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Permanganate Oxidation of Actual Defense Waste Processing Facility (DWPF) Slurry Mix Evaporator Condensate Tank (SMECT) and Offgas Condensate Tank (OGCT) Samples to Remediate Glycolate

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EXECUTIVE SUMMARY

Savannah River Remediation (SRR) tasked Savannah River National Laboratory (SRNL) to examine glycolate destruction in radioactive samples emulating the proposed process. Experiments with radioactive Defense Waste Processing Facility (DWPF) condensates were performed to demonstrate the oxidation of glycolate using sodium permanganate. One test each was performed for Off-Gas Condensate Tank (OGCT) and Slurry Mix Evaporator Condensate Tank (SMECT) samples received. Condensates were blended with Recycle Collection Tank (RCT) heel simulant and Slurry Mix Evaporator (SME) product containing glycolate to represent typical RCT compositions with estimated nominal entrainment of glycolate.

A permanganate to glycolate (P/G) molar ratio targeting 5.7 decreased sample glycolate concentration below Ion Chromatography (IC), glycolate method reporting limits in less than 2 hours for the OGCT sample and in less than 8 hours for the SMECT sample.

Both tests were completed safely with the following observations:

1) The glycolate destruction reaction was faster in the OGCT-RCT mixture than in the SMECT-RCT mixture. This may be due to the insoluble solids present in the OGCT feed solution prior to initiating the experiment. The SMECT feed sample had no solids. Simulant work had suggested that solids improve kinetics. In addition, the current SMECT feed had measurable formate from the DWPF formate flowsheet that consumed some permanganate in the testing. This might have slowed the process for the SMECT batch.
2) Formate concentrations were decreased to below reporting limits within 30 minutes for both mixtures. This result is consistent with basic chemistry literature cited in this report but differs from past observations in SRNL simulant test results.
3) Oxidation of nitrite to nitrate was minimal during the oxidation reactions in alkaline solutions.
4) Soluble mercury decreased for both tests.
5) The SMECT sample was analyzed for methylmercury. Triplicate results were all less than 0.2 mg/L. All mercury in the SMECT solution was likely inorganic. The OGCT sample was not analyzed for methylmercury.
6) For both reactions, oxalate increased by approximately the same molar amount as glycolate decreased, within analytical uncertainty. The stoichiometry is suggested by the chemical equation below. The current work targeted a P/G ratio of 5.7 versus 4.0 of the equation shown here. The increased amount of permanganate over 4.0 would be for kinetic purposes and to cover imperfect chemical yield.

$$\mathbf{C_2H_3O_3^- + 4MnO_4^- + 5OH^- = C_2O_4^{2-} + 4MnO_4^{2-} + 4H_2O}$$

It is recommended to continue the in-progress modeling to assess any impacts of kinetics, formate, and P/G ratio. A small amount of formate is generated during glycolate processing. Formate in the RCT will be much lower than what is found in the formate flowsheet. Future modeling work may adjust the P/G ratio based on needs for faster kinetics or lower required product glycolate content, such as 5 or 10 mg/L glycolate. The needs for faster process time or lower product glycolate are not defined at this time. Chemical and tank farm modeling, including considerations for downstream thermolytic hydrogen formation, are planned to meet such needs.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ALARA	As Low as Reasonably Achievable
AD	SRNL Analytical Development
CSTF	Concentration, Storage, and Transfer Facilities
DMA	Direct Mercury Analysis
DWPF	Defense Waste Processing Facility
IC	Ion Chromatography
ICPES	Inductively Coupled Plasma Emission Spectrometry
ICPMS	Inductively Coupled Plasma Mass Spectrometry
OGCT	Off-gas Condensate Tank
N/A	Not Applicable
P/G	Permanganate to glycolate (initial molar ratio)
RCT	Recycle Collection Tank
RSD	Relative Standard Deviation
SC	Safety Class
SME	Slurry Mix Evaporator
SMECT	Slurry Mix Evaporator Condensate Tank
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SWPF	Salt Waste Processing Facility
TAR	Technical Assistance Request
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
TTQAP	Task Technical and Quality Assurance Plan

1.0 Introduction

The Savannah River Site's Defense Waste Processing Facility (DWPF) flowsheet is being upgraded with the introduction of the Nitric-Glycolic flowsheet. The new flowsheet improves or maintains necessary parameters such as 1) reduction of mercury, 2) adjustment of feed rheology, and 3) adjustment of melter oxidation/reduction potential. Further, the potential for catalytic hydrogen generation in DWPF processing is virtually eliminated.

The Nitric-Glycolic flowsheet significantly improves DWPF's ability to address one of the Savannah River Site's key challenges: the incorporation of effluent received from the Salt Waste Processing Facility (SWPF). SWPF will deliver significant effluent volume to DWPF, resulting in a concurrent increase in DWPF effluent returned to the Concentration, Storage, and Transfer Facilities (CSTF). Testing to develop a process to oxidize glycolate and other organic species was requested by Savannah River Remediation (SRR).[1] A Task Technical and Quality Assurance Plan (TTQAP) describes the testing requested by SRR in the development of a process to oxidize glycolate and other organic species that contribute to thermolysis hydrogen generation.[2] Scoping studies have been completed as requested by a Technical Assistance Request (TAR)[3] to evaluate the feasibility of using sodium permanganate and Fenton's Reagent (iron catalyzed hydrogen peroxide) for destroying glycolate.[4] The results from these scoping studies were summarized in a report.[5]

Tests with simulants to develop the oxidation flowsheet for the Recycle Collection Tank (RCT) are complete. Phases are shown in Table 1. The first phase was additional scoping tests of permanganate and Fenton's reagent.[5] The second phase was a matrix of 23 tests that informed a down select decision by SRR and completed testing needed to understand the impact of various factors in defining the optimum processing conditions for oxidation of organics.[6] From the results of this testing, oxidation by permanganate was chosen over Fenton's reagent with hydrogen peroxide. The third phase of testing demonstrated nominal operation of the permanganate oxidation process under protocols like those used in DWPF. The specific off-normal condition of a Chemical Process Cell foamover into the SMECT was also performed in this phase. A report documents the results of these tests.[7]

Table 1. Test Phase Status and TTQAP Task Activities

Phase	Description	Status	TTQAP Task Activity
1	Scoping Tests	Complete	1 e
2	Down Select Testing	Complete	1 a-g
3	Protocol Testing – Nominal and CPC Foamover Conditions	Complete	Parts of 1 and of 3
4	Actual Waste Testing	Described in this document	2
5	Larger Scale Testing	Complete	1 h
6	Corrosion Testing	Complete	4

(Phases 4-6 not in order of testing)

The Larger Scale Testing (Phase 5) describes experiments to determine the effect of scaling on the oxidation of glycolate with supplemental experiments to investigate the impact of the order of addition of the nitrite corrosion inhibitor, the influence of varied glycolate concentrations, and to further refine the suggested permanganate to glycolate (P/G) molar ratio.[8] Studies, in Phase 6, were also performed with simulants showing accelerated corrosion is not expected for materials of construction of the RCT and downstream components with sodium permanganate.[9]

This report describes experiments and analysis to demonstrate the oxidation of glycolate using sodium permanganate with actual waste from DWPF (i.e., Phase 4). Each test batch contained a blend of OGCT or SMECT samples with simulant representative of a typical RCT heel and condensate mixture. Slurry Mix Evaporator (SME) product sludge containing glycolate was blended into the batches to represent nominal entrainment of glycolate in the RCT. SME product sludge was added to each test to target 125 mg/kg per the protocol runs for nominal foamover conditions.[7] Note that while the reference uses units of mg/kg, current work uses liquid volumes and assumes density close to 1 g/mL consistent with the feeds, thus targeting 125 mg/L.

2.0 Basis for Real Waste Testing

Previous studies on the destruction of glycolate using permanganate in RCT simulants at caustic conditions demonstrated permanganate was effective in converting glycolate to oxalate, but showed negligible reactivity for other species in the simulant solution (e.g., formate, nitrite, etc.).[7] Equation (1)) was found to best describe the observed reaction of glycolate with permanganate under nominal and low glycolate entrainment conditions.

$$\mathbf{C_2H_3O_3^- + 4MnO_4^- + 5OH^- = C_2O_4^{2-} + 4MnO_4^{2-} + 4H_2O} \qquad \mathbf{(1)}$$

The target glycolate concentration for radioactive waste testing was 125 mg/kg and represents a “nominal entrainment” of sludge and is similar to the expected future glycolate concentration in the RCT.[10]

The molar ratio of nitrite to nitrate in the CSTF returns is kept above 1.66 for corrosion control concerns.[11] In the Formic-Acid flowsheet, nitrite and caustic are added to the RCT heel before the addition of condensate to eliminate the need for chemical addition steps prior to transfer out of the RCT. In down-select and protocol tests, nitrite was added after condensate addition to avoid the potential for oxidation of nitrite to nitrate or NO_x by the permanganate intended for glycolate destruction. Results from simulant tests and published reports found nitrite is unreactive towards permanganate in caustic solutions. Tests with radioactive waste were performed with nitrite and caustic added before permanganate to confirm that added nitrite will not be oxidized.

3.0 Experimental Procedure

Two tests were performed to demonstrate nominal operation of the permanganate oxidation process under protocols similar to those to be used in DWPF. A detailed description of the experimental design can be found in the corresponding Run Plan.[12] The RCT mixture was a specified amount of SME product (to simulate glycolate entrainment) combined with a Heel simulant and either SMECT or OGCT sample. A transfer pipette was used for additions of 20 wt % sodium permanganate solution. Additions were not constant but were made drop by drop and required dozens of drops over the 20-minute addition. The DWPF and equivalent lab-scale flowrates were approximately 4.5 gpm and 3 mL/hour, respectively.

3.1 Real Waste Composition

The waste composition represents the transfer from the SMECT or OGCT onto the heel in the RCT. The heel was pre-charged with sodium hydroxide and nitrite to provide 4.4 moles of nitrite per mole of nitrate when combined with condensate.

3.1.1 Source of Glycolate

Glycolate for the testing was sourced from SME product present in SRNL Cell Block A. The product was a slurry of solids and liquid containing glycolate. The SME product used was labeled “SC-18 SME Product NMC&A M016108, Pareizs May 2017”. Additional information can be found in references 10 and 13.[10,13] The target concentration for the simulated entrainment is 125 mg/L glycolate in the waste batches representing approximately 14 gallons of carryover into the RCT.

3.1.2 SMECT and OGCT Samples

SMECT and OGCT liquid samples were transported from DWPF to SRNL (Shielded Cells A Block). A more detailed description of these materials can be found in Section 4.1.

3.1.3 Source of the Heel Component

The process simulation required a liquid heel material since it contributes organics, nitrite, and caustic, which might affect the later parts of the process. Since no preceding test material as a heel was available, a simple solution of nitrate, nitrite, formate, oxalate, and hydroxide sodium salts was used. The recipe was developed from previous experiments with simulants[14] and is reproduced in Table 2 below.

Table 2. RCT heel plus condensate simulant composition (pH ~13)

Species	Conc. (mg/kg)	Conc. (M)
Sodium Formate	27.7	4.07×10^{-4}
Sodium Oxalate	2.48	1.85×10^{-5}
Sodium Nitrate	4070	4.79×10^{-2}
Sodium Nitrite	2640	3.83×10^{-2}
Sodium Hydroxide	8340	2.08×10^{-1}

3.2 Oxidation Stoichiometry

A detailed discussion of oxidation stoichiometry can be found in the simulant study reports.[7,8] The stoichiometry for this process is defined as the molar ratio of permanganate to glycolate (P/G) in the batch. The target stoichiometry for the testing was 5.7 P/G and was anticipated to decrease the glycolate concentration to below IC reporting limits within 3 hours. While reaction equation (1) above indicates a P/G ratio of 4, more would likely be needed for the sake of kinetics and reaction inefficiencies.

3.3 Experiment Scaling

The experiments were performed with 250 mL condensate feed volumes, which is 1/88,000 fraction of the RCT condensate feed volume (5810 gallons). A comparison of scale between DWPF, small-scale testing with simulants, and radioactive waste testing in the Shielded Cells can be found in Table 3. Despite the small size, the chemistry is not expected to be scale dependent, and the main value of radioactive waste testing is to validate results from simulant testing.

Table 3. Chemical scaling of laboratory experiments vs. DWPF Operations

	DWPF (gal)	Large-Scale Laboratory (mL)	Small-Scale Laboratory (mL)	Small-Scale Shielded Cells (mL)
RCT Heel	1400	3470	315	60.2
50 wt% NaOH	75	186	16.9	3.23
Condensate	5810	15600	1310	250
Sludge	14	35	3.15	0.60
6.6M $NaNO_2$ Solution	215	532	48.4	9.25
Permanganate, Targeting P/G 5.7	53	132	12	2.3

3.4 Experimental Apparatus and Parameters

The experimental apparatus was located in SRNL Cell Block A and consisted of an open 500-mL borosilicate glass Erlenmeyer flask on a magnetic stir plate. A photograph of the apparatus used for the OGCT experiment can be seen in Figure 1. An opaque covering for the flask was provided to minimize light exposure, since permanganate and manganate species are known to be affected by visible light. The 500-mL volume was sufficient for the test with reasonable but not excessive freeboard (see volumes in Table 3). The mixture was maintained at the ambient Shielded Cells temperature of 15-17 °C. The mixing rate was not measured but was set visually with no vortex that would entrain air.

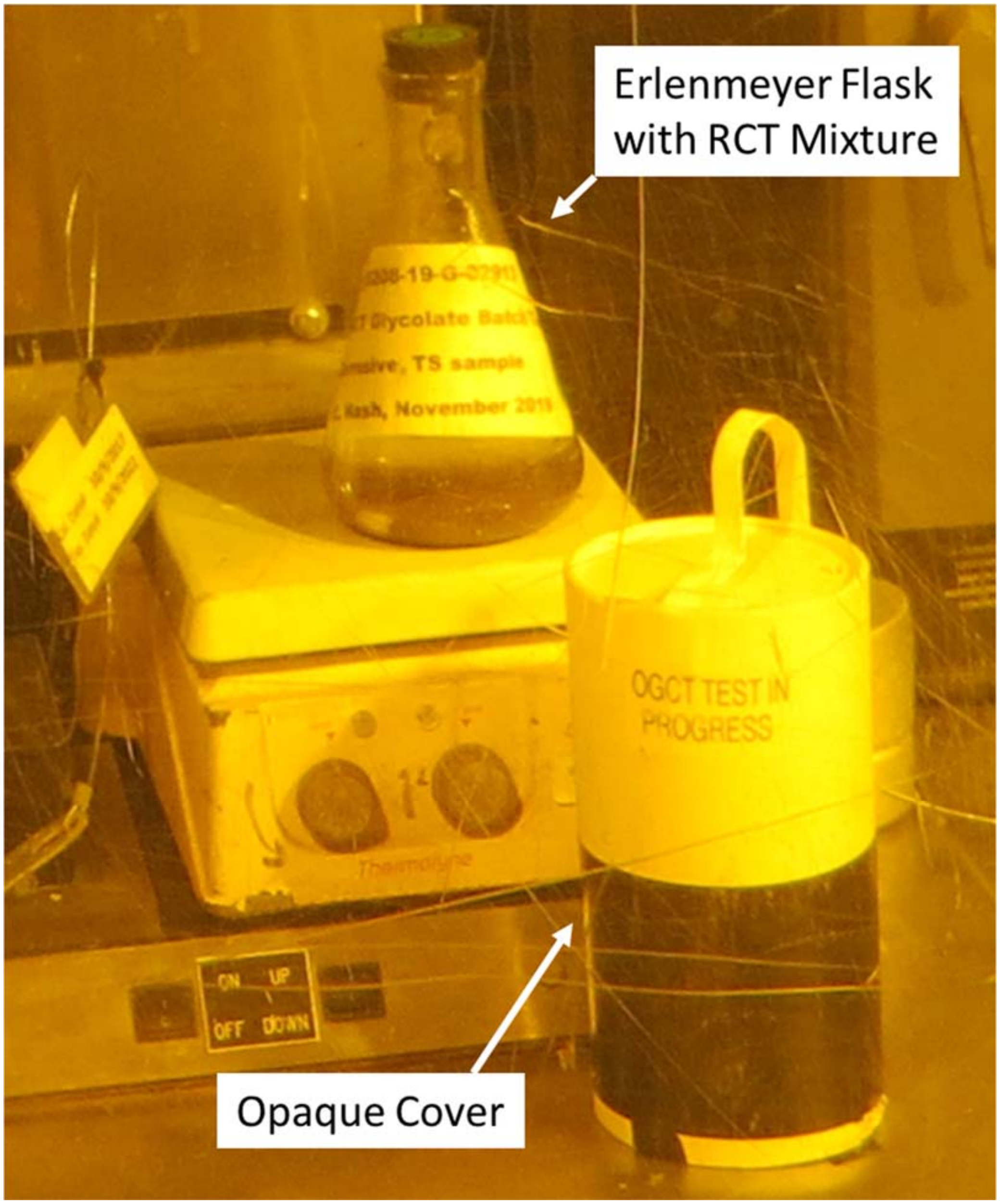


Figure 1: Photograph of the experimental apparatus in the Shielded Cell.

3.5 Sample Preparations and Characterization

Characterizations of the OGCT and SMECT samples received from DWPF were performed as described in the Run Plan.[12] During each experiment, approximately 15 mL samples were drawn from the test vessel. Measurement of the glycolate kinetics requires "quenching" of samples when they are drawn to stop the reaction(s) that are removing glycolate from solution. This is to be distinguished from another process of "caustic quenching" used elsewhere in this work. This work quenched samples with about 0.3 grams of sodium sulfite, a reductant, pre-loaded in each sample vial. Sodium sulfite consumes permanganate, stopping any reaction that permanganate is causing. This method thus allowed tracking of glycolate concentration with time, preserving remaining glycolate in sample solutions.

Outside of alkaline samples quenched with sodium sulfite, acid and water dilutions of samples were performed as in the run plan. Dilutions for OGCT samples were often done at one order of magnitude to help with the concern about as low as reasonably achievable (ALARA) dose from those highly radioactive samples. Cesium removal was not performed for ALARA purposes because of concerns that it could remove desired analytes. For example, crystalline silicotitanate will remove cesium, but it would also target other metals like calcium and iron. Cesium removal was only performed per routine within the Analytical Development (AD) liquid scintillation analysis to help refine alpha and low level beta quantifications.

Acid was used to dilute samples when metal and radioisotope analyses were done, giving best stability for such analytes. Water was used to dilute samples for IC anions, total inorganic carbon (TIC) and total organic carbon (TOC).

3.6 Quality Assurance

This work meets the requested Safety Class (SC) level of quality assurance. SRNL will treat all documents from this work as lifetime retention records. The work is not waste form affecting and does not apply the requirements of RW333P. Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. Information from the experiments was posted in Electronic Laboratory Notebook T7692-00094-08.

4.0 Results and Discussion

4.1 Waste Component Characterization

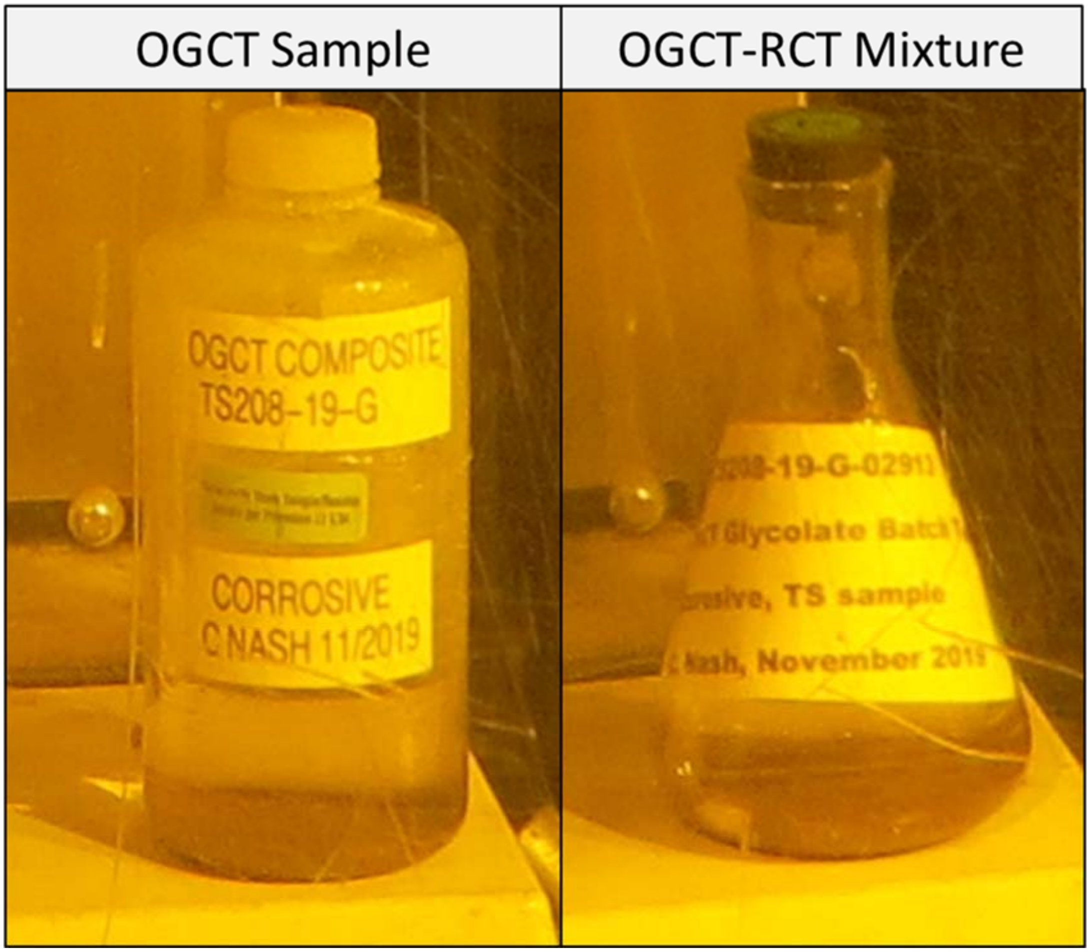
The OGCT and SMECT samples were received from DWPF in sets of six 220 mL stainless steel sample vials. The OGCT sample number was #25484, pulled April 5, 2019. The DWPF melter was being fed and was pouring glass at the time. The SMECT sample number was #25648, pulled May 21, 2019. Melter status was not available, but the sample is shown as Batch number 4573.

Each set of six was composited to provide separate SMECT and OGCT feed samples. The SMECT sample was a clear liquid with no visible solids and is not pictured. The OGCT sample contained very fine gray visible settled solids. A photograph of the composite OGCT sample and the combined OGCT-RCT mixture can be seen in
Figure **2**.

The SME product used for this work contained 18700 mg/L +/- 9% relative standard deviation (RSD) glycolate by triplicate measurement. This work measured out about 10 grams of SME product sludge for treatment with 50 wt % NaOH. The SME product sludges were contacted with sodium hydroxide per the "caustic quench" method used in reference 13. Deionized water and 1 gram of the caustic SME sludge samples were put into the flasks, making caustic sludge water mixtures of 100 mL. AD reported IC anions glycolate results from filtrates to find the amount of glycolate that SME product sludge would add to an alkaline aqueous solution.

SME product sludge was added to the combined RCT samples to simulate glycolate carryover to attain the values listed in in Table 4. The target was 125 mg/L glycolate. The amount of SME product sludge was adjusted based on batch volumes and the measured average glycolate content of the sludge.

Table 4 shows a summary of the measured concentrations of key analytes for the samples and mixtures. The pH of the SMECT was determined by AD using an ion selective electrode. The OGCT sample was estimated using pH paper in cell. This is because dilutions required for ALARA purposes to minimize radiation exposure to AD would invalidate pH measurement of samples.



OGCT Sample

OGCT-RCT Mixture

Figure 2: Photographs of the combined OGCT sample and the combined OGCT, RCT Heel, and SME product.

Formate and oxalate measurements in Table 4 show that SME product was a contributing source of both of these. The heel simulant values show that the simulant contained insufficient amounts of these to account for the combined batch compositions before permanganate was added.

All values of TIC, TOC, and total carbon were less than the reporting limits for both the SMECT and OGCT samples as-received. SMECT reporting limits were <40 mg/L for total carbon and <20 mg/L for each of TIC and TOC. OGCT reporting limits were <117 mg/L for total carbon and <58.6 mg/L for each of TIC and TOC. The 60.4 mg/L of formate in the SMECT sample provided only 16 mg/L carbon content, which is less than the <20 mg/L reporting limit for TOC.

Table 4: Composition of SME, SMECT, OGCT and RCT Samples

	Glycolate (mg/L)	Formate (mg/L)	Oxalate (mg/L)	Nitrate (mg/L)	Nitrite (mg/L)	Mn (mg/L)	Hg (mg/L)	pH
SME Product	18700	--	--	--	--	--	--	--
SMECT	--	60.4	< 10	3600	< 10	3.87	101	1.6
OGCT	--	< 117	< 117	2790	< 117	57.7	213	0-1[a]
RCT Heel Simulant[b]	0	18.3	2.05	2970	1762	0	0	13.3
Combined SME Product, Heel Simulant, and SMECT or OGCT								
Combined SMECT	145	59.3	66.4	2790	9460	< 0.227	--	13.2
Combined OGCT	183	66.8	70.0	2940	9500	576	173	13.2

[a] Estimated
[b] Calculated
Dashes in cells here and other tables indicate no measurement performed.

Table 5 shows ICPES results of the OGCT and SMECT samples from DWPF and composited with the RCT heel and SME product. The increase in sodium content reflects the addition of the Table 2 simulant per the desired volume additions of Table 3. Unexpectedly, an increase in soluble Ca, Fe, Mn, Ni, Si, Th, U, and Zr was observed in the combined OGCT-RCT sample. The solubility of these metal ions is expected to be lower in highly alkaline solutions and the reason for their appearance is unknown. The solids that came in with the acidic OGCT sample are the source of the excess elements, so the exposure of the OGCT solids to the caustic simulated heel must have increased their tendency to dissolve. The resulting batch mixture was alkaline. AD re-checked their various dilutions of the samples and found that ICPES data were consistent in indicating the presence of the elements in the samples. In contrast, minimal soluble manganese, beyond a small amount left from the permanganate reaction, was detected in the liquid from the completed SMECT test batch.

Table 6 provides the few significant masses detected by ICPMS. Masses corresponding to mercury (196, 198, 204) were omitted because ICPMS is not used to quantify mercury. Masses likely indicate technetium-99, cesium-133 and 137, cerium-140, and uranium-238. The OGCT results for cesium show that about 23% of the cesium is Cs-137 versus the stable 133 isotope.

Table 5: SMECT and OGCT Samples and RCT Composite Samples - ICPES Results*

	SMECT Sample		SMECT-RCT		OGCT Sample		OGCT-RCT	
Element	Average (mg/L)	% RSD	mg/L	% RSD	Average (mg/L)	% RSD	mg/L	% RSD
Al	4.26	10	13	10.3	82.3	10	401	10
B	7.75	10	< 23.4	N/A	106	10	< 257	N/A
Ba	< 0.076	N/A	< 0.498	N/A	0.47	10	< 9.2	N/A
Ca	2.45	10.1	< 0.989	N/A	15.0	10	119	10
Cr	< 0.32	N/A	< 1.02	N/A	7.63	10.1	< 19.47	N/A
Cu	< 0.30	N/A	< 2.58	N/A	0.99	10.9	< 15.2	N/A
Fe	6.54	10	< 0.882	N/A	131	10	1410	10.2
Li	< 0.53	N/A	< 3.45	N/A	12.6	10	< 34.5	N/A
Mg	0.35	10	< 0.068	N/A	2.98	10	30.6	10.1
Mn	3.87	10	< 0.227	N/A	57.7	10	576	10.1
Na	37.6	10	10500	10.6	349	10	12800	10
Ni	< 1.21	N/A	< 7.92	N/A	10.7	10.1	122	10
Si	60.2	10.2	76.3	10.1	137	10	283	20
Sr	< 2.84	N/A	< 18.6	N/A	0.24	10	< 2.97	N/A
Th	< 1.81	N/A	< 7.15	N/A	7.68	10.5	78.9	20
Ti	< 0.061	N/A	< 0.324	N/A	0.57	10.1	< 3.96	N/A
U	< 9.66	N/A	< 52.6	N/A	30.6	11.8	231	11.8
Zn	1.80	N/A	< 2.03	N/A	2.52	10	< 8.47	N/A
Zr	< 0.088	10.1	< 0.574	N/A	1.39	10.2	18.1	10.3

Results listed are soluble elemental concentrations. %RSD is 100(std deviation/average of values)

Table 6. Isotopic Masses found in the DWPF Samples

Mass	SMECT		OGCT	
	mg/L	% RSD	mg/L	% RSD
99	<0.003	N/A	1.48	1
133	0.08	0.6	5.45	1
137	0.03	0.5	1.59	2
140	0.08	2.3	1.43	9
238	1.94	0.1	31.1	1

Table 7 provides the radiochemistry characterization for the as-received SMECT and OGCT samples. SMECT samples were submitted with 3x dilution using 1.5 M nitric acid to ensure low pH samples. OGCT samples were acid-diluted by a factor of about 12 mainly for radiation exposure purposes. Initial dose data from DWPF indicated high activity from the OGCT sample. The OGCT sample was found to contain a two orders of magnitude higher concentration of cesium-137 than the SMECT liquid sample. Sample dose is significant to the radiation hood worker at the higher cesium-137 activity, so OGCT samples were diluted and handled in smaller numbers for ALARA purposes. Sample cesium removals were not performed for dose purposes because of the possibility of removing other analytes along with cesium. Radiological hood limits are specified in an SRNL radiological work practices procedure.[15]

Cesium-137 activity summed with two times the strontium-90 activity is in good agreement with total beta for the SMECT sample. This suggests that there are no other significant beta emitters in that sample. Cesium-137 explains most of the total beta found in the OGCT sample, and if that is removed, the double beta decay of the strontium-90 explains much of the remainder. Technetium-99 activities are too low to affect beta measurements. Alpha activity is below quantification concentrations for both SMECT and OGCT samples. The cesium-removed alpha value is often the more reliable of the two types of alpha measurement. This is because the high beta activity of the sample with cesium-137 present will interfere with concurrent alpha quantification by liquid scintillation.

The last row of Table 7 compares technetium-99 by the counting method with that of the ICPMS value converted to activity. The ICPMS value is about 15% lower but is within the uncertainty for the ICPMS method (20%).

Table 7: Summary of Activity from as-received SMECT and OGCT samples

	SMECT		OGCT	
	dpm/mL	**% RSD**	**dpm/mL**	**% RSD**
Total Alpha	< 1.04E+05*	N/A	< 3.15E+06**	N/A
Total Alpha - Cs Removed	--	N/A	< 1.59E+06	N/A
Total Beta	6.28E+06	2.86	4.36E+08	1.32
Total Beta - Cs Removed	--	N/A	5.53E+07	30.8
Cs-134	< 1.23E+04	N/A	< 1.11E+05	N/A
Cs-137	3.42E+06	1.40	3.34E+08	4.18
Sr-90 (beta)	1.42E+06	13.4	1.63E+07	17.1
Tc-99	8.98E+01	15.1	6.56E+04	4.00
Tc-99 calculated from ICPMS	<1.1E+02	-	5.63E+04	20.0

* Upper Limit Value
** Below the Minimum Reporting Amount

4.2 Glycolate Destruction

Results from the glycolate destruction reaction in the SMECT and OGCT batches can be found in Table 8 and Table 9. All results in these tables are reported with an analytical uncertainty of 10%. Water dilutions were performed on the OGCT samples for ALARA purposes prior to analysis resulting in higher IC glycolate reporting limits for the OGCT samples (i.e., 23 mg/L for OGCT vs. 10 mg/L for SMECT samples). The final SMECT analysis was performed 1 month after permanganate addition. An analogous time was not feasible for the OGCT sample because of analytical resource limitations at the end of the calendar year. IC analyses for the OGCT samples were performed 15, 70, and 114 days after the reaction with sodium permanganate. Glycolate concentrations were decreased to below the IC reporting limits in both reactions, and the molar ratio of nitrite to nitrate remained well above the 1.66 ratio required for the corrosion control program after 1 month for both tests.

Table 8: SMECT experimental results – glycolate destruction and nitrite/nitrate ratio

Slurry Mix Evaporator Condensate Tank (SMECT) - P/G 5.6						
Time (min)	**Glycolate (mg/L)**	**Formate (mg/L)**	**Oxalate (mg/L)**	**Nitrite (mg/L)**	**Nitrate (mg/L)**	**Nitrite/Nitrate (mol/mol)**
0	145	59.3	66.4	9460	2790	4.57
32	67.7	< 10	139	9610	3230	4.01
60	46.3	< 10	158	9570	3030	4.26
93	32.2	< 10	197	9550	2950	4.36
121	25.7	< 10	198	9530	3120	4.12
182	16.4	< 10	--	--	--	--
478	< 10	< 10	239	9590	3000	4.31
31 days	< 10	< 10	263	8750	3000	3.93

Cells with dashes show no measurements were taken or available.

Table 9: OGCT experimental results – glycolate destruction and nitrite/nitrate ratio

Offgas Condensate Tank (OGCT) - P/G 4.9						
Time (min)	**Glycolate (mg/L)**	**Formate (mg/L)**	**Oxalate (mg/L)**	**Nitrite (mg/L)**	**Nitrate (mg/L)**	**Nitrite/Nitrate (mol/mol)**
0	183	66.8	70	9500	2940	4.36
31	79.8	< 23	210	9120	2920	4.21
60	40.7	--	--	--	--	--
91	25.2	--	--	--	--	--
122	< 23	--	--	--	--	--
177	< 23	--	--	--	--	--
474	< 23	< 22	294	9000	3000	4.04
15 days*	< 10	< 10	296	9110	3700	3.32
70 days**	< 25	< 10	< 10	5090	2640	2.60
114 days	--***	<10	297	9080	3600	3.40

* Unquenched undiluted sample analyzed 15 days after start of the test.
** Likely a dilution problem with IC anions analysis for this sample
*** Not measured; no increase/return of glycolate assumed

The solution from the OGCT batch was sampled and analyzed by IC anions 114 days after the start of the test because the 70 day analysis appeared to have a dilution problem. The 114 day results in Table 9 show values in line with the 15 day sample, showing that anionic composition was stable through the long duration.

Glycolate concentrations in the combined SMECT/OGCT, RCT Heel, and SME Product mixture were measured prior to the permanganate reaction. Values from this measurement were used to determine the amount of permanganate required to meet the target P/G of 5.7. Sodium permanganate was added dropwise by pipette for 20 minutes, and the mass of permanganate added to the batch was determined using a mass balance. Using these measurements, the actual P/G ratio was determined to be 5.6 and 4.9 for the SMECT and OGCT reactions respectively. Despite the actual ratios being below the targets, the glycolate destructions were still accomplished to below the reporting limit within the day of the tests.

Figure 3 depicts glycolate and oxalate concentrations as a function of time after the start of permanganate addition to the SMECT and OGCT batch solutions. Glycolate concentrations were decreased below reporting limits in both reactions, and greater than 50% of the glycolate was destroyed within 30 minutes of the start of permanganate addition. For both reactions, oxalate increased by approximately the same molar amount as glycolate decreased as in equation (1).

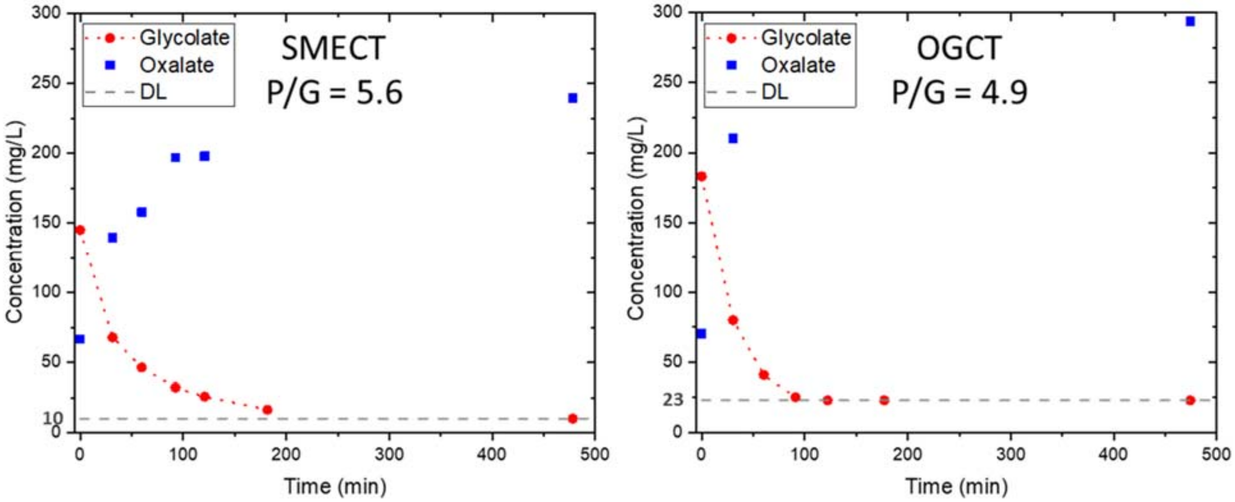


Figure 3: Glycolate and oxalate concentration as a function of time in *(left) SMECT* and *(right)* OGCT solutions during the permanganate reaction. IC reporting limits as shown.

The reaction rate was faster in the OCGT batch even with a lower P/G, and this may be due to the solids observed in the as-received OGCT sample. Testing with simulants revealed a lower P/G ratio was required to destroy glycolate with higher sludge content present in the test.[7,8] While not definitive, one proposed mechanism suggests Mn^{2+} in sludge can be used to generate the oxidant Mn^{3+} from manganate or permanganate.[7] The solids in the OGCT sample likely contained Mn^{2+} that may have contributed to this reaction.

Formate concentrations were decreased to below reporting limits within 30 minutes for both samples. The disappearance of formate differs from simulant studies where formate behaved as a spectator, but was in agreement with published reports of formate and permanganate reacting in alkaline solutions.[16,17,18,19,20] Carbon dioxide (or carbonate in alkaline solution) is the likely product of this reaction because formate is such a simple molecule that no oxidized intermediates exist between it and carbon dioxide. While off-gas analysis was not performed during these experiments, it is expected that the majority of carbon dioxide formed was adsorbed into the highly alkaline (pH > 13) solution as it was generated. It would not have been observed in an off-gas stream. The low formate concentration would contribute so little carbonate that changes in solution would be too low to quantify.

The reason for the difference between reported simulant formate reactivity versus literature and radioactive waste results is unknown. The formate reaction in these tests did not consume enough oxidative reagents to prevent glycolate destruction in the first 8 hours. However, the glycolate flowsheet is considered to convert about 1% of glycolic acid to formate.[21] The P/G ratio used in DWPF may need to be raised for conservatism to cover formate, chemical inefficiencies, uncertainties in glycolate content, and enhanced kinetics. Future modeling work may be able to address any additional questions.

4.2.1 Manganese

Simulant studies confirmed permanganate (Mn^{7+}) was reduced to manganate (Mn^{6+}). The manganese species were found to persist in caustic solutions.[8] The concentration of soluble manganese was measured by ICPES for the SMECT and OGCT tests before and during the oxidation reaction, and approximately 1 month after the completion of the reaction (see Table 10). The sodium permanganate required to achieve a P/G of 5.7 contributes ≈600 mg/L Mn to the solution. The "combined batch & permanganate" column in Table 10 is a combination of the measured manganese concentration in the batch solution with the calculated value of added permanganate.

Table 10: Soluble Manganese concentrations in OGCT and SMECT Solutions

	Soluble Manganese (mg/L)				
	Batch Solution	Added Permanganate	Combined Batch & Permanganate	3 Hours	1 Month
SMECT	< 0.277	594	594*	22.6	11.6
OGCT	576	660	1236*	1190	< 0.277

*Calculated

Measured manganese concentrations were decreased by one or two orders of magnitude for both tests after 1 month, likely the result of manganese dioxide precipitation. For the OGCT test, soluble manganese concentrations were similar to initial concentrations in the hours after the glycolate destruction reaction. The manganese concentration was greatly reduced three hours after the addition of permanganate to the SMECT batch. Gauger and Hallen reported that an equilibrium between permanganate and manganate will exist in alkaline solution.[16] Manganate will precipitate some manganese dioxide in a disproportionation reaction to re-form permanganate. Since permanganate is chemically active to oxidize organic species in solution, soluble manganate is indirectly still useful towards this end. Manganese dioxide solids formation is the end state in any case.

4.2.2 ICPES Results

Results from ICPES analyses for the SMECT and OGCT batch mixtures, and 180 min, 480 min, and 1 month after the start of the permanganate reaction are found in Table 11 and Table 12. All results with initial and final values below reporting limits were omitted. Both the SMECT and OGCT samples contained ≈0.5 M sodium with measurable Al and Si. (Chemical leaching of the Erlenmeyer flask was a likely contributor to Si.) After one month, the solutions still contained 0.5 M Na and had measurable Si and Al, but the aluminum was decreased by a factor of 7 in the OGCT. In addition, the OGCT sample had measurable Ca, Fe, Mg, Ni, Th, U, and Zr in the initial sample and all were decreased during the permanganate reaction and ultimately below or near reporting limits after 1 month. Reduced concentrations of these metals would be expected in alkaline solutions because their solubilities are known to be very low at high pH.

Table 11: SMECT Experimental Results – ICPES

SMECT-RCT Glycolate Oxidation - ICPES Results								
Element	Batch (mg/L)	%RSD	180 min (mg/L)	%RSD	480 min (mg/L)	%RSD	1 mo (mg/L)	%RSD
Al	13	10.3	19.2	10	19.4	10	23.1	10
Li	< 3.45	N/A	0.906	10.2	0.978	10.3	3.13	10.1
Na	10500	10.6	10500	10	10700	10	10700	10
Si	76.3	10.1	85.1	10	85.8	10	141	10.1
U	< 52.6	N/A	< 6.47	N/A	< 6.47	N/A	5.81	12.2

Table 12: OGCT Experimental Results – ICPES

OGCT-RCT Glycolate Oxidation - ICPES Results								
Element	Batch (mg/L)	%RSD	90 min (mg/L)	%RSD	480 min (mg/L)	%RSD	1 mo (mg/L)	%RSD
Al	401	10	172	10	174	10.1	57.2	10
B	< 257	N/A	61.9	10	61.6	10	66.7	10
Ca	119	10	38.9	10	38.9	10	< 0.989	N/A
Cr	< 19.47	N/A	7.59	10.2	7.89	10.3	5.59	10.9
Fe	1410	10.2	456	10.2	464	10	< 0.493	N/A
Li	< 34.5	N/A	15.1	10.1	15.2	10.1	< 9.28	N/A
Mg	30.6	10.1	7.49	10	7.42	10	0.14	10.1
Na	12800	10	11200	10	11200	10	11400	10
Ni	122	10	34.2	10.5	34.8	10.1	< 6.71	N/A
Si	283	20	123	10.2	127	10.2	99.6	12
Th	78.9	20	25.9	11.1	25.8	10.3	< 1.93	N/A
U	231	11.8	60.1	10.2	61.2	10.5	< 21.6	N/A
Zr	18.1	10.3	6.45	10.1	6.37	10	< 0.574	N/A

4.2.3 Mercury

Total mercury by "Direct Mercury Analysis" or DMA was measured in triplicate for SMECT and OGCT samples as received (diluted with acid in the case of OGCT for ALARA purposes). The AD method used a Milestone® Direct Mercury Analyzer (DMA-80) system.[22] Total mercury was also measured once for initial batches before permanganate addition, and at other times as shown in Table 13 below. The 480-minute measurement for the SMECT batch was not performed, though the concentration at that time was likely not much different from the values found earlier in the day.

Ideally the initial batch measurement would be 76% of the as-received sample value due to dilution by simulant heel, 50 wt % NaOH, and 6.6 M $NaNO_2$ solutions (60.2, 3.2, and 9.25 mL respectively). SME product would add about 2.5 mL of volume as well. The SME product may contribute some mercury, though the 21% uncertainty in each case is probably from sampling and measurement uncertainties.

Table 13. DMA Total Mercury (Soluble) for Various Samples

Test Time, SMECT	mg/L Hg	Test Time, OGCT	mg/L Hg
As-received Sample*	101 +/- 21.6 %RSD	As-received Sample*	213 +/- 22.4 %RSD
Initial Batch	102	Initial Batch	173
60 minute	108		--
120 minute	116		--
180 minute	104	480 minute	54.8
1-month	45.1	1-month	13.6

*Triplicate measurements

The OGCT batch showed some soluble mercury reduction measured after eight hours of processing while the SMECT batch showed no significant change in three hours. In the 1 month of aging after that, definite reductions in soluble mercury was seen in both cases. The permanganate process thus does not decrease soluble mercury concentrations rapidly. The effect of the permanganate process to precipitate mercury is not known because no control was run to see if mercury concentration in solution would decline in time on its own.

SRNL measured methylmercury anion (CH_3Hg^+) in triplicate for the SMECT sample, and all three results were less than the 0.2 mg/L reporting limit.[23] The SMECT sample was analyzed "as is" without mercury preservative use. The mercury in the SMECT sample as received thus appeared to be inorganic species.

4.2.4 Weight Percent Solids

Table 14 shows measured total and insoluble solids for both SMECT and OGCT samples. Total solids measurements were performed in triplicate and the average and %RSD are reported. For the SMECT sample, one measurement differed more than expected from the other two and accounts for the relatively large %RSD. Excluding this outlier, the SMECT total solids becomes 3.05 +/- 0.5 %RSD and total solids in both condensates are close to 3 weight percent.

The permanganate process adds solids to the batch because the permanganate goes to lower oxidation states, and probably to manganese dioxide, in alkaline media. An example calculation shows that this process adds less than 0.1 wt % insoluble solids to the RCT batch if the permanganate does convert to manganese dioxide solids. If the glycolate concentration in the adjusted batch is 145 mg/L, that is 0.00193 M, and at a stoichiometry ratio of 5.7, would call for the addition of 0.011 M manganese as permanganate. Converting all this manganese to insoluble manganese dioxide would yield 0.96 grams in one liter, or just less than 0.1 wt %.

Table 14 shows measured insoluble solids gains for both SMECT and OGCT tests. The in-cell method[24] shows greater gains than 0.1 wt %, but all numbers are still very low. Uncertainties are large because of the small amounts of solids present in the samples. Perhaps the solids are hydrated and do not dry completely or lose hydroxide groups at the 130 °C drying temperature. Increases may also include other species that precipitate with the manganese, such as a small amount of mercury. Soluble aluminum, calcium, magnesium, nickel, uranium, thorium, and zirconium also show declines in the one-month OGCT sample – see Table 12. The declines are slow but easily measurable.

Table 14. Weight % Total and Insoluble Solids Measurements

SMECT	Weight % Total Solids	Weight % Insoluble Solids	OGCT	Weight % Total Solids	Weight % Insoluble Solids
As-received	3.49 +/- 21.1 %RSD	Zero*	**As-received**	3.06 +/- 2.22 %RSD	0.043 +/- 43 %RSD
1-month	Not Measured	0.47 +/- 27%RSD	**1-month**	Not Measured	0.36 +/- 55 %RSD

*Observed completely clear solution

4.3 Comparison of radioactive waste and simulant testing

A comparison of glycolate concentrations between the two tests with radioactive waste and analogous experiments with simulants can be found in Figure 4. In addition to the tests using SMECT and OGCT described in this report, two experiments using simulants with a P/G ratio of 5.7 (Sim 1.8 L) and 5.6 (Sim 20 L) are shown. The simulant studies were performed at different volume scales and additional information on these tests can be found in the report.[8] The IC reporting limit of 10 mg/L shown applies to the SMECT and both simulant samples. The OGCT reporting limit of 23 mg/L is not shown, and OGCT data below this value is omitted for clarity.

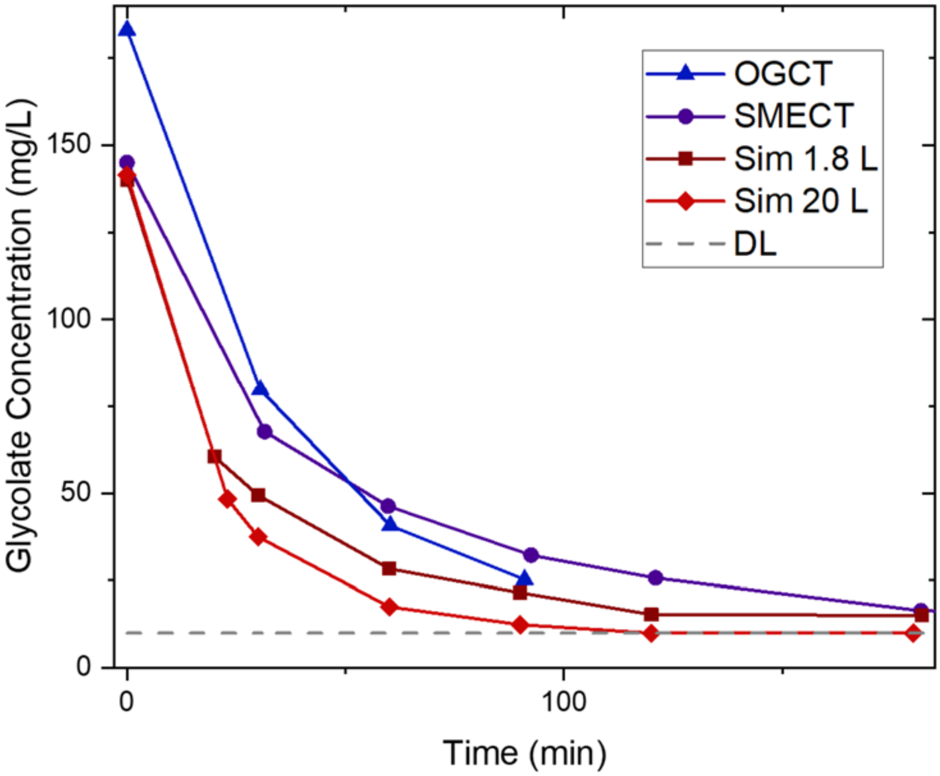


Figure 4: Comparison of glycolate destruction reactions performed in radioactive waste (SMECT and OGCT) and simulants (Sim 1.8 L and Sim 20 L) with similar initial glycolate concentrations and P/G ratios.

Glycolate was similarly destroyed in both radioactive-waste and simulant studies. Where > 80% of the glycolate destruction reaction was complete before 90 minutes in the simulant studies and the OGCT, approximately 78% was destroyed in the SMECT composite possibly suggesting a slower reaction rate in this sample.

5.0 Conclusions and Recommendations

Glycolate destruction tests using sodium permanganate with OGCT and SMECT samples from DWPF have been completed. The following conclusions are drawn.

1) Glycolate at a starting concentration of 145 mg/L was decreased below the IC reporting limit of 10 mg/L for the SMECT test in 8 hours using a P/G ratio of 5.6.
2) Glycolate at a starting concentration of 183 mg/L was decreased below 23 mg/L for the OGCT test within 122 minutes (approximately 2 hours) using a P/G of 4.9.
3) In both experiments, > 75% of the glycolate destruction reaction was complete within 90 minutes of the start of permanganate addition.
4) Oxalate content in both tests increased by approximately the same molar amount as glycolate decreased. This is consistent with equation 1 of Section 2.0.
5) Contrary to simulant studies, formate was decreased to below IC reporting limits within 30 minutes. This assumed reaction with permanganate would consume oxidant and may need consideration if significant formate were in the feed.
6) The glycolate destruction reaction was faster in the OGCT batch than in the SMECT batch, possibly due to manganese bearing solids present in the OGCT feed sample.
7) The soluble manganese concentration for the OGCT product was decreased one or two orders of magnitude after one month.
8) A small amount of nitrite destruction may occur, but nitrite/nitrate ratios remained well above 1.66 for corrosion control concerns after one month. Since permanganate is not present after a month, it is no longer able to oxidize nitrite to nitrate.

The work herein demonstrates permanganate to be an effective oxidant for mitigating glycolate. A current kinetic modeling effort is examining the rate constants from radioactive and simulant results. It is recommended to continue the chemical and tank farm modeling, which will include impacts of thermolytic hydrogen downstream.

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