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A Review of the Literature on the Radiolytic Stability of the Next-Generation Solvent

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EXECUTIVE SUMMARY

The Original Caustic-Side Solvent Extraction (CSSX) solvent (based on the BOBCalixC6 extractant) currently in use at the Salt Waste Processing Facility (SWPF) is set to be replaced by the Next-Generation Solvent (NGS, based on the MaxCalix extractant). Current SWPF flammability safety controls use G-values for volatile gases obtained from radiolysis studies with the Original CSSX solvent. A review of past irradiation tests of both solvent systems was performed to assess the radiolytic stability of NGS with respect to flammable gas generation from radiolysis.

NGS shares the same diluent and modifier as the Original CSSX solvent and appears to have similar radiolytic stability. In an external gamma irradiation study, NGS was stable up to 1 Mrad (equivalent to a thirteen-year dose at SWPF). The new NGS guanidine suppressor, TiDG, was not part of that study, but it is a minor component of the solvent and structurally similar to the former guanidine suppressor (DCiTG). TiDG was also deployed at Modular CSSX Unit (MCU) starting in 2013 as part of the NGS flowsheet. Additionally, the improved extractant of NGS, MaxCalix, has the same structural calixarene backbone as the BOBCalixC6 extractant and would be expected to have similar radiolytic stability. No radiolysis experiments have been performed with NGS to determine volatile gas generation yields. However, the bulk of NGS is the same as the Original CSSX solvent and the new components are structurally similar and present in a small quantity.

There is still a low risk that NGS would not be bounded by the current G-values for flammable gases, particularly hydrogen gas, obtained from studies with the Original CSSX solvent. Without experimental radiolytic G-value measurements with NGS, a bounding (worst case) hydrogen gas G-value would be 5 molecules/100 eV, which is the measured G-value for single chain alkanes of similar length to Isopar® L. However, this is considered an overly conservative estimate and it is believed that the actual hydrogen G-value for NGS would be similar to the Original CSSX solvent. The modifier is believed to lower the hydrogen G-value of the solvent due to its aromaticity, but is present at a lower concentration in NGS. The extractant concentration in NGS is increased, however, leading to an increase in the total aromaticity when compared to the Original CSSX solvent. Currently, a conservative hydrogen G-value of 1.944 molecules/100 eV is used at SWPF for the Original CSSX solvent, which should bound the NGS hydrogen G-value. A confirmatory test to determine the hydrogen G-value for NGS is encouraged.

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LIST OF ABBREVIATIONS

CLFL	Composite Lower Flammability Limit
Cs-7SB	1-(2,2,3,3-tetrafluoropropoxy)-3-(4-sec-butylphenoxy)-2-propanol
CSSX	Caustic-Side Solvent Extraction
CSTF	Concentration, Storage, and Transfer Facilities
DCiTG	1,3-dicyclohexyl-2-(isotridecyl)guanidine
DWPF	Defense Waste Processing Facility
ESI-MS	Electrospray Ionization Mass Spectrometry
ESS	Extraction, Scrub, and Strip
FTIR	Fourier-Transform Infrared Spectroscopy
GC	Gas Chromatography
INL	Idaho National Laboratory
MCU	Modular CSSX Unit
NGS	Next-Generation Solvent
ORNL	Oak Ridge National Laboratory
PISA	Potential Inadequacy in the Safety Analysis
SBP	4-sec-butylphenol
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site
SWPF	Salt Waste Processing Facility
TiDG	N,N',N''-Tris(3,7-dimethyloctyl)guanidine
TOA	tri-n-octylamine
TTR	Technical Task Request

1.0 Introduction

Savannah River Remediation (SRR) has identified technical gaps in the implementation of the Next-Generation Solvent (NGS) at SWPF.[1] Savannah River National Laboratory (SRNL) has been requested by SRR via a Technical Task Request (TTR) to perform a literature review on radiolytic G-values for NGS to address flammability concerns.[2] SWPF currently uses G-values obtained from gas generation radiolysis studies (with the Original CSSX solvent) in setting air purge flowrates and time to the composite lower flammability limit (CLFL) for process vessels.[3,4] The Original CSSX solvent consists of the following:

- 0.007 M extractant BOBCalixC6 (Calix[4]arene-bis(t-octylbenzo-crown-6)),
- 0.75 M modifier Cs-7SB (1-(2,2,3,3-tetrafluoropropoxy)-3-(4-sec-butylphenoxy)-2-propanol),
- 0.003 M suppressor TOA (Tri-n-octylamine), and
- Isopar® L balance.

The Original CSSX solvent is currently in use at SWPF, but it is set to be replaced by NGS, which uses an improved extractant and guanidine suppressor, TiDG, and consists of the following:

- 0.05 M extractant MaxCalix (1,3-alt-25,27-bis(3,7-dimethyloctyl-1-oxy)calix[4]arenebenzo-crown-6),
- 0.65 M modifier Cs-7SB (1-(2,2,3,3-tetrafluoropropoxy)-3-(4-sec-butylphenoxy)-2-propanol),
- 0.003 M suppressor TiDG (N,N′,N″-Tris(3,7-dimethyloctyl)guanidine), and
- Isopar® L balance.

The modifier concentration has been reduced, while the increased extractant concentration allows for greater cesium extraction. Starting in 2013, NGS replaced the Original CSSX solvent at the Modular CSSX Unit (MCU). This NGS formulation used a Cs-7SB concentration of 0.5 M, compared to the 0.65 M Cs-7SB concentration that will be used at SWPF.

SRNL previously performed a literature review of the radiation stability of NGS in 2011 prior to its implementation in MCU to address flammability concerns downstream during Defense Waste Processing Facility (DWPF) processing.[5] Additionally, a more recent 2017 flammable gas generation mechanism report reviewed previous radiolysis gas generation testing of the CSSX solvent.[6]

2.0 Discussion

2.1 Original CSSX Solvent Radiation Stability and Gas Generation Tests

A considerable amount of irradiation tests using both external and internal gamma radiation have been performed with the Original CSSX solvent, reviewing solvent stability and extraction, scrub and strip (ESS) test performance post-irradiation.[7,8,9] These irradiation studies found negligible solvent degradation and no significant impact on solvent performance. Solvent degradation in these studies was mainly limited to the suppressor TOA, and to a lesser extent, the modifier Cs-7SB was also found to degrade to 4-sec-butylphenol (SBP).

Four irradiation tests investigating gas generation have been performed. In a 2007 SRNL study, irradiation tests on the Original CSSX solvent were performed with a cobalt-60 gamma source at 50°C.[10] In these tests, 15 mL of the Original CSSX solvent was washed with 0.01 M NaOH and placed in stainless steel vessels and irradiated with 7.11 Mrad dose (MCU 5-year equivalent dose). The headspace of the samples was then analyzed post-irradiation using gas chromatography (GC). From these tests, an average hydrogen generation rate of 8 x 10^{-8} ft^3 h^{-1} gal^{-1} was found, with a corresponding hydrogen G-value of 1.7

molecules/100 eV. In a follow-up study at SRNL,[11] an additional gas generation study was conducted using irradiated Original CSSX solvent solution in contact with either water, simulated salt solution, or 0.05 M nitric acid. After contact, the solvent was placed in stainless steel sample vessels and irradiated (4 Mrad and 32 Mrad) using a cobalt-60 source at ambient temperature and 50°C. The headspace of the vessels was then sampled using a sample bulb and analyzed by GC. At 50°C, a hydrogen G-value of 0.85 – 1.13 (molecules/100 eV) was measured. In addition, no impact was found on hydrogen generation by the entrained aqueous phase (water, salt simulant, or 0.05 M nitric acid) of the tests.

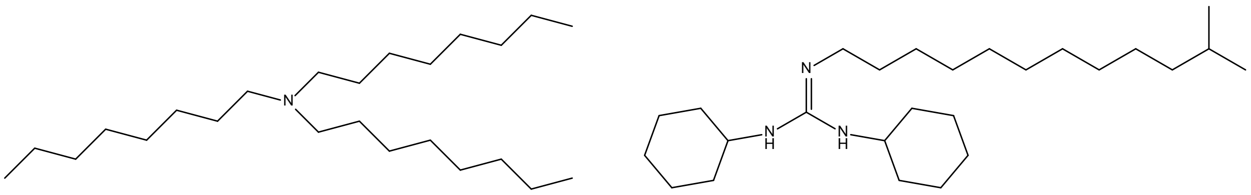
The most significant gas generation radiolysis test for the Original CSSX solvent was carried out at Oak Ridge National Laboratory (ORNL) in 2007.[12] Two radiolysis tests were performed, with the first being a baseline radiolysis test in which 1 L of water-equilibrated Original CSSX solvent was administered a 32.2 Mrad dose of external gamma radiation from a cobalt-60 source. Any volatile species formed in the headspace were periodically sampled and measured using Fourier-Transform Infrared Spectroscopy (FTIR). The G-values found for hydrogen and several other gas species are given in Table 2-1. An additional seven "qualitative" G-values were given for propene, isobutene, methylpropene, methylbutane, pentene, tetrafluoropropane, and tetrafluoroethane due to a lack of calibration data. In the second radiolysis test, sealed ampules of 1 mL of Original CSSX solvent were subjected to various doses of gamma radiation at various temperatures, ~17-43°C. From this test, the concentrations for the various gases analyzed were comparable at all conditions (dose and temperature) except for the hydrogen concentration in the 32.2 Mrad test, which showed a 50% increase in concentration at ~43°C compared to lower temperatures tested. It should be noted that SWPF flammability safety calculations currently take that into account, and additionally increase the hydrogen G-value by 20% to account for any instrument uncertainty; this gives a conservative hydrogen G-value of 1.944 molecules/100 eV, which is an 80% increase over the measured value of 1.08 molecules/100 eV. A follow-up to this study was performed at Idaho National Laboratory (INL).[13] This study just examined the radiolytic hydrogen gas yield over the temperature range 18-48°C for the Original CSSX solvent. In this study, 10 mL of water-equilibrated Original CSSX solvent was sealed in 25-mL stainless steel sample containers and irradiated, up to ~40 Mrad absorbed dose, using an external cobalt-60 gamma source. The headspace was then sampled post-irradiation by GC. In comparison to the ORNL study, no measurable effect on the hydrogen gas yield (0.93 molecules/100 eV) was found over the entire temperature change (18-48°C).

Table 2-1. Measured G-values for Gases Generated from the Irradiation of the Original CSSX Solvent[12]

	G-value (molecules/100 eV)
Hydrogen	1.08
Methane	0.261
Ethane	0.0341
Propane	0.0283
n-butane	0.00378
n-pentane	0.00115
n-hexane	0.00172
CO	0.0591
CO_2	0.0619

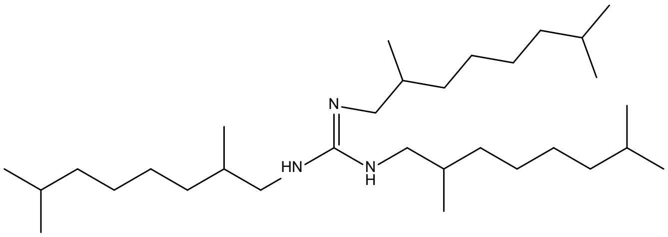
2.2 NGS Radiation Stability

ORNL has performed a radiation stability study with NGS.[14] It should be noted that this testing was done with the NGS formulation that uses the first-generation guanidine suppressor DCiTG (1,3-dicyclohexyl-2-(isotridecyl)guanidine) instead of the current suppressor TiDG. Figure 2-1 contains a structural comparison of the two guanidine suppressors and TOA (the Original CSSX solvent suppressor). All of the suppressors are structurally similar in that they all contain alkyl groups connected to a nitrogen center. In the study, NGS was irradiated using an external cobalt-60 gamma source at up to 5 Mrad. During irradiation, the solvent was in contact with either a salt simulant, 0.025 M sodium hydroxide scrub solution, or 0.01 M boric acid strip solution to mimic the current CSSX process. Following irradiation, an ESS test was performed to check solvent performance. Discoloration was noted following irradiation of NGS in contact with both caustic solutions (salt simulant and scrub solution). ESS was not significantly impacted by irradiation up to 1 Mrad, which is thirteen times the estimated annual absorbed dose for SWPF of 0.075 Mrad. Above 1 Mrad, the concentration of the Cs-7SB degradation product SBP increases while the concentration of the suppressor DCiTG is reduced from irradiation, both of which are suggested to have a negative impact on solvent stripping. From Electrospray Ionization Mass Spectrometry (ESI-MS) data, only a trace degradation product of MaxCalix with one alkyl tail missing was identified. Like the Original CSSX solvent, the suppressor and modifier seem to be the most susceptible to degradation. SRNL has also performed real-waste ESS testing with the NGS formulation that contains the suppressor DCiTG and no solvent degradation was seen after testing.[15] The suppressor TiDG has been used at MCU beginning in 2013, as part of the NGS flowsheet. Its degradation in MCU has been studied by SRNL.[16] A method was derived to predict the TiDG depletion in MCU using data derived from process samples, with the assumption that the TiDG depletion is mainly due to chemical degradation and partitioning into the aqueous phases. SRNL is currently performing experiments to determine the partitioning ratio of these TiDG degradation products.



TOA

DCiTG



TiDG

Figure 2-1. Structure Comparison of the Original CSSX Solvent Suppressor TOA, the First-Generation Guanidine Suppressor DCiTG, and the Second-Generation Guanidine Suppressor TiDG (the Current NGS Suppressor)

For straight chain alkanes, the radiolytic hydrogen gas G-values are typically about 5 molecules/100 eV absorbed dose[17]; in comparison, the hydrogen G-value measured for the Original CSSX solvent was ~1 molecule/100 eV. If we were to assume that the radiolytic G-values obtained are more representative of the bulk species (Isopar® L, which is a C_{11}-C_{13} isoparaffin), then we would expect a higher G-value for hydrogen gas for the Original CSSX solvent. Aromatics have been shown to decrease the radiolytic hydrogen gas yield in mixtures with aliphatic hydrocarbons.[18] This reduction is due to the increased stability of aromatic compounds to radiation as well as the efficient radical scavenger abilities of unsaturated compounds.[19] It can be proposed that the modifier Cs-7SB acts as a radical scavenger in the solvent, decreasing the radiolytic G-value for hydrogen. The modifier concentration is reduced from 0.75 to 0.65 M in NGS. This reduction has the potential to lead to slightly higher hydrogen gas G-values for NGS in comparison to that of the Original CSSX solvent. It should be noted that NGS was used at MCU starting in 2013, with a Cs-7SB concentration of 0.5 M, which would have had a higher hydrogen G-value (following the logic above) than the NGS formulation that will be used at SWPF. Additionally, the extractants MaxCalix and BOBCalixC6 have multiple aromatic phenyl rings, which should have a similar effect on the radiolytic G-value as the modifier. When comparing the two CSSX solvent systems on a molar % aromaticity, NGS actually has a higher mol % aromaticity than the Original CSSX solvent (21.1 compared to 18.1 mol %) due to an extractant concentration that is 7 times higher than the Original CSSX solvent. In could then be inferred from the molar aromaticity that NGS would actually have a similar or smaller radiolytic G-value for hydrogen in comparison to the Original CSSX solvent. It should be noted that this relies on the assumption that the multiple aromatic rings on MaxCalix each have an equivalent effect on the radiolytic G-value as the aromatic ring on Cs-7SB. This appears to be a fair assumption to make; for example, the reaction rate with hydrogen radicals in aqueous solutions for biphenyl is 5.0 x 10^{-09} L/mol·s compared to 1.4 x 10^{-09} L/mol·s for benzene.[20] LaVerne et al. had plotted the effect of benzene mol % on the radiolytic hydrogen G-value, $G(H_2)$, in cyclohexane/benzene mixtures. That graph is given in Figure 2-2 along with the overlay of the $G(H_2)$ value range for the Original CSSX Solvent, the current SWPF $G(H_2)$ value used for the solvent, and a possible $G(H_2)$ value range for NGS if we follow the above logic.[18]

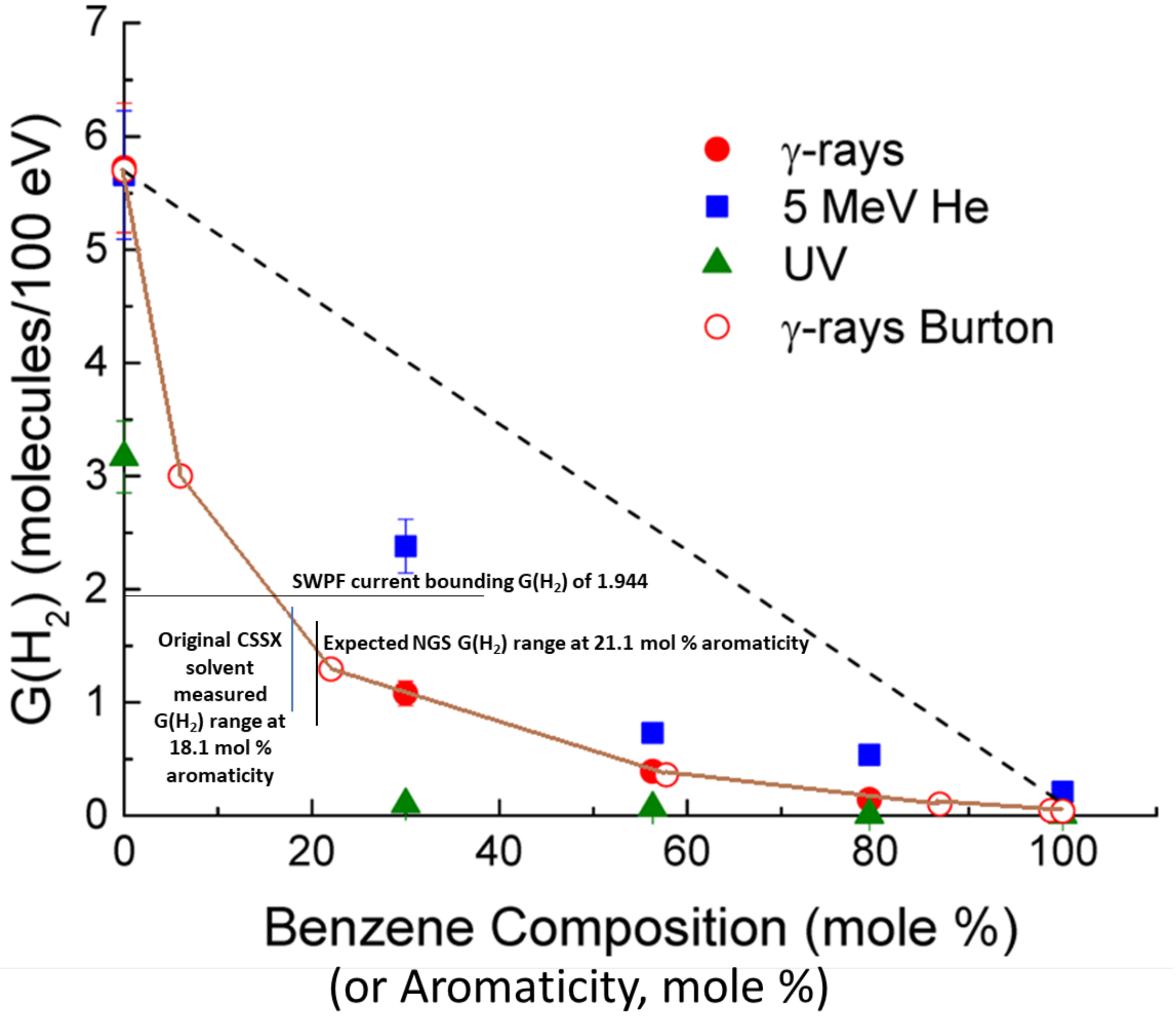


Figure 2-2. Radiolytic Hydrogen Yield as a Function of Molar Percent Benzene in Cyclohexane/Benzene Mixtures.[18] Dashed Line Shows Expected Values Following the Mixture Law. Original Graph is Overlayed with Original CSSX Solvent $G(H_2)$ Range at a Molar Percent aromaticity of 18.1%, Possible NGS $G(H_2)$ Range at a Molar Percent Aromaticity of 21.1%, and Current Bounding $G(H_2)$ Value Used at SWPF.

3.0 Conclusions

There have been numerous radiolytic studies with the Original CSSX solvent, with a few of these focusing on gas generation. In its only irradiation study, NGS was stable up to 1 Mrad (equivalent to a 13-year dose at SWPF). This test was focused on solvent stability and ESS performance, and gas generation was not measured. While there is an inherent risk with a lack of long-term radiolytic stability testing with the TiDG suppressor, it is a minor component of NGS. Additionally, NGS with TiDG had been in use at MCU for several years, and base-catalyzed hydrolysis of TiDG appears to be the main degradation route compared to any radiolytic degradation.

It should be noted that the concentration of MaxCalix in NGS is more than seven times the concentration of BOBCalixC6 in the Original CSSX solvent. However, the extractants have the same structural backbone calixarene, and MaxCalix appears to be fairly stable to radiolysis, based on radiolytic degradation testing and its use at MCU.

Isopar® L and Cs-7SB comprise the bulk of both CSSX solvent systems, and flammable gas produced from the radiolysis of the Original CSSX solvent is expected to be more of a product of the major components of the solvent (specifically the Isopar® L diluent) than of the minor components. There is still a low risk that NGS would not be bounded by the current G-values for flammable gases, particularly hydrogen gas, obtained from studies with the Original CSSX solvent. Without experimental radiolytic G-value measurements with NGS, a bounding (worst case) hydrogen gas G-value would be 5 molecules/100 eV, which is the measured G-value for single chain alkanes of similar length to Isopar® L. However, this is considered an overly conservative estimate and it is believed that the actual hydrogen G-value for NGS would be similar to the Original CSSX solvent. The modifier is believed to lower the hydrogen G-value of the solvent due to its aromaticity, but is present at a lower concentration in NGS. The extractant concentration in NGS is increased, however, leading to an increase in the total aromaticity when compared to the Original CSSX solvent. Currently, a conservative hydrogen G-value of 1.944 molecules/100 eV is used at SWPF for the Original CSSX solvent, which should bound the NGS hydrogen G-value. A confirmatory test to determine the hydrogen G-value for NGS is encouraged.

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