DEVELOPMENT AND VALIDATION OF A SAMPLING STRATEGY FOR ASSESSING THE ENVIRONMENTAL QUALITY OF (REUSABLE) SOIL

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ABSTRACT

The decision upon reuse, treatment or landfilling of soil, largely depends on the environmental quality of the soil. The exact contaminant concentrations are therefore essential for the determination of the (final) destination of soil stockpiles. This paper focuses on the development and validation of technical procedures to accurately determine the mean contaminant concentration in soil stockpiles. The challenge derives from the fact that, both organic and inorganic contaminants are distributed heterogeneously over the soil stockpile. Technical procedures involve sampling, sample pre-treatment and chemical analysis. Since chemical analysis procedures are well-developed and standardised, we focussed on the sampling process itself and the sample pre-treatment. We used the following approach:

Step 1 - Determination of contaminant heterogeneity in a limited number of soil stockpiles.
Step 2 - Policy decision on the basic features of the sampling process.
Step 3 - Performance testing of sample pre-treatment procedures.
Step 4 - Testing of different sampling strategies on 30 digital models incorporating various degrees of contaminant heterogeneity.
Step 5 - Validation of the preferred strategy upon the actual measurements of 2600 soil stockpiles.
Step 6 - Practical application, costs and quality assurance.

The resulting strategy involves gathering 2 composite samples consisting of 50 increments (of 180 g) each. For inorganic contaminants, a reliable estimate of the true mean concentration is obtained for 97 – 98 % of the soil stockpiles. For organic contaminants the sampling procedure results in reliable estimates for 75 – 82 % of the soil stockpiles. These percentages are valid for all soil stockpiles encountered in daily practice in the Netherlands, ranging from clean to highly contaminated.

The procedure can also be used for in-situ “stockpiles”, provided that the excavation process is in line with the (spatial) definition of the sampled in-situ “stockpile”. In The Netherlands the “2 x 50” strategy is well-established. Costs for the total process (from sampling to chemical analysis) are approximately 1.350,= Euro per stockpile. All (sub)processes are carried out under audited quality control and assurance schemes.

1. INTRODUCTION

The Dutch Building Materials Decree regulates the (re)use of soil [1], as is schematically depicted (for inorganic contaminants) in Figure 1.

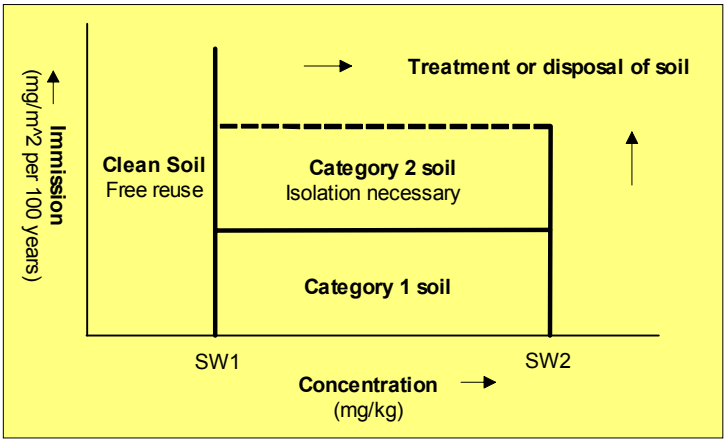

FIGURE 1 – Soil reuse options as a function of contaminant concentration and leachability.

The soil quality, in terms of contaminant concentration and leachability, has to be determined in order to ensure environmentally sound application. In addition, it needs to be assessed whether soil is clean (unrestricted reuse), or should be treated or disposed off in a landfill. This paper, which expands on a previous study by F.P.J. Lamé et al. [2], describes the development of a sampling procedure to determine the mean concentration of contaminants in soil stockpiles. Preferably, this sampling procedure should be valid for the full range of occurring contaminant concentrations for various degrees of heterogeneity. In subsequent order, the following aspects are dealt with:

- Definition of the approach to the fundamental choices on the basic characteristics of the total procedure, which includes sampling strategy, sample pre-treatment and chemical analysis.
- Development of a robust procedure for sample pre-treatment.
- Modelling of various sampling strategies.
- Validation of the derived model (i.e. strategy) with empirical data.

2. APPROACH

Practical experience with sampling of contaminated soils indicates that both organic and inorganic contaminants are heterogeneously distributed over a stockpile. However, at the beginning of our research effort, little was quantitatively known on this phenomenon. It was therefore decided to generate a limited number of sets with basic data. From several soil stockpiles (ca. 2000 ton) large (50 – 100) numbers of individual samples (ca. 180 g) were taken and subsequently analysed for heavy metals, sum-PAH's and mineral oil. Typical results on the distribution of lead and sum-PAH's are shown in Figure 2. Clearly for this specific stockpile a significant spread in the data is observed. Since contaminant heterogeneity is not quantitatively known prior to sampling, a fundamental choice has to be made towards the development of a sampling strategy. Two options exist. The first option is to sample and analyse a (large) number of individual samples. In this case the environmental quality of the soil stockpile can be related to a statistical parameter such as the mean, the median or the 95 % percentile value. The second option is depicted in Figure 3. This option involves collecting a certain number of increments, which are assembled into a composite sample. Essentially the contaminant heterogeneity in the stockpile (e.g. 2000 ton) is transferred to the container (e.g. 10 kg). Subsequent steps in this process are sample
pre-treatment (i.e. homogenisation and sample division into a representative sub-sample) and standardised chemical analysis. The resulting data are representative for the mean

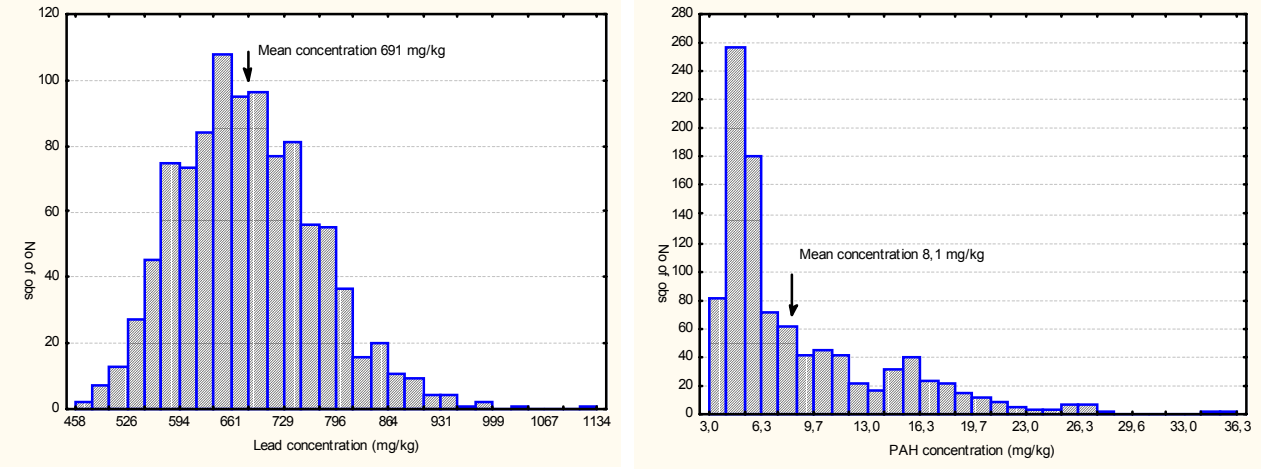

FIGURE 2 – Distribution of lead and sum-PAH's concentrations in a typical soil stockpile.

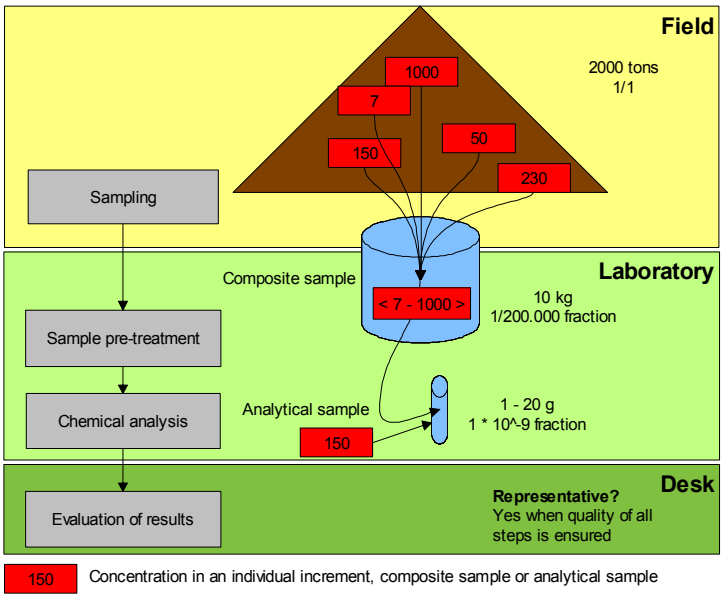

FIGURE 3 – Sampling procedure corresponding with option 2.

concentration of the contaminants in the stockpile. The first option is far more costly (due to the relatively large number of chemical analyses to be performed), but provides information on contaminant heterogeneity. The second option is relatively cheap but details on contaminant heterogeneity are largely lacking. A panel of policy makers decided that option 2 is to be used for the characterisation of soil stockpiles in The Netherlands. Technically, this decision can be translated into two basic questions:

- Are (existing) sample pre-treatment procedures capable to sufficiently homogenise a composite sample consisting of a certain number of individual increments?
- How many individual samples (increments) should be taken to obtain a sufficiently representative impression of the average contaminant concentration in a soil stockpile?

These questions will be addressed in the following paragraphs.

3. SAMPLE PRE-TREATMENT

Sample pre-treatment procedures for organic and inorganic contaminants are, respectively, described in Dutch standard protocols NVN 5730 [3] and NEN 5751 [4]. For organic contaminants sample pre-treatment basically involves cryogenic grinding at temperatures between -196 °C (liquid nitrogen) and approximately -10 °C (the temperature of the sample depends on the process time of grinding). For inorganic contaminants the samples are dried and ground prior to sub-sampling. (Rotating) sample dividers are used for sub-sampling. Performance testing has been extensively described elsewhere [5, 6]. As part of this project the suitability of the procedure for inorganic contaminants was verified. Approximately 10 kg of material consisting of 50 increments was gathered from various soil stockpiles. Prior to pre-treatment random samples (of ca. 2 g) were taken from the composite sample and analysed chemically. The results of which are depicted in Figure 4a. The bulk of the composite sample was then subjected to an intensive drying, grinding and (rotating) dividing process. The results of the chemical analyses of the pre-treated sample are shown in Figure 4b. Clearly, substantial homogenisation has been achieved. Based on more elaborate results as reported elsewhere [5, 6], we were confident that, through adequate pre-treatment of a heterogeneous composite sample (ca. 10 kg) sufficiently representative samples for the chemical analysis of organic (10-20 g) and inorganic (2-10 g) contaminants can be derived. The majority of chemical analyses are covered by Dutch NEN standards, and are therefore no subject for this investigation.

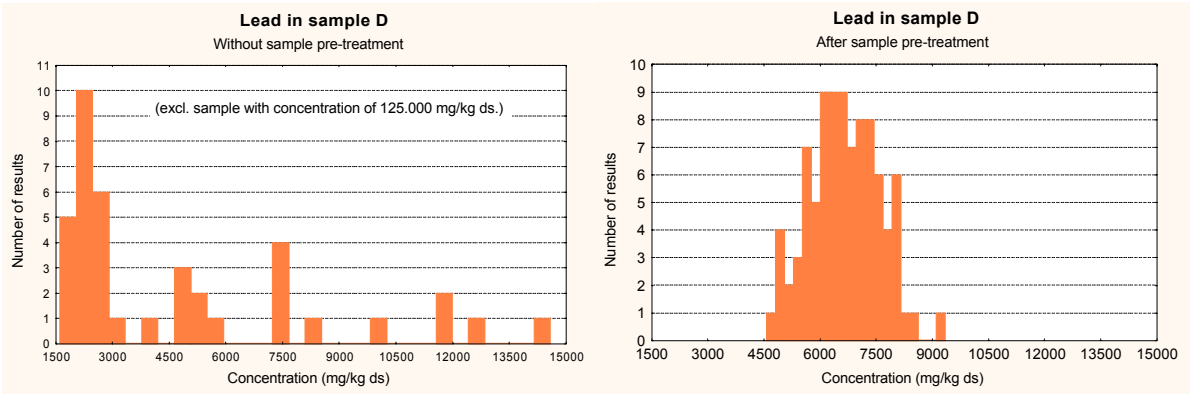

FIGURE 4 – Lead concentrations in 50 randomly taken sub-samples of approximately 2 grams without sample pre-treatment (left) and after sample pre-treatment (right).

4. MODELLING OF SAMPLING STRATEGIES

The research described in this paragraph has been extensively reported elsewhere [7]. Central question towards the development of a sampling strategy is the number of increments to be taken from a soil stockpile in order to obtain a sufficiently reliable estimate of the true mean contaminant concentration. Preferably, this should be carried out by determining the heterogeneity of a substantial number of soil stockpiles. This is an extremely expensive exercise. Therefore it was decided to obtain an impression of the performance of different sampling strategies by testing them on a series of digital models of soil stockpiles. The digital models were based on actual measurements of the spatial distribution of contaminants in soil. Three sets of data were selected for this purpose. By conditional simulation [8] each dataset was converted into a three dimensional digital model. Each model consists of 5.9 million data, representing the distribution of a contaminant on the scale of the increments (of 180 g) in space. Additional to the heterogeneity on the scale of the increments, different degrees of spatial correlation were introduced into the models. This was done by varying the conditions of the simulation, whilst maintaining the original distribution (i.e. the histogram) intact. Thus, 30 digital soil models were derived. A typical example is depicted in Figure 5.

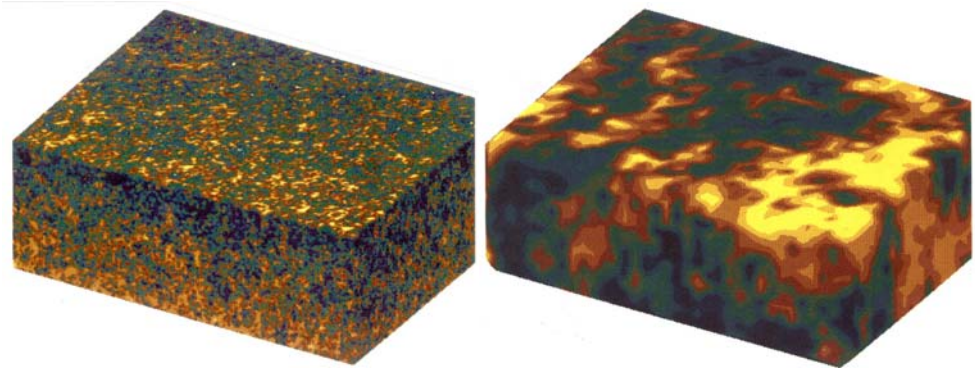

FIGURE 5 – Digital models of soil stockpiles, representing a little (left) and large scale degree of heterogeneity (right).

Various sampling strategies were tested on the 30 models. The main parameter being the number of increments to be taken. A typical example is shown in Figure 6, which essentially demonstrates that the reliability of the estimated mean concentration increases with the number of increments in the composite sample. As shown in Figure 6, the relative width of the distribution of the mean concentration in the composite sample decreases with increasing numbers of increments (in the composite sample). For example, for a soil stockpile with a coefficient of variation of 250 %, the relative width of the distribution of the mean concentration is 1.1 when 50 increments are assembled. For 200 increments the relative width of the distribution decreases to 0.75. It is beyond the scope of this paper to go into further details on the modelling study.

By expert judgement is was decided that the strategy involving 100 increments appeared to produce a sufficiently reliable estimate of the mean contaminant concentration. It was also decided that per soil stockpile, 2 composite samples consisting of 50 increments each should be gathered. By analysis of 2 composite samples, additional information is obtained on soil heterogeneity and (potential) errors in sampling, sample pre-treatment and chemical analysis.

The drawback of the modelling process is the fact that the heterogeneity (on the scale of the increments) of only 3 soil stockpiles is taken into account. In order to validate the “2 x 50” strategy, it is necessary to include the heterogeneity of a multitude of soil stockpiles. This is described in the next paragraph.

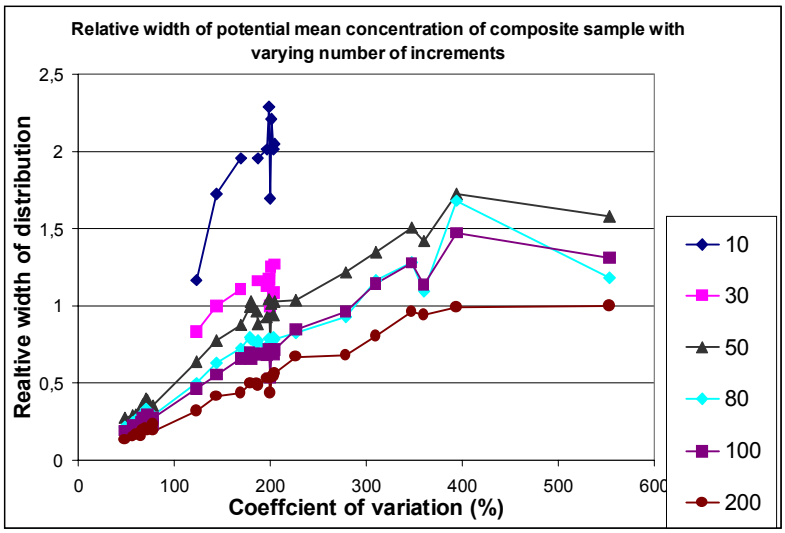

FIGURE 6 – Relative width of the distribution (resulting form digital sampling) as a function of soil heterogeneity, for sampling strategies incorporating varying numbers of increments (from 10 to 200).

5. VALIDATION OF THE "2 x 50" STRATEGY

The research described in this paragraph has been extensively reported elsewhere [9]. The strategy incorporating 2 composite samples, each consisting of 50 increments (of ca. 180 g), was employed on ca. 2600 soil stockpiles over a period of three years. Thus, for each soil stockpile 2 sets of data are produced for the following contaminants: 8 heavy metals (As, Cd, Cu, Cr, Hg, Ni, Pb, Zn), mineral oil, sum-PAH's and extractable halogenated organics (EOX). From Table 1 it is inferred that the database incorporates all soil qualities in sufficiently large percentages, and can therefore be considered as representative for The Netherlands.

Environmental quality	Percentage
Clean soil	26
Lightly contaminated (reusable) soil	57
Heavily contaminated soil	17

TABLE 1 - Soil qualities represented in the database.

Based on the number of increments (in this case 50) in the composite sample, the heterogeneity of the contaminants in the soil stockpile can be mathematically calculated, according to:

$$CV^2_{measured} = CV^2_{analytical} + CV^2_{stockpile} /50$$

Where:

$CV_{measured}$ is the measured coefficient of variation (as derived from the 2 sets of data per soil stockpile).

$CV_{analytical}$ is the coefficient of variation resulting from the errors in sample pre-treatment and chemical analysis.

$CV_{stockpile}$ is the heterogeneity of the contaminant in the soil stockpile on the level of the individual increments.

For each soil stockpile the calculated degree of heterogeneity is highly inaccurate because it is only based on 2 sets of data. However, this is compensated for by the large number of soil stockpiles and contaminants investigated. A typical dependence of the measured coefficient of variation on concentration is shown in Figure 7. From this Figure it is inferred that the heterogeneity of zinc is

largely independent on concentration. This phenomenon was also observed for all other contaminants in this investigation. Other factors – such as soil type, organic matter content, silt content, contaminant type - potentially influence the observed coefficient of variation. These factors were therefore also statistically analysed. Of these factors, only the physical nature (organic or inorganic) of the contaminant appeared to be of any (statistical) relevance. Despite a number of extreme values for all contaminants, the measured coefficient of variation for the group of inorganic contaminants was significantly lower than for the group of organic contaminants, as is shown in Figure 8. In order to determine the true contaminant heterogeneity ($VC_{stockpile}$) the analytical error needs to be known. Since this error was not determined experimentally (which would have been extremely costly), we used estimates. From laboratory operational experience it was deduced that for inorganic contaminants the analytical error is in the 5 to 10 % range, whilst for organic contaminants analytical errors from 5 to 20 % are commonly observed. These values are used as (additional) variables in the above equation. The final result, for which the full derivation is given elsewhere [9], is shown in Table 2. Also, the calculated results for an alternative “2 x 6” strategy are given.

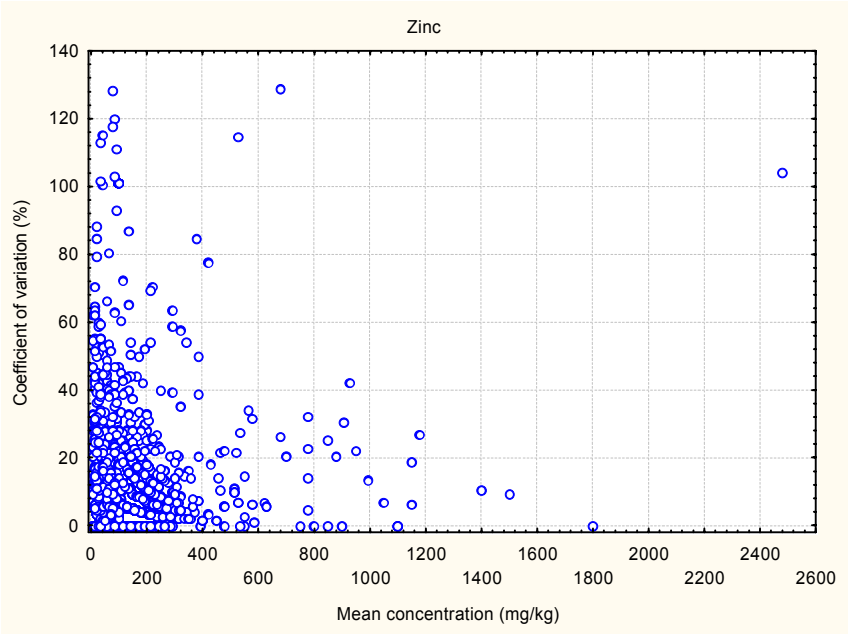

FIGURE 7 – Coefficient of variation for zinc as a function of the mean concentration in 2600 soil stockpiles.

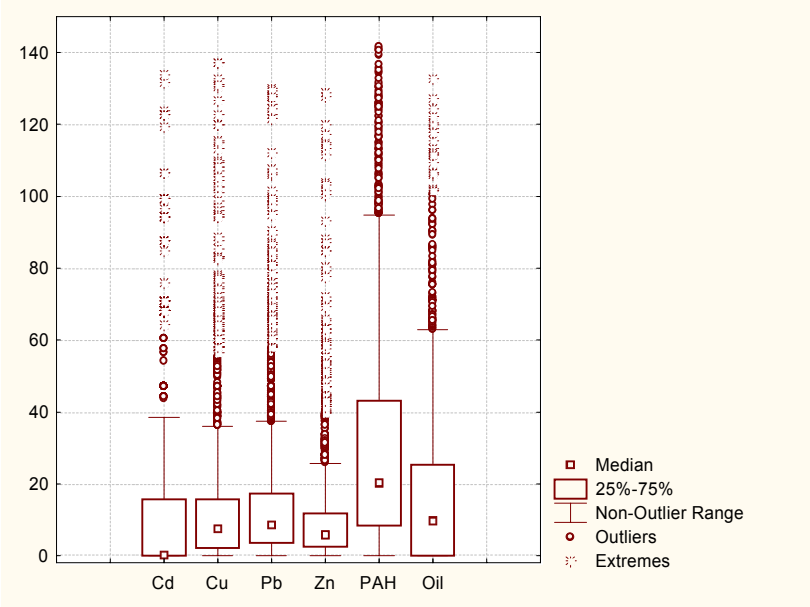

FIGURE 8 – Measured coefficient of variation of some inorganic and organic contaminants in 2600 soil stockpiles based on two samples per stockpile.

	Inorganic contaminants		Organic contaminants	
Estimated analytical error	*5 %*	*10 %*	*5 %*	*20 %*
Strategy 2 x 50	97 %	98 %	75 %	82 %
Strategy 2 x 6	79 %	85 %	48 %	69 %

TABLE 2 - Percentage of the soil stockpiles (of the 2600 investigated) for which a sampling strategy results in a sufficiently reliable estimate for the true mean contaminant concentration.

Application of the “2 x 50” strategy yields:

- A reliable estimate of the true mean concentration for **inorganic** contaminants for 97 – 98 % of the stockpiles occurring in daily practice.
- A reliable estimate of the true mean concentration for **organic** contaminants for 75 - 82 % of the stockpiles occurring in daily practice.

From these results it was concluded by a panel of policy makers that the “2 x 50” strategy was robust enough to be used for assessing the environmental quality of soil stockpiles. In addition, it was concluded that the alternative “2 x 6” strategy – which is legally still permitted – does not fulfill the required reliability standards.

6. COST FACTORS AND QUALITY ASSURANCE

The “2 x 50” strategy is since a couple of years fully established in the operational practice for assessing the environmental quality of soil stockpiles in The Netherlands. It should be emphasised that this strategy not only applies to ex-situ stockpiles but can also be used for the characterisation of in-situ “stockpiles”, provided that the excavation process follows the pattern of the sampling points. To further ensure the execution of proper procedures and customer satisfaction, the relevant processes of sampling [10, 11] and sample pre-treatment and chemical analysis [12] are carried out under various audited quality control and assurance schemes.

The market tariff for sampling is approximately 450,= Euro, whilst the tariff for sample pre-treatment and chemical analysis (for 1 composite sample) is also 450,= Euro. This translates to the following graduation:

SOIL STOCKPILE [ton]	COSTS	
	Euro	Euro/ton
100	1350	13.5
500	1350	2.7
1000	1350	1.4
2000	1350	0.7

TABLE 3 - Market tariffs for soil quality assessment.

These costs compare with the following tariffs for soil treatment and reuse [13]:

PROCES	COSTS [Euro/ton]
Reuse	2-7
Thermal treatment	55
Soil washing	35
Biological treatment	20
Landfilling	40-70

TABLE 4 - Market tariffs for soil treatment, landfilling and reuse.

In practice, for soil stockpiles in excess of 500 ton, the costs of soil quality assessment are considered appropriate in comparison with follow-up costs for reuse, treatment and disposal. Soil lots smaller than 500 ton, but typically smaller than 100 ton, are generally lumped together prior to sampling. Proper lumping is based on semi-quantitative or even qualitative information. To ensure proper compliance with environmental standards, the lumping process is generally carried out under a quality control and assurance scheme for reusable [14,15] and for treatable [16] soil.

7. EPILOGUE

By employing a combination of experimental, modelling and statistical methods, a sampling strategy for the assessment of the environmental quality of, both ex-situ and in-situ, soil stockpiles has been developed and validated. The strategy involves gathering 2 composite samples consisting of 50 increments each. Adequate pre-treatment of the composite sample prior to chemical analysis is a prerequisite. The strategy yields a sufficiently reliable estimate of the mean contaminant concentration. This applies for the large majority of soil stockpiles in The Netherlands over a wide range of contaminant concentrations for a wide range of occurring heterogeneity. The sampling process has gained wide market acceptance and is carried out under audited quality control and assurance schemes, against acceptable costs.

8. REFERENCES

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