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TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP) TESTING OF WASTE GLASS AND K-3 REFRACTORY: REVISITED

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ABSTRACT

The U. S. Environmental Protection Agency (EPA) issued revised Resource Conservation and Recovery Act (RCRA) Phase IV Land Disposal Restrictions (LDR's) on May 26 1998. The new regulation requires that any waste characteristically hazardous for the metals As, Ba, Cd, Cr, Pb, Hg, Se, and Ag will have to be treated to meet the LDR Universal Treatment Standards (UTS) for each metal prior to land disposal. Since EPA regulations continue to become more stringent, here-to-fore unpublished TCLP data generated during testing of simulated High Level Waste (HLW) glass, including the Environmental Assessment glass and K-3 melter refractory, will be reviewed. The refractory TCLP data compilation includes K-3 refractory in contact with DWPF

simulated glass in a pilot scale melter and K-3 refractory in contact with actual mixed waste glass in a 5 ton a day GTS Duratek melter.

INTRODUCTION

High-level liquid nuclear waste (HLLW) at the Savannah River Site (SRS) is being immobilized by vitrification into borosilicate glass. The glass is produced and poured into stainless steel canisters in the Defense Waste Processing Facility (DWPF) for ultimate geologic disposal. The canistered borosilicate waste glass must comply with the Waste Acceptance Product Specifications (WAPS) established by the U.S. Department of Energy. WAPS Specification 1.3 relates to the ability of the vitrification process to consistently control the final waste form durability, i.e., the stability of the glass against attack by water.

In addition, the Environmental Protection Agency (EPA) is concerned about the release of Cr^{+6} to the environment [1]. The feed sludge to the DWPF melter is considered characteristically hazardous waste under the Resource Conservation and Recovery Act (RCRA), because the raw sludge is considered to fail the EPA Toxic Characteristic Leaching Procedure (TCLP) for Cr ion release, e.g. all of the Cr in the waste, ~0.14 wt. % or 1400 ppm, is considered to be Cr^{+6} and completely (100%) soluble. The DWPF waste is not ignitable, reactive or corrosive and does not contain listed wastes.

In the 1990-1993 time frame, the Savannah River Technology Center (SRTC) fabricated a set of simulated high level waste (HLW) glasses doped at 1-3 times the concentrations of the RCRA metals [2] of concern to the EPA. The SRTC subcontracted a South Carolina EPA certified laboratory to perform both the monolithic Extraction Procedure Toxicity Test (EPTox) and the Toxic Characteristic Leaching Procedure (TCLP) test on these glasses. Chemical analysis of the glass confirmed that the elevated concentrations of the RCRA metals were indeed atomically bonded in the glass and had not vaporized during sample fabrication. The EPTox/TCLP testing demonstrated that the vitrified product would perform ~100X better for Cr release than the TCLP regulatory limits for characteristically hazardous waste (see Table I) [2]. The EPTox/TCLP testing [2] was part of the basis upon which EPA declared HLWIT treatment of HLW as the Best Demonstrated Available Technology (BDAT) [3].

The DWPF melter, the West Valley Nuclear Services HLW melter, the GTS Duratek mixed waste glass melter operating at the Savannah River Site, and the proposed Hanford melters for both HLW and Low Activity Waste (LAW) glass processing are lined with high Cr_2O_3 containing K-3 refractory [4]. The refractory is reducing, and the chromium is present primarily as Cr^{+3} oxide, e.g. Cr_2O_3 [4]. In the DWPF, failed glass melters are to be stored in the Failed Equipment Storage Vaults (FESV). The EPA, and therefore the DWPF, were concerned as to whether the ceramic refractory lining in a failed glass melter might contain hazardous concentrations of residual hexavalent chromium and mercury. In the January, 1993, the DWPF requested that SRTC perform TCLP testing on K-3 refractory from a failed 1/100th scale DWPF mini-melter in order to determine whether failed melters could be stored in the FESV as non-RCRA hazardous material. TCLP testing was performed and the K-3 refractory was shown not be RCRA hazardous. Recently, GTS Duratek performed TCLP testing on K-3 refractory which was in contact with a mixed waste glass being processed in the Savannah River Site M-Area facility. The GTS Duratek data was confirmatory of the TCLP testing performed on the failed mini-melter by SRTC.

Since 1993 additional TCLP testing has been performed on simulated DWPF glass made on both the laboratory scale and full scale in the DWPF during startup and checkout. This data confirmed the earlier TCLP testing of the simulated waste glass. The current study documents here-to-fore unpublished TCLP testing performed on simulated HLW glass by SRTC from 1990 to present. This includes testing of glasses made with both oxidizing and reducing flowsheets and testing of the DWPF Environmental Assessment benchmark glass [5]. This report also summarizes the SRTC and GTS Duratek TCLP testing of the high Cr₂O₃ containing K-3 refractory. This report includes the analytic detection limits not cited in the previous reports and indicates whether an EPA certified laboratory conducted the testing.

REGULATORY BACKGROUND

The U. S. Environmental Protection Agency (EPA) revised the Resource Conservation and Recovery Act (RCRA) Phase IV Land Disposal Restrictions (LDR's) on May 26, 1998 which requires characteristically hazardous wastes be treated to meet the LDR Universal Treatment Standards (UTS) prior to land disposal. The revised RCRA LDR's historically evolved from treatment standards for listed hazardous wastes that were initially proposed on June 1, 1990 [3]. The treatment standards for ignitable and corrosive hazardous wastes were revised on May 24, 1993 when an interim final rule was issued. The May, 1993 rule added the definition of additional hazardous species designated as "underlying hazardous constituents," e.g. constituents whose concentration exceeds the treatment standard [6] to the 1990 regulatory constraints. This rule also stated that prior to disposal as a non-hazardous waste, a waste must be treated to remove the hazardous characteristic, and that any underlying hazardous constituents - as well as the individual constituent(s) responsible for the hazardous waste designation - must also be treated to the concentration based standards. In 1994 the EPA formalized Universal Treatment Standards (UTS), which is a list of concentration limits to which listed hazardous wastes must be treated [7]. The 1994 rule also indicated that all the characteristically hazardous waste constituents, except for the RCRA metals As, Ba, Cd, Cr, Pb, Hg, Se, and Ag must be treated to the UTS.

In 1996 and 1997 the Environmental Protection Agency proposed revised Phase IV Land Disposal Restrictions (LDR) [8]. These LDR's were finalized May 26, 1998 [9]. The final Phase IV LDR's require that any waste which is shown to be, or declared to be, characteristically hazardous for the Resource Conservation and Recovery Act (RCRA) metals As, Ba, Cd, Cr, Pb, Hg, Se, and Ag will have to be treated to meet the LDR Universal Treatment Standards (UTS) prior to land disposal. In addition, if the waste is characteristically hazardous and contains underlying hazardous constituents, those constituents will also have to be treated to meet the UTS.

The May, 1998 regulations have not specifically discussed or addressed the treatment standards for radioactive high level waste generated during the reprocessing of fuel rods, which are hazardous for one (or more) of the RCRA metals. These wastes were defined to meet the LDR's if they are treated by High Level Vitrification (HLVIT), a specified treatment technology [1]. The most recent South Carolina Hazardous Waste Management Regulations, June 27, 1997, states that "a waste identified in the table entitled Treatment Standards for Hazardous Wastes, may be land disposed only if it meets the requirements found in Table 268.40 (R.61-79.268

Subpart D, Treatment Standards).¹ The treatment standard identified in the table for High Level Waste containing the above mentioned RCRA metals is “HLVIT,” not a specific Toxic Characteristic Leaching Procedure (TCLP) leachate concentration value. This allows HLW wastes, to be disposed to a surface disposal facility, after it has been treated by HLVIT.²

DWPF VITRIFIED PRODUCT TCLP RESULTS

The DWPF is exempted from the UTS standards, since the waste sludge is being treated by a specified technology, e.g. HLVIT, which has been declared by EPA to be BDAT. If this were not so, and if any of the constituents listed in Table I failed the UTS limit in the original waste, then those constituents would be defined as “underlying hazardous constituents” and the DWPF would have to meet the new UTS for each of those constituents in the final vitrified waste form - rather than the characteristic TCLP limits against which the DWPF glass was originally assessed [2]. This scenario could someday apply as EPA regulations become more stringent and/or if the state of Nevada imposes additional constraints on the DWPF product.³

In the 1990-1994 time frame, the Defense Waste Processing Facility (DWPF) was able to maintain that Cr^{+6} would not be released from DWPF glass, since chromium speciation at the vitrification temperature of 1150°C is primarily present as Cr^{+3} , (providing that the measured glass redox, as indicated by the measured $\text{Fe}^{+2}/\Sigma\text{Fe}$ is between 0.09 and 0.33).⁴ The DWPF redox limits of 0.09 to 0.33 $\text{Fe}^{+2}/\Sigma\text{Fe}$ were set in 1986 [12] to minimize foaming in the melter and eliminate the formation of metallic species and sulfides in the melt which could short out the electrodes in the Joule heated DWPF melter.

Currently, the DWPF operates with a flowsheet that adds considerable amounts of nitrate, (a strong oxidant) to the melt pool. The glasses produced have measured $\text{Fe}^{+2}/\Sigma\text{Fe}$ redox ratios of <0.09. Since these redox ratios are near to the detection limit of the redox measurement technique [13], the lower redox limit of 0.09 $\text{Fe}^{+2}/\Sigma\text{Fe}$ was relaxed for DWPF operations. DWPF

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- 1 The table entitled “Treatment Standards for Hazardous Wastes” in 268.40 R.61-79.268 Subpart D supercedes the treatment standards that hertofore appeared in tables in 268.41, 268.42 and 268.43 per a major revision of the regulations in May, 1996.
 - 2 Treatment by HLVIT does not, in itself, determine that the treated waste is no longer hazardous, or demonstrate that the treated waste meets the UTS. The Nuclear Regulatory Commission and the EPA recently formatlized guidance on the testing of mixed (radioactive and hazardous wastes [10]. This guidance is primarilly concerned with Low Level Waste (LLW), but the guidance should also be applicable to High Level Waste (HLW) The guidance [10] clarifies that both process knowledge, and/or surrogate testing, can be used in place of testing the actual treated radioactive waste.
 - 3 A state with RCRA purview to enact more stringent regulations than the Federal regulations
 - 4 Based on experimental data generated at the Virginia Military Institute (VMI) under subcontract to the Savannah River Technology Center an Electro Motive Force (EMF) series was developed [11] which determined the redox speciation of all of the redox sensitive elements in DWPF glass, e.g. $\text{Cr}^{+3}/\text{Cr}^{+6}$, $\text{Ni}^{\circ}/\text{Ni}^{+2}$, $\text{Pb}^{\circ}/\text{Pb}^{+2}$, $\text{Mn}^{+3}/\text{Mn}^{+4}$, $\text{Mn}^{+2}/\text{Mn}^{+3}$, etc., vs. the $\text{Fe}^{+2}/\text{Fe}^{+3}$ or $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratios . Maintaining the DWPF redox in the 0.09-0.33 range insures that >97% of the chromium present in the glass is present as Cr^{+3} .

operation below the lower redox limit of $0.09 \text{ Fe}^{+2}/\Sigma\text{Fe}$ will allow additional Cr^{+6} to form. The DWPF Qualification Runs and Proficiency Runs were performed with the oxidizing flowsheet.

In order to ascertain whether the total Cr in the DWPF glasses is being released at an elevated concentration from the glass poured using the oxidizing flowsheet, a comparison was made of TCLP tests for DWPF glasses poured under reference conditions [2] (oxygen fugacities between 10^{-2} and 10^{-7} atmospheres corresponding to $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratios of 0.09 to 0.33) and those poured during Proficiency Run 1 (Canisters S000134 and S000144; oxygen fugacities $<10^{-2}$ atmospheres corresponding to $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratios of <0.09 with the DWPF oxidized flowsheet (Table I).

The glasses poured under reference conditions passed both the current and proposed EPA/TCLP/LDR/UTS regulations, e.g. <5 ppm total Cr to meet the toxicity limit, or <0.60 ppm total Cr to meet the UTS (Table I). The glasses poured during Waste Qualification Runs with the oxidized flowsheet demonstrated a 0.0154 ppm total Cr release during TCLP testing (Table I) indicating that the use of the strong oxidant is not altering the Cr oxidation state or the TCLP response.

All of the DWPF glasses tested, including the Environmental Assessment (EA) glass and canistered simulated DWPF glass from Proficiency Run 1 (Canisters S00134 and S00144) indicates the DWPF glass is capable of meeting all of the current EPA regulations even though this is not required.

DWPF MELTER REFRACTORY

At the DWPF, failed glass melter are to be stored in the Failed Equipment Storage Vaults (FESV). The ceramic refractory lining in a failed glass melter may contain hazardous concentrations of residual hexavalent chromium and mercury. In 1993, refractory was taken from the 774-A mini-melter near the melt line where maximum oxidation of Cr^{+3} in the refractory to Cr^{+6} would have occurred. The SRTC provided a report to DWPF which listed the concentration of the RCRA contaminants in the TCLP leachates from the melter refractory. The TCLP testing and leachate analyses were completed at SRTC by the Analytic Development Section (ADS). The report cited many of the leachate concentrations as Below Detection Limit (BDL) but the analytical detection limits were not provided. In addition, the data generated on the leaching of Ni was not given. The original data, including the data for Ni are reproduced in Table II below. The SRTC data indicate that the K-3 refractory is not characteristically hazardous for all constituents.

In 1994 another set of refractory samples were collected from the 774-A mini-melter after the melter had processed Batch 1 feed/glass from Facilities Acceptance testing (FA-10.02), and high iron containing (Purex 4 and 5) feeds from the Integrated DWPF Melter System (IDMS), n-methyl pyrrolidone, and feeds with copper nitrate catalyst. These feeds were highly oxidizing in nature. The refractory samples were sent to RUST Remedial Services in Clemson, South Carolina, an South Carolina EPA certified laboratory. The report cited many of the leachate concentrations as Below Detection Limit (BDL) but the detection limits were not provided. The original data were unobtainable for usage in this study. The data for Cr and Pb generated by RUST Remedial Services indicate that the K-3 refractory is not characteristically hazardous, e.g. the Cr and Pb releases are <5 mg/L. This confirms the 1993 data generated by SRTC of a failed

melter that had produced only reducing feeds. Therefore, there was no effect of the oxidizing type feeds on the refractory Cr oxidation state and the TCLP response.

In 1997, data was provided by GTS Duratek, a subcontractor operating a Joule heated melter at 5 tons glass/day (nominal production rate) in the SRS M-Area, which is also lined with K-3 refractory. This melter processed high nitrate containing feeds for ~ 6 months before the melter was shut down for design modifications. TCLP analysis of K-3 refractory core drilled from the bottom of the melter at the K-3/glass interface by General Engineering Laboratory in Charleston, SC (a SC certified laboratory) confirmed the previous SRTC and RUST Remedial Services results, i.e.; that the K-3 refractory is not characteristically hazardous, even after vitrification of highly oxidized feeds.

**Table I
Compilation of DWPF TCLP Test Results for Simulated HLW Waste Glass**

Element	RCRA Universal Treatment Standards (UTS) Limits	RCRA Toxicity Limits (ppm)	TCLP Response [‡] of DWPF glass made under reference redox conditions	EP-Tox Response [‡] of DWPF glass made under reference redox conditions	TCLP Response [#] of DWPF glass made under reference redox conditions (Batch 1 Glass) ^{††}	TCLP Response [#] of DWPF glass made under reference redox conditions (Batch 2 Glass) [†]	TCLP Response [#] of DWPF glass made under reference redox conditions (Batch 3 Glass) [†]	TCLP Response [#] of DWPF glass made under reference redox conditions (Batch 4 Glass) [†]	TCLP Response [#] of DWPF glass made under reference redox conditions (Blend Glass) [†]	TCLP Response [#] of DWPF glass made under reference redox conditions (HM Glass) [†]	TCLP Response [#] of DWPF glass made under reference redox conditions (Purex Glass) [†]	TCLP Response [#] of DWPF glass made under reference redox condition (EA Glass) ^{*§}	DWPF Canister Glasses S00134 and S00144 made under oxidizing redox conditions
	[8] mg/L	[3] mg/L	[2] mg/L	[2] mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
As	5.0	5.0	<5	<0.5	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.11 [§]
Ba	21	100	<5	<0.005	0.32 0.31	0.32 0.24	<0.20 0.38	0.36 0.74	0.32 0.28	<0.20 0.8	0.65 0.76	0.42 0.56	0.213
Cd	0.11	1	<1	<0.02	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.02 [§] <0.02 [§]	<0.002 [§]
Cr	0.60	5	<5	<0.05	<0.09 [§] <0.09 [§]	<0.09 [§] <0.09 [§]	<0.09 [§] <0.09 [§]	<0.09 [§] <0.09 [§]	<0.09 [§] <0.09 [§]	<0.09 [§] <0.09 [§]	<0.09 [§] <0.09 [§]	<0.09 [§] <0.09 [§]	0.0154
Pb	0.75	5	<5	<0.5	<0.14 [§] <0.14 [§]	<0.09 [§] <0.14 [§]	<0.14 [§] <0.14 [§]	<0.14 [§] <0.14 [§]	<0.14 [§] <0.14 [§]	<0.14 [§] <0.14 [§]	<0.14 [§] <0.14 [§]	<0.14 [§] 0.21	<0.056 [§]
Hg	0.025	0.2	Not tested*	Not tested*	<0.001 [§] <0.001 [§]	<0.001 [§] <0.001 [§]	<0.001 [§] <0.001 [§]	<0.001 [§] <0.001 [§]	<0.001 [§] <0.001 [§]	<0.001 [§] <0.001 [§]	<0.001 [§] <0.001 [§]	<0.001 [§] <0.001 [§]	<0.00007 [§]
Ni	11	---	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested
Se	5.7	1	<1	<0.5	<0.01 [§] <0.01 [§]	<0.01 [§] <0.01 [§]	<0.01 [§] <0.01 [§]	<0.01 [§] <0.01 [§]	<0.01 [§] <0.01 [§]	<0.01 [§] <0.01 [§]	<0.01 [§] <0.01 [§]	<0.01 [§] <0.01 [§]	<0.096 [§]
Ag	0.14	5	<1	<0.1	<0.05 [§] <0.05 [§]	<0.05 [§] <0.05 [§]	<0.05 [§] <0.05 [§]	<0.05 [§] <0.05 [§]	<0.05 [§] <0.05 [§]	<0.05 [§] <0.05 [§]	<0.05 [§] <0.05 [§]	<0.05 [§] <0.05 [§]	0.0051 [§]
Zn ††	4.3	---	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested

- * removed from waste prior to vitrification
- ‡ data generated from SC certified laboratory (Environmental & Chemical Science, Inc., ECS, New Ellenton, SC)
- †† not an “underlying hazardous constituent”
- † data generated from EPA certified laboratory (Weston, Lionville, PA)
- § number given is the analytical detection limit; concentrations measured were below the detection limit of the instrumentation employed
- # data generated from SC certified laboratory (Rust Remedial Services, Inc.)

†† glasses from the DWPF Waste Compliance Plan (WCP) representative of the range of glass compositions to be processed in the DWPF, the first TCLP release is for glasses which were doped at the DWPF reference levels of the RCRA hazardous constituents, the second TCLP release is for glasses doped at 3X the DWPF reference levels of the RCRA hazardous constituents

** DWPF Environmental Assessment Glass [5]

Table II.

Compilation of DWPF and GTS Duratek TCLP Test Results for K-3 High Cr₂O₃ Containing Refractory

Element	RCRA Universal Treatment Standards (UTS) Limits [8] mg/L	RCRA Toxicity Limits [3] mg/L	TCLP Response of DWPF K-3 Refractory†† (Replicate A) mg/L	TCLP Response of DWPF K-3 Refractory†† (Replicate A) mg/L	TCLP Response of DWPF K-3 Refractory (side of meter with adhering glass) mg/L	TCLP Response of DWPF K-3 Refractory (sidewall, no adhering glass) mg/L	TCLP Response of DWPF K-3 Refractory (bottom, replicate 1) mg/L	TCLP Response of DWPF K-3 Refractory (bottom, replicate 2) mg/L	TCLP Response of K-3 Refractory from bottom of Duratek production melter mg/L
As	5.0	5.0	<0.20 [§]	<0.20 [§]	bdl	bdl	bdl	bdl	Not tested
Ba	21	100	13.002 13.364 [†]	7.044 8.246 [†]	bdl	bdl	bdl	bdl	Not tested
Cd	0.11	1	<0.010 [§] <0.010 ^{§†}	<0.010 [§] <0.010 ^{§†}	bdl	bdl	bdl	bdl	Not tested
Cr	0.60	5	0.900 0.984 [†]	0.466 0.560 [†]	0.145	0.233	0.140	0.192	0.391 [‡]
Pb	0.75	5	<0.200 [§] 0.225 [†]	<0.200 [§] <0.200 ^{§†}	bdl	bdl	0.153	bdl	<0.0013 [‡]
Hg *	0.025	0.2	0.0160	0.0128	bdl	bdl	bdl	bdl	Not tested
Ni **	11	---	Not Analyzed 0.128 [†]	Not Analyzed 0.114 [†]	Not tested	Not tested	Not tested	Not tested	1.09 [‡]
Se	5.7	1	0.017	-0.229	bdl	bdl	bdl	bdl	Not tested
Ag	0.14	5	<0.020 [§]	<0.020 [§]	bdl	bdl	bdl	bdl	Not tested
Zn	4.3	---	Not Analyzed 5.947 [†]	Not Analyzed 5.169 [†]	Not tested	Not tested	Not tested	Not tested	Not tested

* removed from waste prior to vitrification and not present in the DWPF K-3 refractory (see Reference 4 and 5)

** not present in DWPF K-3 refractory (see Reference 4)

† data generated from standard Inductively Coupled Plasma (ICP) Spectroscopy scan versus the corresponding number above which was generated from an ICP scan for RCRA metals only

§ number given is the analytical detection limit; concentrations measured were below the detection limit of the instrumentation employed

‡ personnel communication, from Innocent Joseph of Dratek to J.B. Pickett, "Results of TCLP Analysis on K-E and AZS Refractories from the M-Area Melter," on August 13,

CONCLUSIONS

A compilation of all the available Toxicity Characteristic Leaching Procedure (TCLP) data from glasses made over the full range of DWPF operating conditions (oxidized and reduced flowsheets) and over the full range of DWPF waste compositions indicates that the DWPF glasses are extremely durable and leach RCRA hazardous constituents at less than the 1998 UTS limits when tested with the EPA TCLP test. Data is given for the DWPF Waste Compliance Plan (WCP) glasses and the DWPF Environmental Assessment (EA) glass.

A review of the available TCLP data from high Cr₂O₃ containing K-3 refractory tested after processing of both reduced and oxidized wastes indicates that the K-3 refractory will not be characteristically hazardous and, therefore, does not require additional treatment to the LDR UTS standards.

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