

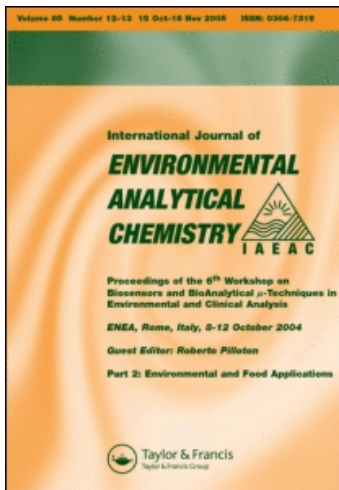
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### Characterization of Compost with Respect to its Content of Heavy Metals

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# CHARACTERIZATION OF COMPOST WITH RESPECT TO ITS CONTENT OF HEAVY METALS Part III: Precision of the Total Analytical Procedure

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This paper concludes a study aimed at the development of a procedure for the determination of the content of heavy metals in compost. It deals with the different error sources in the total analytical procedure. Using analysis of variance, values for the components of variance for the determination of the heavy metal content of garbage compost have been assessed. It could be shown that these values have general validity, regardless of the compost lot examined and the nature of the element. Autocorrelation did not appear to be important when sampling compost. The values for components of variance assessed, served as the basis for designing total analytical procedures—comprising sampling, sample preparation and analysis—for the determination of the heavy metal content of garbage compost.

**KEY WORDS:** Compost, heavy metals, analysis of variance, total analytical procedure.

## 1. INTRODUCTION

A Decree is in preparation in which the Dutch Government will specify values for maximum tolerable levels of heavy metals in compost. For the implementation of the Decree analytical procedures have to be developed. For the design of a suitable total analytical procedure, it is necessary to know the contributions of the different steps in the procedure to the error in the final result.<sup>1</sup>

As compost is a highly heterogeneous material it can be expected that sampling, i.e. withdrawal of increments from a lot, constitutes the major source of error. However, the sampling error can only be established if the errors of the other steps

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\*Deceased 8 November 1990.

in the total analytical procedure, viz. sample preparation and analysis, are known. Therefore, the errors associated with analysis and sample preparation were the subjects of previous studies.<sup>1,2</sup>

The present paper completes our study on the problems associated with the characterization of compost with respect to its content of heavy metals. It contains the results of a large-scale experiment in which the different sources of error, i.e. sampling, subdivision and analysis, are studied simultaneously. The hierarchical arrangement of these sources of error is taken into consideration. In addition, it has been assessed whether autocorrelation occurs for the sampling error.

The aim of the study is to derive values for the components of variance for the determination of the heavy metal content of garbage compost, which have a general validity. These values of general validity are the point of departure in designing suitable total analytical procedures for the determination of heavy metals in compost.

The paper closes with the schematic description of some suitable procedures. These descriptions include the nature and the number of operations—withdrawal of increments, sample preparation and analysis—that have to be performed to obtain an estimate of the heavy metal content in compost lots with a known degree of uncertainty at a specified confidence level. The design of the procedures has been performed in such a way, that the total error is less than 10% of the observed final result, at a confidence level of 5%.

The suitable procedures proposed are evaluated on the basis of the effort required.

## EXPERIMENTAL

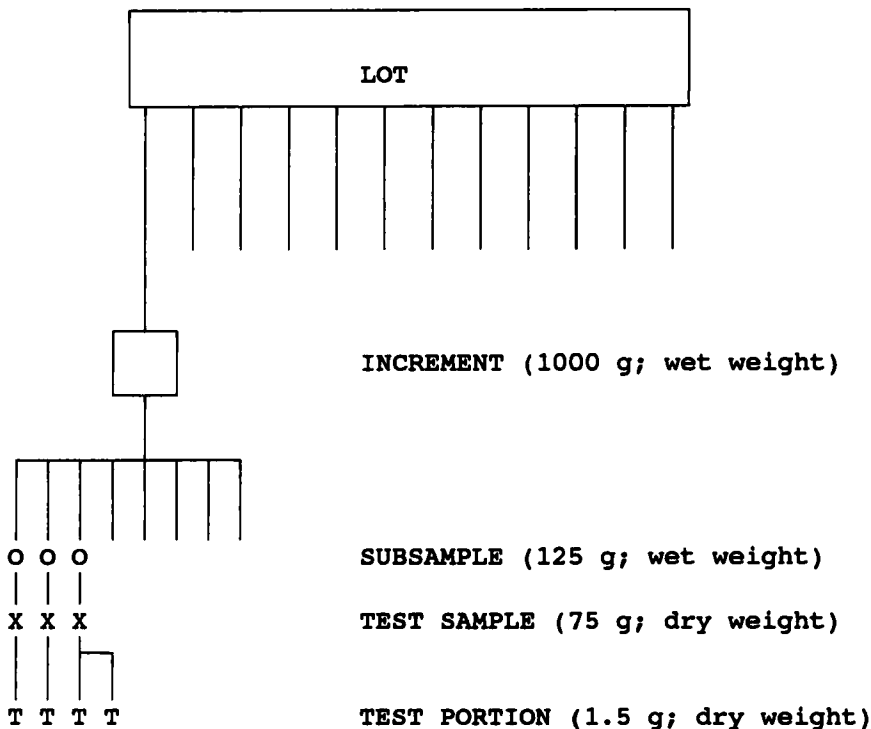
### *Experimental Set-up*

The study of components of variance was based on the expected hierarchical arrangement of the sources of error. It is assumed that the errors due to subdivision and to analysis are subordinate to the sampling error. The analysis error is expected to be subordinate to the subdivision error, which is supported by the results presented in ref. 2. Thus, an experimental set-up can be chosen in which the observates can be treated employing nested analysis of variance, in which the error sources are:

- variance between increments within a lot;
- variance between subsamples within an increment;
- variance between test portions within subsamples.

The design actually used was adopted from ref. 3. The design was chosen so that it leads to similar precision for the estimates of all three components of variance to be studied. Design number 4 in ref. 3 meets this requirement.

In the design of choice (see Scheme 1) 12 increments are taken from a lot. The increments are received in the laboratory as laboratory samples. Each laboratory sample is divided into eight subsamples. Three, randomly selected subsamples, are



Scheme 1 Design for analysis of variance

subsequently prepared to obtain test samples.<sup>2</sup> From two of the resulting test samples one test portion is analysed and from the third test sample two test portions. The total number of analyses per element per lot is 48, which in this study was considered as a feasible effort.

Homogeneity of variances for  $s_{anal}^2$  and for  $s_{subd}^2$  was checked using Cochran's C-test ( $P=0.01$ ).

*Sampling*

Sampling objects were lots of mature ready-to-use compost. When possible, sampling took place under moving-stream conditions. Otherwise, static piles were sampled. The mass of the increments withdrawn was about 1000 g. When sampling moving streams, a systematic strategy was followed, which means that—divided over the whole period of time for handling the lot—equal time intervals are taken between the withdrawal of increments.

Increments were collected by moving a container through the whole width of a falling stream.

For static piles, a two-stage sampling procedure was performed. Firstly, a sampling hole was made using a 18 cm (diameter) Edelman auger, after which the eventual sampling took place with a root auger. Increments were taken at a depth of about 1 m perpendicular to the surface. A stratified sampling strategy was

applied, at which the surface of the compost pile has been divided imaginarily in equal strata. Within each stratum one increment was withdrawn randomly.

Increments were packed in plastic bags for transportation to the laboratory.

### *Autocorrelation*

The content of heavy metals in a compost lot may be internally correlated. When an object is internally correlated, distribution is not random and the sample size, the sample number and the sampling error are related and depend on the size of the object.<sup>4</sup> In that case the execution of the sampling stage in the total analytical procedure is dependent on the size of the compost lot, which complicates the set-up of general valid procedures.

Internal correlation, or autocorrelation, can be described by  $T_x$ , the time constant or correlation constant. Assessment of  $T_x$  was performed for two lots that were sampled by a systematic sampling strategy, using the method described by Limonard and Pijpers.<sup>5</sup> For this, analytical results were combined in pairs in the order in which increments were taken. From the variances within pairs and between pairs,  $T_x$  can be derived. The dimension of  $T_x$ , in this case, is the length of time between successive increments. When  $T_x$  is larger than 3, autocorrelation should be taken into account in the design of sampling procedures.

### *Sample preparation*

The received packed increments, i.e. the laboratory samples, were subdivided using a riffle splitter. Three out of eight subsamples were randomly selected and were subsequently dried and milled.

For milling a quartz planet ball mill was used. As was shown in a previous study,<sup>2</sup> this type of mill yields a more homogeneous test sample than the cross beater mill. Thus the milling procedure of choice will lead to a smaller value for  $RSD_{anal}$ , which is favourable for the assessment of values for the components of variance that are higher in the hierarchical order.

### *Determination of heavy metals*

The determination of heavy metals in compost was performed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) according to the procedure described in ref. 1.

### *Types of composts analysed*

The following types of garbage compost were considered.

*Compost Wijster*, a compost produced from the compostable fraction of household waste obtained after mechanical separation and magnetic removal of iron objects. Two lots for this compost, denoted as "25" and "27", were examined. Both lots were sampled from the falling stream which resulted from sieving the

mature compost. "Wijster 25" consisted of the sieve fraction smaller than 10 mm, "Wijster 27" of the sieve fraction 5–12 mm.

*Compost Soest–Baarn*, a compost produced by the DANO process from mixed household waste. Sampling took place from the static pile. The lot consisted of the sieve fraction smaller than 23 mm.

*Compost Nuenen*, a compost produced from vegetable, fruit and garden waste from households, separated at the source in the community of Nuenen. The lot was sampled from the falling stream which resulted from sieving mature compost through a 6 mm sieve.

*Compost Amsterdam*, a compost produced from vegetable, fruit and garden waste from households, separated at the source in the outskirts of the city of Amsterdam. Sampling was performed from the static pile. The lot was not sieved.

Basically, non-compostable components were not discarded from the compost laboratory samples.

The content of heavy metals of the compost materials mentioned above was presented in ref. 1.

### *Instrumentation*

#### *Analytical equipment:*

- Microwave oven,*
- Philips Compact AVM702, 1100 W,  $2450 \pm 50$  Hz.*
- ICP-AES equipment: Jarrell-Ash Division AtomScan 2000 (sequential ICP system), Jarrell-Ash model 975 Plasma AtomComp (simultaneous ICP system); for experimental conditions, see ref. 1.*

*Milling equipment:* quartz planet ball mill Pulverisette 5 (Fritsch GmbH, Germany) beaker volume 250 ml, ball diameter 30 mm, 4 balls per beaker.

### *Reagents*

All reagents used were of analytical grade (Merck, Darmstadt, Germany).

## RESULTS AND DISCUSSION

### *Components of Variance*

As an example, the statistical treatment of the experimental results for lead in the lot "Wijster 25" are presented. Table 1 lists the analytical results observed. Results of the analysis of variance are summarized in Table 2. The value for  $s_{\text{anal}}^2$  in increment 5 is an outlier according to Cochran's C test ( $P=0.01$ ). Comparison of the analytical results shows that this is due to a high result for 5, 1a (see Table 1). In general it was found that the deviation from homogeneity of variance was caused by a high individual test result, probably due to the presence of a metallic

**Table 1** Analytical results for the determination of the lead content of lot "Wijster 25" (mg/kg dry matter)

Increment	Subsample			
	1	2	3	
	Test portion			
	1a	1b	2a	3a
1	347.0	343.3	328.0	323.8
2	393.8	414.5	360.6	436.8
3	418.1	410.4	434.0	564.9
4	364.5	362.8	357.9	350.8
5	514.6	411.2	419.1	452.6
6	418.5	383.7	378.8	383.6
7	356.1	368.2	362.3	350.1
8	419.7	420.7	420.1	442.5
9	414.0	446.7	420.7	374.5
10	357.1	353.6	416.9	355.9
11	374.9	380.5	413.4	396.8
12	387.0	382.0	400.3	512.2

**Table 2** Analysis of variance table for lead in lot "Wijster 25"<sup>a</sup>

Source of variation	Degrees of freedom	Mean square
Between increments	10	5005 ( $M_2$ )
Between subsamples within increments	22	1699 ( $M_1$ )
Between test portions	11	136 ( $M_0$ )
Total	43	

<sup>a</sup>The results for increment 5 are left out as  $s_{anal}^2$  is an outlier (Cochran's C-test;  $P=0.01$ ).

**Table 3** Estimates for the components of variance in the determination of lead in lot "Wijster 25"

Error source	$s^2$	RSD (%)
Withdrawal of increments	748	7.0
Subdivision	1251	9.0
Analysis	136	3.0

particle in the test portion concerned. Discarding those outliers was of little influence on the mean content of the lot. Means with and without the outlier generally differed less than 5%.

The quantities estimated by the mean squares for the design used<sup>3</sup> are:

$$M_2 = s_{anal}^2 + 1.5s_{subd}^2 + 4s_{incr}^2 \quad (1)$$

$$M_1 = s_{anal}^2 + 1.25s_{subd}^2 \quad (2)$$

$$M_0 = s_{anal}^2 \quad (3)$$

**Table 4** Survey of components of variance associated with analysis, subdivision and withdrawal of increments, expressed as RSD

<i>Element</i>	<i>RSD<sub>anal</sub></i> (%)	<i>RSD<sub>subd</sub></i> (%)	<i>RSD<sub>incr</sub></i> (%)
<i>Lot: Wijster 25</i>			
Cd	7.2	8.7	5.9
Cr	5.4	6.9	4.2
Cu	4.9	11.0	9.0
Ni	7.9	8.0	14.6
Pb	3.0	9.0	7.0
Zn	1.5	5.0	9.8
<i>Lot: Wijster 27</i>			
Cd	11.1	4.6	3.2
Cr	5.7	11.2	16.0
Cu	10.3	17.7	14.5
Ni	6.5	5.4	10.3
Pb	6.7	7.1	13.0
Zn	2.1	9.9	14.2
<i>Lot: Soest-Baarn</i>			
Cd	4.5	13.5	10.4
Cr	6.5	0	9.0
Cu	2.8	6.0	18.7
Ni	7.2	0	10.6
Pb	2.1	7.1	13.0
Zn	1.7	2.6	12.5
<i>Lot: Nuenen</i>			
Cd	27.4	0	0
Cr	6.1	5.0	4.2
Cu	4.0	5.5	4.7
Ni	9.2	15.9	6.8
Pb	5.9	9.0	0
Zn	5.1	4.8	15.6
<i>Lot: Amsterdam</i>			
Cd	7.4	6.7	21.7
Cr	3.6	9.5	19.0
Cu	2.1	10.8	16.6
Ni	5.6	8.5	13.7
Pb	11.0	0	33.0
Zn	3.3	6.5	8.8

The coefficients in Eqs. (1) and (2) are functions of the number of increments, the number of subsamples per increment and the numbers of tests on the test samples. The estimates for the components of variance calculated from Eqs. (1)–(3) are presented in Table 3.

An overview of all results of the analysis of variance for the lots considered is presented in Table 4. In order to make the results obtained for the five different lots mutually comparable, the components of variance examined are presented as the relative standard deviation, RSD.



**Table 5** Chi-square values from Friedman's test for observed components of variance in determining heavy metals in compost

RSD	Factor	
	Element	Lot
RSD <sub>anal</sub>	14.0*	6.1
RSD <sub>subd</sub>	6.5	4.1
RSD <sub>incr</sub>	4.0*	7.4

\*Significant ( $P = 0.05$ ).**Table 6** Median values for components of variance in the total analytical procedure for determining heavy metals in compost

Component of variance	Median value (%)
RSD <sub>anal</sub>	6.1*
RSD <sub>subd</sub>	7.0
RSD <sub>incr</sub>	10.5

\*Without values for Zn (see text).

**Table 7** Autocorrelation factor calculated in sampling compost for the determination of the content of heavy metals<sup>a</sup>

Element	Compost:	
	Wijster 27	Nuenen
Cd	0	0
Cr	1.2	0
Cu	2.2	0
Ni	0	0
Pb	1.3	0
Zn	2.4	0.3

<sup>a</sup> $T_x = 1$  corresponds with the interval between successive increments.

The hierarchy of the error sources manifests itself in the order of magnitude of the values of the mean squares. If there is a hierarchy as expected, values should decline from  $M_2$  to  $M_0$ . Eqs. (1)–(3) show that in such a case positive estimates of all three variances will result. When a negative estimate is found, the hierarchy obviously does not occur for the component of variance involved. In such a case the value zero is assigned to that particular component of variance. The other components of variance are then recalculated employing analysis of variance with two sources of error. When a second negative estimate for a component of variance results, likewise the value zero is assigned to that component of variance. The variance associated with the remaining source of error is then calculated directly from all the experimental results. Table 4 shows that the value zero appears only in 6 out of 90 observations, indicating that in general the expected hierarchy exists.

Statements about the design of a generally valid total procedure for the

**Table 8** Characteristics of analytical procedures that are in agreement with the criterion that  $t_{s_{lot}}$  is smaller than 10% of the observed value ( $P=0.05$ )<sup>a</sup>

<i>Planet ball mill</i>			<i>Cross beater mill</i>		
<i>INDV</i>	<i>P</i>	<i>N</i>	<i>INDV</i>	<i>P</i>	<i>N</i>
a	1	8	a	1	8
b	2	7	b	2	7
c	3	7	c	3	6
<i>COMP</i>	<i>P</i>	<i>N</i>	<i>COMP</i>	<i>P</i>	<i>N</i>
a	2	13	a	4	14
b	3	10	b	5	10
c	4	9	c	6	9
d	5	8	d	7	8

<sup>a</sup>*P*: number of test portions per test sample; *N*: number of increments per lot.

**Table 9** Weighing factors associated with operations in the total analytical procedure<sup>a</sup>

<i>Operation</i>	<i>Weighing factor (a.u.)</i>			
	<i>Pl. ball mill</i>		<i>Cr. beat mill</i>	
	<i>A</i>	<i>B</i>	<i>A</i>	<i>B</i>
Withdrawal of increments (INCR)	15	5	15	5
Subdivision (SUBD)	5	5		
Drying (DRY)	1	1	1	1
Milling (MILL)	5	5	5	5
Mixing (MIX)	1	1	5	5
Analysis (ANAL)	15	15	15	15

<sup>a</sup>For cases A and B, see text.

determination of the content of heavy metals in compost can be made when values for the components of variance are available which have general validity. Such general values can only be derived if the components of variance are independent of the compost lot to be examined as well as of the element considered. This aspect is discussed below.

In order to be independent of the distribution of the values observed, in the further statistical treatment non-parametric methods are used. The assessment of the effect of the factors "lot" and "element" on the components of variance is performed using Friedman's method for randomized blocks.<sup>6</sup> The null hypothesis of this test is, that values of RSDs do not differ significantly for alternative lots or elements. The results for the test are presented in Table 5. It appears that the null hypothesis has to be rejected only in one out of six cases. Due to the lower values for zinc,  $RSD_{anal}$  is affected by the factor "element". An explanation of this behaviour may be found in the combined effect of a relatively high analyte content and the high precision of the determination of Zn by ICP-AES. If Friedman's test is performed after discarding the values for Zn, the observed chi-square statistic is

**Table 10** Number of operations to be performed and estimated total effort for specific analytical procedures mentioned in Table 8<sup>a</sup>

<i>Anal procedure</i>	<i>Operation</i>						<i>Total effort (a.u.)</i>	
	<i>INCR</i>	<i>SUBD</i>	<i>DRY</i>	<i>MILL</i>	<i>MIX</i>	<i>ANAL</i>	<i>A</i>	<i>B</i>
<i>Planet ball mill</i>								
<i>INDV a</i>	8	8	8	8		8	328	248
<i>INDV b</i>	7	7	7	7		14	392	322
<i>INDV c</i>	7	7	7	7		21	497	427
<i>COMP a</i>	13	13	13	13	1	2	369	239
<i>COMP b</i>	10	10	10	10	1	3	306	206
<i>COMP c</i>	9	9	9	9	1	4	295	205
<i>COMP d</i>	8	8	8	8	1	5	284	204
<i>Cross beater mill</i>								
<i>INDV a</i>	8		8	8		8	288	208
<i>INDV b</i>	7		7	7		14	357	287
<i>INDV c</i>	6		6	6		18	396	336
<i>COMP a</i>	14		14	14	1	4	359	219
<i>COMP b</i>	10		10	10	1	5	290	190
<i>COMP c</i>	9		9	9	1	6	284	194
<i>COMP d</i>	8		8	8	1	7	278	198

7.2, which is less than the critical value of 9.5 (d.f. = 4;  $P=0.05$ ) which means that the null hypothesis should not be rejected. That is, when the values for zinc are left out, for all three components of variance general values can be derived and the design of generally valid analytical procedures is possible.

The median values were considered as the general values, thus avoiding the influence of outliers. The results are presented in Table 6. From the results it can be concluded that withdrawal of increments from a lot is the predominant source of error in the procedure. This confirms our expectation that garbage compost is a highly heterogeneous material.

The errors due to subdivision and to analysis are of about the same magnitude. These errors were the subjects of previous papers.<sup>1,2</sup> It was found that  $RSD_{anal}$  (use of a planet ball mill) ranged from 2 to 8% and that values for  $RSD_{subd}$  ranged from 5 to 8%. The values found in the present study are in good agreement with these observations.

#### *Autocorrelation*

The possible occurrence of autocorrelation was studied for compost lots "Wijster 25" and "Nuenen".  $T_x$  values, derived as described in the experimental section, are presented in Table 7. The results show that autocorrelation is either absent or values for  $T_x$  are small; values of  $T_x$  are all under the critical value of 3. So, the conclusion is justified that autocorrelation is not expected to be important in sampling compost lots. Therefore, for the design of analytical procedures, the number of increments to be withdrawn is independent of the size of the compost lot and must be based on random distribution of heavy metals in a compost lot.

*Total analytical procedure*

In the previous section, general values for components of variance for the determination of the heavy metal content of compost have been derived. With these values suitable analytical procedures—comprising sampling, sample preparation and analysis—can be designed.

However, for  $RSD_{anal}$  it has to be taken into account that the value depends on the milling procedure. The use of a planet ball mill results in a more homogeneous test sample than the use of a cross beater mill. On the other hand, the cross beater mill can be used for the complete dried laboratory sample, thus avoiding the error due to subdivision.<sup>2</sup> The choice of the milling procedure thus affects the characteristics of the analytical procedure. In order to conclude on the procedure to be preferred, both ways of milling have been taken into account.

To design suitable total analytical procedures, two types of procedures have been set up. The first type is based on individual test samples for each laboratory sample (*INDV*), whereas in the second a composite test sample is used (*COMP*). For both types, milling by a planet ball mill as well as by a cross beater mill has been addressed.

The total analytical procedures can be described schematically as follows:

*Planet ball mill*

—*INDV*:  $N$  increments are withdrawn from a lot and transported to the laboratory. The resulting laboratory samples are individually subdivided into eight subsamples, one of which is dried and milled. The milled subsamples, one for every increment, are considered as individual test samples. From each test sample  $P$  test portions are removed for analysis.

—*COMP*:  $N$  increments are withdrawn from a lot and transported to the laboratory. The resulting laboratory samples are individually subdivided into eight subsamples, one of which is dried and milled. The milled subsamples are mixed to gain a composite test sample from which  $P$  test portions are removed for analysis.

The total variance for the analytical procedures is presented in Eqs. 4 and 5.

$$s_{tot, INDV}^2 = s_{incr}^2/N + s_{subd}^2/N + s_{anal}^2/N*P \quad (4)$$

$$s_{tot, COMP}^2 = s_{incr}^2/N + s_{subd}^2/N + s_{anal}^2/P. \quad (5)$$

*Cross beater mill*

—*INDV*:  $N$  increments are withdrawn from a lot and transported to the laboratory. The resulting laboratory samples are completely dried and milled to obtain test samples. From each test sample  $P$  test portions are removed for analysis.

—*COMP*:  $N$  increments are withdrawn from a lot and transported to the laboratory. The resulting laboratory samples are completely dried and milled. Subsequently, the milled samples are mixed to gain a composite test sample from which  $P$  test portions are removed for analysis.

The total variance for the analytical procedures is presented in Eqs. 6 and 7.

$$s_{\text{tot}, INDV}^2 = s_{\text{incr}}^2/N + s_{\text{anal}}^2/N*P \quad (6)$$

$$s_{\text{tot}, COMP}^2 = s_{\text{incr}}^2/N + s_{\text{anal}}^2/P. \quad (7)$$

The total analytical procedures were evaluated on the basis of precision and total effort. Evaluation took place from the starting point that a total error of 10% is acceptable in testing observed levels against specified maximum values. Arrangements for the types of analytical procedure presented above have been determined, so that they just meet the 10% criterion. This was done by calculating values for  $N$  at given values of  $P$  so that  $ts_{\text{tot}}$  was less than 10% of the observed content for the lot.

Calculations were based on the general values derived for the components of variance (see Table 6). However, the value of  $RSD_{\text{anal}}$  derived in the foregoing only holds for particle size reduction using a planet ball mill. For the case of the cross beater mill the median value from our previous investigations (1 mm sieve) for  $RSD_{\text{anal}}$  is used, i.e.  $RSD_{\text{anal}} = 9.7\%$  (ref. 2).

Table 8 gives an overview of the characteristics of analytical procedures that are in agreement with the above specified 10% criterion at a level of confidence of  $P=0.05$ . Among the procedures presented are the most optimal combinations of  $P$  and  $N$ . For *INDV* it can be seen that procedures having more than three test portions per test sample only require an increased total effort. To achieve the precision aimed at, the number of increments per lot to be taken will remain about the same. For the *COMP* a lesser number of test portions per test sample than presented will lead to an impractical number of increments per lot, while an extra test portion per test sample will not reduce the number of increments per lot required to achieve the precision wanted.

The analytical procedures presented in Table 8 are similar with respect to the precision of the procedures. The choice of the procedure to be carried out eventually will be based on the effort required. For both milling by a planet ball mill and milling by a cross beater mill, the individual operations of interest for the total effort are presented in Table 9. To each operation a weighing factor is attributed reflecting the cost per operation, based on general practice of several Dutch laboratories. Two cases are distinguished:

- case A, where the effort of analysis and sampling are comparable, and
- case B, where analysis requires more effort than sampling.

Table 10 presents the number of operations that have to be performed for the analytical procedures presented in Table 8 and the total effort estimated for each procedure, using the weighing factors given in Table 9. From Table 10, a number of conclusions can be drawn. For both milling procedures, due to the number of analyses to be performed, the total effort for *INDV* is larger than for *COMP*. The difference between *INDV* and *COMP* is caused by the predominance of the error associated with the withdrawal of increments over that of subdivision and analysis.

With *INDV* the number of test portions per test sample should be restricted to one. For this type less effort is required when using the cross beater mill. For both mill types, the most favourable analytical procedures can be found in the series

COMP, b to d. Both for case A and case B the difference between the estimated total effort within this series does not seem to be significant. Also it can be concluded that, although execution of the total analytical procedure is different, the total effort required for the planet ball mill procedure is about the same as for the cross beater mill procedure.

## CONCLUSIONS

Values for the components of variance associated with the determination of the heavy metal content of garbage compost appear to be independent of the compost lot examined and the nature of the element, which enables the assessment of values of general validity. Comparison of these general values—i.e. the median values of the observates for each component of variance—shows that withdrawal of increments from a compost lot is the predominant source of error in the total analytical procedure. The errors due to subdivision and analysis are of about the same magnitude.

Autocorrelation is not expected to be important in sampling compost lots: autocorrelation is either absent or values for the correlation factor are small. Hence, for the design of total analytical procedures, the number of increments to be withdrawn is independent of the size of the compost lot.

From the viewpoint of precision in relation to total effort, when using individual test samples for each laboratory sample it is useless to take more than one test portion per test sample.

Due to the predominant contribution of the error associated with the withdrawal of increments to the total error, the precision of the total analytical procedure is more affected by the number of increments than by the number of test portions. The most favourable analytical procedures involve composite test samples.

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