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Applying Incremental Sampling Methodology to Soils Containing Heterogeneously Distributed Metallic Residues to Improve Risk Analysis

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Abstract
This study compares conventional grab sampling to incremental sampling methodology (ISM) to characterize metal contamination at a military small-arms-range. Grab sample results had large variances, positively skewed non-normal distributions, extreme outliers, and poor agreement between duplicate samples even when samples were co-located within tens of centimeters of each other. The extreme outliers strongly influenced the grab sample means for the primary contaminants lead (Pb) and antimony (Sb). In contrast, median and mean metal concentrations were similar for the ISM samples. ISM significantly reduced measurement uncertainty of estimates of the mean, increasing data quality (e.g., for environmental risk assessments) with fewer samples (e.g., decreasing total project costs). Based on Monte Carlo resampling simulations, grab sampling resulted in highly variable means and upper confidence limits of the mean relative to ISM.

Keywords Soil sampling · Spatial statistical analysis · Heterogeneity · Metallic residue · Small-arms range · Upper confidence limit

There have been no previous studies comparing conventional grab and incremental sampling methodology (ISM) for metal contaminants from the same military small-arms range (SAR) despite the increasing applicability of ISM to characterize environmental loading of contaminants at a variety of sites (Hadley and Mueller 2012; ITRC 2012; Brewer et al. 2016a, b; Wroble et al. 2017). Studies on sampling of military sites with heterogeneous distribution of energetics has shown the ISM approach yields higher quality data representative of site conditions (Jenkins et al. 2001, 2005a; Hewitt et al. 2005, 2009; Walsh et al. 2005). This manuscript addresses this data gap by applying both sampling procedures to surface soil sampling at a site where heterogeneous distributions of metallic residues are expected.

Characterization of surface soils for environmental purposes typically uses conventional grab sampling conducted in a judgmental (e.g., biased) or random manner (USEPA 1995). Research has shown energetic residues released into the environment occur as particulates distributed in a heterogeneous manner (Jenkins et al. 2001, 2005a; Hewitt et al. 2009). Results from these studies indicate that when particulates are present, grab soil sampling is ineffective (Hewitt et al. 2005; Walsh et al. 2005). Recently, this work was extended from energetics to metallic residues at SAR sites (Clausen 2015).

An outcome of the recent research on the particulate nature of energetic residues (Hewitt et al. 2007, 2009) was modification to U. S. Environmental Protection Agency (USEPA) Method 8330 (USEPA 1996) for explosives, resulting in the updated Method 8330B (USEPA 2006a). Collectively, the modifications to the field sampling and sample processing techniques are referred to as ISM (ITRC 2012), multi-increment sampling (MIS)™ or incremental

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sampling. The Department of Defense (DoD), the regulatory community, and environmental consultants are now using ISM for other analytes such as metals (Hawaii 2008; Alaska 2009; Hewitt et al. 2012; ITRC 2012; Florida 2013). However, there has been no published research supporting the guidance for use of ISM versus conventional grab sampling for sites with heterogeneous distributions of metallic residues, such as at SARs.

This paper demonstrates the use of ISM to estimate mean metal concentrations at a SAR and compares the results with conventional grab sampling. Estimating the population mean is a typical goal of environmental studies such as background comparisons and risk assessments. The 95% upper confidence limit (UCL) of the mean is typically used for human and ecological risk assessments or comparisons with cleanup thresholds (USEPA 2002) except for lead where the arithmetic mean is used due to Integrated Exposure Uptake Biokinetic model assumptions (USEPA 2007).

Materials and Methods

Soil samples were collected from a 300 m^2 SAR berm at range 4–3 at Camp Ethan Allen, Vermont. The sand and gravel soil contains visible bullet fragments consisting of Pb and Sb with Cu and Zn present in the bullet jacket (Clausen et al. 2004; Clausen and Korte 2009). In addition, a background location approximately one-half mile from the range was sampled with the ISM approach in triplicate. Clausen et al. (2012) presents additional information on the characteristics of the range.

To obtain sufficient data for performing a statistical analysis, 30 grab samples were collected using systematic random sampling based on the recommendations from Matzke et al. (2010) and USEPA (1995). Grab soil sampling followed procedures typically utilized by the environmental industry such as steel scoops to obtain sufficient material to fill a 4 oz glass container. (Clausen et al. 2012). Grab sample processing followed USEPA Method 3050B (USEPA 1996), which typically involves collecting a single 1–2 g from the top of the sample container for aliquot digestion.

Collection of ISM surface soil samples followed the methodology outlined in Clausen et al. (2013a). To address compositional and distributional heterogeneity, this sampling strategy requires acquisition of an adequate number of contaminant particles. In other words, the particles must be present in the sample in roughly the same proportion as in the decision unit (DU) for the results to be accurate (ITRC 2012). The DU is the smallest area of interest for which one plans to make a decision based on the outcome of the soil contaminant concentration data. In this case, the DU was the entire berm face. The ISM involved the collection of evenly spaced increments (Clausen et al. 2012) to a depth of 5 cm with a 2-cm diameter corer. Multiple increments from the same DU were combined to form a single sample with a total mass > 1 kg, consistent with USEPA Method 8330B (USEPA 2006a) and recommendations by Hewitt et al. (2005, 2007, 2009, 2012); ITRC (2012). Seven replicate ISM samples consisting each of 5, 10, 20, 30, 50, and 100 increments in addition to a single 200-increment sample were collected. This is more sampling than typical but we wanted to assess whether the number of increments effects data quality. In order to statistically evaluate the data we collected seven replicates for each different number of increment samples. We would have preferred to collect 20 replicates to have more power in our statistical analysis but that was not feasible. The ISM samples were processed using a modified method in which samples were air-dried, passed through a 10-mesh sieve prior to milling, milled, and then subsampled (Clausen et al. 2013a). Milling involved grinding the sample to < 2 mm using a Lab Tech Essa chrome steel ring mill grinder (Model LM2, Belmont, Australia). For subsampling, the milled soil was spread over a sheet of aluminum foil as a thin layer 1–2 cm thick; and 20 increments were collected using a flat spatula in a manner similar to field sampling and combined to yield a 2 g digestion aliquot.

All metal analyses were performed by inductively coupled optical emission using a Thermo Scientific ICAP 6000 Series and Method 6010C (USEPA 2006b). Method and quality assurance/quality control details are provided in Clausen et al. (2013a).

Results and Discussion

Table 1 presents descriptive statistics for Pb and Sb for the 30 grab sample results; the six sets of ISM results; a set of ISM results pooled from the 30, 50, and 100 increment samples; and laboratory replicate analyses of the 200-increment ISM sample. Cu and Zn, often present as SAR contaminants had concentrations only slightly higher than background. As such, Zn, in particular, had similar variance as the native metals (e.g., Al, Fe, Ni, etc; see Clausen 2015). At sites where Cu and Zn concentrations exceed background levels their behavior mimicked that of Pb and Sb. It should be noted that Cr had elevated concentrations in the ISM samples (Clausen 2015), but is not discussed further because the samples were believed to be contaminated by the milling equipment that contains chrome-steel grinding surfaces (Clausen et al. 2012).

Table 1 provides the descriptive statistics for the grab and ISM Pb and Pb results (left side) and the mean and RSD for the ISM samples with varying number of increments (right side). The pooled mean value includes those ISM samples with RSD < 20%, i.e. samples with > 30

Table 1 Statistical summary of grab and ISM samples collected at Camp Ethan Allen

	Grab	ISM	Grab	ISM	Increments	Mean[a]		RSD	
						Pb	Sb	Pb	Sb
	Pb	Pb	Sb	Sb	Mean 5-inc.[a,b]	2989	23.5	25	25
n	30	21	30	21	Mean 10-inc.[a,b]	2132	18.5	32	63
Mean[a]	5060	2583	87.8	21	Mean 20-inc.[a,b]	2689	23.1	30	50
Median[a]	1238	2539	10	20.2	Mean 30-inc.[a,b]	2664	22.7	14	15
Minimum[a]	43.9	1835	0.898	15	Mean 50-inc.[a,b]	2156	17.6	11	11
Maximum[a]	79,020	3595	2072	28.9	Mean 100-inc.[a,b]	2929	22.8	3	2
STD[a]	14,438	488	375	3.85	Mean 200-inc.[a,e]	2717	22.6	NA	NA
RSD	285	19	427	18	$\text{Mean}_{\text{Pooled}}$[a,c]	2589	21.1	NA	NA
					RPD[d]	1.2	1.7	NA	NA

NA not applicable, *STD* standard deviation, *RSD* percent relative standard deviation

[a]Units of mg/kg

[b]ISM samples consisting of x increments with field replicates n = 7

[c]Pooled DU Mean calculated from 22 ISM field samples and 100 analyses

[d]RPD = relative percent difference between mean of the pooled mean and mean for the 200-increment field sample which was analyzed 30 times

[e]Single sample with n = 30 laboratory replicate analyses

increments. The grab sample means for Pb and Sb were 4–7 times greater than the medians; and the percent relative standard deviations (RSD) values were 285% and 427%, respectively (Table 1). The large outliers (e.g., Pb at 79,020 mg/kg, suggesting the presence of Pb particles in the sample) and the larger sample means relative to the medians are indicative of positively skewed Pb and Sb distributions. The median Pb grab sample concentration (1238 mg/kg) was several times smaller than the mean (5060 mg/kg), indicating a small number of grab samples at a site such as this will likely underestimate the DU mean. In some cases, a grab sample will significantly underestimate the mean (e.g., the minimum Pb value of 43.9 mg/kg suggests no anthropogenic Pb present). In contrast, means and medians were nearly equal for the metals assumed to be naturally occurring (Al, Ba, Cd, Co, Fe, Mg, Mn, Ni, P, Sr, and V) with RSDs < 30% (Clausen 2015).

Unlike the grab results for Pb and Sb, the ISM results (consisting of 30, 50, and 100 increments, and the pooled dataset) had estimated means similar to the medians, and the RSDs were < 25% for all anthropogenic and native metals (Clausen 2015). As shown in Table 1, the ISM results exhibited much higher precision than the grab sample results (e.g., Pb and Sb ISM RSD results ranged from about 2%–63% whereas Pb and Sb grab results ranged from 285% to 427%). Consistent with our prior work on explosives and Gy's sampling theory a minimum of 30 increments are necessary to yield acceptable data quality. As a general rule of them we prefer RSDs of < 10%, which would suggest for this particular site and the level of heterogeneity 100-increment samples would be appropriate.

As discussed in Clausen (2015) if the set of grab samples is repeatedly resampled using a bootstrap method, a wide range of different estimates of the Pb mean concentration occur. This raises several questions: (1) Which grab sample estimate of the mean provides the "best" estimate of the "true" Pb concentration within the DU? (2) How many grab samples are needed for a representative statistical sample? and (3) Would an alternative sampling approach such as ISM provide an estimate of the mean with less uncertainty? Clausen (2015) investigated the number of grab samples necessary to yield reproducible estimates of the mean; the results suggested > 35 samples are needed for a 300 m^2 area.

To estimate the DU mean, the single 200-increment, seven 30-increment, seven 50-increment, and seven 100-increment ISM data were pooled. This pooled dataset consisted of 100 analyses from 22 ISM samples with 1460 increments in total (e.g., the pooled ISM Pb mean was 2589 mg/kg).

ISM RPDs were generally < 20% for all samples, except the 1 consisting of 10 increments. The high Cu and Zn RPDs suggests the presence of Cu-jacket material in the sample.

The previous work at SARs (Clausen 2015) has shown that a reliable estimate of the mean requires at least 35 grab samples to obtain an RSD ≤ 30%. The RSD target of ≤ 30% for total precision is based on the work of Walsh et al. (2005) for energetics. Prior resampling simulations showed each independent grab sampling event yielded significantly different estimates of the mean based on two-tailed Kruskal Wallis tests for the medians and Levene's test for the variances (Clausen 2015). These findings are of concern because environmental practitioners are often pressured to collect the fewest number of samples possible to save sampling time and analytical costs (ITRC 2012). Typically fewer than 35 grab samples would normally be collected (Jenkins et al. 2005b; Hadley et al. 2011) for this particular size of DU.

To estimate how many grab samples are necessary to achieve the same level of data quality obtained with ISM, a bootstrap (resampling) method for different numbers of grab samples *m* was performed. Three hundred bootstrap simulations were performed for each value of *m*. Figure 1 is a plot of the standard error of the mean (SE) (from the simulations) versus the number of grab samples (*m*). The simulations indicate the SE declines with an increasing number of grab samples, and the trend of the data can be fitted with a power curve with a coefficient of determination $R^2 = 0.9851$. If the fitted curve for the grab samples is extrapolated to the SEs for the ISM samples prepared from 30 to 100 increments, the plot suggests approximately 100 grab samples would be needed to achieve the same data quality (i.e., the same SE) as a single ISM sample of 30-increments. For many projects, the necessity of collecting 100 grab samples would be cost prohibitive. The time and cost to collect three ISM samples is 5%–50% lower than for 7–15 grab samples (Clausen et al. 2013b). In contrast, collecting three to seven 100-increment samples from a DU is sufficient for most situations (ITRC 2012).

Increasing the number of increments decreased the range in Pb grab sample values, resulting in distributions more Gaussian shaped, and presumably improved the estimates of the mean (Clausen 2015). Such results are consistent with the central limit theorem in statistics, which states that even when the distribution of individual results are non-normal, the distribution of means will approach a normal distribution as the sample size *n* increases. As was observed for small numbers of grab samples, a small number of increments tended to underestimate the mean for Pb, Cu, Sb, and Zn. This observation is consistent with the findings for impact

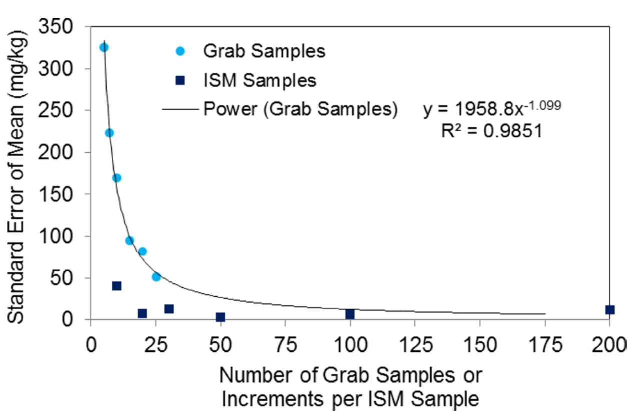

Fig. 1 SE of the mean versus number of grab samples or increments per ISM sample

areas containing energetic particulates from the detonation of military munitions (Jenkins et al. 2004, 2005b).

For this reason, previous studies on the use of ISM for soils with energetic residues recommended the collection of a minimum of 30 increments per sample and preferably 50–100 (Hewitt et al. 2005, 2007, 2009, 2012; Jenkins et al. 2004, 2005b; Walsh et al. 2005). The present study supports this recommendation of collecting a minimum of 30 increments and preferably ≥ 100 to properly address the distributional heterogeneity. Our earlier work (Clausen and Korte 2009) suggested the metal distribution at SARs may not be as heterogeneous as the distribution of energetics at artillery and mortar, anti-tank, and grenade impact areas, but it is still significant.

The data presented in this paper demonstrate the likelihood that grab samples will yield a negative bias when estimating the mean for sites with heterogeneous contamination—values that are often used to assess risk and to determine whether remedial actions are necessary. Table 2 presents 95% UCLs calculated using USEPA's ProUCL Version 5.0 software (USEPA 2013) to assess the impact that the sampling method had on the estimates of risk. The calculated UCLs are greater for the Pb and Sb grab data as compared with the ISM data. See earlier discussion about Pb. Pb UCL values calculated for illustrative purpsoses. The differences are a consequence of the large variability and positively skewed distributions of the grab sample concentrations that ProUCL identified as consistent with lognormal distributions.

ProUCL selected the (H-UCL) Land's H-statistic as the most appropriate UCL for Pb and the Chebyshev lognormal UCL for Sb. For comparison purposes, Table 2 presents the Student's *t*, Student's *t* test modified for skewness, gamma distribution, and Chebyshev UCLs for all of the data sets (though the last three UCLs are more appropriate of positively skewed distributions). Multiple distributions are presented in Table 2 as the results fit several distributions in addition to the one recommended by ProUCL. ProUCL evaluates fits using three theoretical distributions: normal (N), lognormal (L) and gamma distributions (G).

Because the calculated Pb 95% UCLs are greater than the USEPA screening level (RSL) for residential soil, both grab and ISM data would result in the same decision: contaminant concentrations exceeded the RSL and remediation is required. If, however, the environmental driver at the site was Sb under an industrial scenario (Sb RSL = 47 mg/kg), then different outcomes are possible depending on the sampling method. In the present study, grab sample results (92–387 mg/kg Sb) indicated remediation is necessary and ISM samples (19–29 mg/kg Sb) suggested no-action.

In conclusion, the advantages of ISM over conventional grab sampling are its excellent precision for estimating the DU mean and its ability to represent site chemical conditions

Table 2 Summary of ProUCL 95% UCLs calculated using set of 30 grab samples and the 30, 50, and 100 increment samples consisting of 7 replicates each

Test	Sample				
	Type	Cu[a]	Pb[a]	Sb[a]	Zn[a]
Distribution	Grab	N, S, G, L	L	L	N, S, G, L
Distribution	ISM 30		N, G, L	N, G, L	N, G, L
Distribution	ISM 50	N, G, L	N, G, L	N, G, L	N, G, L
Distribution	ISM 100	N, G, L	N, G, L	N, G, L	N, G, L
Student's *t*	Grab	341[b]	NA	NA	71.6[a]
H (lognormal)	Grab	365	9429[b]	92	72.5
Chebyshev (lognormal)	Grab	429	9457	387[b]	80.5
Student's *t*	Grab	341	NA	NA	89.8
Gamma distribution	Grab	351	NA	NA	72.3
Student's *t*	ISM 30	636[b]	2933[b]	25.1[b]	70.5[b]
Chebyshev	ISM30	714	3268	28.1	74.1
Student's *t*	ISM 50	527[b]	2335[b]	19.0[b]	72.0[b]
Chebyshev	ISM50	615	2557	20.7	78.0
Student's *t*	ISM 100	772[b]	3287[b]	25.6[b]	92.9[b]
Chebyshev	ISM 100	936	3746	29.1	103
EPA RSL for residential soil	NA	310	400	3.1	2300

N normal distribution, *S* skewed distribution, *G* gamma distribution, *L* lognormal distribution, *NA* not applicable, *UCL* upper confidence limit, *H-UCL* UCL based on Land's H-statistic, *ISM* incremental sampling methodology, *EPA* Environmental Protection Agency, *RSL* recommended screening level

[a]Units of mg/kg

[b]UCL selected as most representative by ProUCL

with fewer samples than grab samples would support. This is critical for environmental work because inferences about human and ecological risk depend on the means of the DUs, which are typically estimated using 95% UCLs. Reliable estimates of DU means are problematic for grab sampling, when the sample sizes are small, especially when there is large variability and positively skewed distributions. A small set of replicate ISM samples (3–5), with a minimum of 30 increments provides a comparable or better estimate of the DU mean than a much larger number (> 30) of grab samples, reducing the total cost of sampling collection, processing, and analysis. Unless a large number of grab samples are collected, unreliable estimates of the mean are likely when the contaminant of interest is a solid distributed heterogeneously. This error due to compositional and distributional heterogeneity will not be apparent when collecting a few grab samples. This is of particular concern for energetic compound residues and metallic fragments deposited on DoD training lands, as grab sample results will likely be highly variable.

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