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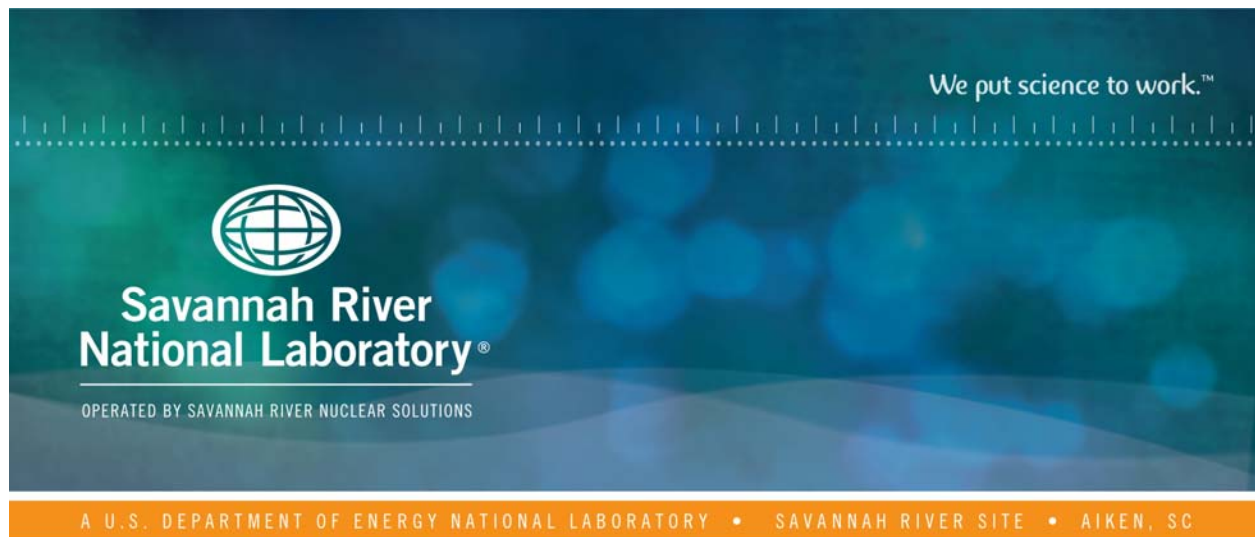
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The Effect of TiDG, DCiTG (Lix[®]79), and Other Suppressors on the Compatibility of NGS with SWPF Polymers

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EXECUTIVE SUMMARY

This document provides a screening calculation of the potential miscibility of specific polymers proposed for use in the SWPF, notably polyether-etherketone (PEEK), ultra-high molecular weight polyethylene (UHMWPE), polyvinylidene fluoride (PVDF), Viton[®] B fluoroelastomer, and Tefzel[®] ethylene-tetrafluoroethylene (ETFE) copolymer in different suppressors including trisisodecyl guanidine (TiDG), dicyclohexane-isotrisdecyl guanidine (DCiTG), and two other dicyclohexane guanidine related suppressors. The evaluation suggests that Tefzel[®] ETFE may mildly interact with the Next Generation Solvent (NGS) regardless of the suppressor used. Based on this approach (calculations), the other listed polymers appear compatible with the NGS containing the four different suppressors. There is evidence of chemical affinity between Tefzel[®] ETFE, Viton[®] B with aliphatic amines (which are degradation by-products from the TiDG). In addition, the calculation suggests that drawing miscibility conclusions from exposing polymers to a single pure component may give misleading or incorrect results due to the potential for synergistic effects or other interactions.

Based on this review, the Savannah River National Laboratory (SRNL) / Environmental, Materials, and Energy Sciences (EMES) recommends testing Tefzel[®] ETFE, Kalrez[®], and Viton[®] B fluoropolymers for compatibility with NGS solvent containing different formulation of the four suppressors listed in this report. Specifically, the same compounds and grades of the polymers used in the facility should be evaluated if such details are known. Minor variations in processing and compounding may affect interactions. Different grades of polymers should also be investigated (with similar chemical resistance and mechanical response) that may include silicon based polymers (such as fluorosilicon, methyl silicone, methyl vinyl silicone, and possibly methyl phenyl silicone). Miscibility studies should last at least 24 days to capture kinetics and steady state loading. In addition, thermal studies should include at least one compatibility at 35°C. Finally, gamma irradiation tests should be conducted on Viton B and Kalrez under humid air. Irradiated polymers should be measured for samples dimensions and hardness (ASTM D2240).

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LIST OF ABBREVIATIONS

CSSX	Caustic Side Solvent Extraction
DCHU	1,3-dicyclohexylurea
DCiTG	dicyclohexylisotrisdecyl guanidine
DiDU	diisodecylurea
DSS	Decontaminated Salt Solution
DTDCHG	2-decyl-1-tetradecyl dicyclohexane guanidine
EMES	Environmental, Materials, and Energy Sciences
ETFE	ethylene tetrafluoroethylene copolymer
HDCHG	11-heneicosyl dicyclohexane guanidine
HDPE	high-density polyethylene
HFP	hexafluoropropylene
IDA	isodecyl amine
MCU	Modular Caustic-Side Solvent Extraction Unit
NGS	Next Generation Solvent
PEEK	polyether ether ketone
PTFE	polytetrafluoroethylene
PVDF	polyvinylidene fluoride
RED	Relative Energy Difference (a ratio of two numbers)
SRNL	Savannah River National Laboratory
SWPF	Salt Waste Processing Facility
TiDG	tris(isodecyl) guanidine
UHMWPE	Ultra High Molecular Weight Polyethylene

1.0 Introduction

The recently constructed SWPF is in the final stages of readiness assurance for initial operations. The higher throughput SWPF facility replaced the Modular Caustic-Side Solvent Extraction Unit (MCU) Process that successfully processed more than 7.2 million gallons of liquid waste (from 2008 to 2019). Initially, MCU operated with the BOBCalixC6 based solvent and later added an improved Next Generation Caustic-Side Solvent Extraction (NG-CSSX or NGS) solvent to create a blended solvent. During processing, it was shown that the suppressor in NGS (TiDG) was depleted in the solvent over time. With SWPF nearing operations, and with plans to transition to use of NGS, there is an opportunity to assess current materials of construction for the planned operations and solvent transition.

Materials of construction at SWPF were chosen on the initial assumption of using the BOBCalixC6 based solvent as the liquid-liquid extractor. With the current plans of using the improved NGS solvent, an in-progress review of possible technical gaps with MCU revealed similar materials of construction between SWPF and MCU.ⁱ A recommended list of materials that may contact the NGS include Kalrez[®] (FFKM) perfluoroelastomer, Tefzel[®] ethylene-tetrafluoroethylene (ETFE), PEEK, polyvinylidene fluoride (PVDF) fluoropolymer, Viton[®] B (FKM) fluoroelastomer, and UHMWPE (see Fig. 1). Of these, Kalrez[®] is very stable and there are no published solubility data due to its high chemical stability making it difficult to estimate its Hansen solubility parameters. Therefore, this elastomer is amenable to the analysis method within this document. Some of these materials were previously screened for compatibility with the NGS solventⁱⁱ but with the current plans for changing the suppressor and its proposed concentration, this report documents a screening analysis of these polymers. Another condition the proposed materials for SWPF would face during their lifetime is gamma irradiation. All polymers suffer radiation damage to different extent (and damages are accentuated in the presence of oxygen and/or humidity). For a given polymer material, radiation damage may be better tolerated when the polymer is deployed as a gasket rather than as a valve seat. Polymers like PVDF, Teflon, and Viton[®] A will readily give off HF and embrittle.ⁱⁱⁱ Others will experience chain scission, crosslinking (resulting in hardness and cracking) and increase in crystallinity like in UHMWPE.^{iv} While this is a serious concern for material selection, this document focuses on chemical compatibility of the proposed materials for SWPF.

2.0 Assumptions

There are several grades of Kalrez[®], Tefzel[®], Kynar[®] (PVDF), and Viton[®] polymers commercially available. These are also trademarked materials, with other generic equivalents available. It is important to note the generic polymer type as well as the tradename and grade in specifications, as some tradenames may be used for more than one polymer type and vendors may offer substitutes that are not the same exact polymer type, can affect compatibility.

Since no specific polymer grades were identified in the documents provided by the Gap Analysis Team (other than Viton[®] B), the results of the calculations below are for a generic ETFE, PVDF, and Viton[®] B polymers. The PVDF data is assumed to be for PVDF homopolymer, though PVDF copolymers are sometimes used for specific applications. Viton[®] A is a grade of fluoroelastomer, being a copolymer of hexafluoropropylene (HFP) and vinylidene fluoride (VF₂) with a nominal fluorine content of 66%. Viton[®] B is a terpolymer of VF₂, HFP and tetrafluoroethylene (TFE) polymers, with a nominal fluorine content of 68%.

In specific cases where the specific grade is identified such as in Figure 1, there was no solubility data available. In Figure 1, Viton[®] B is specified as the gasket for the Tk-201 (Decontaminated Salt Solution Tank) Coalescer Media gasket. As with most FKM fluoroelastomers, Viton[®] B terpolymer has limited compatibility with 1.91M hydroxide.^v The degree of compatibility or allowable degradation is unknown and varies for specific applications. Since the elastomer is deployed as a gasket under compression, the bulk of the gasket is not likely exposed directly to the caustic Decontaminated Salt Solution (DSS). Most FKM elastomers have limited resistance to high pH solutions due to sensitivity of the VF₂ group, depending on concentration and temperature. The material of construction list from SWPF listed Viton[®] B as a gasket to be used at the DSS coalescer where the majority liquid phase is high caustic salt solution. An alternate material that can resist high caustic salt solution may be the Viton Extreme ETP grades with improved resistance to high pH solutions. Similarly, PVDF fluoropolymer has limited resistance to strong caustic/high pH solutions, particularly the homopolymer grades and depending on the degree of crystallinity.

The calculation results in this report are primarily considered for screening purposes, not for absolute determination of compatibility. Compatibility results (discovery, postulated or confirmation) can only come from testing. Given this caveat, calculations used the group contribution method.^{vi} For a given polymer (or elastomer), the repeating unit was broken into groups and each group dispersive (δ_d), polar (δ_p), and hydrogen bonding (δ_h) contribution was added up to compute the solubility of the polymer. A similar exercise was done with the NGS solvent components. Table 1 lists the Hansen's parameters for the suppressors in consideration for NGS. These parameters were calculated using the tables listed in Reference vi. All suppressors have significant dispersion and hydrogen bond capabilities. Table 2 lists the recommended set of polymers that may contact the NGS solvent (contactors, coalescers, pipes, and tanks). Except for UHMWPE and Tefzel[®] ETFE, the rest of the polymers have significant polarity and hydrogen bonding capabilities. The root sum square of the difference between the polymer and the NGS solvent components was ratioed against the polymer interaction radius (R_o) to yield an index number (*RED* or Relative Energy Difference) (see equations 1 and 2). If the RED number is larger than one, the polymer is compatible with the NGS solvent.

APPLICATION	MATERIALS OF CONSTRUCTION	
	RECOMMENDED	DO NOT USE
	Wetted parts:	
Contactors	<ul style="list-style-type: none"> • 316 SST • 316L SST for welded connections 	-
Piping, Tubing, Flanges, and Fittings	<ul style="list-style-type: none"> • 304 SST • 304L SST for welded connections 	-
O-Ring, Gaskets, Packings, Seals	<p>Polymers</p> <ul style="list-style-type: none"> • PEEK (PolyEtherEtherketone) • PVDF (Polyvinylidene Difluoride) • ETFE (EthyleneTetrafluoroethylene) • Rytan[®] PPS (Poly Phenylene Sulfide) • UHMW-PE (Ultra High Molecular Weight Polyethylene) <p>Elastomers</p> <ul style="list-style-type: none"> • Kalrez 	<ul style="list-style-type: none"> • Vespel • Kapton • Teflon PTFE Fluoropolymer • Delrin/acetal • Polypropylene • Cellulosics • Butyl rubber • Teflon PTFE • Nitrile rubber (NBR/Buna-N) • Neoprene • EPDM • Viton

IDENTIFICATION

DESCRIPTION: TK-201 COALESCER MEDIA

- Manufacturer: Franken Filtertechnik
- Phase Separation Element Type: FTC IV/1250-CM-FF
- Part No: P001595
- Rating: 3 micron
- Body & Mesh Material: 316L Stainless Steel
- Gasket Material: Viton B
- To be installed in TPS 900 Phase Separation Stage (Reference Franken Drawing 2-PA-812166.7)
- Reference Franken Drawing 2-PA-912985

Figure 1. Proposed material of construction and gasket material to be installed in the TK-201 coalescer media for the DSS

Table 1. A List of Suppressors (and Their Degradation Products) Considered for NGS

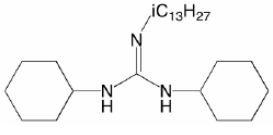
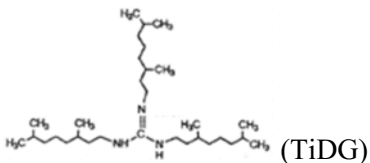
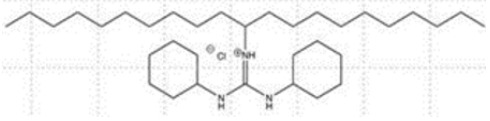
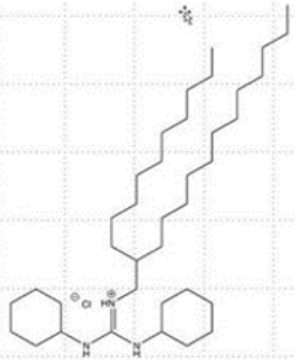
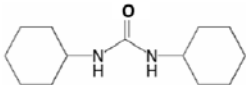
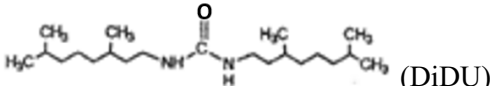
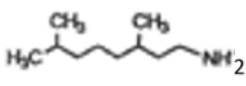

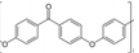
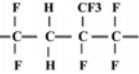

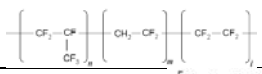
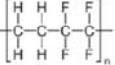

Suppressors and possible degradation products	Molecular Weight (g)	Density (g/mL)	δ_d MPa ^{1/2}	δ_p MPa ^{1/2}	δ_h MPa ^{1/2}
 <p>(DCiTG) Commercially known as LIX[®]79</p>	405	1.01	18.2	2.2	5.3
 <p>(TiDG)</p>	479	0.814	16.1	1.5	4.4
 <p>(HDCHG)</p>	517	0.971	17.9	1.7	4.6
 <p>(DTDCHG)</p>	559	0.960	17.8	1.6	4.4
 <p>(DCHU)</p>	224	1.22	21.0	4.9	6.7
 <p>(DiDU)</p>	340	0.915	11.1	2.4	4.7
 <p>(IDA)</p>	157	0.809	10.5	3.1	5.4
δ_d is the dispersion force, δ_p is the polar force, and δ_h is the hydrogen bond force (see Ref. vi)					

Table 2. Polymers Proposed for Use in SWPF for Contact with NGS

Polymer/Structure	g/mL	δ_d MPa ^{1/2}	δ_p MPa ^{1/2}	δ_h MPa ^{1/2}	R_o MPa ^{1/2}
UHMWPE 	0.97	18	0	2	2
PEEK 	1.32	18.7	7.82	7.72	2
Viton® A 	1.8	15.6	9.6	7.8	7.1
PTFE 	2.2	17.1	8.1	1.3	4.7
Viton® B 	1.85	15.6 ^s	9.6 ^s	7.8 ^s	7.7
ETFE (Tefzel®) 	1.7	17.6*	4.0*	1.6*	4*
PVDF 	1.78	17	12.1	10.2	4.1
R _o is the interaction radius of the polymer in the δ_d , δ_p , and δ_h space. *Obtained from the average of the PTFE and UHMWPE data since it is a block copolymer of the units of these two polymers. ^s Obtained from the average of the PTFE and Viton® A parameters.					

$$R_a^2 = 4(\delta_{d1} - \delta_{d2})^2 + (\delta_{p1} - \delta_{p2})^2 + (\delta_{hb1} - \delta_{hb2})^2 \quad 1)$$

$$RED = \frac{R_a}{R_o} \quad 2)$$

2.1 Calculation Results

The *RED* numbers (from Equation 2) are listed in Tables 3 through 9. Except for Tefzel® ETFE copolymer, all polymers are compatible with the suppressors based on these data. The calculation indicates that Tefzel® ETFE is expected to interact with the NGS solvent regardless of the suppressor used. This finding is consistent with the swelling and softening observed when Tefzel® ETFE was tested against NGS.ⁱⁱ The interaction is perhaps due to a larger radius of interaction (R_o) and the -CH₂-CH₂-CF₂- sequence that lowers the polarity of the polymer to the point such that total solubility occurs with the NGS solvent. Note that Tefzel® ETFE is more resistant to the pure suppressors. This behavior indicates that performing compatibility tests with pure components of a mixture to determine compatibility of a given polymer to the mixture may be misleading; rather testing should include the full solvent formulation – as well as aqueous solutions – due to potential synergistic interactions. Similarly, one can see the reverse trend as in the case of PEEK where more interaction is expected with a very polar pure component versus a mixture that dilutes that polar component.

Table 3. The Effect of TiDG on the Polymers Considered in SWPF

Polymer	3mM	10mM	15 mM	100% TiDG
Tefzel® (ETFE)	0.81	0.85	0.85	1.19
PVDF	3.38	3.40	3.40	2.98
PTFE	1.44	1.46	1.46	1.60
PEEK	4.96	5.03	5.02	4.44
VITON® B	1.32	1.33	1.33	1.14
HDPE	1.58	1.64	1.64	2.40

All suppressor concentrations in NGS solvent. Numbers >1 indicates no interaction with the polymer. The closer the number is to zero the more interaction (swelling).

Table 4. The Effect of LIX®79 on the Polymers Considered in SWPF

Polymer	3mM	10mM	15 mM	100% LIX®79
Tefzel® (ETFE)	0.85	0.85	0.84	1.06
PVDF	3.41	3.41	3.40	2.75
PTFE	1.46	1.46	1.45	1.57
PEEK	5.04	5.03	5.02	3.09
VITON® B	1.32	1.32	1.32	1.22
HDPE	1.64	1.64	1.63	1.99

All suppressor concentrations in NGS solvent. Numbers >1 indicates no interaction with the polymer. The closer the number is to zero the more interaction (swelling).

Table 5. The Effect of HDCHG on the SWPF Polymers

Polymer	3mM	10mM	15 mM	100%
Tefzel® (ETFE)	0.85	0.84	0.84	0.93
PVDF	3.41	3.40	3.40	2.96
PTFE	1.46	1.46	1.45	1.57
PEEK	5.04	5.03	5.02	3.66
VITON® B	1.32	1.32	1.32	1.27
HDPE	1.64	1.63	1.63	1.44

All suppressor concentrations in NGS solvent. Numbers >1 indicates no interaction with the polymer. The closer the number is to zero the more interaction (swelling).

Table 6. The Effect of Suppressor DTDCHG on the SWPF Polymers

Polymer	3mM	10mM	15 mM	100%
Tefzel® (ETFE)	0.85	0.84	0.84	0.96
PVDF	3.41	3.40	3.40	2.92
PTFE	1.46	1.46	1.45	1.57
PEEK	5.04	5.03	5.02	3.52
VITON® B	1.32	1.32	1.32	1.26
HDPE	1.64	1.63	1.63	1.55

All suppressor concentrations in NGS solvent. Numbers >1 indicates no interaction with the polymer. The closer the number is to zero the more interaction (swelling).

Similarly, Tefzel® ETFE appears to interact with the urea byproduct from the degradation of a dicyclohexane guanidine type of suppressor diluted in NGS solvent (see Tables 7 and 8 for numbers less than one). Again, this behavior could be driven by the effect of the NGS solvent alone. Tefzel® and Viton® A both appear to have affinity for a pure long chain aliphatic amine (see Table 9). This finding is consistent with published data from the producer of Tefzel® ETFE (Dupont) showing that dibutyl amine can plasticize the Tefzel® ETFE copolymer.^{vii} The organic amine effect on Tefzel® and Viton® is mild (based on the RED number equals one) perhaps due to the decyl group attached the amine introducing a large volume and dispersion such that this molecule appears more like a hydrocarbon than an organic base. The concentration of degradation products is expected to be small. Therefore, no significant effect on the polymers is expected.

Table 7. The Effect of the Urea byproduct from LIX®79 on the SWPF Polymers

Polymer	1000 mg/L*	100%
Tefzel® (ETFE)	0.81	2.1
PVDF	3.38	2.8
PTFE	1.44	2.1
PEEK	4.96	2.8
VITON® B	1.17	1.7
HDPE	1.58	4.5

*Urea concentration in NGS solvent. Numbers >1 indicates no interaction with the polymer

Table 8. The Effect of the Urea from TiDG on the SWPF Polymers

Polymer	1000 mg/L Urea*	100% Urea
Tefzel® (ETFE)	0.81	3.4
PVDF	3.38	4
PTFE	1.44	2.9
PEEK	4.96	8.2
VITON® B	1.17	2.0
HDPE	1.59	7.2

*Urea concentration in NGS solvent. Numbers >1 indicates no interaction with the polymer.

Table 9. The Effect of an Amine Byproduct from TiDG

Polymers	1000 mg/L Amine*	100% Amine
Tefzel® (ETFE)	0.96	1.0
PVDF	3.14	2.5
PTFE	1.50	1.4
PEEK	4.60	3.1
VITON® B	1.11	1.0
HDPE	1.90	2.5

*Amine concentration in NGS solvent. Numbers >1 indicates no interaction with the polymer.

3.0 Conclusions

A screening calculation of the potential miscibility of PEEK, UHMWPE, PVDF, Viton® B fluoroelastomer, and Tefzel® ETFE copolymer in different suppressors that include TiDG, dicyclohexaneisodecyl guanidine, and two other dicyclohexane guanidine related suppressors was performed. It was found that Tefzel® ETFE copolymer may mildly interact with the NGS regardless of the suppressor used. Based on the screening calculation, all other identified polymers for SWPF were compatible with the NGS containing the four different suppressors. There is evidence of chemical affinity between Tefzel® and Viton® B with aliphatic amines (a degradation by-product from the TiDG). Amines and other bases are known to interact with Viton FKM fluoroelastomers. In addition, the calculation suggests that drawing miscibility conclusions from exposing polymers to a single pure component may not fully consider the overall effects of a mixture.

Based on this screening calculation, SRNL recommends testing Tefzel® ETFE and Viton® B materials for compatibility with NGS solvent containing different formulations of the four suppressors listed in this report. Specifically, the same compounds and grades of the polymers used in the facility should be evaluated if such details are known. Minor variations in processing and compounding may affect interactions. Different grade of polymers should also be investigated (with similar chemical resistance and mechanical response) that may include silicon based polymers (such as fluorosilicone, methyl silicone, methyl vinyl silicone, and possibly methyl phenyl silicone). Miscibility studies should last at least 24 days to capture kinetics and steady state loading. In addition, thermal studies should include at least one compatibility at 35°C. Finally, gamma irradiation tests should be conducted on Viton B and Kalrez at two different dose rates (rad/h) for two different total doses (for example 10 and 15 Mrad) under humid air. Irradiated polymers should be measured for samples dimensions and hardness (ASTM D2240).

4.0 References

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- ⁱ D. D. Larsen and M A. Pelicone, “MCU Task Requirements and Criteria Design Constraint Verification”, LWO-SPT-2007-00149, July 2007.
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