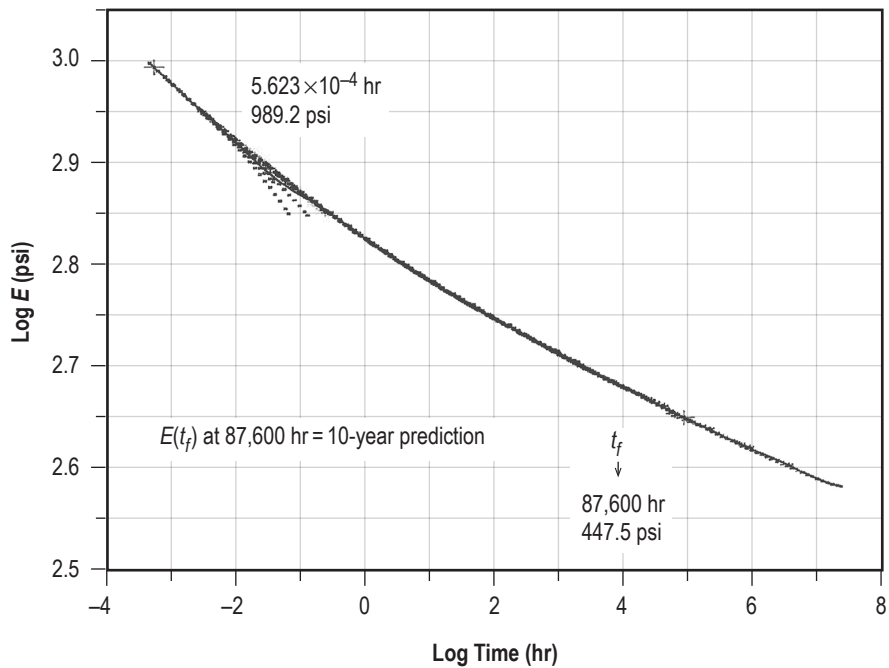


Figure 9. TTS master curve of $\log E$ versus \log time from DMA data on a sample of Norprene A-60-F thermoplastic elastomer after immersion in pretreated urine for 84 days. The value of E at $t_f = 87,600$ hours is a 10-year prediction.

3.2.2.2 Intermediate- to High-Modulus Materials. Teflon® polytetrafluoroethylene (PTFE) is an intermediate-modulus, semicrystalline polymer that produced mostly smooth, continuous TTS master curves. Figure 10 shows a plot of $\log E$ versus \log time for a sample of PTFE after immersion in pretreated urine for 90 days. A 10-year prediction at 87,600 hours (and beyond) was made for E .

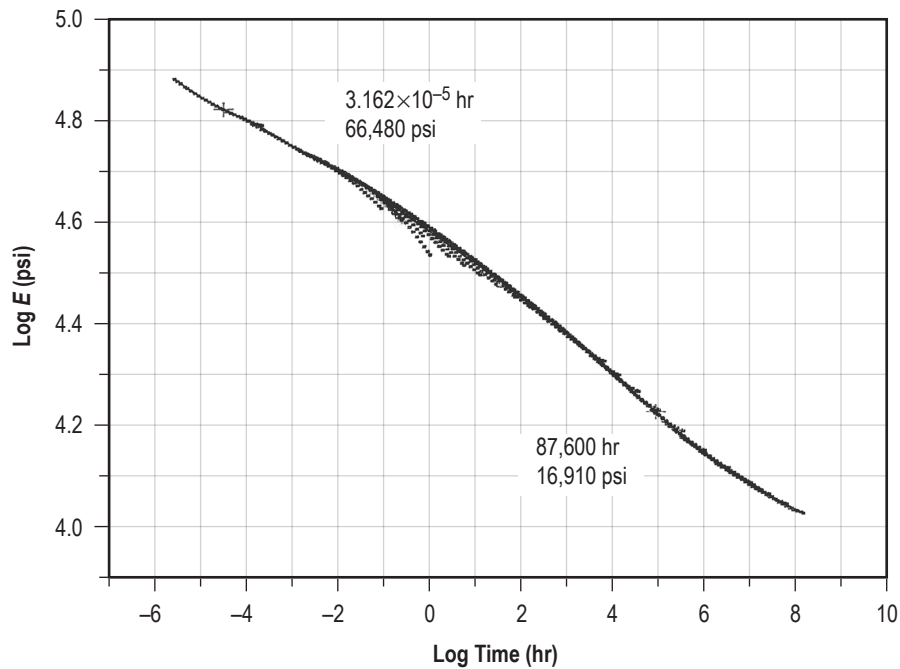


Figure 10. TTS master curve of $\log E$ versus \log time from DMA data on a sample of Teflon PTFE after immersion in pretreated urine for 90 days.

3.2.2.3 High-Modulus Materials. Figures 11 and 12 are plots of $\log E$ versus \log time for two amorphous polymers (no crystallinity): polysulfone and Ultem 2300 polyetherimide (PEI), respectively. Both of these materials allowed a 10-year prediction (and beyond) of E , but both materials produced rather different TTS curves. In figure 11, for polysulfone after immersion in brine solution for 86 days, stress relaxation modulus E was fairly constant over decades of time before it decreased rapidly. In figure 12, for Ultem 2300 after immersion in brine solution for 182 days, there was a more steady decrease in E over decades of time before it decreased rapidly. For Ultem 2300, there was also more ‘feathering’ of the isothermal relaxation segments in the TTS master curve as compared to polysulfone material.

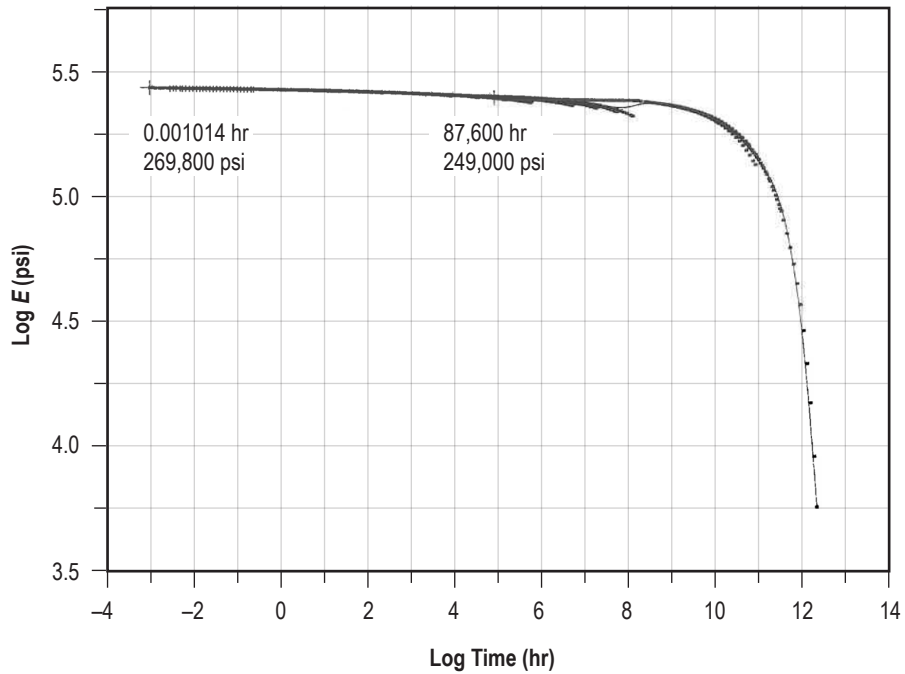


Figure 11. TTS master curve of $\log E$ versus $\log t$ from DMA data on a sample of polysulfone after immersion in brine solution for 86 days.

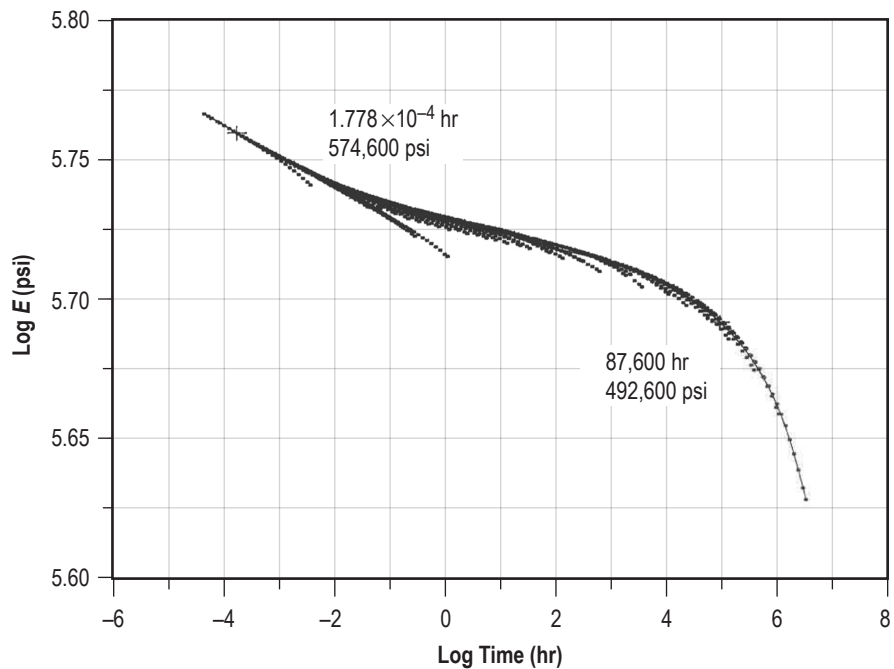


Figure 12. TTS master curve of $\log E$ versus $\log t$ from DMA data on a sample of Ultem 2300 after immersion in brine solution for 182 days.

Vespel SP-21 is also a high-modulus polymer, but is semicrystalline. However, the DuPont™ Vespel polymer family is unique in that they are not thermoplastic, but are closer to a thermoset and have no measurable melting point. Figure 13 is a plot of $\log E$ versus \log time for a sample of Vespel SP-21 after immersion in pretreated urine for 42 days. Even though the master curve allowed a 10-year prediction (and beyond) of E , there was considerable ‘feathering’ of the isothermal relaxation segments.

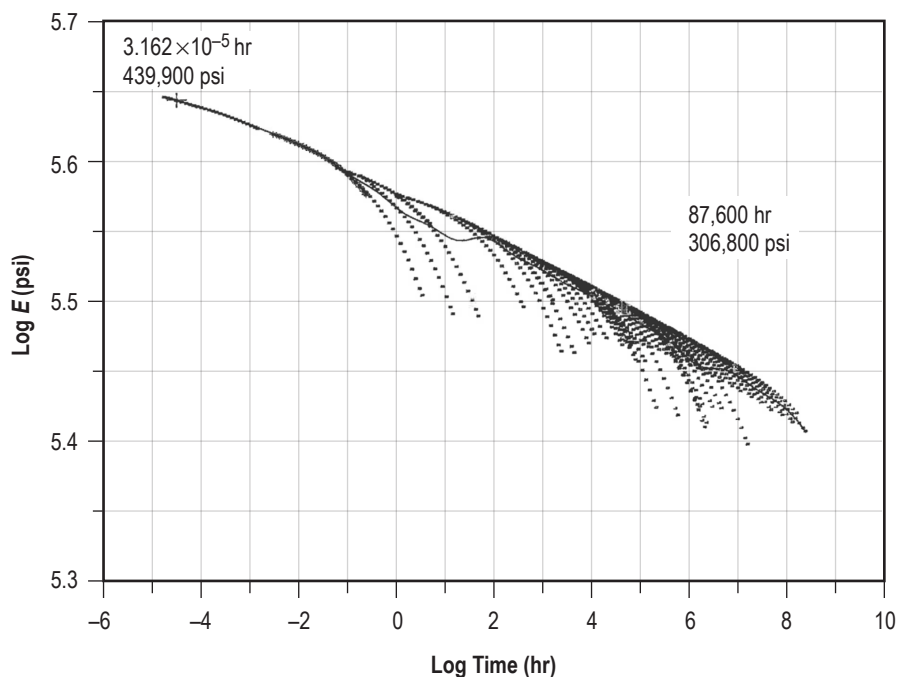


Figure 13. TTS master curve of $\log E$ versus \log time from DMA data on a sample of Vespel SP-21 after immersion in pretreated urine for 42 days.

3.2.3 Summary of Averaged Dynamic Mechanical Analysis Stress Relaxation Data From Time-Temperature Superposition Master Curves

3.2.3.1 Calculated and Tabulated Data. Tables 5–7 summarize averaged DMA stress relaxation data from TTS master curves for nonmetallic materials immersed in pretreated urine and brine solutions for times up to ≈ 180 days. The percent of the virgin value for $\Delta E(t_o - t_f)$ represents an average of two DMA samples tested per material for each solution and time of immersion. Two DMA samples were also tested for virgin material (not immersed in solution). Table 5 represents intermediate- to high-modulus materials, and table 6 is for elastomeric/rubber materials (including Scotch-Brite).

Table 5. Summary of averaged DMA stress relaxation data for nonmetallic materials with intermediate to high modulus.

Nonmetallic Material	Immersion Time in Solution (Days)		Percent of Virgin Value for $\Delta E (t_0 - t_f)$		E at t_f Determined for 10-Year Prediction?
	Pretreat	Brine	Pretreat	Brine	
Teflon PTFE	45	47	104.5	98.8	Yes
	90	92	105.0	106.5	Yes
	180	182	92.5	96.0	Yes
Kynar 740 PVDF	42	44	107.5	101.2	Yes
	85	87	105.5	107.4	Yes
	180	182	97.9	101.2	Yes
Vespel SP-21 polyimide	42	44	106.5	112.5	Yes
	84	86	106.0	101.6	Yes
	180	182	92.7	86.8	Yes
Ultem 2300 polyimide with 30% glass fiber	40	42	140.2	158.3	Yes
	82	84	130.8	145.1	Yes
	180	182	124.0	143.6	Yes
Polysulfone	40	42	96.5	81.5	Yes
	84	86	76.6	62.2	Yes
	180	182	53.4	45.4	Yes
Polypropylene	44	46	94.1	90.0	Yes
	84	86	106.1	110.5	Yes
	180	182	126.0	132.4	Yes

Table 6. Summary of averaged DMA stress relaxation data for elastomeric/rubber materials (including Scotch-Brite).

Nonmetallic Material	Immersion Time in Solution (Days)		Percent of Virgin Value for $\Delta E (t_0 - t_f)$		E at t_f Determined for 10-Year Prediction?
	Pretreat	Brine	Pretreat	Brine	
Norprene A-60-F thermoplastic elastomer	44	46	92.2	92.3	Yes
	84	86	93.7	91.8	Yes
	180	182	86.5	84.7	Yes
Parker V747-75 fluorocarbon elastomer	42	44	99.1	93.2	No
	86	88	88.5	90.7	No
	180	182	92.4	88.0	No
Parker E751-65 EPDM elastomer	46	48	59.6	63.0	No
	90	92	72.0	63.2	No
	180	182	60.3	55.2	No
Parker E515-80 EPDM elastomer	44	46	65.1	68.0	No
	86	88	76.0	67.7	No
	180	182	64.8	60.4	No
Neoprene elastomer with Kevlar fibers	42	44	76.8	63.7	No
	82	84	66.4	61.2	No
	180	182	68.0	61.6	No
Scotch-Brite	44	46	138.5	97.2	No
	86	88	108.7	129.3	No
	180	182	145.6	92.1	No

Table 7. Summary of averaged DMA stress relaxation data for nonmetallic materials, including data obtained after at least one year of immersion.

Nonmetallic Material	Immersion Time in Solution (Days)		Percent of Virgin Value for $\Delta E (t_0 - t_f)$		E at t_f Determined for 10-Year Prediction?
	Pretreat	Brine	Pretreat	Brine	
Polysulfone	40	42	96.5	81.5	Yes
	84	86	76.6	62.2	Yes
	180	182	53.4	45.4	Yes
	375	377	48.0	53.5	Yes
Ultem 2300 polyimide with 30% glass fiber	40	42	140.2	158.3	Yes
	82	84	130.8	145.1	Yes
	180	182	124.0	143.6	Yes
	368	370	167.5	181.6	Yes
Parker E751-65 EPDM elastomer	46	48	59.6	63.0	No
	90	92	72.0	63.2	No
	180	182	50.3	55.2	No
	380	382	62.9	60.1	No
Neoprene elastomer with Kevlar fibers	42	44	76.8	63.7	No
	82	84	66.4	61.2	No
	180	182	68.0	61.6	No
	370	372	72.5	58.0	No

3.2.3.2 Intermediate- to High-Modulus Materials. All of the materials in table 5 yielded values of stress relaxation modulus E at time t_f that represented a 10-year prediction. The data in table 5 are plotted graphically in figures 14 and 15.

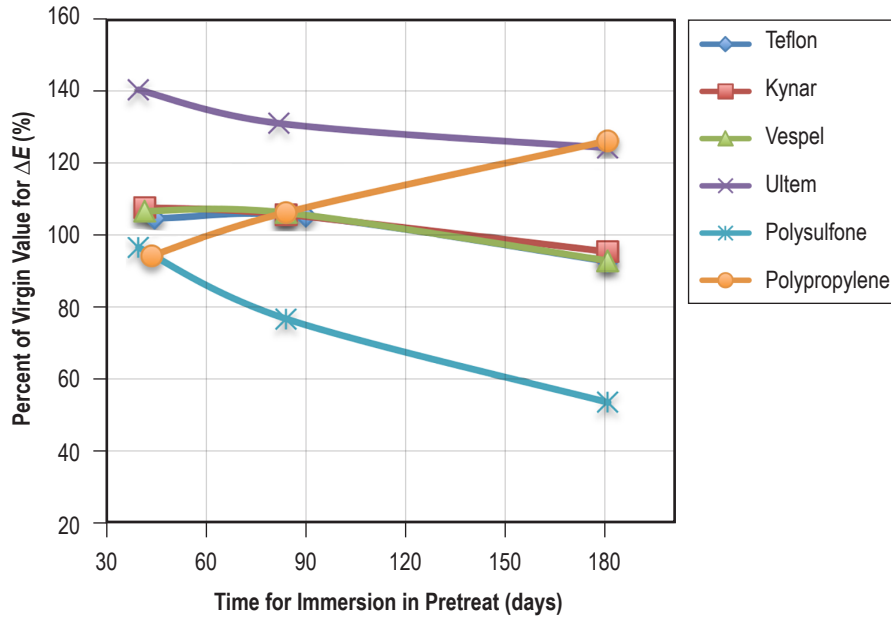


Figure 14. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time for immersion in pretreated urine to ≈ 180 days for several non-metallic materials with intermediate to high modulus.

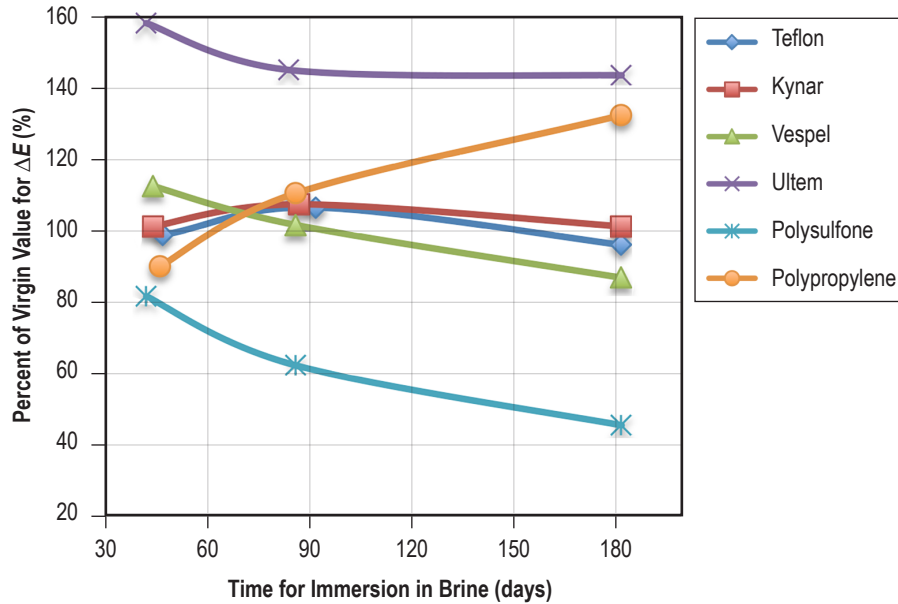


Figure 15. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time for immersion in brine solution to ≈ 180 days for several nonmetallic materials with intermediate to high modulus.

In figure 14, for immersion in pretreated urine, Ultem 2300 and polysulfone clearly had ΔE values considerably higher and lower, respectively, than 100% of the virgin value for ΔE (considered no change). Polypropylene was the only material that showed an increase in ΔE (stiffness) with increased immersion time in pretreated urine. Teflon, Kynar, and Vespel showed the least change in ΔE with increased immersion time in pretreated urine.

In figure 15, for immersion in brine solution, the trends in ΔE with increasing immersion time were similar to those shown in figure 14. The Ultem 2300 and polysulfone had slightly higher and lower ΔE values with increasing immersion time, respectively, than the ΔE values in figure 14. This perhaps shows that the brine solution yields slightly greater change in a material property such as modulus with increasing immersion time than pretreated urine solution.

3.2.3.3 Elastomeric/Rubber Materials (Including Scotch-Brite). With the exception of Norprene thermoplastic elastomer, none of the materials in table 6 yielded values of stress relaxation modulus E at time t_f that represented a 10-year prediction. This was due to the relaxation segments for E tending to ‘bunch’ together and not creating a smooth, continuous TTS master curve (as described in sec. 3.2.2.1). The data in table 6 are plotted graphically in figures 16 and 17.

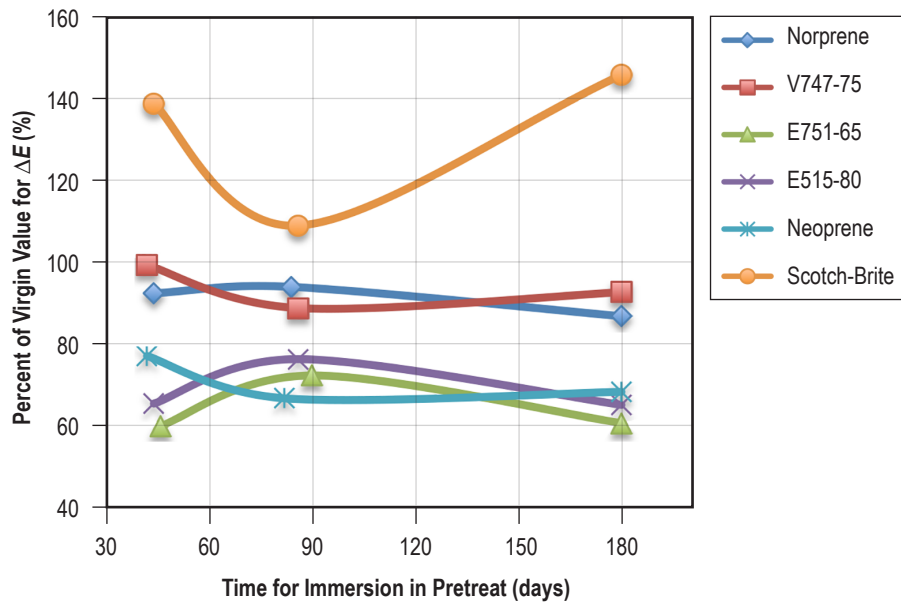


Figure 16. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time for immersion in pretreated urine to ≈ 180 days for several non-metallic materials with low modulus (elastomer/rubber materials and Scotch-Brite).

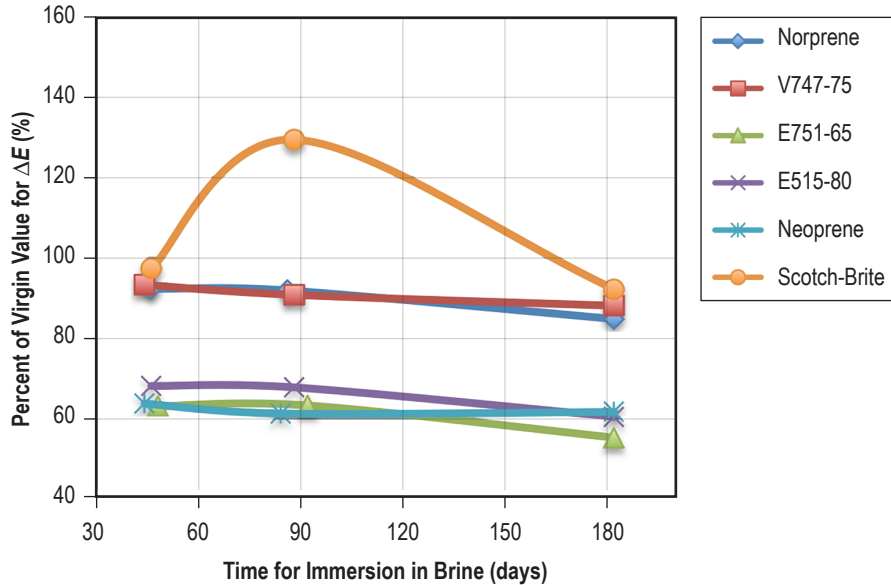


Figure 17. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time for immersion in brine solution to ≈ 180 days for several nonmetallic materials with low modulus (elastomer/rubber materials and Scotch-Brite).

In figure 16, for immersion in pretreated urine, neoprene and the two EPDMs clearly had the greatest decrease in ΔE ($\approx 35\%$ to $\approx 40\%$) compared to virgin material, with increasing immersion time. The Scotch-Brite material clearly had the greatest increase in ΔE ($\approx 45\%$) with increasing immersion time. However, it is very difficult to make accurate measurements of DMA sample dimensions for Scotch-Brite because of its porosity and softness. Because of this, it is also difficult to obtain accurate DMA modulus values for a material like Scotch-Brite. In figure 16, the V747-75 and Norprene had the least change in ΔE (compared to virgin material) with increasing immersion time.

In figure 17, for immersion in brine solution, the trends in ΔE with increasing immersion time were fairly similar to those shown in figure 16. For soft materials like elastomers and Scotch-Brite, it appeared that a plot of ΔE versus immersion time tended to ‘flatten’ the curve more for immersion in brine solution than it did for immersion in pretreated urine solution.

3.2.3.4 Nonmetallic Materials With Large ΔE Values Subjected to Longer Immersion Times.

There were two high-modulus materials (Ultem 2300 and polysulfone) that showed large changes in ΔE with increasing immersion time in both pretreated urine and brine solutions. The Ultem 2300 and polysulfone became much stiffer and less stiff (compared to virgin material), respectively, with increasing immersion time. There were three low-modulus materials (E751-65, E515-80 EPDMs, and neoprene drive belt) that showed a large decrease in ΔE (i.e., more softening) with increasing immersion time.

It was desirable to test these materials for longer immersion times (at least one year) in both pretreated urine and brine solutions to look for undesirably large changes in ΔE . Table 7 summarizes DMA stress relaxation data from TTS master curves for four of these five materials with original data to ≈ 180 days of immersion, plus additional data to >1 year of immersion. The data in table 7 are plotted graphically in figures 18 and 19. As before, data in table 7 represent the average of two DMA samples tested per material for each solution and time of immersion.

In figure 18, for immersion in pretreated urine, the most noticeable change was for Ultem 2300, which increased in ΔE (stiffness compared to virgin material) by an additional 43.5% from 180 to 368 days of immersion. The next most noticeable change in ΔE was for EPDM (E751-65), which increased in ΔE by an additional 12.6% from 180 to 380 days of immersion.

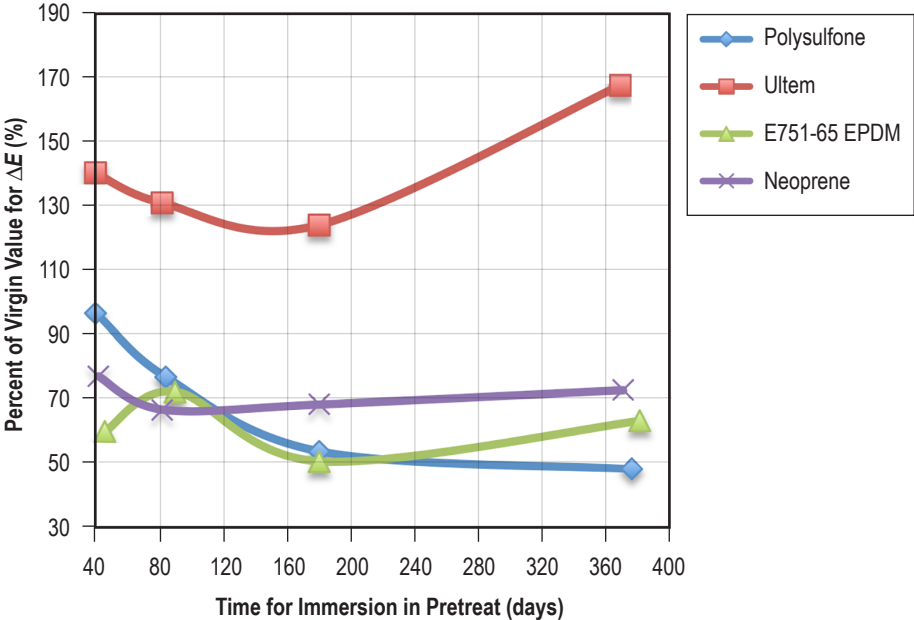


Figure 18. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time for immersion in pretreated urine to >1 year for several nonmetallic materials with significant \pm changes in percentage of ΔE after ≈ 180 days of immersion.

In figure 19, for immersion in brine solution, the most noticeable change was still for Ultem 2300, which increased in ΔE by an additional 38% from 182 to 370 days of immersion. For the other three materials (polysulfone, E751-65 EPDM, and neoprene), overall, there were fewer changes in ΔE with an increase in immersion time (>1 year) than for similar immersion times in pretreated urine.

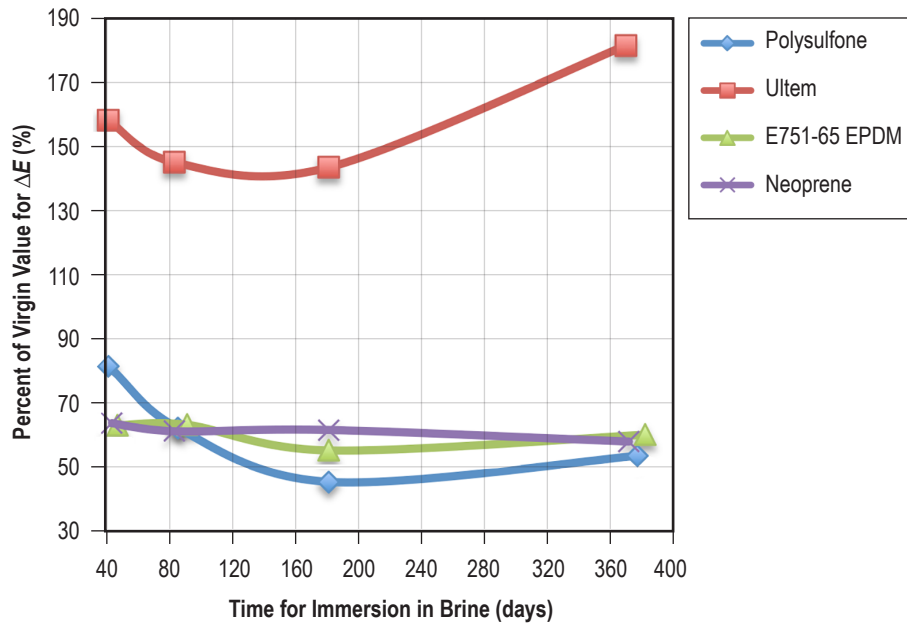


Figure 19. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time for immersion in brine solution to >1 year for several nonmetallic materials with significant +/- changes in percentage of ΔE after ≈ 180 days of immersion.

3.2.3.5 Dynamic Mechanical Analysis Stress Relaxation Data Compared Between 2013–2014 and 2002–2003. In 2002–2003, DMA stress relaxation data were obtained on a number of UPA nonmetallic materials that were immersed in the currently used Russian pretreat and brine solutions containing CrO_3 oxidizer and H_2SO_4 pretreat. For the same or similar nonmetallic materials tested by DMA in 2013–2014 and 2002–2003, it was desirable to compare the stress relaxation data for any possible changes due to the change in pretreat from H_2SO_4 to newly proposed H_3PO_4 . The CrO_3 oxidizer remained the same (and in the same concentration) for both the H_2SO_4 and H_3PO_4 pretreated urine solutions.

For many materials tested with the different pretreats, changes in ΔE (compared to virgin material) with increasing immersion time to ≈ 180 days yielded fairly similar ΔE values and trends. This is shown—for immersion in pretreated urine only—in figures 20–25 for the following materials: PTFE, Vespel, Norprene, EPDM, and neoprene. In 2002–2003, it was uncertain what type/grade of EPDM was tested, but the results were fairly similar to those of the two EPDMs (E751-65 and E515-80), as shown in figure 23.

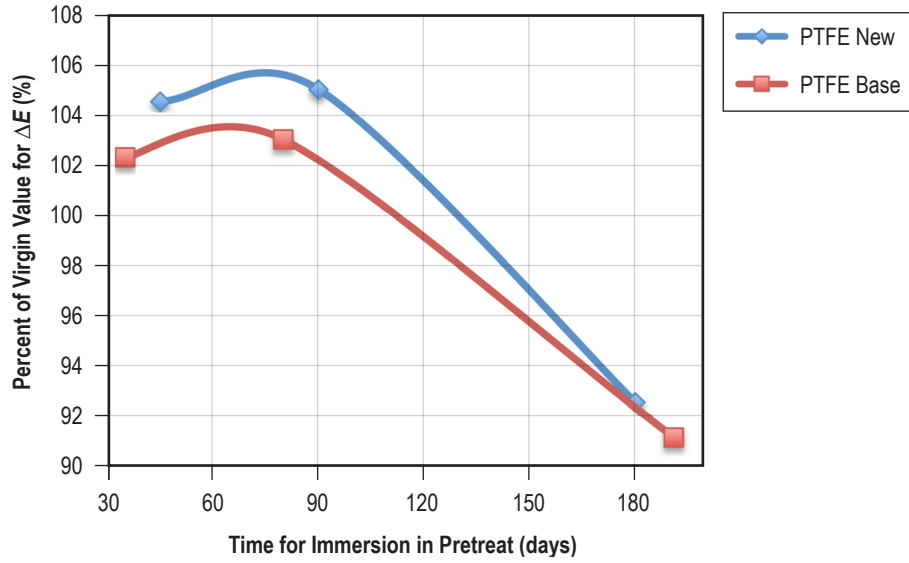


Figure 20. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for Teflon PTFE material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline).

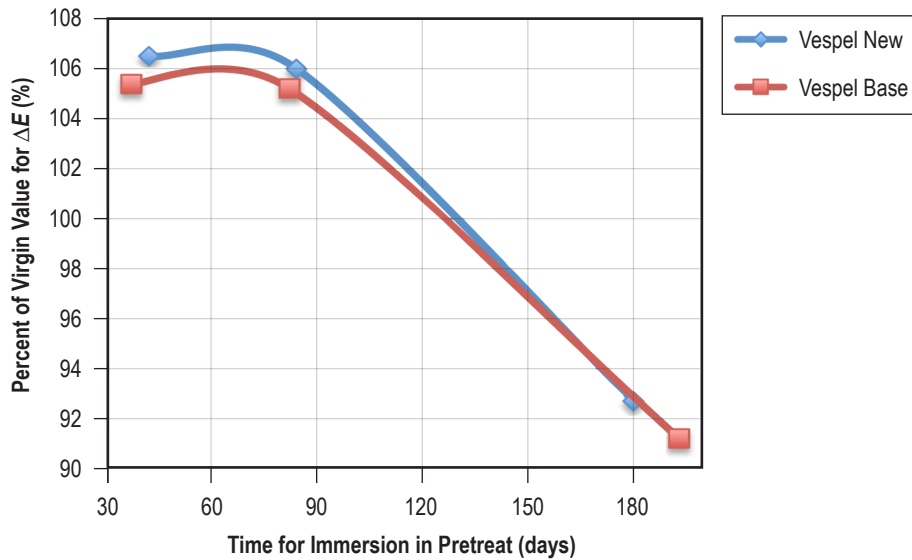


Figure 21. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for Vespel SP-21 material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline).

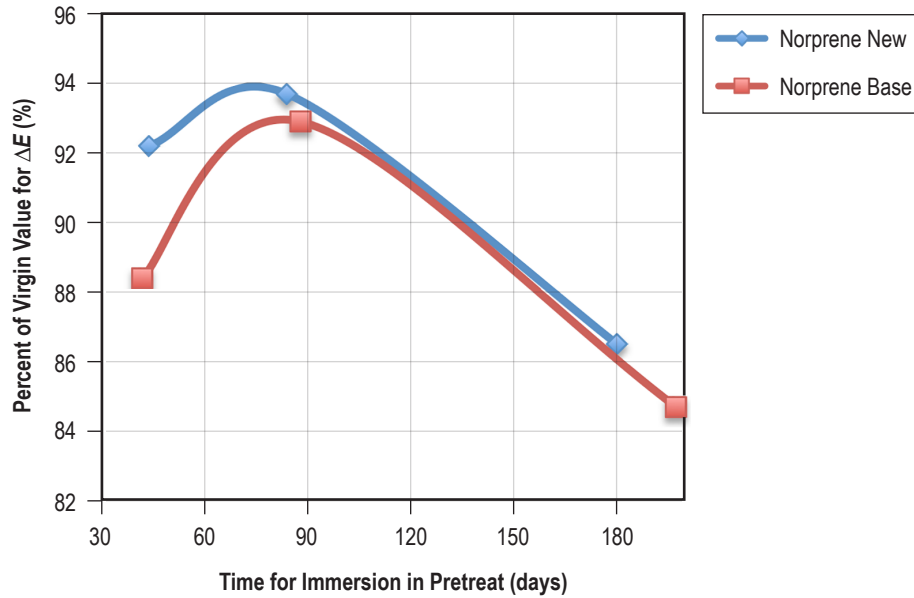


Figure 22. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for Norprene A-60-F material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline).

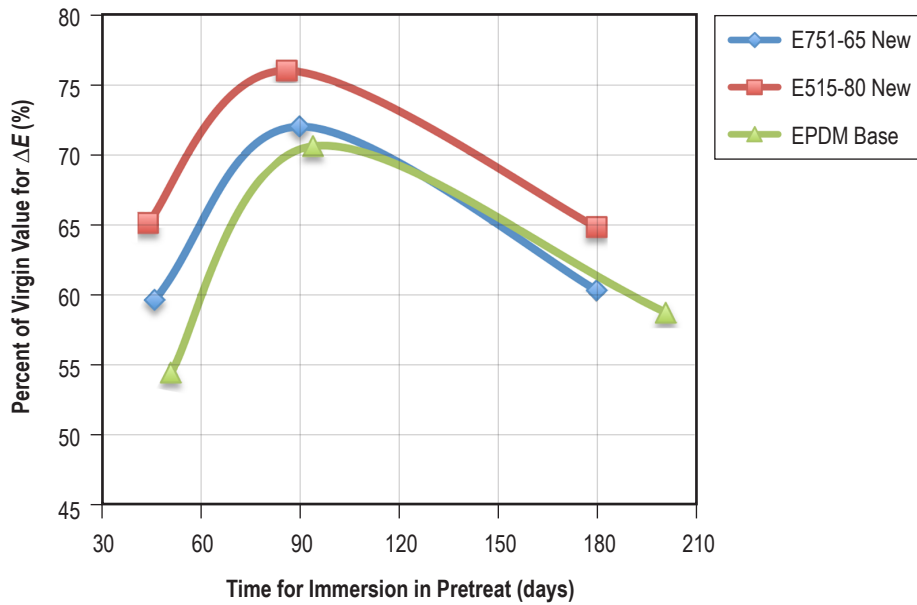


Figure 23. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for EPDM rubber material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline). Two Parker formulations of EPDM were tested for this work (E751-65 and E515-80).

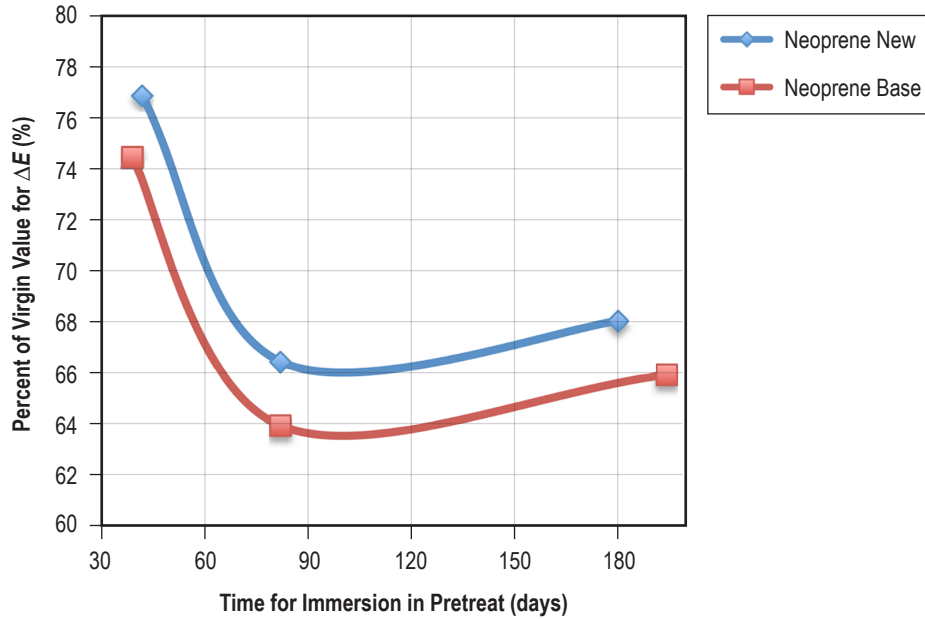


Figure 24. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for neoprene rubber drive belt material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline).

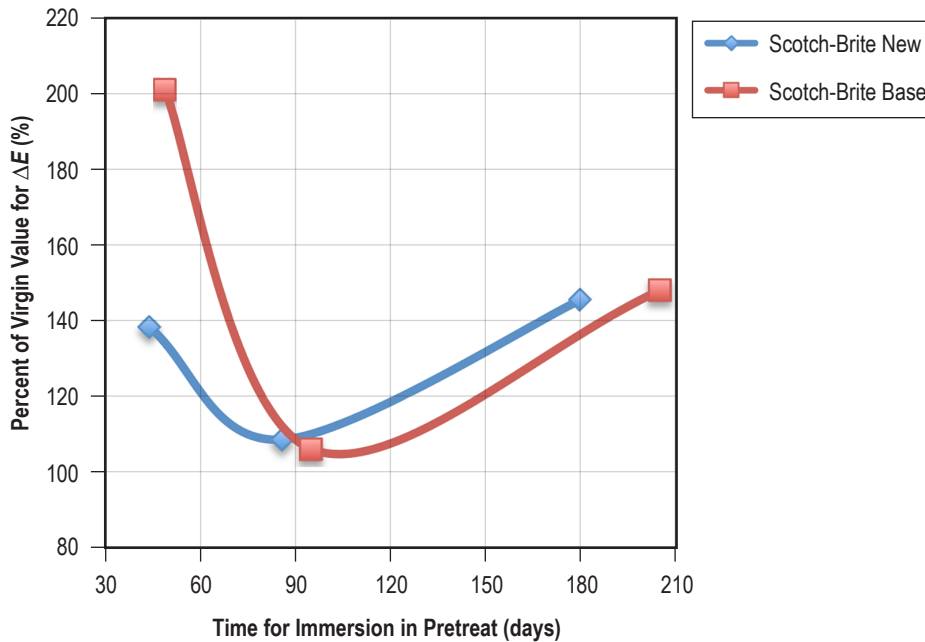


Figure 25. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for Scotch-Brite material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline).

There were three materials (Ultem 2300, polysulfone, and V747-75 elastomer) that showed significant changes in ΔE with increasing immersion time to ≈ 180 days for the two different pre-treated acid solutions. These changes are shown in figures 26–31 for both the pretreated urine and brine solutions.

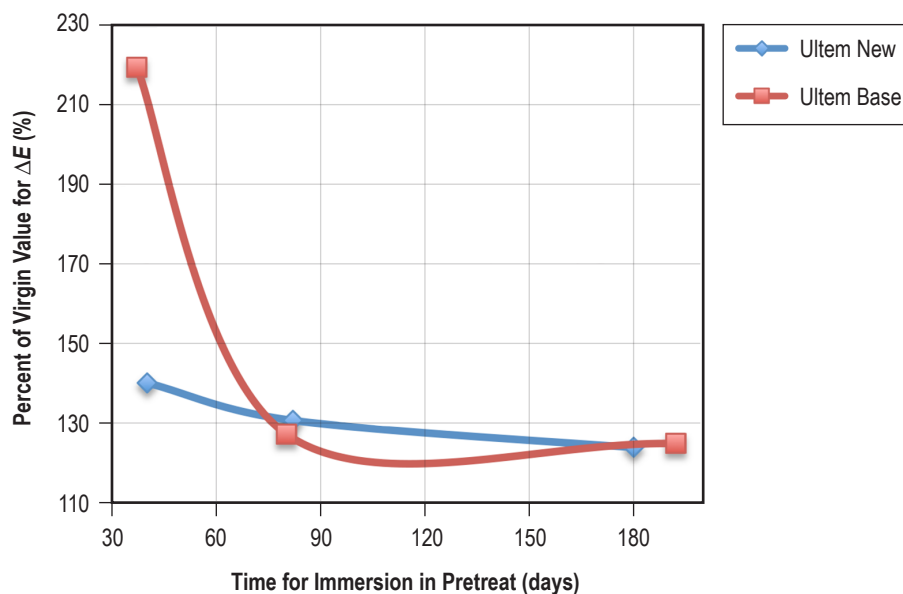


Figure 26. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for Ultem 2300 material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline).

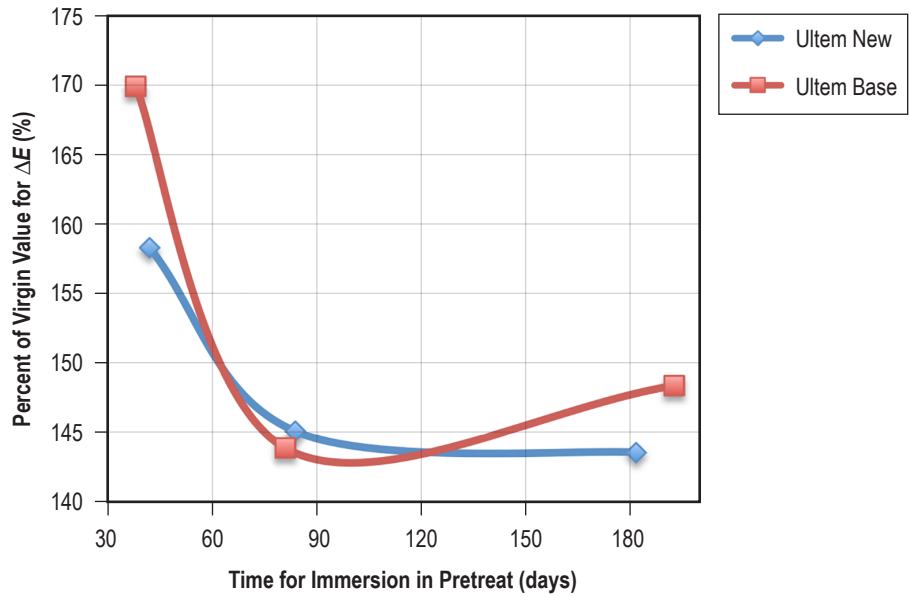


Figure 27. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in brine solution to 180+ days for Ultem 2300 material: comparison of brine with H_3PO_4 (new) and H_2SO_4 (baseline).

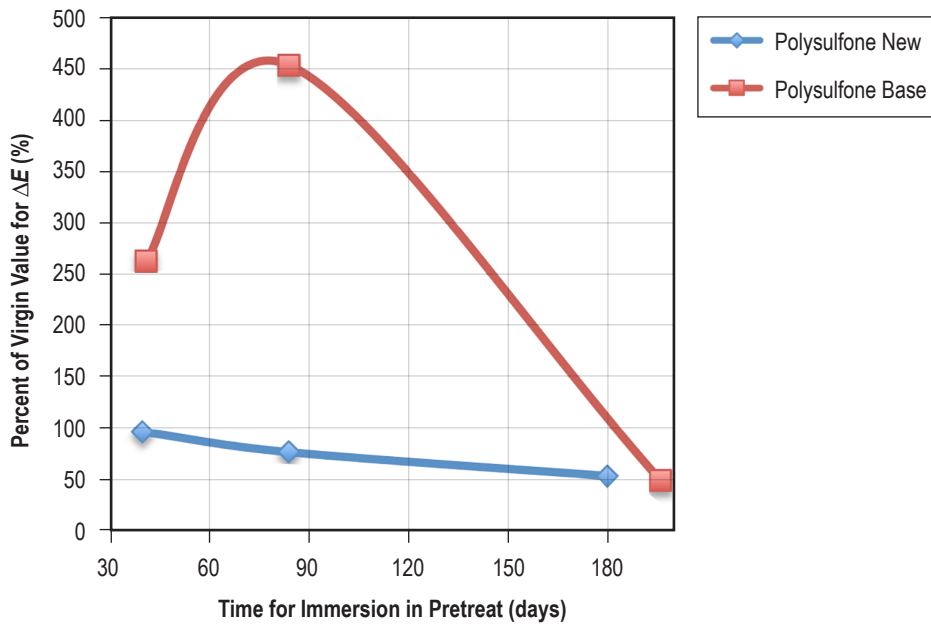


Figure 28. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for polysulfone material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline).

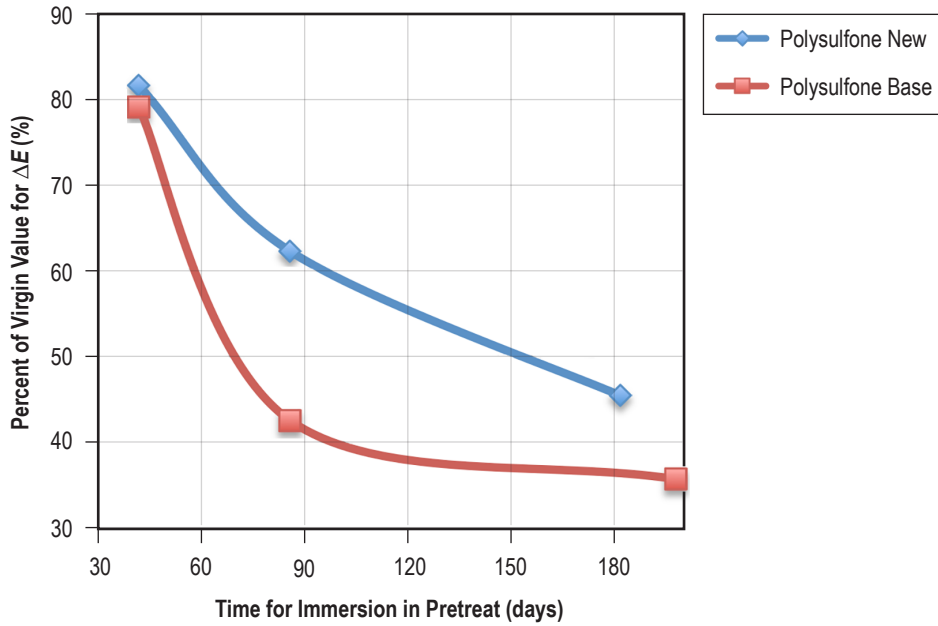


Figure 29. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in brine solution to 180+ days for polysulfone material: comparison of brine with H_3PO_4 (new) and H_2SO_4 (baseline).

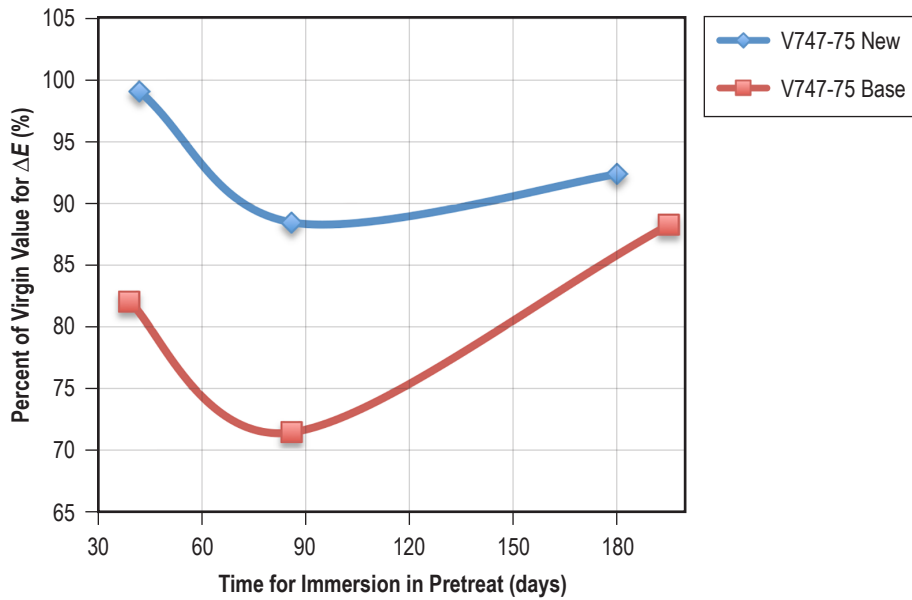


Figure 30. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in pretreated urine to 180+ days for V747-75 fluorocarbon rubber material: comparison of pretreat with H_3PO_4 (new) and H_2SO_4 (baseline).

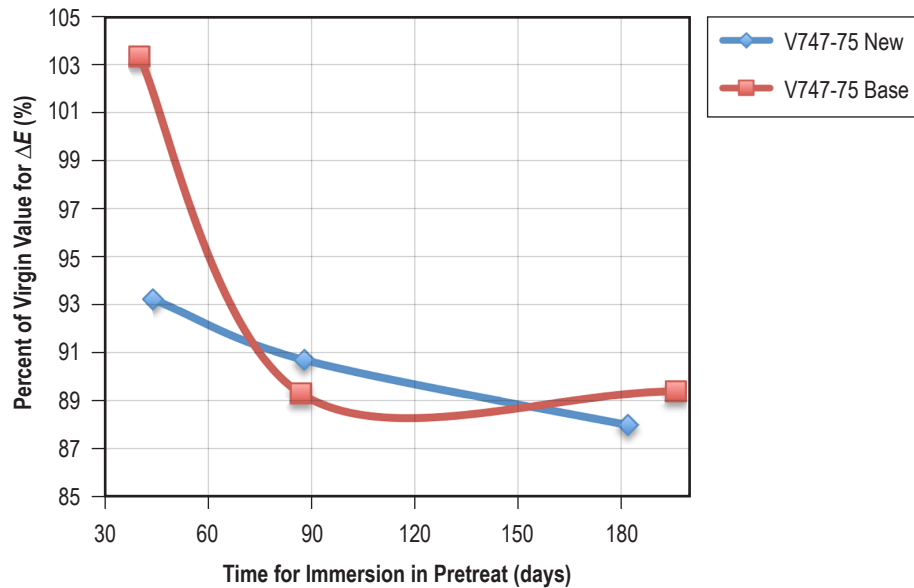


Figure 31. Percent of virgin value for change in DMA stress relaxation modulus (ΔE) versus time in immersion in brine solution to 180+ days for V747-75 fluorocarbon rubber material: comparison of brine with H_3PO_4 (new) and H_2SO_4 (baseline).

In figure 26, Ultem shows a very high ΔE at $\approx 220\%$ of the virgin value for 37 days immersion in pretreated urine containing H_2SO_4 . However, in figure 27, there was much better agreement in ΔE versus immersion time for brine solution containing H_3PO_4 (new) and H_2SO_4 (baseline).

In figure 28, polysulfone shows very high ΔE at $\approx 263\%$ and $\approx 454\%$ of the virgin value for 41 and 84 days immersion, respectively, in pretreated urine containing H_2SO_4 . However, in figure 29, there was much better agreement in ΔE versus immersion time for brine solution containing H_3PO_4 (new) and H_2SO_4 (baseline).

In figure 30, V747-75 fluorocarbon elastomer shows a difference in ΔE versus immersion time of up to 17% between the curves for pretreated urine containing H_3PO_4 (new) and H_2SO_4 (baseline). However, in figure 31, there was better agreement in ΔE versus immersion time for brine solution containing H_3PO_4 (new) and H_2SO_4 (baseline).

In figure 26 for Ultem and figure 28 for polysulfone (both for immersion in pretreated urine), it appears the curves for the new and baseline acid pretreatment were in much better agreement for longer immersion times >180 days. For the baseline pretreated urine tested in 2002–2003, it appears that a few of the TTS master curves did not yield a smooth overlap of isothermal relaxation segments, leading to an erroneously high percentage of ΔE values compared to virgin material for both Ultem and polysulfone.

4. SUMMARY AND CONCLUSIONS

4.1 Background and Approach for Compatibility Testing

Twelve nonmetallic materials—wetted directly or indirectly by pretreated urine—were tested in 2013–2014 for compatibility with pretreated urine and brine solutions. The testing was performed as part of a proposed change from H_2SO_4 to H_3PO_4 used to pretreat urine as part of the UPA used on ISS to convert urine into potable water.

Rectangular samples of the 12 materials were tested by DMA to determine changes in stress relaxation modulus E after immersion in pretreated urine and brine solutions (pretreated with H_3PO_4) at room temperature for times up to ≈ 180 days. There were six materials with intermediate to high modulus, and six with low modulus (elastomeric/rubber and Scotch-Brite).

4.2 Methodologies for Dynamic Mechanical Analysis Compatibility Testing and Data Analysis

Methodologies for DMA compatibility testing and data analysis are summarized as follows:

- Isothermal stress relaxation segments were obtained from 25 °C to an elevated temperature roughly equal to the maximum recommended continuous operating temperature of the material.
- The isothermal segments were shifted on the timescale to yield a continuous master curve of E over decades of time. Lower temperature segments were shifted to shorter times, and higher temperature segments were shifted to longer times.
- The goal for each material was to predict E after 10 years of continuous use.
- For each master curve, values of E were chosen at a short time (t_o) and low temperature and at a long(er) time (t_f) and high(er) temperature.
- For each material, E was calculated and evaluated as $\Delta E = E(t_o) - E(t_f)$ after immersion in pretreated urine and brine solutions for a specified time. The ΔE for each immersed sample was evaluated as a percentage of ΔE for virgin material (not immersed).
- For each material, two DMA samples were tested per solution and immersion time, as well as for virgin samples, and the percentage of ΔE was reported as an average value.

4.3 Results and Discussion for Compatibility Testing

4.3.1 Testing for up to Approximately 180 Days of Immersion in Pretreated Urine and Brine Solutions

For intermediate- to high-modulus materials, Teflon, Kynar, and Vespel did not show considerable changes in percentage of ΔE for immersion in pretreated urine and brine solutions up to ≈ 180 days. However, Ultem and polysulfone showed considerable changes in ΔE of up to $\approx 58\%$ and $\approx -55\%$, respectively, in the two solutions. Polypropylene was the only material in this group to show an increase in percentage of ΔE with increasing immersion time in the two solutions, with percentage of ΔE of up to $\approx 32\%$ after 180 days.

For low-modulus materials, Norprene thermoplastic elastomer and V747-75 fluorocarbon elastomer did not show considerable changes in percentage of ΔE for immersion in pretreated urine and brine solutions up to ≈ 180 days. However, neoprene and two EPDM elastomers showed considerable changes in ΔE of up to $\approx -45\%$ in the two solutions after ≈ 180 days. Scotch-Brite showed changes in ΔE of up to $\approx 46\%$ in the two solutions after ≈ 180 days. The softness and porosity of Scotch-Brite makes it difficult to obtain accurate modulus values for this material.

4.3.2 Testing for Approximately One Year of Immersion in Pretreated Urine and Brine Solutions

Because of considerable increases and decreases in percentage of ΔE for some of the materials described in section 4.3.1, it was desirable to perform DMA testing on these materials after at least one year of immersion in the pretreated urine and brine solutions. The following four materials were tested: (1) Polysulfone, (2) Ultem 2300, (3) neoprene drive belt material, and (4) E751-65 EPDM elastomer. Although both EPDM elastomers showed similar test results, only one was chosen for further testing. There were some slight increases and decreases in percentage of ΔE for three of the materials (polysulfone, E751-65, and neoprene) for the prolonged immersion in both solutions. However, Ultem 2300 showed a considerable change in ΔE of up to 43.5% in the two solutions between ≈ 180 and ≈ 370 days of immersion.

4.3.3 Comparison of Dynamic Mechanical Analysis Stress Relaxation for Solutions With a Different Acid Pretreat

DMA stress relaxation data on nonmetallic materials in 2013–2014 with the H_3PO_4 pretreat were compared to the same type of data on the same or similar materials tested in 2002–2003 with the H_2SO_4 pretreat. In both timeframes, material samples were immersed in both pretreated urine and brine solutions for times of at least 180 days. For the most part, data obtained with the different acid pretreatments agreed fairly well for most materials immersed in both solutions. The exceptions were Ultem 2300 and polysulfone, which showed some unusually large increases in percentage of ΔE with increasing immersion time in urine pretreated with H_2SO_4 . The large increases in percentage of ΔE did not occur for immersion in brine solution pretreated with H_2SO_4 . These anomalous increases in percentage of ΔE were likely due to some of the TTS master curves not yielding smooth overlap of relaxation segments.

4.3.4 Conclusion of Dynamic Mechanical Analysis Stress Relaxation Data

There were changes in DMA stress relaxation modulus (ΔE) greater than $\pm 50\%$ for some nonmetallic materials tested, indicating a significant increase or decrease in stiffness for immersion in pretreated urine and brine solutions for ≈ 180 days to at least one year. Other than discoloration due to the solutions, it appeared that none of the materials were chemically attacked by the solutions during immersion. While significant changes in modulus are not necessarily a prerequisite for failure of a material part on the UPA, such changes are an indicator that performance of the material parts should be monitored for prolonged operating use on the UPA.

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14. ABSTRACT <p>On International Space Station (ISS), the Urine Processor Assembly (UPA) converts human urine and flush water into potable water. The urine is acid-pretreated primarily to control microbial growth. In recent years, the sulfuric acid (H₂SO₄) pretreatment was believed to be largely responsible for producing salt crystals capable of plugging filters in UPA components and significantly reducing the percentage of water recovery from urine. In 2012, ISS management decided to change the acid pretreatment for urine from sulfuric to phosphoric with the goal of eliminating or minimizing formation of salt crystals. In 2013-2014, as part of the qualification of the phosphoric acid (H₃PO₄) formulation, samples of 12 nonmetallic materials used in UPA components were immersed for up to one year in pretreated urine and brine solutions made with the new H₃PO₄ formulation. Dynamic mechanical analysis (DMA) was used to measure modulus (stiffness) of the immersed samples compared to virgin control samples. Such compatibility data obtained by DMA for the H₃PO₄-based solutions were compared to DMA data obtained for the H₂SO₄-based solutions in 2002-2003.</p>								
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