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2	1	Cog. Mgr. J.W. Winters	<i>[Signature]</i>	7/2/96	R2-12	J.B. Schaffer	<i>[Signature]</i>	6-13-96		1	1
		QA				W.I. Winters	<i>[Signature]</i>	6/5/96	76-30	1	1
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1	1	R.F. Eggers	<i>[Signature]</i>	7/2/96							
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Technical Basis and Spreadsheet Documentation for Correcting Waste Tank Core Samples for Water Intrusion Based on a LiBr Tracer

Wayne D. Winkelman

Westinghouse Hanford Company, Richland, WA 99352
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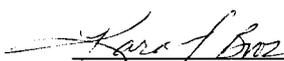
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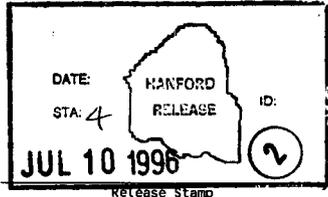
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Technical Basis and Spreadsheet Documentation for Correcting Waste Tank Core Samples for Water Intrusion Based on a LiBr Tracer

W. D. Winkelman
Westinghouse Hanford Company

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Westinghouse
Hanford Company

P.O. Box 1970
Richland, Washington

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LIST OF ACRONYMS

DSC	Differential Scanning Calorimetry
HHF	Hydrostatic Head Fluid
IC	Ion Chromatography
ICP	Inductive Coupled Plasma
OTC	Onsite Transport Cask
RPD	Relative Percent Difference
TGA	Thermogravimetric Analysis
TSAP	Tank Sample Analysis Plan
TWRS	Tank Waste Remediation System

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Westinghouse Hanford requires that software used to calculate parameters and base decisions on be documented. That is the intent of this document. Information in this document is from the former work of Dave Bechtold (Bechtold 1995), Dan Reynolds, and Robert Eggers. The ideas and concepts developed in this prior work are integrated into a tool that can be used by the Tank Waste Characterization project coordinators.

1.0 INTRODUCTION

The Tank Waste Characterization Project is responsible for providing all Tank Waste Remediation System (TWRS) programs information describing the physical, chemical, and radiological properties of the contents of the 177 waste storage tanks at the Hanford Site. These tanks contain radioactive waste generated from the production of nuclear materials throughout the Hanford Site's history. The method for providing this information is to remove samples from the tanks and analyze those samples for the appropriate chemical and physical properties of interest. Currently three sampling methods are used at the Hanford Site. These are referred to as grab, auger, and core. Grab sampling removes samples by lowering a bottle into the tank and allowing it to fill with tank material which is then removed. Auger samples are taken by screwing an auger type device into the waste form. The auger is then removed and the waste that has been retained on the auger flutes is analyzed. Core sampling is capable of removing vertically oriented cylindrical "core" of waste material from the tank. The core is taken in 19-inch segments and by using sufficient segments, a full-depth profile of the tank can be achieved. The sampling process sometimes requires the use of LiBr traced water which has the potential for contaminating the sample. This document focuses on how the characterization process detects when the sample has been contaminated with this fluid, how the impact of the contamination can be compensated for or, when the contamination is sufficient to destroy the integrity of the sample.

2.0 CORE SAMPLING PROCESS

Currently four sampling trucks can be used to obtain waste tank core samples. The trucks are equipped with commercial drilling platforms. The drill assembly hydraulically lowers the drill rod assembly into the waste with a core sampler latched inside the bottom of the drill rod assembly. A downward 19-inch stroke loads the sampler with waste material. The hydrostatic head of the drill string is adjusted to prevent waste from entering the drill string. Then the drill assembly and the shielded receiver, which are mounted on a rotating platform, are rotated 180° to position the shielded receiver above the drill rods for sampler retrieval. The sampler is pulled into the receiver and the platform is then rotated with shielded receiver and positioned above the onsite transport cask (OTC). The sampler is then lowered into the OTC for shipment to the laboratory. A clean sampler is retrieved from another OTC, the truck platform is rotated to lower the next sampler into the drill rod assembly, and the process is repeated.

Samples can be taken in either rotary mode or push mode. In rotary mode a cutting bit is attached to the bottom of the drill rod assembly and the drill rod assembly is rotated as it is pushed into the waste. This process cuts a core sample out of the waste and draws it into the sampler. Push mode simply pushes the drill string into the waste using a tapered push bit. The waste is subsequently pushed into the sampler.

The four trucks use either nitrogen gas or 0.3 Molar LiBr traced water to balance the hydrostatic head of the waste. Core sample truck #1 cannot use nitrogen purge gas to cool the bit and clean the hole during rotary sampling. It is therefore used exclusively for push-mode sampling. In addition, it is used exclusively with LiBr solution as a hydrostatic head fluid (HHF). Trucks 2, 3, and 4 use nitrogen to balance the hydrostatic head but LiBr traced water may still be used, and potentially contaminate the sample, to unplug or unstick a drill bit.

Rotary-Mode Sampler: The rotary-mode sampler was designed to obtain saltcake, sludge, and liquid samples. It is used in both push and rotary mode core sampling. The sampler is loaded into the drill string and either pushed or rotated through the waste using the commercial drill rig. This sampler can obtain a complete profile of tank waste in 19-inch (or less) segments. The maximum volume is 300 mL for liquids and sludges and 245 mL for saltcake samples. At the discretion of the sampling crew cognizant engineer, several gallons of LiBr traced water can be poured into the drill string. This is usually the case when a plugged drill bit is the suspected cause of poor recovery. Softening of the waste at the bottom of the drill string so that push-mode operations can be accomplished is also a possibility.

Push-mode Sampler: The push-mode sampler is a modified version of the rotary mode sampler used in push-mode core sampling. When the sampler is removed from the drill string, the HHF can flow into the tank out the bottom of the drill string. This will happen until the pressure of the HHF at the end of the drill string equals the head pressure of the tank at that

elevation. When the new sampler is lowered and latched into position at the bottom of the drill string, HHF located in the bit can be forced into the tank.

Once the samplers are removed from the tank, they are transported to the laboratory for analysis. In the laboratory, samples are removed from the shipping cask and inserted into the hot cell. Core samplers are loaded into the extruder where the sample is removed by using a piston to push it out of the sampler and onto a sample tray.

Any drainable liquid is separated and the volume measured. The liquids are retained for further processing. Each sample is divided and homogenized according to the tank sample analysis plan (TSAP). Homogenization is performed so that aliquots removed for analysis will represent the subsegment or composite. Aliquots of the solid portion of the sample material are taken in duplicate to do the required analysis. Any liquid portion of the sample is handled separately.

Moisture (water wt%) is used to correct the differential scanning calorimetry (DSC) estimate of fuel content to a dry weight basis. Thermogravimetric Analysis (TGA) is the method used to make this measurement. TGA measures the changes in sample weight as the sample is heated from 35° to 500 °C at a constant rate. The percentage weight loss evaporated off the sample (up to about 200 °C) is calculated from the thermogram, and assumed to be primarily water. Gravimetric methods may also be used to estimate the moisture content of the waste.

For cases where contamination of the sample was possible either by a hydrostatic head fluid (HHF) or the need to unblock/unstick a drill bit, Li and Br analyses are done. Li is measured by inductive coupled plasma (ICP) analysis and Br is measured by ion chromatography (IC). In addition a sample of the HHF used during the sampling event is shipped to the laboratory and also analyzed for Li and Br concentrations.

3.0 CORRECTING FOR CONTAMINATION OF LiBr TRACED WATER

If either Li or Br is detected in the sample then the sample has been contaminated by water traced with LiBr during the sampling operation. The amount of intrusion needs to be calculated so that the system guidance can be applied. If less than 10% of the water present is from HHF, no correction needs to be performed. If greater than 50% of the water present is from HHF, then the sample must be considered suspect.

The calculation to determine the amount of water from HHF is based on the LiBr tracer that is placed in the water used for sampling. From analyzing the concentration of Li and Br in the HHF sample and the concentration of Li or Br in the waste sample, the amount of water in the waste sample due to HHF can be computed.

To simplify the equation derivations, the following nomenclature will be used.

Primary Descriptors

Wt=Weight
C=Concentration
Wt%=Weight percent
Wt_f=Weight fraction
ρ=Density
Mwt=Molecular Weight

Subscripts

s=sample
w=water
L=Lithium
B=Bromide
t=tracer (Li or Br)
h=hydrostatic head fluid
c=corrected value
a=actual value of tank contents

The weight percent water which is measured by TGA or gravimetry is the weight of water divided by the weight of the sample. The equation would read as: The weight fraction of the sample water is equation to the weight of the sample water divided by the weight of the sample.

$$Wt_{f_{sw}} = \frac{Wt_{sw}}{Wt_s} \tag{1}$$

To correct for the influence of the contamination we must subtract the contribution of water from the numerator and the total weight of the water and tracer from the denominator. Therefore the weight fraction of the corrected sample water is equal to the weight of the sample water minus the weight of the HHF water and divided by the weight of the sample minus the weight of the HHF.

$$Wf_{csw} = \frac{Wt_{sw} - Wt_{hw}}{Wt_s - Wt_h} \quad (2)$$

The weight of the sample water (w_w) can be easily calculated by multiplying the weight of sample(s) by the percent water measurement made by TGA.

$$Wt_{sw}(g) = Wt_s(g) * \frac{Wt\%_{sw}}{100} \quad (3)$$

To calculate the weight of the water in the sample contributed by HHF, a standard mass balance equation is used for a solid or liquid sample. The concentration of the tracer measured in the sample multiplied by the weight of the sample should be equal to the concentration of the tracer in the tank waste times the weight of the tank waste material plus the concentration of the tracer in the HHF time the weight of the hhf divided by the density. For liquid samples, the density of the sample and waste is also used for the balance.

$$\text{Solids} \quad \frac{C_{ts} \left(\frac{\mu\text{g}}{\text{g}} \right) * Wt_s(g)}{10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)} = \frac{C_{ta} \left(\frac{\mu\text{g}}{\text{g}} \right) * Wt_a(g)}{10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)} + \frac{C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right) * Wt_h(g)}{\rho_h \left(\frac{\text{g}}{\text{mL}} \right) * 10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)} \quad (4)$$

$$\text{Liquids} \quad \frac{C_{ts} \left(\frac{\mu\text{g}}{\text{mL}} \right) * Wt_s(g)}{\rho_s \left(\frac{\text{g}}{\text{mL}} \right) * 10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)} = \frac{C_{ta} \left(\frac{\mu\text{g}}{\text{mL}} \right) * Wt_a(g)}{\rho_a \left(\frac{\text{g}}{\text{mL}} \right) * 10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)} + \frac{C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right) * Wt_h(g)}{\rho_h \left(\frac{\text{g}}{\text{mL}} \right) * 10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)} \quad (5)$$

LiBr was used as a tracer because these elements are not expected to be found in the waste. Therefore, if we make C_u zero and solve for the weight of the HHF we get the following.

Solids	Liquids
$Wt_h(g) = \frac{C_{ts} \left(\frac{\mu g}{g} \right) * Wt_s(g) * \rho_h \left(\frac{g}{mL} \right)}{C_{th} \left(\frac{\mu g}{mL} \right)}$	$Wt_h(g) = \frac{C_{ts} \left(\frac{\mu g}{mL} \right) * Wt_s(g) * \rho_h \left(\frac{g}{mL} \right)}{C_{th} \left(\frac{\mu g}{mL} \right) * \rho_s \left(\frac{g}{mL} \right)} \quad (6)$

The same derivation can be performed to calculate the Weight of water contributed by the HHF. Instead of the density of the HHF we use the concentration of water in the HHF.

Solids	Liquids
$Wt_{wh}(g) = \frac{C_{ts} \left(\frac{\mu g}{g} \right) * Wt_s(g) * C_{wh} \left(\frac{g}{mL} \right)}{C_{th} \left(\frac{\mu g}{mL} \right)}$	$Wt_{wh}(g) = \frac{C_{ts} \left(\frac{\mu g}{mL} \right) * Wt_s(g) * C_{wh} \left(\frac{g}{mL} \right)}{C_{th} \left(\frac{\mu g}{mL} \right) * \rho_s \left(\frac{g}{mL} \right)} \quad (7)$

We now have all of the data required to calculate the correction to the water concentration in the sample except for the density of the HHF and the concentration of water in the HHF. These numbers can be derived from the analysis of the HHF sample which is sent to the Laboratory from the field and analyzed for Li and Br concentrations. The density of this mixture is calculated by using numbers in the Handbook of Chemistry (Lange 1967).

The first two columns are copied from the reference. The other columns are calculated using the following equations.

$$Mwt_L = 6.939 \left(\frac{\text{g}}{\text{mole}} \right) \quad Mwt_B = 79.909 \left(\frac{\text{g}}{\text{mole}} \right) \quad Mwt_{LB} = Mwt_L + Mwt_B$$

$$Molarity_{LB} \left(\frac{\text{mole}}{\text{L}} \right) = \frac{Wt\%_{LB}}{100} * \rho_s \left(\frac{\text{g}}{\text{mL}} \right) * \frac{1000 \left(\frac{\text{mL}}{\text{L}} \right)}{Mwt_{LB} \left(\frac{\text{g}}{\text{mole}} \right)}$$

$$C_{LB} \left(\frac{\text{g}}{\text{mL}} \right) = \frac{Molarity_{LB} \left(\frac{\text{mole}}{\text{L}} \right) * Mwt_{LB} \left(\frac{\text{g}}{\text{mole}} \right)}{1000 \left(\frac{\text{mL}}{\text{L}} \right)} \quad (8)$$

$$C_L \left(\frac{\mu\text{g}}{\text{mL}} \right) = \frac{Molarity_{LB} \left(\frac{\text{mole}}{\text{L}} \right) * Mwt_L \left(\frac{\text{g}}{\text{mole}} \right) * 10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)}{1000 \left(\frac{\text{mL}}{\text{L}} \right)}$$

$$C_B \left(\frac{\mu\text{g}}{\text{mL}} \right) = \frac{Molarity_{LB} \left(\frac{\text{mole}}{\text{L}} \right) * Mwt_B \left(\frac{\text{g}}{\text{mole}} \right) * 10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)}{1000 \left(\frac{\text{mL}}{\text{L}} \right)}$$

Concentration of Br and Li related to HHF Density					
Wt. % _{LB} (Tabulated)	ρ_r @ 20C (g/ml) tabulated	Molarity	C_{LB} (g/ml)	C_B (ug/ml)	C_L (ug/ml)
0.0	0.9982	0.0000	0.0000	0.00	0.00
1.0	1.0055	0.1158	0.0101	9,251.62	803.38
2.0	1.0128	0.2332	0.0203	18,637.58	1,618.42
4.0	1.0277	0.4733	0.0411	37,823.54	3,284.46
6.0	1.0429	0.7205	0.0626	57,574.45	4,999.55
8.0	1.0585	0.9750	0.0847	77,914.22	6,765.78
10.0	1.0746	1.2373	0.1075	98,874.14	8,585.86
12.0	1.0910	1.5075	0.1309	120,459.73	10,460.27
14.0	1.1079	1.7859	0.1551	142,713.31	12,392.69
16.0	1.1253	2.0731	0.1800	165,662.49	14,385.51
18.0	1.1432	2.3694	0.2058	189,334.87	16,441.13
20.0	1.1616	2.6750	0.2323	213,758.05	18,561.95
22.0	1.1806	2.9907	0.2597	238,979.88	20,752.12
24.0	1.2002	3.3167	0.2880	265,033.48	23,014.52
30.0	1.2629	4.3624	0.3789	348,598.96	30,271.04
35.0	1.3204	5.3213	0.4621	425,215.84	36,924.16
40.0	1.3836	6.3725	0.5534	509,221.13	44,218.87
45.0	1.4535	7.5313	0.6541	601,815.58	52,259.42

If we fit a quadratic equation to the density of the LiBr solution using the concentration of Li or Br as the operand we get two equations by which we can calculate the density of the HHF from the concentration of either Li or Br. The concentration of the tracer in $\mu\text{g/mL}$ was used so that conversion from the measured units would not be necessary. The curve fit was performed so that the output would be in g/mL and consistent with other density measurement units.

$$\rho_h \left(\frac{\text{g}}{\text{mL}} \right) = .9993 + 8.728 * 10^{-6} * C_L \left(\frac{\mu\text{g}}{\text{mL}} \right) - 1.559 * 10^{-12} * \left(C_L \left(\frac{\mu\text{g}}{\text{mL}} \right) \right)^2 \quad (9)$$

$$\rho_h \left(\frac{\text{g}}{\text{mL}} \right) = .9993 + 7.579 * 10^{-7} * C_B \left(\frac{\mu\text{g}}{\text{mL}} \right) - 1.175 * 10^{-14} * \left(C_B \left(\frac{\mu\text{g}}{\text{mL}} \right) \right)^2 \quad (10)$$

The concentration of water in the HHF can likewise be calculated by subtracting the concentration of LiBr from the density of the HHF. The concentration of LiBr can be calculated either from the Li analysis or from the Br analysis. The concentration is calculated in g/m : so that it can be directly subtracted from the density measurements.

$$\text{Lithium Analysis} \quad C_{LBh} \left(\frac{\text{g}}{\text{mL}} \right) = \frac{C_{Lh} \left(\frac{\mu\text{g}}{\text{mL}} \right)}{Mwt_L \left(\frac{\text{g}}{\text{mole}} \right)} * \frac{Mwt_{LB} \left(\frac{\text{g}}{\text{mole}} \right)}{10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)} \quad (11)$$

$$\text{Bromine Analysis} \quad C_{LBh} \left(\frac{\text{g}}{\text{mL}} \right) = \frac{C_{Bh} \left(\frac{\mu\text{g}}{\text{mL}} \right)}{Mwt_B \left(\frac{\text{g}}{\text{mole}} \right)} * \frac{Mwt_{LB} \left(\frac{\text{g}}{\text{mole}} \right)}{10^6 \left(\frac{\mu\text{g}}{\text{g}} \right)} \quad (12)$$

$$C_{wh} \left(\frac{\text{g}}{\text{mL}} \right) = \rho_h \left(\frac{\text{g}}{\text{mL}} \right) - C_{LBh} \left(\frac{\text{g}}{\text{mL}} \right) \quad (13)$$

Now if we refer back to equation 2 and substitute in the derived equations for each of the unknowns we get the following. The density of HHF and the concentration of water in the HHF are left unexpanded to simplify the equation.

$$\text{Solid Samples} \quad Wt\%_{wc} = \frac{Wt_s(g) * \frac{Wt\%_w}{100} - \frac{C_{iw} \left(\frac{\mu g}{g} \right) * Wt_s(g) * C_{wh} \left(\frac{g}{mL} \right)}{C_{ih} \left(\frac{\mu g}{mL} \right)}}{Wt_s(g) - \frac{C_{is} \left(\frac{\mu g}{g} \right) * Wt_s(g) * \rho_h \left(\frac{g}{mL} \right)}{C_{ih} \left(\frac{\mu g}{mL} \right)}} * 100 \quad (14)$$

$$\text{Liquid Samples} \quad Wt\%_{wc} = \frac{Wt_s(g) * \frac{Wt\%_w}{100} - \frac{C_{iw} \left(\frac{\mu g}{mL} \right) * Wt_s(g) * C_{wh} \left(\frac{g}{mL} \right)}{\rho_s \left(\frac{g}{mL} \right) * C_{ih} \left(\frac{\mu g}{mL} \right)}}{Wt_s(g) - \frac{C_{is} \left(\frac{\mu g}{mL} \right) * Wt_s(g) * \rho_h \left(\frac{g}{mL} \right)}{\rho_s \left(\frac{g}{mL} \right) * C_{ih} \left(\frac{\mu g}{mL} \right)}} * 100 \quad (15)$$

Simplification and term cancellation yields the following equations used to calculate the original weight percent water in the waste.

$$\text{Solid Samples} \quad Wt\%_{wc} = \frac{\frac{Wt\%_s}{100} * C_{ih} \left(\frac{\mu g}{mL} \right) - C_{is} \left(\frac{\mu g}{g} \right) * C_{wh} \left(\frac{g}{mL} \right)}{C_{ih} \left(\frac{\mu g}{mL} \right) - C_{is} \left(\frac{\mu g}{g} \right) * \rho_h \left(\frac{g}{mL} \right)} * 100 \quad (16)$$

Liquid Samples

$$Wt\%_{wc} = \frac{\frac{Wt\%_s}{100} * C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right) * \rho_s \left(\frac{\text{g}}{\text{mL}} \right) - C_{ts} \left(\frac{\mu\text{g}}{\text{mL}} \right) * C_{wh} \left(\frac{\text{g}}{\text{mL}} \right)}{C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right) * \rho_s \left(\frac{\text{g}}{\text{mL}} \right) - C_{ts} \left(\frac{\mu\text{g}}{\text{mL}} \right) * \rho_h \left(\frac{\text{g}}{\text{mL}} \right)} * 100 \quad (17)$$

Since the weight percent correction is specific to the starting weight percent water measured by analysis, another useful calculation is the maximum concentration that the tracer could be if all of the water was from HHF. This equation is created by setting $Wt\%_{wc}$ to zero and solving for the concentration of tracer in the waste.

Solid Samples

$$MaxC_{tw} \left(\frac{\mu\text{g}}{\text{g}} \right) = \frac{\frac{Wt\%_s}{100} * C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right)}{C_{wh} \left(\frac{\text{g}}{\text{mL}} \right)} \quad (18)$$

Liquid Samples

$$MaxC_{tw} \left(\frac{\mu\text{g}}{\text{mL}} \right) = \frac{\frac{Wt\%_s}{100} * C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right) * \rho_s \left(\frac{\text{g}}{\text{mL}} \right)}{C_{wh} \left(\frac{\text{g}}{\text{mL}} \right)} \quad (19)$$

Current guidance concerning HHF intrusion indicate that if less than 10% of the water present is from HHF, the correction does not need to be made. However, if greater than 50% of the water present is from HHF, the sample is considered to be too highly contaminated to be useful. Therefore, being able to calculate what the concentration of the tracer would be for a given percentage of the water being HHF is useful. The percentage of water from HHF is derived from the weight of HHF water divided by the total water. Taking equations 3 and 7 we arrive at the following equations.

$$\begin{aligned}
 \text{Solids} \quad Wt\%_{whw} &= \frac{C_{ts} \left(\frac{\mu\text{g}}{\text{g}} \right) * Wt_s(\text{g}) * C_{wh} \left(\frac{\text{g}}{\text{mL}} \right)}{C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right)} \\
 &= \frac{Wt\%_w}{100} * Wt_s(\text{g}) \\
 \text{Liquids} \quad Wt\%_{whw} &= \frac{C_{ts} \left(\frac{\mu\text{g}}{\text{mL}} \right) * Wt_s(\text{g}) * C_{wh} \left(\frac{\text{g}}{\text{mL}} \right)}{C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right) * \rho_s \left(\frac{\text{g}}{\text{mL}} \right)} \\
 &= \frac{Wt\%_w}{100} * Wt_s(\text{g})
 \end{aligned} \tag{20}$$

If we simplify this equation and solve for the concentration of the tracer in the waste we get the following equations for solids and liquids.

$$\begin{aligned}
 \text{Solids} \quad C_{ts} \left(\frac{\mu\text{g}}{\text{g}} \right) &= \frac{Wt\%_{whw}}{100} * \frac{Wt\%_{ws} * C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right)}{C_{wh} \left(\frac{\text{g}}{\text{mL}} \right)} \\
 \text{Liquids} \quad C_{ts} \left(\frac{\mu\text{g}}{\text{mL}} \right) &= \frac{Wt\%_{whw}}{100} * \frac{Wt\%_{ws} * \rho_s \left(\frac{\text{g}}{\text{mL}} \right) * C_{th} \left(\frac{\mu\text{g}}{\text{mL}} \right)}{C_{wh} \left(\frac{\text{g}}{\text{mL}} \right)}
 \end{aligned} \tag{21}$$

4.0 CALCULATION OF ERROR IN CORRECTED VALUE

A summary of the data required to perform the analysis is as follows:

- Weight fraction water by TGA
- Concentration of tracer in sample
- Concentration of tracer in HHF
- Density of sample (if liquid).

Each of these tests is performed with a primary and duplicate analysis. If the average of these values is used to perform the correction calculation, the error or standard deviation between the primary and duplicate analysis can be used to estimate the standard deviation of the correction. The calculation of propagating errors can be studied in Mood et al. (1974). For a function of two variables $f(x,y)$ the calculated answer and variance of that answer is given by:

$$\begin{aligned} \xi[f(x,y)] \approx & f(\mu_x, \mu_y) + \frac{1}{2} \text{var}[x] \frac{\delta^2}{\delta x^2} f(x,y) \Big|_{\mu_x, \mu_y} \\ & + \frac{1}{2} \text{var}[y] \frac{\delta^2}{\delta y^2} f(x,y) \Big|_{\mu_x, \mu_y} + \text{covar}[x,y] \frac{\delta^2}{\delta x \delta y} f(x,y) \Big|_{\mu_x, \mu_y} \end{aligned}$$

and (22)

$$\begin{aligned} \text{Var}[f(x,y)] \approx & \left\{ \frac{\delta}{\delta x} f(x,y) \Big|_{\mu_x, \mu_y} \right\}^2 \text{Var}[x] + \left\{ \frac{\delta}{\delta y} f(x,y) \Big|_{\mu_x, \mu_y} \right\}^2 \text{Var}[y] \\ & + 2\text{cov}[x,y] \left\{ \frac{\delta}{\delta y} f(x,y) \Big|_{\mu_x, \mu_y} * \frac{\delta}{\delta x} f(x,y) \Big|_{\mu_x, \mu_y} \right\} \end{aligned}$$

This method requires taking the first and second partial derivative of the function with respect to each variable involved. These partial derivatives are used in combination with the standard deviations of the variables to estimate the standard deviation of the result. For the equation derived above (15,16) to correct for the intrusion of HHF, the second partial derivatives evaluate to zero. If we assume that the covariance of the variables is zero and extrapolate the equation to include three and four variables, we can derive the following equations to estimate

the variance of the result for both the liquid and solids calculations.

$$\begin{aligned}
 \text{Solids} \quad \text{Var}[f(Wt\%_{ws}, C_{ls}, C_{th})] &\approx \left\{ \frac{\delta}{\delta Wt\%_{ws}} f(Wt\%_{ws}, C_{ls}, C_{th}) \right\}^2 \text{Var}[Wt\%_{ws}] \\
 &+ \left\{ \frac{\delta}{\delta C_{ls}} f(Wt\%_{ws}, C_{ls}, C_{th}) \right\}^2 \text{Var}[C_{ls}] \\
 &+ \left\{ \frac{\delta}{\delta C_{th}} f(Wt\%_{ws}, C_{ls}, C_{th}) \right\}^2 \text{Var}[C_{th}]
 \end{aligned} \tag{23}$$

$$\begin{aligned}
 \text{Liquids} \quad \text{Var}[f(Wt\%_{ws}, \rho_s, C_{ls}, C_{th})] &\approx \left\{ \frac{\delta}{\delta Wt\%_{ws}} f(Wt\%_{ws}, \rho_s, C_{ls}, C_{th}) \right\}^2 \text{Var}[Wt\%_{ws}] \\
 &+ \left\{ \frac{\delta}{\delta \rho_s} f(Wt\%_{ws}, \rho_s, C_{ls}, C_{th}) \right\}^2 \text{Var}[\rho_s] \\
 &+ \left\{ \frac{\delta}{\delta C_{ls}} f(Wt\%_{ws}, \rho_s, C_{ls}, C_{th}) \right\}^2 \text{Var}[C_{ls}] \\
 &+ \left\{ \frac{\delta}{\delta C_{th}} f(Wt\%_{ws}, \rho_s, C_{ls}, C_{th}) \right\}^2 \text{Var}[C_{th}]
 \end{aligned} \tag{24}$$

Performing these partial derivatives produces the following equations

$$\text{For Solids} \quad \frac{\delta}{\delta Wt\%_{ws}} f(Wt\%_{ws}, C_{ls}, C_{th}) = \frac{C_{th}}{C_{th} - \rho_h C_{ls}} \tag{25}$$

$$\frac{\delta}{\delta C_{ls}} f(Wt\%_{ws}, C_{ls}, C_{th}) = C_{th} \frac{(Wt\%_{ws} \rho_h - C_{wh})}{(C_{ls} \rho_h - C_{th})^2} \tag{26}$$

$$\frac{\delta}{\delta C_{th}} f(Wt_{ws}, C_{is}, C_{th}) = -C_{is} \frac{Wt_{ws} \rho_h - C_{wh}}{(C_{is} \rho_h - C_{th})^2} \quad (27)$$

For Liquids

$$\frac{\delta}{\delta Wt_{ws}} f(Wt_{ws}, C_{is}, C_{th}) = \rho_s \frac{C_{th}}{C_{th} \rho_s - \rho_h C_{is}} \quad (28)$$

$$\frac{\delta}{\delta C_{is}} f(Wt_{ws}, C_{is}, C_{th}) = \rho_s C_{th} \frac{Wt_{ws} \rho_h - C_{wh}}{(C_{is} \rho_h - \rho_s C_{th})^2} \quad (29)$$

$$\frac{\delta}{\delta C_{th}} f(Wt_{ws}, \rho_s, C_{is}, C_{th}) = -\rho_s C_{is} \frac{Wt_{ws} \rho_h - C_{wh}}{(C_{is} \rho_h - \rho_s C_{th})^2} \quad (30)$$

$$\frac{\delta}{\delta \rho_s} f(Wt_{ws}, C_{is}, C_{th}) = -C_{is} C_{th} \frac{Wt_{ws} \rho_h - C_{wh}}{(C_{is} \rho_h - \rho_s C_{th})^2} \quad (31)$$

5.0 REFERENCES

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APPENDIX A

SINGLE-SAMPLE SPREADSHEET DOCUMENTATION

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APPENDIX A

SINGLE SAMPLE SPREADSHEET DOCUMENTATION

The excel spreadsheet program "LIBRCORR.XLS" contains the equations and functions to calculate the original waste tank sample water percentage based on Li and Br laboratory analysis. The program consists of three spreadsheet pages and a module page. The three spreadsheet pages are shown as Figures A1, A2, and A3. Example printouts are also included in Appendix D. The module page contains the custom functions which emulate the equations developed to calculate the correction for sampling water sample intrusion. A listing of the module code is included in Appendix C.

There were 27 custom functions developed to implement the equations required to correct for sampling water intrusion. Many of the functions are similar with only the necessary changes required to differentiate whether Li or Br was being used to make the correction and whether the sample was a solid or a liquid. The functions are defined in detail in Appendix C. HHF is used to indicate sample water laced with LiBr regardless of how the water was used in the sampling process.

Figure A1 shows the data input page which is where the analysis data and sample information is entered. The spreadsheet cells are all protected from change by the user except for those which need user input. These input cells are colored for easy identification.

The row and column indicators are included in the figures so that they may be referenced.

Cells B3, D3, and F3 are strictly text entry cells for identifying the tank, core, and segment the correction is on. Likewise cells B8 and B15 are text cells for entering the sample number for the solid and liquid sample portions of the segment.

The spreadsheet is divided into three sets of data input. These are the analytical data for any solids in the segment, or any liquid portion of the segment and the data from the water sample from the field. Column B, C, and D are set up to receive the primary, duplicate, and if appropriate, triplicate analysis. Rows 9-12 are for the solids analysis as labeled in column A. Rows 16-20 are for the liquid analysis and correspond to the labels in column A. Rows 24 and 25 are for the Li and Br analysis of the water sample from the field.

For each of the analysis data sets Columns E, F, G, and H, calculate statistics on the analysis results. Column H data will be used later to determine the error of the correction.

Column E simply calculates the data average. An if function is used to prevent a calculation error from showing in the spreadsheet if only one data point is entered. The equation used is listed below.

=IF(COUNT(B10:D10)>0,SUM(B10:D10)/COUNT(B10:D10),"")

If there is at least one data entry, then the sum of the entries is divided by the number of entries; otherwise, a blank cell is left. If only one entry exists then the average is listed as the value of the entry.

Column F calculates the difference in the data in a similar way with the equation below.

=IF(COUNT(B10:D10)>1,MAX(B10:D10)-MIN(B10:D10),"")

If there are at least two data entries, then the difference can be calculated by taking the maximum value and subtracting the minimum value.

Column G calculates the relative percent difference (RPD) by the equation below.

=IF(COUNT(B10:D10)>1,F10/E10,"")

Again, if there are at least two data points, the RPD is calculated by dividing the difference by the average value.

Column H calculates the standard deviation of the average by the equation below.

=IF(COUNT(B10:D10)>1,STDEV(B10:D10)/COUNT(B10:D10)^0.5,"")

If there are at least two data entries, the internal Excel function STDEV is used to return the population standard deviation. The standard deviation of the average is the value desired so this value is divided by the square root of the number of values.

Figure A1. Data Input Page of Spreadsheet.

Tank #	Core #	Segment #	Laboratory Analysis			Statistical Analysis		
			Primary Analysis	Duplicate Analysis	Triplicate Analysis	Average Value	Difference	% Relative Difference
Solids Portion								
	Sample #							
9	1515							
10	323	345			31	9.00%	15.50	
11	390	3050			100	3.28%	50.00	
12	211%	23.5%			1.20%	5.11%	0.60%	
Liquids Portion								
	Sample #							
16	100							
17	115	1.150						
18	535.4	840			4	0.42%	1.75	
19	14003	14003						
20	75.5%	77.1%			1.20%	1.54%	0.60%	
HHF Blank								
23								
24	2100	2050			100	4.88%	50.00	
25	23000	24000			2,000	8.33%	1,000.00	
26								
27	Based on Li	Based on Br						
28	HHF Density Calculation							
29	Density of HHF (g/mL)	1.017	1.017					
30	Conc of H ₂ O in HHF (g/mL)	0.992	0.991					
31					297.0E-6	0.03%	148.5E-6	
32					129.5E-6	0.01%	64.7E-6	
LiBr Correction								

On the second page of the spreadsheet, the values are calculated to indicate the corrected weight percent water in the sample for the liquid and solid samples and based on either Li or Br analysis. This accounts for four columns of information that are identical in function. The differences are that the liquid samples use a slightly different function to account for density of the sample and the reference values for whether Li or Br is being used to make the correction.

	A	B	C
37			
38			Solids Portion
39			
40			
41			Lithium
42			Analysis
43	Weight H ₂ O from HHF (g)	=WatSol(B9,E10,E24,E31)	
44	Weight H ₂ O in tank (g)	=B9*E12-B43	
45	% Water from HHF	=Pws(E12,E10,E24,E31)	
46			
47	Corrected Wgt (g)	=B9-WatSol(B9,E10,E24,E30)	
48	Weight% H ₂ O in tank	=CorWtpLiSol(E12,E10,E24)	
49	95% Confidence Low	=IF(COUNT(B12:D12)>1,IF(COUNT(B12:D12)>2,B48-B52*2.9,B48-B52*3.314),"")	
50	Weight% H ₂ O Difference	=+B48-E12	
51	Variance	=(B52*100)^2/100	
52	Std Dev of Corrected % H ₂ O	=sdCWtpS(E12,E10,E24,E30,E31,H12,H10,H24)	

Each value is labeled on the left side of the sheet with appropriate headings for each of the columns.

	A	C	D
37			
38			Solids Portion
39			
40			
41			Bromine
42			Analysis
43	Weight H ₂ O from HHF (g)	=WatSol(B9,E11,E25,E31)	
44	Weight H ₂ O in tank (g)	=B9*E12-D43	
45	% Water from HHF	=Pws(E12,E11,E25,E31)	
46			
47	Corrected Wgt (g)	=B9-WatSol(B9,E11,E25,E30)	
48	Weight% H ₂ O in tank	=CorWtpBrSol(E12,E11,E25)	
49	95% Confidence Low	=IF(COUNT(B12:D12)>1,IF(COUNT(B12:D12)>2,D48-D52*2.9,D48-D52*3.314),"")	
50	Weight% H ₂ O Difference	=+D48-E12	
51	Variance	=(D52*100)^2/100	
52	Std Dev of Corrected % H ₂ O	=sdCWtpS(E12,E11,E25,E30,E31,H12,H11,H25)	

	A	F	B
37			
38			Liquid Portion
39			
40			
41		Lithium	
42		Analysis	
43	Weight H ₂ O from HHF (g)	=WatLiq(B16,E18,E17,E24,E31)	
44	Weight H ₂ O in tank (g)	=B16*E20-F43	
45	% Water from HHF	=Pw(E20,E18,E17,E24,E31)	
46			
47	Corrected Wgt (g)	=B16-WatLiq(B16,E18,E17,E24,E30)	
48	Weight% H ₂ O in tank	=CorWtpLLiq(E20,E18,E24,E17)	
49	96% Confidence Low	=IF(COUNT(B17:D17)>1,IF(COUNT(B17:D17)>2,F48-F52*2.9,F48-F52*6.314),****)	
50	Weight% H ₂ O Difference	=+F48-E20	
51	Variance	=(F52*100)^2/100	
52	Std Dev of Corrected % H ₂ O	=sdCWtplliq(E20,E18,E24,E17,E30,E31,H20,H18,H24,H17)	

	A	G	H
37			
38			Liquid Portion
39			
40			
41			Bromine
42			Analysis
43	Weight H ₂ O from HHF (g)	=WatLiq(B16,E19,E17,E25,E31)	
44	Weight H ₂ O in tank (g)	=B16*E20-H43	
45	% Water from HHF	=Pw(E20,E19,E17,E25,E31)	
46			
47	Corrected Wgt (g)	=B16-WatLiq(B16,E19,E17,E25,E30)	
48	Weight% H ₂ O in tank	=CorWtpBrLiq(E20,E19,E25,E17)	
49	96% Confidence Low	=IF(COUNT(B17:D17)>1,IF(COUNT(B17:D17)>2,H48-H52*2.9,H48-H52*6.314),****)	
50	Weight% H ₂ O Difference	=+H48-E20	
51	Variance	=(H52*100)^2/100	
52	Std Dev of Corrected % H ₂ O	=sdCWtplliq(E20,E19,E25,E17,E30,E31,H20,H19,H25,H17)	

The weight of water in the sample from HHF is calculated on Row 43. The WatSol and WatLiq custom function was used. Documentation for each of the custom functions is found in Appendix C.

Row 44 shows the weight of water in the sample that is estimated to be in the tank is calculated by finding the total water in the sample and subtracting out the HHF contribution.

Row 45 shows the percentage of the water present that is from HHF is calculated with the Pws and Pwl functions depending on whether a solid or liquid sample was used.

Row 47 shows the corrected weight of the sample. This is the original sample weight minus the weight of the HHF. The weight of HHF is calculated using the same function (Watsol and Watliq) used to calculate the weight of water from HHF, except that the density of the HHF is used instead of the concentration of water in the HHF. This gives the total weight of HHF in the sample.

The corrected weight percent of water that would be found in an uncontaminated sample is given on Row 48. The correct form of the function is used for each of the four combinations. The functions used are CorrWtpLiLiq, CorrWtpBrLiq, CorrWtpLiSol, CorrWtpBrSol.

Row 49 calculates the one sided 95% low confidence level based on the student T distribution. The decision on whether to use a distribution for duplicate or triplicate analysis is based on the number of analysis performed to measure weight percent water. If triplicate analysis was performed, a distribution value of 2.92 is used. If duplicate analysis was performed, 6.314 is used.

Row 50 calculates the difference between the TGA measured weight percent water of the sample and the estimated corrected value. This is done by subtracting the two values.

Row 51 calculates the variance of the result. This is the square of the standard deviation on Row 52. Converting the number from a decimal fraction to a whole number is necessary to get a correct answer.

Row 52 Calculates the estimated standard deviation of the corrected value. This is done by using either the sdCWtpS or sdCWtpL custom function depending on whether a liquid or solid sample is being used. The complete workings of this function are described in section 4.0 and implemented in Appendix C.

Figure A2. Spreadsheet Correction Data.

	A	B	C	D	E	F	G	H
39								
40								
41								
42		Solids Portion			Liquid Portion			
43		Litium	Bromine		Litium	Bromine		
44		Analysis	Analysis		Analysis	Analysis		
45	Weight H ₂ O from HHF (g)	25.575	19.341		35.333	50.292		
46	Weight H ₂ O in tank (g)	10.497	16.732		42.367	27.408		
47	% Water from HHF	70.90%	53.62%		45.47%	64.73%		
48	Corrected Wgt (g)	127.267	133.666		63.746	46.396		
49	Weight% H₂O in tank	8.26%	12.62%		66.46%	66.66%		
50	95% Confidence Low	0.76%	6.89%		69.92%	42.78%		
51	Weight% H ₂ O Difference	-15.3%	-10.98%		-11.25%	-21.05%		
52	Variance	1.41%	0.79%		1.07%	4.83%		
53	Std Dev of Corrected % H ₂ O	1.19%	0.89%		1.04%	2.20%		
54								
55								
56								
57								
58								
59								
60								
61								
62								
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67								
68								
69								
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100								

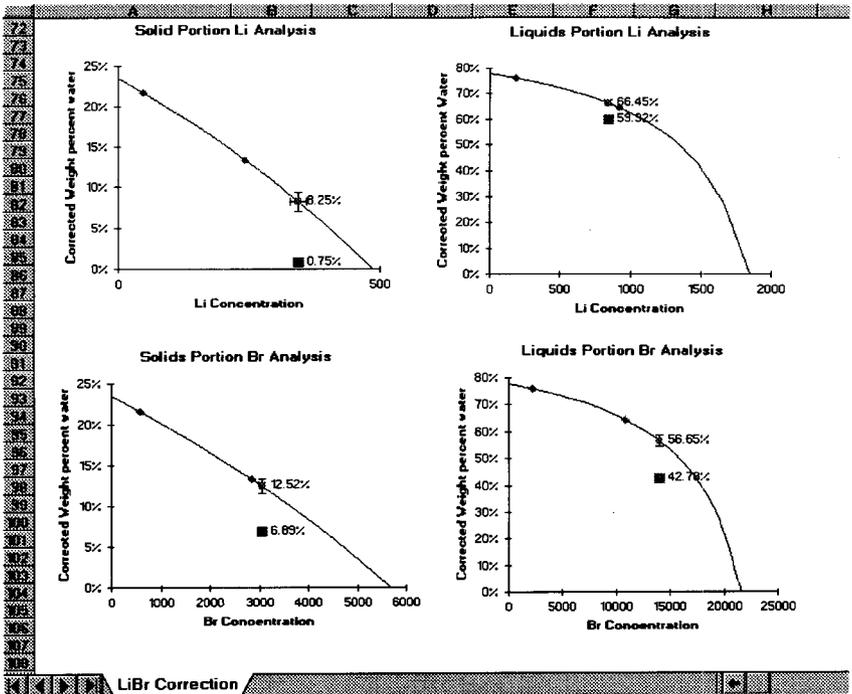
The last page of the spreadsheet gives a graphical representation of where the corrected results fall on a curve. The curve is representative of the range between no contamination and that of all of the water from HHF. The data for the graph is generated in columns J through R and rows 39 through 57. It is divided into four sections and represents the four graphs. Figure A7 is the equations for one of the four sections. The other sections are similar with different references to represent the other data. The graphs are X, Y data pairs. The X data is in Column J and the Y data is in Column K. Rows 42 to 52 represent 10 equal increments of tracer concentration and are used to generate the primary curve. Row 63 is used to plot the corrected value. Row 54 is used to plot the 95% Low confidence value. Rows 56 and 57 are used to calculate the values to plot the points where 10% and 50% of the water present would be from HHF. The error bars in the Y direction on the corrected value data

points are drawn from the standard deviation of the corrected result. The X direction error bars are the standard deviation of the tracer measurement. Actual value labels are provided for the corrected value and the 95% low confidence level.

Figure A7 Graph Information Generation.

	I	J	K
37			
38			
39			Solids
40		Li	
41		Conc	Corrected
42	0		=CorrWtpLiSol(\$E\$12,J42,\$E\$24)
43	=MaxCLiS(\$E\$12,\$E\$24)/10		=CorrWtpLiSol(\$E\$12,J43,\$E\$24)
44	=MaxCLiS(\$E\$12,\$E\$24)/10*2		=CorrWtpLiSol(\$E\$12,J44,\$E\$24)
45	=MaxCLiS(\$E\$12,\$E\$24)/10*3		=CorrWtpLiSol(\$E\$12,J45,\$E\$24)
46	=MaxCLiS(\$E\$12,\$E\$24)/10*4		=CorrWtpLiSol(\$E\$12,J46,\$E\$24)
47	=MaxCLiS(\$E\$12,\$E\$24)/10*5		=CorrWtpLiSol(\$E\$12,J47,\$E\$24)
48	=MaxCLiS(\$E\$12,\$E\$24)/10*6		=CorrWtpLiSol(\$E\$12,J48,\$E\$24)
49	=MaxCLiS(\$E\$12,\$E\$24)/10*7		=CorrWtpLiSol(\$E\$12,J49,\$E\$24)
50	=MaxCLiS(\$E\$12,\$E\$24)/10*8		=CorrWtpLiSol(\$E\$12,J50,\$E\$24)
51	=MaxCLiS(\$E\$12,\$E\$24)/10*9		=CorrWtpLiSol(\$E\$12,J51,\$E\$24)
52	=MaxCLiS(\$E\$12,\$E\$24)		=CorrWtpLiSol(\$E\$12,J52,\$E\$24)
53	=+E10		=B48
54	=+H10		=B52
55	10% Limit		50%limit
56	=Ctws(\$E\$12,E24,\$E\$31,0.1)		=Ctws(\$E\$12,E24,\$E\$31,0.5)
57	=CorrWtpLiSol(\$E\$12,J56,\$E\$24)		=CorrWtpLiSol(\$E\$12,K56,\$E\$24)

Figure A3. Graphical representation of results.



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APPENDIX B

**TABULAR SPREADSHEET
DOCUMENTATION**

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The spreadsheet "LIBRCORR.XLS" provides for the correction of a single sample along with its drainable liquid. If many samples have a HHF contamination problem, it is desirable to allow multiple sample data input and correction. The spreadsheet "TABLIBR.XLS" provides the ability to input multiple data sets into a tabular arrangement and simultaneously get a tabular set of corrections.

The multiple page capability of Excel is used so that the input of data for each analytical value in on a separate page. Each page is selected by clicking the mouse on the tab at the bottom of the screen for that page. Therefore there are 11 pages in this Excel workbook. One of these pages has been hidden to the user. This is the module page and is the same as in the other spreadsheet and is described in Appendix C. The 10 pages visible to the user are described below. The page is given then the label on the page tab and then a short description of the page contents.

- Page 1. "HHF Data" data from analysis of the HHF field sample and calculation of HHF density and the concentration of water in the HHF.
 - Page 2. "Liq Density" data input page for density measurements of liquid samples
 - Page 3. "Li Solids" Li analysis data for solid samples.
 - Page 4. "Li Liquids" Li analysis data for liquid samples.
 - Page 5. "Br Solids" Br analysis data for solid samples.
 - Page 6. "Br Liquids" Br analysis data for liquid samples.
 - Page 7. "H2O Solids" weight percent water analysis for solid samples.
 - Page 8. "H2O Liquids" weight percent water analysis for liquid samples.
 - Page 9. "Corrected Solids" corrections analysis based on Li and Br concentrations for solid samples.
 - Page 10. "Corrected Liquids" corrections analysis based on Li and Br concentrations for liquid samples.
- Page 1 is shown in Figure B1.

The data is entered in columns B, C and D or Rows 10 and 11. These areas are highlighted for easy identification. A place at the top to enter the tank number for documentation purposes is provided. Columns E, F, G and H calculate the average value, difference, percent relative difference and standard deviation of average with the following Excel equations. These are basically the same as the description of the "LIBRCORR.XLS" program discussed in Appendix A.

Column E simply calculates the data average. An if function is used to prevent a calculation error from showing in the spreadsheet if only one data point is entered. The equation used is listed below.

$$=IF(COUNT(B10:D10)>0,SUM(B10:D10)/COUNT(B10:D10),"")$$

If there is at least one data entry, then the sum of the entries is divided by the number of entries; otherwise, a blank cell is left. If only one entry exists, then the average is listed as the value of the entry.

Figure B1. HHF Data Calculation Page.

Li and Br analysis for HHF Blank							
Tank #							
	Laboratory Analysis			Statistical Analysis			
	Primary Analysis	Duplicate Analysis	Triplicate Analysis	Average Value	Difference	% Relative Difference	Standard Deviation of Average
HHF Blank Data							
Lithium Conc (µgm/gm)				2,050	100	4.88%	50
Bromine Conc (µgm/gm)				24,000	2,000	8.33%	1,000
HHF Density Calculation	Based on Li	Based on Br					
Density of HHF (gm/mL)	1.017	1.017		1.017	2.97E-04	0.03%	1.48E-04
Conc of H ₂ O in HHF(gm/mL)	0.992	0.991		0.991	1.29E-04	0.01%	6.47E-05

Column F calculates the difference in the data is a similar way with the equation below.

$$=IF(COUNT(B10:D10)>1,MAX(B10:D10)-MIN(B10:D10),"")$$

If there are at least two data entries, then the difference can be calculated by taking the maximum value and subtracting the minimum value.

Column G calculates the relative percent difference by the equation below.

$$=IF(COUNT(B10:D10)>1,F10/E10,"")$$

Figure B2. Example Data Entry Page.

	A	B	C	D	E	F	G	H
1								
2		Tank # B-106						
3		Density Analysis for Liquids Portion of Sample						
4		Laboratory Analysis			Statistical Analysis			
5		Primary Analysis	Duplicate Analysis	Triplicate Analysis	Average Value	Difference	% Relative Difference	Standard Deviation of Average
6		ug/mL	ug/mL	ug/mL	ug/mL			
7	Sample Number							
8	95-8354	1.235	1.235		1.235	0.00	0.00%	0.00
9								
10								
11								
12								
13								

Again, if there are at least two data points, the relative percent difference is calculated by dividing the difference by the average value.

Column H calculates the standard deviation of the average by the equation below.

$$=IF(COUNT(B10:D10) > 1,STDEV(B10:D10)/COUNT(B10:D10)^0.5,"")$$

If there are at least two data entries, the internal Excel function STDEV is used to return the population standard deviation. The standard deviation of the average is the value desired, so this value is divided by the square root of the number of values.

Row 16 Column B and C calculates the density of the HHF based on Li and Br analysis. The RhoBr and RhoLi custom functions are used. These functions are documented in Appendix C. Row 17 Column B and C calculates the concentration of water in the HHF based on Li and Br analysis. The CWHLi and CWHBr functions are used. These functions are documented in Appendix C. Columns E, F, G and H of Rows 16 and 17 provide the same statistical analysis as performed on the analytical data using the same equations.

Pages 2 through 8 are basically the same differing only in the data which is placed on them. The liquid density page is shown in Figure B2 as an example.

Figure B3. Percent Water Correction Page for Solid Samples

	A	B	C	D	E	F	G	H	I	J	K	L	M
2	Tank # B-106												
3													
6		Solids Wt% corrected with Li Analysis						Solids Wt% corrected with Br Analysis					
7	Sample Number	20-20 from HHF	Corrected Wt% H2O in tank	95% Conf Interval Low	Wt% Difference	Variance of result	Sd of Result	20-20 from HHF	Corrected Wt% H2O in tank	95% Conf Interval Low	Wt% Difference	Variance of result	Sd of Result
8		37.69%	28.19%	0.30%	-10.31%	19.51%	4.42%	27.36%	31.36%	20.13%	-7.14%	3.16%	1.78%

Rows 8 through 35 are available for data entry. If more rows are needed, the equations contained in Columns E, F, G and H will need to be copied to the additional rows in each of the data entry pages. Column A is used to input the sample number. This will be used on the liquids corrections page to ensure that the same sample is being used for each of the analysis. On the solids correction page, the three different sample numbers used are placed in this column. Columns B, C, and D are used for data input in the units specified for that column. Columns E, F, G, and H perform the same data analysis of each data set as described for the HHF data page.

The two pages used to make the percent water corrections are basically the same. They differ only in the data sources used, the custom function forms to account to Li or Br analysis and the inclusion of sample density for the Liquid samples. One additional difference is that three different sample numbers are required for solid samples. This is because of the different fusion techniques required for each different analysis. Figure A3 shows the spreadsheet form for the solids correction page.

The following example equations are for correcting the weight percent of a sample using Li analysis on a solid sample. Similar equations would be used for the Br analysis and liquid samples. Refer to the spreadsheet for details and reference data locations.

The percent in the sample from HHF is calculated on column B and H. The IF statement checks to see if valid data exists on Li Solids analysis page. If the data is of TYPE 1 or numerical type data, then the calculation is performed. A similar check is made for each of the following functions. If no data is found, a blank is left in the cell. The nomenclature of the references is the label of the page, a ! and, then the cell reference. The PWS custom function was used. Documentation for each of the custom functions is found in Appendix C.

$$=IF(TYPE('Li Solids'!E8)=1,Pws('H2O Solids'!E8,'Li Solids'!E8,'HHF Data'!E10,'HHF Data'!E17), "")$$

The corrected weight percent of water that would be found in an uncontaminated sample is

given on Column C and I. The correct form of the function is used for each of the four combinations. The functions used are CorrWtpLiLiq, CorrWtpBrLiq, CorrWtpLiSol, CorrWtpBrSol for each of the four possible combinations. The example below is for Li analysis of a solid sample.

=IF(TYPE('Li Solids'!E8)=1,CorrWtpLiSol('H2O Solids'!E8,'Li Solids'!E8,'HHF Data'!\$E\$10),"")

Column D and J calculates the one sided 95% low confidence level based on the student T distribution. The decision on whether to use a distribution for duplicate or triplicate analysis is based on the number of analysis performed to measure weight percent water. If triplicate analysis was performed a distribution value of 2.92 is used. If duplicate analysis was performed, 6.314 is used.

=IF(TYPE('Li Solids'!E8)=1,IF(COUNT('H2O Solids'!B17:'H2O Solids'!D17)>2, C8 - G8 * 2.92,C8-G8*6.314),"")

Columns E and K calculate the difference between the TGA measured weight percent water of the sample and the estimated corrected value. This is done by simply subtracting the two values.

=IF(TYPE('H2O Solids'!E8)=1,C8-'H2O Solids'!E8,"")

Columns F and L calculate the variance of the result. This is simply the square of the standard deviation in Columns G and M, respectfully. Converting the number from a decimal fraction to whole number is necessary to obtain a correct answer.

=IF(TYPE('Li Solids'!E8)=1,(G8*100)^2/100,"")

Columns G and M calculate the estimated standard deviation of the corrected value. This is done by using either the sdCWtpS or sdCWtpL custom function depending on whether a liquid or solid sample is being used. The complete workings of this function are described in Section 4.0 and implemented in Appendix C.

=IF(TYPE('Li Solids'!E8)=1,sdCWtpS('H2O Solids'!E8,'Li Solids'!E8,'HHF Data'!\$E\$10,'HHF Data'!\$E\$16,'HHF Data'!\$E\$17,'H2O Solids'!H8,'Li Solids'!H8,'HHF Data'!\$H\$10),"")

Once again the equations have been copied to rows 8 through 35. This is the amount of data that will fit on one landscape page. If more samples are needed, the spreadsheet protection will need to be turned off, and the equations will need to be copied to sufficient rows to cover the additional data.

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APPENDIX C
EXCEL CUSTOM FUNCTIONS
MODULE LISTING

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The following is a table that connects the equations developed in the spreadsheet module page to the derivation equations listed in the body of the document. The listing of codes located in the module page of the spreadsheets follows.

Table C1. Listing of Special Functions

Function Name	Description	Equation Number being Implemented
RhoLi RhoBr	Calculate the density of the LiBr traced sampling water based on Li or Br.	9 10
CWHLi CWHBr	Calculate the concentration of water in the HHF based on Li or Br	11-13
MaxCBrS MaxCLiS MaxCBrL MaxCLiL	Calculate the maximum concentration of Br or Li possible in a solid or liquid sample.	18 19
CorrWtpBrSol CorrWtpLiSol CorrWtpBrLiq CorrWtpLiLiq	Calculate the corrected weight percent water in a solid or liquid sample based on Br or Li.	16 17
WatSol WatLiq	Calculate the weight of the HHF water in solid or liquid samples.	6
dCtwliq dCthliq dCrwliq dCWtpliq dCthS dCtwS dWtpS	- The partial derivative of the correction with respect analytical variables. Ctw -- Concentration of tracer in the waste Cth -- Concentration of tracer in the HHF Crw - Density of the waste Wtp - Weight percent water in the sample S - Solid sample L - Liquid sample.	25 26 27 28 29 30 31
sdCWtpliq - sdCWtpS	Standard deviation of the corrected weight percent for a liquid or solid sample.	23 24
Ctws Ctwl	Calculate the concentration of the tracer in the sample of a given percentage of the water in the sample which is HHF for a solid or liquid sample.	21
Pws Pwl	Calculate the percentage of water percent that is from HHF for solid samples.	20

Option Explicit

'Functions to calculate corrections to water concentration of
 'Waste samples do to intrusion of .3Molar LiBr traced waster
 'used during push-mode waste tank core sampling
 'Written by Wayne Winkelman
 'Created on February 1996
 'Last modified April 17, 1996

'Calculate the density (Rho) of the HHF by Br and Li analysis
 ' CLi is the concentration of Li in ug/g
 ' CBr is the concentration of Br in ug/g

Function RhoLi(CLi)

$$\text{RhoLi} = 0.9993 + 8.728 * 10^{-6} * \text{CLi} - 1.559 * 10^{-12} * \text{CLi}^2$$

End Function

Function RhoBr(CBr)

$$\text{RhoBr} = 0.9993 + 7.579 * 10^{-7} * \text{CBr} - 1.179 * 10^{-14} * \text{CBr}^2$$

End Function

'calculate the concentration of Water in the HHF by Br and Li analysis

Function CWHLi(CLi)

Dim MWTLiBr, MWTLi

$$\text{MWTLiBr} = 86.848$$

$$\text{MWTLi} = 6.939$$

$$\text{CWHLi} = \text{RhoLi}(\text{CLi}) - \text{CLi} * \text{MWTLiBr} / \text{MWTLi} / 10^6$$

End Function

Function CWHBr(CBr)

Dim MWTLiBr, MWTLi

$$\text{MWTLiBr} = 86.848$$

$$\text{MWTLi} = 79.909$$

$$\text{CWHBr} = \text{RhoBr}(\text{CBr}) - \text{CBr} * \text{MWTLiBr} / \text{MWTLi} / 10^6$$

End Function

'Calculate the Maximum Br or Li concentration possible in the sample that would constitute
 'that all of the water present was from HHF. S or L stands for solid or liquid sample
 'Used to create graphic plots.

Function MaxCBrS(Wtp, CBrhhf)

$$\text{MaxCBrS} = \text{Wtp} * \text{CBrhhf} / \text{CWHBr}(\text{CBrhhf})$$

End Function

Function MaxCLiS(Wtp, CLihhf)

$$\text{MaxCLiS} = \text{Wtp} * \text{CLihhf} / \text{CWHLi}(\text{CLihhf})$$

End Function

Function MaxCLiL(Wtp, RhoW, CLihhf)

$$\text{MaxCLiL} = \text{Wtp} * \text{RhoW} * \text{CLihhf} / \text{CWHLi}(\text{CLihhf})$$

End Function

Function MaxCBrL(Wtp, RhoW, CBrhhf)

$$\text{MaxCBrL} = \text{Wtp} * \text{RhoW} * \text{CBrhhf} / \text{CWHBr}(\text{CBrhhf})$$

End Function

' Calculate the corrected Weight percent water in the solid sample from Br concentration

' Wtp is the total weight % water in the sample by analysis

' CBr, CLi is the concentration of the tracer (Br or Li) in the sample ug/g

' CBrhhf, CLihhf is the concentration of the tracer (Br or Li) in the HHF ug/g

Function CorrWtpBrSol(Wtp, CBr, CBrhhf) ' Concentrations in micro gram per gram

Dim Ctw, Cth, rh, rwh

$$\text{Ctw} = \text{CBr} / 10 \wedge 6 \quad \text{'convert to g/g}$$

$$\text{Cth} = \text{CBrhhf} / 10 \wedge 6 \quad \text{'convert to g/g}$$

$$\text{rh} = \text{RhoBr}(\text{CBrhhf}) \quad \text{'calculate density of HHF}$$

$$\text{rwh} = \text{CWHBr}(\text{CBrhhf}) \quad \text{'calculate concentration of water in HHF}$$

$$\text{CorrWtpBrSol} = (\text{Wtp} * \text{Cth} - \text{Ctw} * \text{rwh}) / (\text{Cth} - \text{Ctw} * \text{rh})$$

End Function

'Calculate the corrected weight percent water in the solid sample based on Li analysis

Function CorrWtpLiSol(Wtp, CLi, CLihhf) ' Concentrations in micro gram per gram

```

Dim Ctw, Cth, rh, rwh
Ctw = CLi / 10 ^ 6
Cth = CLihhf / 10 ^ 6
rh = RhoLi(CLihhf)
rwh = CWHLi(CLihhf)
CorrWtpLiSol = (Wtp * Cth - Ctw * rwh) / (Cth - Ctw * rh)

```

End Function

'Calculate the corrected weight percent water in the Liquid sample based on Br analysis

Function CorrWtpBrLiq(Wtp, CBr, CBrhhf, RhoW) ' Concentrations in micro gram per milliliter density in grams per milliliter

```

Dim Ctw, Cth, rh, rwh
Ctw = CBr / 10 ^ 6
Cth = CBrhhf / 10 ^ 6
rh = RhoBr(CBrhhf)
rwh = CWHBr(CBrhhf)
CorrWtpBrLiq = (Wtp * RhoW * Cth - Ctw * rwh) / (Cth * RhoW - Ctw * rh)

```

End Function

'Calculate the corrected weight percent water in the liquid sample based on Li analysis

Function CorrWtpLiLiq(Wtp, CLi, CLihhf, RhoW) ' concentrations in micro gram per milliliter density in grams per milliliter

```

Dim Ctw, Cth, rh, rwh
Ctw = CLi / 10 ^ 6
Cth = CLihhf / 10 ^ 6
rh = RhoLi(CLihhf)
rwh = CWHLi(CLihhf)
CorrWtpLiLiq = (Wtp * RhoW * Cth - Ctw * rwh) / (Cth * RhoW - Ctw * rh)

```

End Function

' Calculate weight of water from HHF for solid and liquid sample
 ' Wt is the weight of the sample
 ' Ctw is the concentration of the tracer in the waste
 ' Cth is the concentration of the tracer in the HHF
 ' rwh is the concentration of water in the HHF

Function WatSol(Wt, Ctw, Cth, rwh)

$$\text{WatSol} = \text{Wt} * \text{rwh} * \text{Ctw} / \text{Cth}$$

End Function

Function WatLiq(Wt, Ctw, rw, Cth, rwh)

$$\text{WatLiq} = \text{Wt} * \text{rwh} * \text{Ctw} / \text{Cth} / \text{rw}$$

End Function

'Derivative functions to calculate
 'standard deviation of error on result for Lig samples
 'Wtp is the weight % water in the sample by analysis
 'Ctw is the concentration of the tracer in the waste
 'Cth is the concentration of the tracer in the HHF
 'rw is the density of the waste for liquid samples
 'rh is the density of the HHF
 'rwh is the concentration of water in the HHF

Function dCtwliq(Wtp, Ctw, Cth, rw, rh, rwh)

$$\text{dCtwliq} = \text{rw} * \text{Cth} * (\text{Wtp} * \text{rh} - \text{rwh}) _ \\ / (\text{Ctw} * \text{rh} - \text{rw} * \text{Cth}) ^ 2 _$$

End Function

Function dCthliq(Wtp, Ctw, Cth, rw, rh, rwh)

$$\text{dCthliq} = -\text{rw} * \text{Ctw} * (\text{Wtp} * \text{rh} - \text{rwh}) _ \\ / (\text{Ctw} * \text{rh} - \text{rw} * \text{Cth}) ^ 2 _$$

End Function

Function dCrwliq(Wtp, Ctw, Cth, rw, rh, rwh)

$$\text{dCrwliq} = \frac{-\text{Ctw} * \text{Cth} * (\text{Wtp} * \text{rh} - \text{rwh})}{(\text{Ctw} * \text{rh} - \text{rw} * \text{Cth})^2}$$

End Function

Function dCWtqliq(Wtp, Ctw, Cth, rw, rh, rwh)

$$\text{dCWtqliq} = \text{rw} * \text{Cth} / (\text{Cth} * \text{rw} - \text{rh} * \text{Ctw})$$

End Function

'standard deviation of result for liquid samples

Function sdCWtqliq(Wtp, Ctw, Cth, rw, rh, rwh, sdWtp, sdCtw, sdCth, sdRW)

$$\begin{aligned} \text{sdCWtqliq} = & \text{Sqr}(\text{dCtwliq}(\text{Wtp}, \text{Ctw}, \text{Cth}, \text{rw}, \text{rh}, \text{rwh})^2 * \text{sdCtw}^2 + \\ & \text{dCthliq}(\text{Wtp}, \text{Ctw}, \text{Cth}, \text{rw}, \text{rh}, \text{rwh})^2 * \text{sdCth}^2 + \\ & \text{dCrwliq}(\text{Wtp}, \text{Ctw}, \text{Cth}, \text{rw}, \text{rh}, \text{rwh})^2 * \text{sdRW}^2 + \\ & \text{dCWtqliq}(\text{Wtp}, \text{Ctw}, \text{Cth}, \text{rw}, \text{rh}, \text{rwh})^2 * \text{sdWtp}^2) \end{aligned}$$

End Function

'Derivative functions to calculate standard deviation of error or result
'for solid samples

Function dCthS(Wtp, Ctw, Cth, rh, rwh)

$$\text{dCthS} = \frac{-\text{Ctw} * (\text{Wtp} * \text{rh} - \text{rwh})}{(\text{Ctw} * \text{rh} - \text{Cth})^2}$$

End Function

Function dCtwS(Wtp, Ctw, Cth, rh, rwh)

$$\text{dCtwS} = \frac{\text{Cth} * (\text{Wtp} * \text{rh} - \text{rwh})}{(\text{Ctw} * \text{rh} - \text{Cth})^2}$$

End Function

Function dWtpS(Wtp, Ctw, Cth, rh, rwh)

$$\text{dWtpS} = \text{Cth} / (\text{Cth} - \text{Ctw} * \text{rh})$$

End Function

Standard deviation of result for solid samples

Function sdCWtpS(Wtp, Ctw, Cth, rh, rwh, sdWtp, sdCtw, sdCth)

$$\begin{aligned} \text{sdCWtpS} = & \text{Sqr}(\text{dWtpS}(\text{Wtp}, \text{Ctw}, \text{Cth}, \text{rh}, \text{rwh})^2 * \text{sdWtp}^2 + \\ & \text{dCthS}(\text{Wtp}, \text{Ctw}, \text{Cth}, \text{rh}, \text{rwh})^2 * \text{sdCth}^2 + \\ & \text{dCtwS}(\text{Wtp}, \text{Ctw}, \text{Cth}, \text{rh}, \text{rwh})^2 * \text{sdCtw}^2) \end{aligned}$$

End Function

'Calculate the concentration of the tracer in the waste for a specific percentage
'of water that is from the HHF. This is used for graphing the acceptable range
'of corrections

Function Ctws(Wtp, Cth, rwh, Pr)

$$\text{Ctws} = \text{Pr} / \text{rwh} * \text{Wtp} * \text{Cth}$$

End Function

Function Ctwl(Wtp, rw, Cth, rwh, Pr)

$$\text{Ctwl} = \text{Pr} / \text{rwh} * \text{Wtp} * \text{Cth} * \text{rw}$$

End Function

' Calculate the percent of the water that is from HHF

Function Pws(Wtp, Ctw, Cth, rwh)

$$\text{Pws} = \text{Ctw} * \text{rwh} / \text{Wtp} / \text{Cth}$$

End Function

Function Pwl(Wtp, Ctw, rw, Cth, rwh)

$$\text{Pwl} = \text{Ctw} * \text{rwh} / \text{Wtp} / \text{Cth} / \text{rw}$$

End Function

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