Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Tris(isodecyl)guanidine Degradation in the MCU System

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June 2016

SRNL-STI-2015-00372
Revision 0

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Printed in the United States of America

Prepared for
U.S. Department of Energy

Keywords: *MCU, NGS, ISDP*

Retention: *Permanent*

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Savannah River National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

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EXECUTIVE SUMMARY

The current solvent blend consists of four components; an extractant, the modifier, a suppressor, and the diluent. Of the four components, only the suppressor – tris(isodecyl)guanidine (TiDG) has exhibited an appreciable depletion rate during facility operations.[∇]

Using data derived from MCU process samples, SRNL derived a method to predict the TiDG depletion based upon time and volume of feed processed. With the current data set, the following formula can estimate the TiDG concentrations after processing:

$$A_t = [A_0 \times \exp(-0.000900 \times t)] - [5.39\text{E-}06 \times \text{volume}]$$

where A_t is the TiDG concentration (mM) at time t (days), A_0 is the TiDG concentration (mM) at time = 0, and volume is the amount of salt solution processed in gallons from time 0 to time t. The ability to use this formula as a predictive tool is limited due to the number of data points obtained for this scope of work. As such, this formula should not be used to precisely predict future TiDG concentrations.

[∇] The more technically correct name for this compound is *N*,*N*',*N*"-tris(3,7-dimethyloctyl)guanidine, and is typically called "TiDG".

LIST OF ABBREVIATIONS

MCU	Modular Caustic-Side Solvent Extraction Unit
NGS	Next Generation Solvent
NMR	Nuclear Magnetic Resonance
ORNL	Oak Ridge National Laboratory
RSD	relative standard deviation
SHT	Solvent Hold Tank
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TiDG	N,N’,N”-tris (3,7-dimethyloctyl) guanidine, tris(isodecyl)guanidine
TOA	trioctylamine

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1.0 Introduction

In late 2013, Modular Caustic-Side Solvent Extraction Unit (MCU) switched to the Next Generation Solvent (NGS) flow sheet. Facility personnel implemented the switch by adding a non-radioactive, NGS "cocktail" containing the new extractant (MaxCalix), modifier (Cs-7SB), Isopar-L ™, and a new suppressor (TiDG) to the Solvent Hold Tank (SHT) heel. SHT samples are regularly sent to Savannah River National Laboratory (SRNL) to examine solvent composition changes over time.[1] Analysis of these samples shows that, of the six chemicals in the resulting blend solvent, only three of them are known to deplete at a rate faster than that the whole solvent. Isopar L ™ is known to suffer evaporative losses due to its relatively high volatility. Trioctylamine (TOA) is known to more rapidly deplete and has been previously studied.[2] Tris(isodecyl)guanidine (TiDG) is also more rapidly depleting than the bulk solvent as evidenced by recent solvent sample results.

Isopar L ™ losses are routinely replaced by semi-routine additions. Losses of TOA are not important as it plays no role in the function of the blend solvent, nor are there any downstream impacts.[2] The TiDG losses must be carefully monitored as the material is made by a single provider and has an appreciable lead time for procurement.

Therefore, it is desirable to be able to predict the rate of loss of the TiDG to forecast demand, and minimize the chances of an interruption in supply of the material. Oak Ridge National Laboratory (ORNL) examined partitioning ratios of TiDG as well as the effects of temperature on the degradation of TiDG,[3,4] while this document employs a more holistic review of operational data.

2.0 Experimental Procedure

2.1 Routine SHT Samples

SHT samples are received from MCU on a nominal monthly basis, since late 2013, when the use of the blend solvent started. These samples are subject to an extensive battery of analyses. To analyze for the TiDG content, both a titration method and a nuclear magnetic resonance (NMR) method are used. These methods use an internal standard of laboratory prepared blend solvent as a point of comparison.

2.2 Assumptions

The chemical makeup of the aqueous feed has been approximately the same over the duration of use of the blended solvent; the variations in the chemical makeup for Salt Batches 7A, 7B, 8A and 8B are considered to be not important. The target composition of the solvent remained constant over the time period of samples for all components except TOA and BOBCalixC6 (calix[4] arene-bis-(tert-octylbenzo)-crown-6), each of which are allowed to slowly deplete. The relative minor variations in concentration of other solvent components are assumed to not influence the depletion or degradation rates of TiDG.[5]

The blended solvent has been used for processing of salt batches 7A, 7B, 8A, and 8B. While batches 7A and 7B possessed a cesium activity of 0.192-3 Ci/gal, [6] this increased to 0.432 Ci/gal for Salt Batch 8A and 0.742 Ci/gal for Salt Batch 8B.[7] There are too few data points for salt batches 8A and 8B to discern a difference in the degradation rates and this estimate assumes that the change in the cesium activity does not alter the degradation rates to a noticeable degree at the radiation rates experienced at MCU.

Previous work from ORNL has studied the effects of radiation exposure on solvent performance.[8] This work shows that serious degradation in solvent performance (implied to be due to loss of the guanidine) does not occur until doses are received that are orders of magnitude greater than the received dose at MCU.[⊕]

At this time, no attempt is made to include the temperature dependence of the guanidine degradation. Prior studies examined the thermal degradation of TiDG and could be used to incorporate a temperature dependency functionality for degradation.[3,8] However, the facility operating temperature remained within tight control bands during the period of collection for these samples. The solvent spends most of its time at ambient conditions (~25 °C). While the solvent spends a small portion of its life at higher temperatures (33±3 °C) in the strip contactors, the majority of its lifetime is spent either at 23±3 °C (extraction and scrub contactors) or at ambient temperature in the SHT. For simplicity, this effort assumes thermal degradation rates for TiDG remained relatively constant during the operations.

The ratio of aqueous to organic phases within the operating banks of the contactors remained relatively constant at set points of 4 aqueous (A) to 1 organic (O) (by volume) in the extraction contactors, and 1A to 3.75O in the scrub and strip contactors.

Prior testing [3] shows an appreciable partitioning ratio (>1000) for TiDG from the solvent to the boric acid in the strip solution.[Σ] Partitioning to the strip acid is presumably much larger than to the other aqueous streams. For simplicity, this effort does not include a term for the aqueous to organic phase ratio during operations.

Information such as volume of processed feed, or volume in the solvent hold tank was provided by the Savannah River Remediation, LLC (SRR) customer.

Therefore, this document proposes the depletion rate of the TiDG is a function of time and the amount of processed feed.

[⊕] While the guanidine used in this study was not TiDG, it was structurally similar and expected to have the same degree of radiation sensitivity as TiDG. Furthermore, while the study did not examine the specific radiation decay of TiDG alone, the study implies that radiation exposure leads to lipophilic anion generation and loss of guanidine, which in turn leads to solvent performance degradation.

[Σ] The partitioning ratio is defined as the guanidine concentration in the organic phase divided by the guanidine concentration in the aqueous phase.

2.3 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7 2.60. For SRNL documents, the extent and type of review using the SRNL Technical Report Design Checklist is outlined in WSRC-IM-2002-00011, Rev. 2.[9]

3.0 Results and Discussion

3.1 Time-Based Degradation of TiDG•HCl Stored Standards

As-received from the vendor, the TiDG exists as the hydrochloride salt, TiDG•HCl. Solvent prepared in lab, or prepared for MCU operations uses this material. Once the TiDG•HCl contacts a strong base (such as the caustic feed), the TiDG•HCl converts into the "freebase" form. Previous ORNL work indicates that while the protonated (i.e., the TiDG•HCl material as it is initially added to the system) guanidinium is relatively stable; contact with caustic media accelerates degradation.[4] This is initially due to the deprotonation to the neutral freebase form, which in turn is more likely to undergo chemical reactions. In order to confirm this SRNL examined previously prepared standards to the TiDG•HCl material that had been in storage for some time.

It is important to note that the analytical method does not analyze TiDG•HCl itself. The analysis detects, or titrates, the freebase form. The measurement protocol requires the MCU sample be converted into the freebase form first.[10] As part of the titrimetric determination of the MCU samples, standards are prepared and analyzed in parallel. These standards are prepared in the TiDG•HCl form of the guanidine, and small portions are analyzed during titrations. The same standards are used over varying periods of time, and so the time-base degradation of the TiDG•HCl can be determined by examining the titration results of the standards.

Over time, SRNL used multiple standards. Records from three of these standards were reviewed. The first standard was used from October 2013 to April 2014, the second from June 2014 to November 2014, and the third from December 2015 to February 2015. The standards were not washed or contacted with caustic which would convert the guanidine into the freebase form. The time from the date of preparation was noted as well as the resulting titration result. See Table 1. The "Time" is the number of days elapsed since preparation of the standard, while the "Measurement" is in mM. The analytical uncertainty for single measurements is 10% (1-sigma). While there was no active temperature control of the samples, the temperature in the lab units where the samples were stored is typically 23±3 ˚C, and the samples were kept in sealed glass vials.

For each standard, the values for the time = 0 standard (as prepared gravimetrically) can be compared for the values at later times. In this way, it is determined that the average of the samples is 98.7% of the values as-prepared (well within the 10% analytical uncertainty). Therefore, we can say that over periods of 200+ days, SRNL cannot discern any evidence of degradation.

Table 1. Results from Titrations of Standards

Standard	Time (days)	Measurement (mM)	% of Prepared
A	0	3.02	-
A	4	3.17	105%
A	75	3.26	108%
A	102	3.17	105%
A	126	2.91	96.2%
A	146	2.91	96.2%
A	193	3.01	99.8%
A	215	3.07	102%
B	0	3.01	-
B	23	3.05	102%
B	84	2.95	97.9%
B	119	2.81	93.4%
B	141	2.83	93.9%
B	159	2.97	98.7%
B	194	2.74	91.0%
C	0	2.97	-
C	1	2.95	99.3%
C	30	2.80	94.4%
C	51	2.87	96.7%
Average of % As-Prepared			98.7%

The analytical uncertainty for the TiDG titrations is 10%.

3.2 Time-Based Degradation of the Archived (i.e., Aged after Analysis) TiDG Standards

After analysis, the samples of standards used for quality control are archived and retained for a period while personnel develop the reports and close with customers on the findings. This study, at later dates, repeated the analysis of the "used" standards via the titration method to determine the remaining concentration of the TiDG. This data is compiled in Table 2. The set of archived "used" standards each give a single data point at varying duration of storage. As each of these standards was prepared in an identical fashion, stored in the same conditions and analyzed by the same instrument, this analysis treats the series of standards as if it were from a single source, generating a single decay curve.

Once the points on the decay curve are known, it is necessary to assume a single rate law decay order. The most likely candidates are zero, first, and second order decay. As zero order decay is chemically unlikely[∇], the choice is between first and second order decay. Previous ORNL work assumes that the TiDG decay is first order.[4] To determine which rate law was most appropriate, SRNL fitted the titration data to both first and second order graphs. ln[A] (first order) and 1/[A] (second order) were calculated (Table 2).

A_0 is the TiDG concentration (in mM) of the freshly prepared solvent standard. "A" is defined as the concentration (in mM) of TiDG at the time of analysis.

Table 2. Results from Re-Titrations of TiDG Standards

Time (days)	**A_0 (mM)**	**A (mM)**	**ln[A]**	**1/[A]**
0	2.837	2.837	1.0429	0.3524
73	2.735	2.574	0.9454	0.3885
82	3.00	2.726	1.0028	0.3669
108	2.967	2.405	0.8776	0.4158
299	3.011	2.219	0.7970	0.4507
350	2.901	2.095	0.7393	0.4774
367	2.905	2.022	0.7041	0.4946
394	3.092	1.670	0.5129	0.5987
418	3.256	2.068	0.7267	0.4835

The data in Table 2 can be graphed in both first and second order decay formats (Figures 1 and 2). The better fit of the data is provided by the first order plot, although only marginally so. Therefore, this report assumes that the time based decay is first order.

In summary, a solution of blend solvent containing TiDG in the freebase form, at ~room temperature, will decay in a first order fashion. The slope of the line (-0.000900) is the rate order constant in mM/day.

$$A_t = A_0 \times \exp(-0.000900 \times t)$$

The time=394 days data point was tested to determine whether or not it could be removed from the data set as a statistical flier. However, when analyzing the Studentized Residuals, the data point in question was just within the 95% confidence region, so we could not dismiss it.

[∇] For zero-order reactions, the reaction rate is independent of the concentration of a reactant, so that changing its concentration has no effect on the speed of the reaction.

Figure 1. Plot of Data for First Order Decay

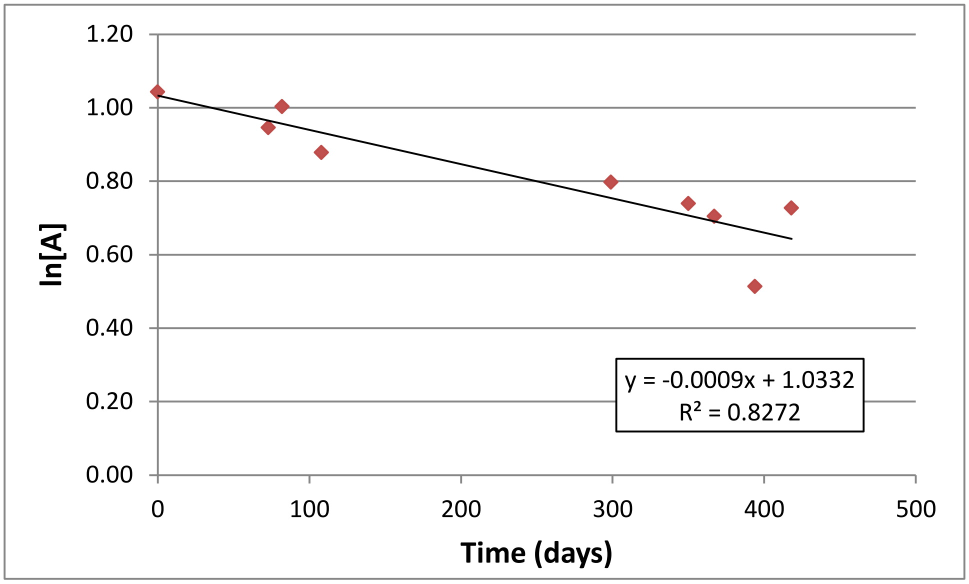
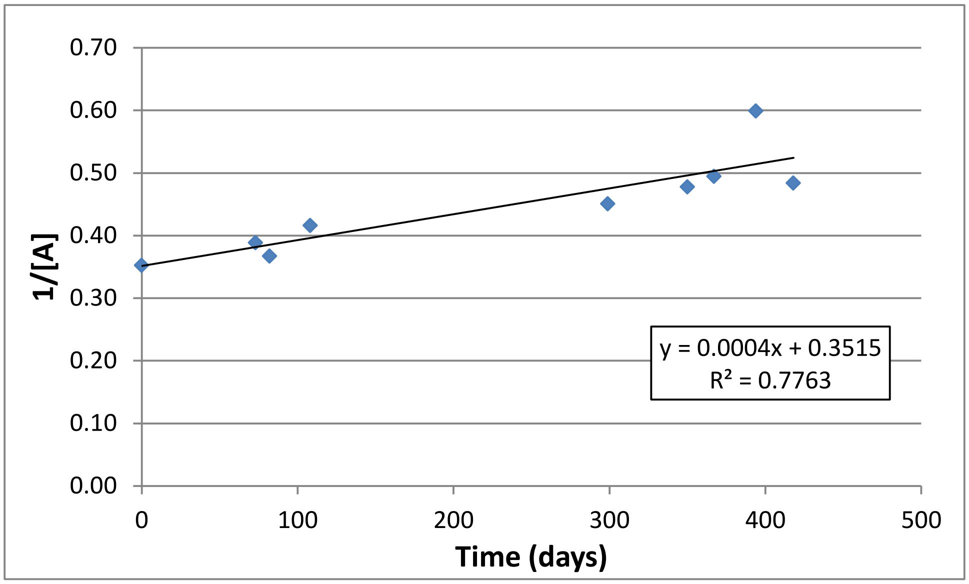


Figure 2. Plot of Data for Second Order Decay



3.3 Consideration of Data from MCU Samples

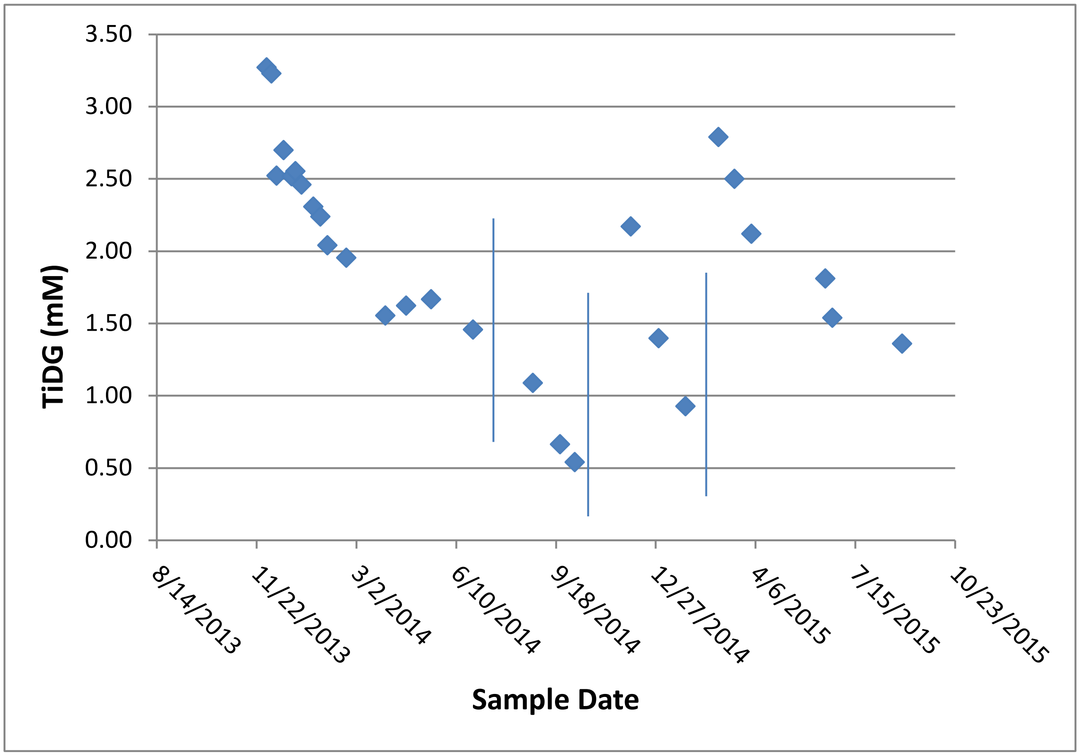
It is known that the depletion of the TiDG from the solvent is not just a process of chemical or radiological degradation over time. Partitioning losses to the aqueous phases, and especially to the strip effluent, also play a function. Partitioning losses should be proportional to the amount of feed processed.

If the loss rates from time alone are known, then it is possible to estimate losses due to processing. By tabulating the MCU sample data, the total losses can be determined. If the total losses are due to time and processing volume, then by subtracting the time based losses (estimated from work in previous sections), then it is possible to derive a loss rate associated only with processing volumes.

SRNL tabulated all the results from the SHT samples over the time period that the blend solvent was in use (12/2/2013 to 8/31/2015). See Appendix A for a tabulation of the sample results used. In addition, further information was collected, such as the date of the sample, the solvent volume in the SHT, the time since the solvent was trimmed, and the volume of processed feed. With all this data, SRNL can estimate the losses due to processing feed.

Although a large number of data points have been collected, the data set as a whole is split up into four sub-sets. This is due to additions of TiDG to replenish the solvent to the nominal operating range. While it is possible to account for the Isopar L ™ additions by normalizing the volume in the SHT, the TiDG that is occasionally added requires the data set to be analyzed in separate sets. For each set we assign the time=0 data points as the very next data point after an addition of TiDG. For example, a TiDG measurement for a sample from 1/26/2015 is available. On 2/22/2015, a TiDG•HCl trim, or addition, of 838 grams occurred. The next analysis was from a sample on 2/25/2015. We treat the 2/25/2015 sample as the new time=0 data point. See Figure 3.

Figure 3. Volume Normalized TiDG Concentrations



3.4 Example of Methodology

To help illustrate the methodology, an example is given. See Table 3. At MCU, a TiDG trim was added on 2/22/2015. This means the very next sample data point (sample MCU-15-389/390) is treated as the new time=0 data point. The TiDG measurement for that sample was 2.844 mM, with the reported SHT volume being 212 gallons. All further TiDG results in this series were volume normalized to this time=0 volume.

Table 3. Example of Methodology

Sample	Result (mM)	Result, Norm. (mM)	Sample Date	Cumul. Time (d)	Predicted Conc. (mM)	Cum. Proc. Vol. (gal)	Loss due to Proc. (mM)	Loss/Vol. (mM/gal)
Feb-389/390	2.844	2.84	2/25/2015	0	2.844	[redacted]	[redacted]	[redacted]
Feb-439/440/441	2.842	2.79	2/28/2015	3	2.836	28617	0.048	1.68E-06
March -556/557/558	2.356	2.50	3/16/2015	19	2.796	126812	0.295	2.33E-06
April-661/662/663	1.921	2.12	4/2/2015	36	2.753	206758	0.633	3.06E-06
June-710/711/712	1.64	1.81	6/15/2015	110	2.58	216535	0.766	3.54E-06
June-750/751/752	1.43	1.54	6/22/2015	117	2.56	239075	1.022	4.27E-06
August-802/3/4/5/6/7	1.34	1.36	8/31/2015	187	2.4	239262	1.045	3.81E-06

Later samples have a cumulative time (in days) set against the time=0 data point. From the cumulative time and calculated first order rate constant, the predicted concentration is calculated. This calculated value is based solely on time-based degradation. Note that in all cases, the predicted concentration is greater than what was actually measured, implying that there are factors other than time that influence the TiDG degradation. Once the difference between the measured and predicted TiDG concentration is known, this value is assigned to partitioning losses due to processing. Processing losses should be a function of the volume of salt solution processed and this relationship is expected to be linear. The losses that are assigned to processing ("Loss due to Proc" in Table 3) are divided by the cumulative amount of salt solution processed ("Cum Proc Vol" in Table 3) to generate a mM loss per gallon processed ("Loss/vol" in Table 3).

The loss per gallon processed is derived for each sample data point, except for the time=0 points, where the volume processed is zero. After the entire data set was completed, all of the "loss per gallon processed" terms were averaged into a single value; 5.39E-06 mM/gallon. This value is consistent with the ORNL partitioning coefficients into strip of >1000. This average value had a 37% relative standard deviation (RSD). The relatively small number of data points, and the fact that the entire data set has to be separated into smaller sets leads to a high standard deviation in the loss-per-gallon predictive formula. As further data points are received, this predictive tool will continue to be refined which should decrease the standard deviation.

A number of values were not included in this average. A review of the results shows that for operating MCU with very small batch volumes (< 10000 gallons) in a non-continuous mode, the loss per gallon processed was typically much higher (~10×) than times that were typical of continuous operations. As this non-continuous mode of operations was atypical, data points corresponding to MCU running in a non-continuous mode (at very low overall amounts of <10000 gallons) are not included in the loss/gallon calculation.

Second, we should note that there is an interesting divergence in the loss-per-gallon results. Results before 2/22/2015 gave an average result of 6.46E-06 mM/gallon (20.1% RSD), while values after 2/22/215 gave an average result of 3.03E-06 mM/gallon (34.9% RSD).

The only change in operations around that time was a solvent trim that happened on 2/22/2015. This trim was unusual in that it was high in density, (0.908 g/mL), and in fact the highest density trim ever added to the system. SRNL is uncertain as to how this trim could affect the TiDG loss rates, but there seems to be a clear correlation. At this time, the data sets before and after the trim are still combined, which is one reason for the high RSD for the data set as a whole.

If, in the future, MCU changes the O:A volume ratios in the contactors, this would lead to changes in the partitioning behavior. In such case, the loss-per-gallon prediction would likely change.

Now that both the time and processing volume terms are known, a predictive formula can be assembled:

$$A_t = [A_0 \times \mathrm{EXP}(-0.000900 \times t)] - [5.39\mathrm{E}{-06} \times \mathrm{volume}]$$

where A_t is the TiDG concentration (mM) at time t (days), A_0 is the TiDG concentration (mM) at time = 0, and volume is the amount of salt solution processed in gallons. This can be used as a predictive tool to forecast TiDG demand. For example, if MCU plans on running for 90 days while processing 250,000 gallons of feed and assuming a starting TiDG concentration of 3 mM, then we would predict a final TiDG concentration of 1.42 mM.

An examination of the two components of the predictive formula shows that the losses are predominantly due to partitioning.

4.0 Conclusions and Path Forward

The current solvent blend consists of four components; an extractant, the modifier, a suppressor, and the diluent. Of the four components, only the suppressor – tris(isodecyl)guanidine (TiDG) is known to have an appreciable degradation rate.

Using data derived from process samples, this analysis derived a method to predict the TiDG losses based upon time and volume of feed processed. This work assumes that the degradation is due to two reasons; time (chemical degradation) and partitioning into aqueous phases (processing salt solution).

While other factors such as temperature or feed radiation dose could influence the degradation rates, this work does not examine these factors.

SRNL recommends that as further solvent sample data is received, the predictive tool is continuously updated. In addition, it is recommended that a detailed study into the decomposition pathways of the TiDG are explicitly studied and decomposition products are identified. Once probable decomposition products are identified, a detailed analysis of a SHT sample and a wash sample should be performed in order to confirm the presence of these materials.

Finally, SRNL recommends investigating strategies that could lead to the TiDG being converted back to the quaternized guanidinium form for part of the solvent extraction cycle. This would slow the decomposition reaction.

APPENDIX A. List of Data Points Used for this Work

Batch		TiDG (mM)		Date
prelim		3.27		12/2/2013
1		2.995		12/7/2013
2a		2.49		12/12/2013
2		2.71		12/19/2013
3		2.442		12/27/2013
4		2.466		12/31/2013
5		2.45		1/6/2014
6a		2.166		1/18/2014
6b		2.111		1/25/2014
6c		1.989		2/1/2014
Feb-135/136		1.954		2/20/2014
March-214/215/216		1.452		3/31/2014
April-259/260/261		1.524		4/21/2014
May-315/316/317		1.571		5/16/2014
June-395/396		1.385		6/27/2014
July-497/498		1.913		7/22/2014
Aug 667/668/669/670/671/672		1.174		8/26/2014
Sept-846/847		0.809		9/22/2014
Oct-913		0.66		10/7/2014
Oct#2 986/987		2.526		10/27/2014
Nov-943/1061		2.287		12/2/2014
Dec-1172/1173		1.453		12/30/2014
Jan 2015-129/130/131		1.031		1/26/2015
Feb-389/390		2.844		2/25/2015
Feb-439/440/441		2.842		2/28/2015
March -556/557/558		2.356		3/16/2015
April-661/662/663		1.921		4/2/2015
June-710/711/712		1.64		6/15/2015
June-750/751/752		1.43		6/22/2015
August-802/3/4/5/6/7		1.34		8/31/2015

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