Whitepaper

Stockpile Sampling for Soil Reuse at a Site

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1.0 INTRODUCTION

Soil stockpiles are often used as fill material for a site. To ensure that chemical concentrations will not pose a risk to human health, these stockpiles must be adequately sampled before reuse. The definition of adequate sampling varies by state and government agency. Therefore, a wide variety of stockpile sampling approaches exist. The goal of each of these methodologies is to generate a representative sample for each volume of soil sampled. This will protect human health and ensure that informed and accurate decisions are made regarding future use of the stockpile soil.

This whitepaper includes an evaluation of the appropriate number and type of samples that should be collected from a stockpile of soil of a given size for soil reuse at a site. As requested by the Florida Department of Environmental Protection (FDEP), the evaluation considers the appropriateness of discrete versus incremental composite sampling, the number of increments for each composite, sample locations in the stockpile, and the number of samples that should be taken per cubic yard. To identify sampling methodologies for inclusion in this whitepaper, we reviewed the literature for stockpile sampling protocols that were validated in the field or by simulation studies. Additionally, we searched for current stockpile sampling methods utilized by state and federal agencies. These searches were not limited to the United States to ensure that a diverse range of approaches was represented.

2.0 BASIS FOR SAMPLING PROTOCOLS

There are two general types of stockpile sampling protocols: 1) protocols instituted by policy and 2) protocols that utilize a statistically validated approach to estimate the mean concentration for a specified volume of soil. Our search identified nine different stockpile sampling approaches. Four of these approaches utilized sampling theory and/or field validated studies to produce the recommendations. The other five approaches utilized sampling approaches based on policy decisions and historical sampling methodologies. These approaches are summarized in Table 1.

3.0 DISCRETE AND COMPOSITE SAMPLING

3.1 Discrete sampling

Two of the sampling approaches utilize discrete sampling, including those implemented by the Environmental Protection Agency (EPA) of Victoria, Australia and the Department of Toxic Substances Control (DTSC) in California. These discrete sampling protocols are not statistically-based. The DTSC recommends one discrete sample per 250 yd^3 with a smaller sampling density for stockpiles greater than 1,000 yd^3 (Table 2). The guidance states that if contaminants exceeding acceptable criteria are identified in the stockpile, the material is deemed unacceptable and new fill material will be necessary. Composite sampling may be accepted depending on the homogeneity of the contaminant in the stockpile. The DTSC does not accept composite sampling for volatile and semivolatiles compounds (DTSC, 2001). The Victoria EPA recommends one discrete sample per 25 m^3 (32.7 yd^3) with a minimum of three samples. The number of samples can be reduced if a 95% upper confidence limit (UCL) is utilized for the soil. If a 95% UCL is utilized only 10 samples are required to calculate a UCL and this sampling density can be utilized on stockpiles up to 3,000 m^3. When stockpiles are greater than 3,000 m^3, one sample should be taken for every 250 m^3 and incorporated into the 95% UCL calculation (Table 3). Similar to the DTSC, soils are categorized based on the maximum sampling result. However, the Victoria EPA

sampling density is approximately eight times greater than the DTSC. The guidance notes that a 95% UCL calculation is best suited for homogenous soils and that a higher number of samples may be required if the stockpile is heterogeneous. Whether using discrete or composite samples, sampling should be uniformly distributed throughout the stockpile both horizontally and vertically. A 3-dimensional systematic grid sampling design should be applied to the stockpile to account for variability in contaminant distribution (Victoria EPA, 2009). The Canadian Council of Ministers of the Environment (CCME) recommends discrete sampling for volatile chemicals. Fifteen discrete samples are taken from locations uniformly distributed throughout the stockpile (CCME, 2016).

3.2 Conventional composite sampling

Composite sampling of stockpiles is recommended by the majority of the agencies/organizations included in this review. Composite sampling includes non-structured (conventional composite sampling) as well as structured composite sampling (i.e., incremental sampling). For clarity, incremental sampling approaches are summarized separately from conventional composites. Conventional sampling approaches are summarized in Table 4.

3.2.1 Decision unit size

Decision unit (DU) size for conventional composite sampling approaches varies considerably among the agencies/organizations reviewed in this document. The Netherlands' National Institute for Public Health and the Environment (RIVM) and Minnesota Department of Agriculture (MDA) recommend assessment on the scale of the whole stockpile. Therefore, the decision unit size will vary depending on the size of the stockpile (Lamé et al., 2005; MDA, 2005; MDA, 2011). The Ministry of the Environment in British Columbia (BCME) and the CCME define the decision unit based on the volume of soil. These agencies state stockpiles should have a maximum size of 250 m^3. Each stockpile is divided into five equal cells that are individually compared against criteria (BCME, 2009; CCME, 2016). The BCME states that the stockpile can be managed as a whole or on a cell-by-cell basis. If managed as a whole stockpile, all of the individual cell composite samples need to be below criteria or the stockpile is classified as exceeding soil quality criteria. If managed on a cell-by-cell basis, cells that exceed soil quality criteria are segregated from the stockpile. The BCME also requires one additional representative cell sample from an uncontaminated cell to confirm chemical concentrations (BCME, 2009). The International Organization for Standardization (ISO) does not specify a decision unit size. The guidance is written on the scale of the entire stockpile. However, it also specifies that stockpile size can be based on policy (ISO, 2018).

3.2.2 Sampling unit size

Sampling unit size also varies both within and among the agencies reviewed. The BCME specifies a sampling unit size of 10 m^3. For the BCME the sampling unit size is consistent and the number of sampling units required for each stockpile varies depending upon the soil volume of the stockpile (BCME, 2009). The CCME and RIVM utilize the whole stockpile as the sampling unit. For these agencies, the number of sampling units per stockpile is consistent (one) and the size of the sampling unit varies depending of the size of the stockpile. (Lamé et al., 2005; CCME, 2016). Both the sampling unit size and number of sampling units varies for the MDA depending upon the volume of the stockpile (Table 5).

3.2.3 Number of increments

The RIVM recommends 50 increments per composite sample. This recommendation is based on digital modeling. For the analysis, 30 models of simulated 2,000-ton soil stockpiles were created based on three genuine stockpile datasets. The simulated stockpiles were sampled using different numbers of increments varying from 10 to 200. Each stockpile model was sampled a large number of times and a distribution of results was obtained. It was concluded that two replicates of 50 increments would be sufficient for obtaining a reliable estimate of the true mean of the soil stockpile. This methodology is estimated to predict the true mean of inorganic contaminants with 97-98% confidence and of organics with 75-82% confidence (Lamé et al., 2005). The ISO recommendation for 50 increments is based on the study by RIVM (ISO, 2018). The MDA recommends 4-18 increments for each composite sample. The soil sampling guidance states that each composite sample should be created using 4-6 soil boring locations and 1-3 subsamples collected from each soil boring. The guidance states that all increments should be collected from a depth of greater than one foot below the stockpile surface (MDA, 2011). Additionally, the stockpile should be sampled in a manner that results in a representative sample including the area of suspected highest concentration (MDA, 2005). The BCME and the CCME do not specify the number of increments required to form a composite stockpile sample (BCME, 2009; CCME, 2016). The BCME guidance states that multiple increments (termed specimens) may be incorporated into the composite sample; however, a recommended number is not provided.

3.2.4 Number of replicates

The MDA, BCME, and CCME only recommend one composite sample per sampling unit (MDA, 2005; BCME, 2009; CCME, 2016). The BCME states that the recommended sampling methodology assumes that the stockpile is homogenous and that one sample will be representative. The RIVM recommends two replicate samples. The second sample is considered necessary to obtain an estimate of the heterogeneity of the stockpile. The ISO guidance describes three possible sampling approaches (ISO, 2018). In the first sampling approach, one replicate is taken from the stockpile and the increments are taken throughout the stockpile to obtain a representative sample. If the increments are distributed throughout the stockpile, the composite sample should provide an adequate estimate of the mean concentration. This approach provides an estimate of the mean, but not the variability. It is recommended for stockpiles where the variability is assumed to be minimal (i.e., homogenous). The second approach divides the stockpile into sampling units that are uniform in size. A composite sample is obtained from a representative number of sampling units. The number of sampling units that are required to obtain a representative sample is not provided. The composite samples for the sampling units are averaged to obtain an estimate of the mean concentration for the stockpile. In order to obtain an accurate estimate of the mean concentrations in the stockpile, a composite sample should be obtained for each sampling unit. The third sampling approach is equivalent to the first except more than one replicate is taken. Each of the replicates is an estimate of the mean concentrations in the stockpile. This approach provides an estimate of the variability of the stockpile. For both the RIVM and ISO sampling approaches that utilize more than one replicate to estimate variability, it is unclear how the replicates are analyzed to obtain the mean concentration of the DU.

3.3 Incremental sampling

Incremental sampling is a form of structured composite sampling. This type of sampling has a specific methodology that must be followed in order for the composite sample to be considered

an incremental sample. The methodology including placement of DUs, location of increments, and sampling pattern is summarized in the Interstate Technology & Regulatory Council (ITRC) incremental sampling methodology (ISM) guidance (ITRC, 2020). Use of this type of sampling methodology allows statistical inferences to be made about the sampled population as well as producing a defensible estimate of the mean concentration. Sampling of stockpiles using ISM is recommended by the ITRC and the Hawaii Department of Health (HDOH). Incremental sampling approaches are summarized in Table 6.

3.3.1 Decision unit size

The ITRC states stockpiles should be divided into volume-based DUs representative of exposure areas for future use. For example, if the soil is being reused in a residential development where lots are 0.25-acres each, the DU should be 200 yd^3. This represents a ¼-acre lot covered with six inches of fill material from the stockpile (ITRC, 2012; ITRC, 2020). The HDOH recommends DU volumes based on the receiving site land use and the chemical of concern (HDOH, 2017). These recommendations are summarized in Table 7. For volatile compounds, the recommended DU size is 20 yd^3. This is equal to six inches of fill material under a 1,000 ft^2 building (the default building size recommended in vapor intrusion models). The recommended DU size is also 20 yd^3 for highly leachable compounds. The HDOH states this is the minimum size of a spill area that could potentially cause significant leaching hazards. The DU size for unrestricted use is 100 yd^3, which is equivalent to a default residential exposure area in Hawaii (5,000 ft^2) covered in six inches of fill. The recommended DU size for schools, high-density residential developments, and commercial areas is 400 yd^3. This volume equates to six inches of fill material over a 20,000 ft^2 area (0.46 acres). If the fill material is from a former agricultural area, the HDOH requires that future use be commercial or industrial. A minimum of 18 DUs are recommended for characterization of the fill material. The DU volume will vary based on the size of the stockpile and the number of DUs chosen for characterization. When dredge material stockpiles are used for beach replenishment projects, the DU size is 800 yd^3. This equates to an exposure area size of 1 acre over a depth of six inches (HDOH, 2017). Sampling units were not utilized by the ITRC or the HDOH in the recommended stockpile sampling procedures.

3.3.2 Number of increments

The ITRC recommendation of 30-100 increments is based on statistical simulations and over a decade of ISM sampling in the field. Thirty increments are considered the minimum number of increments necessary to obtain a representative estimate of the mean concentration. More than 30 increments are necessary when the distribution of contaminants in soil is highly heterogeneous. Increments should be collected from the surface as well as the interior of the stockpile. The ITRC recommends a minimum of 30 systematic random or random increments within grids placed in both vertical and horizontal locations throughout the stockpile (ITRC, 2020). The HDOH recommends 30-75 increments per ISM sample. The recommendation by the ITRC is similar (30-100 increments). The HDOH states the recommendation for the number of increments is based on incremental sampling theory, ten years of field work in Hawaii, and other published information. Thirty increments are recommended for soil where the variability is expected to be low. The number of increments should increase as the variability increases. Seventy-five increments are used in areas of high variability or where contamination is present as small nuggets in soil. The HDOH also emphasizes that the increments should be collected from the entire three-dimensional mass of the stockpile (HDOH, 2020).

3.3.3 Number of replicates

The ITRC incremental sampling guidance states replicates are important to evaluate the precision of stockpile sampling. Replicates should be collected in the same manner as the original sample, but in separate random locations. A minimum of three replicates are required to calculate a 95% UCL estimate on the mean concentration (ITRC, 2020). The HDOH recommends three replicate samples in 10% of the DUs. The remainder of the DUs only require one increment sample to assess the mean concentration. For very large stockpile volumes, it may be impractical to sample every DU. Therefore, the HDOH recommends the sampling of a minimum of 59 DUs from large stockpiles. This represents the number of samples necessary to assume the stockpile is uncontaminated with 95% confidence if all the tested DUs are below criteria. The DUs chosen for sampling should be systematically, randomly selected from the stockpile. If one or more sampled DUs exceed criteria, then additional testing of the fill material will be necessary to determine which DUs are unacceptable for use as fill (HDOH, 2017).

4.0 EXPOSURE POINT CONCENTRATION

For discrete sampling, the DTSC and Victoria EPA utilize the maximum concentration as the exposure point concentration (EPC). If the maximum concentration exceeds the soil criteria, then the stockpile is not suitable for use as fill material. The Victoria EPA also allows 95% UCL for homogenous soil. For volatile samples, the BCME and CCME utilize discrete sampling. The maximum discrete concentration of the volatile is compared to regulatory criteria.

The BCME and CCME recommend one composite sample per sampling unit. The BCME compares the mean concentration in each sampling unit to regulatory criteria. This methodology assumes that the stockpile is homogenous and that replicates are not needed to obtain an accurate estimate of the mean. As stated above, each stockpile is divided into five cells and one composite is taken per cell. The composite for each cell is split into two subsamples and a stockpile composite sample is created from half of the split sample. This composite is used to represent the mean concentrations in the stockpile. The other half of the sample is used to represent the mean concentrations in the cell. To account for variability, the following equation may be used:

$$Calculated\ Value = C_S + [abs(C_s - C_x)]$$

The calculated value in the stockpile is equal to the mean concentration in the stockpile (C_s) plus the absolute value of the difference between the mean stockpile concentration and the mean concentration in that cell (C_x). Both the individual cell composites and the calculated value should be below regulatory criteria. The BCME provides a table for interpretation of the results when one or more cells or the calculated value is above criteria. The chart lays out a detailed procedure that allows the stockpile to be managed as a whole or cell by cell depending on the number of cells that exceed criteria (BCME, 2009). The CCME recommends a similar procedure of dividing the stockpile into five cells and taking a composite from each cell. A stockpile composite is also made from split cell samples. For stockpiles smaller than 50 m^3, the individual cell and stockpile composites are compared to regulatory criteria. For stockpiles larger than 50 m^3, a quasi-statistical approach is recommended. The BCME approach described above is cited as one possible quasi-statistical approach (CCME, 2016).

The MDA uses the average concentration obtained from a single composite sample as the exposure point concentration. A composite sample is taken for each subsection of the

stockpile as described in Table 5. The composite sample for each subsection is compared to regulatory criteria. Alternatively, a weighted average can be calculated for the entire stockpile using the composite samples for each subsection. If a stockpile average is used, the pile must be mixed thoroughly before reuse to ensure homogeneity (MDS, 2005). RIVM recommends two composite samples taken throughout the entire stockpile. Two samples are recommended to obtain an estimate of the heterogeneity in sampling as well as possible information on procedural error. The two composites are averaged together to estimate a mean concentration of contaminants in the stockpile (Lamé et al., 2005). The ISO guidance recommends three sampling approaches (summarized in Section 3.2.4). For each method, the ISO utilizes the mean of the composite samples as the exposure point concentration for the stockpile (ISO, 2018).

The HDOH and the ITRC utilize incremental sampling methods to estimate a mean concentration, which is used as exposure point concentration. As stated in Section 3.3.3, the HDOH obtains incremental samples in triplicate from 10% of the DUs to evaluate the precision of field sampling methods and one incremental sample from the remainder of the sampled DUs. The calculation of a 95% UCL is not required if the relative standard deviation (RSD) of the triplicate samples is less than 35%. If a 95% UCL is used for these data, the Student's t-test is recommended for the calculation. If the RSD is greater than 35%, a 95% UCL is calculated from the triplicate data using the Chebyshev method. For DUs where replicate data are not available, the concentration of contaminant i in the DU is multiplied by the ratio of the 95% UCL and the mean for the replicate data set to estimate the 95% UCL:

$$Estimated\ 95\%\ UCL\ =\ C_i + \left(C_i \times \frac{95\%UCL}{X}\right)$$

where C_i is the concentration of contaminant i and X is the mean concentration of the replicate data set used to calculate the initial 95% UCL (HDOH, 2020). The ITRC recommends using the 95% UCL concentration on the mean as the exposure point concentration. The 95% UCL is calculated using a minimum of three replicates and a 95% UCL calculation tool developed for the ITRC (ITRC 95% UCL calculation tool, 2020). The calculation tool chooses the correct equation for the calculation of a 95% UCL (Chebyshev or Student's t-test). The Chebyshev method produces a more accurate estimate of the 95% UCL when the coefficient of variation of the increments is above 0.23 or the coefficient of variation of the replicates is above 1.5. In some cases, triplicate sampling may not be feasible for every DU. For DUs where only one incremental sample is taken, the 95% UCL can be extrapolated based on the variance obtained from the DUs sampled in triplicate if the site conceptual model is the same for both DUs. The 95% UCL for the DU with one ISM sample is calculated using the equation:

$$95\%\ UCL\ =\ \bar{x} + 4.36(s_{pooled})$$

where $\bar{x}$ is the mean contaminant concentration in the singly sampled DU and s_{pooled} is the square root of the mean variance from all triplicate sampled DUs (ITRC, 2020).

5.0 RECOMMENDATIONS

Based on our review of stockpile guidances, we recommend sampling using ISM. ISM is a structured composite sampling methodology that reduces data variability and provides an accurate estimate of the mean concentration. Typically, an ISM sample is obtained by dividing the stockpile into DUs. The DU is divided into a number of grids equivalent to the number of

increments in the sample. Then, one increment is taken from each grid at a location determined using a random number generator. The location can be the same or a new location can be determined for each grid. This type of sampling is more representative of the stockpile than discrete or traditional composite sampling (ITRC, 2020).

We recommend a minimum of 30 increments and three replicates for each ISM sample. Thirty increments were shown to achieve 95% coverage of the mean in simulation studies when three replicates are used. This recommendation is also supported by a decade of practitioner experience that concludes 30 increments is sufficient to control the random heterogeneity in most instances. The sample mean from one ISM sample is more likely to underestimate the mean than overestimate it due to the lognormal distribution of environmental datasets (ITRC, 2020). In order to be health protective and provide an accurate estimate of the mean concentration, a 95% UCL should be used. For the calculation of a 95% UCL, a minimum of three replicates are required. All DUs should be sampled in triplicate. We do not recommend extrapolation of variance between DUs to calculate a 95% UCL unless it can be reasonably assumed that the distribution of contaminants in the stockpile is homogenous.

Decision units are defined as the volume of soil over which decisions should be made. For ease of analysis, we recommend setting the DU size equivalent to the exposure area of the receptor of concern. For example, we recommend a 200 yd^3 for residential reuse. This is approximately equal to the volume of soil needed to cover a 0.25-acre yard, six inches deep. Because stockpiles are various shapes and sizes, accessing DUs in the interior of a stockpile could be difficult or unsafe. The HDOH (2017) recommends two possible approaches for stockpile sampling. Stockpiles can be flattened or spread to a specified depth and grids are assigned across the surface of the stockpile. The soil is sampled similar to surface soil ISM sampling. If the stockpile cannot be flattened, increments can be sampled from a vertical side of the stockpile to a depth equivalent to a volume of the DU. These samples would be taken horizontally into the stockpile. The sampled DU is then removed to reveal another vertical soil wall for sampling. This method is repeated until the stockpile is depleted. Regardless of the stockpile sampling method, increments should be distributed across the surface of the DU and at depth so that increments accurately represent the entire DU volume.

5.0 REFERENCES

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Table 1 – Summary of stockpile sampling approaches

Agency or Organization	Sampling Theory or Policy Based	Discrete or Composite Samples	ISM or Conventional Composites	Reference
Hawaii Department of Health	Sampling theory	Composite	ISM	HDOH, 2017
International Organization for Standardization	Sampling theory	Composite	Conventional	ISO, 2018
Netherlands' National Institute for Public Health and the Environment	Sampling theory	Composite	Conventional	Lamé et al., 2005
Interstate Technology & Regulatory Council	Sampling theory	Composite	ISM	ISM, 2020
Minnesota Department of Agriculture	Policy	Composite	Conventional	MDA, 2011
Victoria, Australia Environmental Protection Agency	Policy	Discrete	NA	Victoria EPA, 2009
Department of Toxic Substances Control, California	Policy	Discrete	NA	DTSC, 2001
Ministry of the Environment, British Columbia	Policy	Composite	Conventional	BCME, 2009
Canadian Council of Ministers of the Environment	Policy	Composite	Conventional	CCME, 2016

EPA – Environmental Protection Agency; ISM – Incremental Sampling Methodology; NA – not applicable

Table 2 – Discrete stockpile sampling approach for the Department of Toxic Substances Control, California

Volume of stockpile (yd^3)	Samples
<1,000	1 sample per 250 yd^3
1,000 to 5,000	4 samples for first 1,000 yd^3 plus 1 sample for each additional 500 yd^3
>5,000	12 samples for first 5,000 yd^3 plus 1 sample for each additional 1,000 yd^3

Source: DTSC, 2001

Table 3 – Discrete stockpile sampling approach for the Victoria, Australia Environmental Protection Agency

Soil volume (m^3)	Number of samples (1:25 m^3)[a]	Minimum number of samples for the 95% UCL
≤25	3	10
50	3	10
75	3	10
100	4	10
125	5	10
150	6	10
175	7	10
200	8	10
300	12	10
400	16	10
500	20	10
600	24	10
700	28	10
800	32	10
900	36	10
1,000	40	10
1,500	60	10
2,000	80	10
2,500	100	10
3,000	120	12 (1:250 m^3)
4,000	160	16 (1:250 m^3)
5,000	200	20 (1:250 m^3)
>5,000	1:25 m^3	1:250 m^3

Source: Victoria EPA, 2009; [a] – One sample per 25 m^3 of soil

Table 4 – Conventional composite sampling approaches

Agency or Organization	Decision Unit Size	Sampling Unit Size	Number of Increments	Number of Replicates	Reference
International Organization for Standardization	Not specified[a]	Not specified[a]	50	1 or more	ISO, 2018
Netherlands' National Institute for Public Health and the Environment	Whole stockpile[b]	Whole stockpile[c]	50	2	Lamé et al., 2005
Minnesota Department of Agriculture	Whole stockpile[b]	Varies[d]	4-18	1	MDA, 2005; MDA, 2011
Ministry of the Environment, British Columbia	10 to 50 m^3	10 m^3	Not specified	1	BCME, 2009
Canadian Council of Ministers of the Environment	2 to 50 m^3	2 to 50 m^3 [c]	Not specified	1	CCME, 2016

[a] – May represent the whole stockpile or defined volumes specified by the agency; [b] – Decision unit size will vary based on the size of the stockpile; [c] – Sampling unit size is equal to decision unit size; [d] – Sampling unit size is based on the size of the stockpile, see Table 5

Table 5 – Number of sampling units per stockpile recommended by the Minnesota Department of Agriculture

Volume of Soil in Stockpile (yd^3)	Number of Sampling Units
<200	1
200-500	2
500-1,000	3
1,000-5,000	4
Each additional 2,000 yd^3	1

Source: MDA, 2011

Table 6 – Incremental sampling approaches

Agency or Organization	Decision Unit Size	Sampling Unit Size	Number of Increments	Number of Replicates	Reference
Hawaii Department of Health	Based on exposure area[a]	NA[b]	30-75	1 or 3[c]	HDOH, 2017
Interstate Technology & Regulatory Council	Based on exposure area[d]	NA[b]	30-100	3 or more	ITRC, 2012 ITRC, 2020

[a] – Decision unit size is dependent upon the receiving site land use and the contaminant of concern, see Table 7; [b] – Not applicable, sampling units were not utilized for sampling stockpiles; [c] – Three replicates are recommended for 10% of the decision units. The remaining decision units only require one replicate; [d] – Decision unit size is equivalent to the exposure area for the receptor of concern at the receiving site

Table 7 – Decision unit sizes recommended by the Hawaii Department of Health

Contaminant of Concern	Receiving Site Land Use	Recommended Decision Unit (yd^3)
Volatile Compounds	Any	20
Highly Leachable, Non-Volatile Contaminants	Any	20
Low Mobility Contaminants	Unrestricted use	100
	Schools and high-density residential developments	400
	Commercial or industrial use	400
	Commercial or industrial use (agricultural field source)	Varies[a]
	Beaches (replenishment projects)	800

[a] – The stockpile is divided into a minimum of 18 decision units. Decision unit size is dependent upon size of the stockpile

Source: HDOH, 2017