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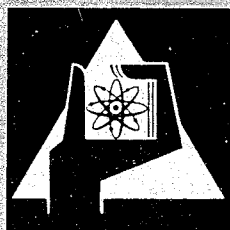
Oktober 1968

KFK 802

Institut für Angewandte Reaktorphysik

Use of Statistical Analysis for the Establishment of Material  
Balance in a Reprocessing Plant

W. Gmelin, D. Gupta, W. Häfele



GESELLSCHAFT FÜR KERNFORSCHUNG M. B. H.  
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K F K 8 0 2

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1. INTRODUCTION

In carrying out fissile material balance in a reprocessing plant considerable amounts of information and data are required. Since the collection and use of these data may cause a heavy burden of work on the control authority it is desirable to find out ways and means in reducing this work. In this paper two possibilities of reduction have been discussed and analysed. The first possibility deals with compositing individual samples obtained from tanks or containers at different strategic points for the determination of fissile material content. The second one deals with the use of data obtained by the operator himself for the determination of fissile material balance in the plant. Possible applications for the two methods have been shown with the help of numerical examples. Throughput data used for the first strategic point in the reference case for high plutonium containing fuel, as used in [1] form the basis for the numerical examples.

## 2. COMPARISON OF INDIVIDUAL AND COMPOSITE SAMPLING METHODS FOR INDEPENDENT MEASUREMENTS

Measurements of throughput form a part of the material balance. As an example it can be assumed that at the first strategic point in a reprocessing plant, the feed tank will be filled with the fissile material solution  $n$ -times per day ( $n=15$  for the reference case considered here). It is then possible to carry out the throughput measurement according to the following two methods:

- (i) For every filling  $i$  ( $1 \leq i \leq n$ ) the volume  $v$  and the concentration  $c$  will be determined individually and the result for each filling will be summed up to obtain the total throughput per day.
- (ii) From each filling  $i$  a sample will be taken, then all the samples for the  $n$  fillings will be mixed and from the mixture a single sample will be taken out to determine the concentration. The throughput through the feed tank per day will be obtained with the help of this averaged concentration and the individual volume measurements.

The conditions, number of chemical analyses required, the differences in the attainable accuracies and the advantages and disadvantages of these two methods are discussed below.

### 2.1 Individual sampling method

The extent of efforts for this method is determined by the quality of homogeneity assumed for the samples. It can either be taken for granted that an *á priori* homogeneity exists for all the samples or that the effort to test the homogeneity of the samples is negligible. It can also be assumed that certain amount of effort will be required to determine the homogeneity. Both the cases are considered here.

#### 2.1.1 The first case:

In this case it is only required to obtain a single sample and determine the concentration by analysing it twice, for obtaining the estimates for the meanvalue and the variance. With the results of the analysis  $c_1$  and  $c_2$  one

can calculate the 68 % interval  $\sigma$  after making the t-correction:

$$\sigma_c = 1.82 \left[ \frac{1}{2} (c_1^2 + c_2^2) - \frac{1}{4} (c_1 + c_2)^2 \right]^{1/2} \quad (1)$$

For subsequent consideration the term relative standard deviation or relative error  $\delta_c$  is defined with the help of the following expression:

$$\delta_c = \sigma_c / \frac{1}{2} (c_1 + c_2) \quad (2)$$

In case the accuracy of the volumetric determination can be calculated in a similar way then the fissile material content of the filling  $i$  can be calculated as follows:

$$m_i = \bar{c}_i v_i \pm \left[ \delta_i^2 + \delta_v^2 + \delta_i^2 \delta_v^2 \right]^{1/2} \bar{c}_i v_i \quad (3)$$

where

$$\bar{c}_i = \frac{1}{2} (c_1 + c_2) \quad (4)$$

is the average concentration and  $v_i$  is the volume of the solution of the filling.

Thus one can calculate the fissile material throughput  $M$  per day for all the  $n$ -fillings:

$$M = \sum_{i=1}^n \bar{c}_i v_i \pm \left[ \sum_{i=1}^n (\delta_{ci}^2 + \delta_v^2) (c_i v_i)^2 \right]^{1/2} \quad (5)$$

In equation (5) all the terms with  $\delta_{ci}^2 \cdot \delta_v^2$  have been neglected. The absolute standard deviation is given then:

$$\sigma_{11} = \left[ \sum_{i=1}^n (\delta_{ci}^2 + \delta_v^2) (\bar{c}_i v_i)^2 \right]^{1/2} \quad (6)$$

The effort in that case will be

$$A > 2 n \sqrt{\text{Analyses per day}}$$

The effort for determining the volumes can be neglected for this consideration as in both the cases i.e. for single as well as for composite sampling methods they will be the same.

### 2.1.2 Second case:

In case the test for homogeneity requires efforts that means one has to make some chemical analyses to determine the homogeneity of the sample, then the following method may be used: This method is divided into three steps

(a) One takes two samples from each filling and analyses each of these samples twice. One can then obtain the values  $c_{11}$ ,  $c_{12}$  and  $c_{21}$ ,  $c_{22}$ .

(b) One then calculates the terms

$$\bar{c}_1 = \frac{1}{2} (c_{11} + c_{12}) \text{ and } S_{c1} = \sqrt{\frac{1}{2} (c_{11}^2 + c_{12}^2) - \bar{c}_1^2} \quad (7a)$$

and

$$\bar{c}_2 = \frac{1}{2} (c_{21} + c_{22}) \text{ and } S_{c2} = \sqrt{\frac{1}{2} (c_{21}^2 + c_{22}^2) - \bar{c}_2^2} \quad (7b)$$

when  $S_{c1}$  is the deviation for the samples without t-correction.

(c) One tests then the terms  $\bar{c}_1$  and  $\bar{c}_2$  for equality with each other (for example with the help of t-test).

In case no significant deviation is obtained between these two results one can then take the solutions to be homogeneous. Then it is possible to utilize all the 4 results obtained under (a) and the resulting relative standard deviation is given in that case by  $\delta_c^{(2)} = \delta_c / \sqrt{2}$ . (8)

One can then calculate the fissile material amount M by the same method as in 2.1.1:

$$M = \sum_{i=1}^n c_i v_i + \left[ \sum_{i=1}^n (\delta_{c_i}^2 / 2 + \delta_v^2) (\bar{c}_i v_i)^2 \right]^{1/2} \quad (9)$$

The absolute standard deviation is then given by:



$$\sigma_{12} = \left[ \sum_{i=1}^n (\delta_{c_i}^2 / 2 + \delta_v^2) \cdot (\bar{c}_i v_i)^2 \right]^{1/2} \quad (10)$$

One can see that the absolute standard deviation in this case will be slightly better but the effort required will be higher and is given by

$$A \geq 4n.$$

In case the results of the two samples deviate significantly, the test has to be repeated with new samples.

## 2.2 Composite sampling method

In this case following points have to be considered in some more detail.

2.2.1 The influence of the differences in the individual volumes of the solution in the tank.

2.2.2 The influence of homogeneity in case additional chemical analyses are required.

2.2.1 The influence of differences in the volumes of solution

The total amount of fissile material passed through a tank in a day can be obtained as below:

$$M = \sum_{i=1}^n c_i v_i \quad (11)$$

This sum has to be expressed as a function of the average concentration  $\bar{c}$  which has been estimated from the mixture of all the samples.

This sum can be expressed with the Tschebyschew's inequality:

$$\sum_{i=1}^n c_i v_i \leq \frac{1}{n} \sum_{i=1}^n c_i \sum_{i=1}^n v_i = n \bar{c} \bar{v} \quad (12)$$

This equation is however only valid if

$$c_1 \leq c_2 \leq \dots \leq c_n \text{ and } v_1 \geq v_2 \dots \geq v_n \quad (13)$$

The other version of the Tschebyschew's inequality is given by

$$\sum_{i=1}^n c_i v_i \geq n \bar{c} \bar{v}, \quad \text{if } c_1 \leq c_2 \leq \dots \leq c_n \quad \text{and} \quad (14)$$

$$v_1 \leq v_2 \leq \dots \leq v_n .$$

Since the exact sequence of the volumes and the concentration are not known it is not possible to say whether the fissile material amounts calculated by equations 12 - 14 represent an upper or a lower limit of the actual value. This is because of the fact, that the individual concentrations are not known. Therefore it is not possible to utilize the method of composite sampling in general. However, this method can be utilized in case the following two conditions are fulfilled:

- (a) All the volumes  $v_i$  must be constant  $v_i = v$ .

The volume can however be measured with the relative standard deviation of  $\delta_v$  just as the measurement of the concentrations may also be associated with the relative standard deviation of  $\delta_c$ .

- (b) The volume of all the samples must be the same.

In case these two conditions are fulfilled the total fissile material flow can be measured by:

$$M = \sum_{i=1}^n c_i v_i = n v \bar{c} + \left[ \left( \delta_c^2 + \delta_v^2 \right) (n v \bar{c})^2 \right]^{1/2} \quad (15)$$

The effort in this case will then be remarkably low, namely

$$A \geq 2,$$

as the representative sample from the mixture has to be analysed twice.

### 2.2.2 The influence of homogeneity

In case it is required to test the homogeneity of the samples the same method as discussed under 2.1.2 can be utilized to test it. In case there is no significant deviation as a result of the t-test the total fissile

material flow can be calculated:

$$M = n v \bar{c} \pm \left[ (\delta_c^2 / 2 + \delta_v^2) (n v \bar{c})^2 \right]^{1/2} \quad (16)$$

In this case the effort will be

$$A \geq 4.$$

In case there is a significant deviation as a result of the two tests the method of composite sample can not be utilized for the estimation of the material balance without the use of data from the operator.

This will be discussed in detail under chapter 3 of this paper.

### 2.3 Numerical results

To compare the various efforts and the attainable absolute standard deviations for the two cases discussed above a numerical example has been calculated with the reference case mentioned in [1]. Following assumptions have been made:

- (a)  $n = 15$  fillings
- (b)  $M = 70$  kg Pu/day
- (c) The relative standard deviations for the concentration and the volumetric measurements will vary in such a manner that the following three overall relative standard deviations  $\delta$  will be obtained:

$$\begin{aligned} \text{c.1 } \delta_c &= 0.7 \% & \delta_v &= 0.7 \% \\ \text{thus } \delta &= \sqrt{\delta_c^2 + \delta_v^2} & &= 1 \% \end{aligned} \quad (17)$$

$$\text{c.2 } \delta_c = 1.4 \% ; \quad \delta_v = 1.4 \% ; \delta = 2 \%$$

$$\text{c.3 } \delta_c = 0.45 \% ; \quad \delta_v = 0.35 \% ; \delta = 0.5 \%$$

With these assumptions the values on the effort on analyses and the absolute standard deviations in kg Pu/day have been calculated for the reference case and given in table I.

## 2.4 Conclusions

On the basis of the numerical values presented in table I following conclusions can be drawn:

- (i) The advantage of the composite sampling method over the single sampling method lies in a considerably lower effort for the former. This effort is approximately 1/15 of that required for the single sampling method.
- (ii) One disadvantage of the composite sampling method lies in the fact that it has got a higher absolute standard deviation than that obtained in the single sampling method. For the reference case this is 3.8. In general the reduction in the accuracy is given by  $\frac{1}{\sqrt{n}}$ , where n represents the number of fillings. An increase in the number of samples from the mixture gives only a small improvement in the absolute standard of deviation as only the absolute deviation of the concentration measurement is improved.
- (iii) The main disadvantage of the composite sampling method lies in the fact that in case of inhomogeneity among the various samples no proper statement can be made. In that case a reproduction of the samples cannot be undertaken as all the fillings would have already been sent into the plant. However, as will be shown in the next chapter this method in combination with the data from the operators can be quite an effective one for the establishment of the material balance.

## 3. USE OF OPERATOR'S DATA

The control authority can establish a material balance according to the following two methods:

- (i) Through independent measurement without considering the data obtained by the operator.
- (ii) After consideration of the operator's data. As can be expected this method requires a less effort on the part of the control authority and may even be more accurate. However, these data have to be controlled by statistical means.

The first case was considered under 2. The possibility of using the operator's data has been discussed below. In developing the relevant equations the following assumptions have been made:

- (i) One tank which may be at the first strategic point will be filled  $n$ -times per day with a fissile material solution having a volume of  $v_i$  ( $1 \leq i \leq n$ ) and with a concentration  $c_i$  ( $1 \leq i \leq n$ ).
- (ii) The operator carries out a material balance for his own purpose according to the single sampling method discussed in 2.1 with the standard deviations and the efforts discussed there.
- (iii) The objective of the control of the operator's data will be to state with a probability  $P$  (at level of significance) of:
  - (a)  $P = 80 \%$
  - (b)  $P = 90 \%$
  - (c)  $P = 99 \%$

that these data do not coincide with the data obtained by the controller for the same purpose.

A significant deviation of the operator's data from the controller's data would mean that the operator has tried to falsify his own data to camouflage a scheduled diversion of fissile material.

In case the operator would plan a diversion he would either try to reduce the values at the strategic point at the entrance of the plant or try to increase the values at the strategic point at the exit of the plant.

Therefore to control the operator's data it will be necessary and sufficient to carry out a one-sided test namely

- (i) To test for a lower limit at the strategic point at the entrance of the plant
- (ii) and to test for the upper limit at the strategic point at the exit of the plant.

The problem is to find out the extend of effort required in analysing the samples and also to establish a method for determining the possibility with which the operator can falsify his data expressed in kg Pu/day as a function of the standard deviation and the confidence level (which may be considered to be an index for the reliance placed on the operator's data) before the control authority can state that falsification has taken place and that the operator's data can be rejected.

In case the inspector proposes to utilize the operator's data he has to bring forth two types of reliance towards the operator. These two reliances are discussed below. For the single sampling method both the reliances are required, whereas for the composite sampling method only the second type is necessary.

In developing the controlling method two cases have to be distinguished from each other:

3.1 Control with the help of single sampling methods

3.2 Control with the help of composite sampling methods

### 3.1 Single sampling method

In this method  $m$  samples from  $n$  tank fillings ( $m \leq n$ ) will be controlled. With  $m = n$  samples controlled, the inspector performs an independent material balance as shown in 2.1 and with  $m = 0$  the inspector only uses the operator's data without any control and any effort. The number  $m$  of samples controlled by the inspector depends of course on the reliance of the first type which the inspector places on the operator's data.

#### 3.1.1 Reliance of the first type

Two methods can be considered to define the reliance of the first type:

- (i) First model: Under the assumption, that the operator falsifies  $q$  from  $n$  tank fillings, the reliance  $R_{11}$  may be defined:

$$R_{11} = 1 - P_{11}(n, m, q) \quad (18)$$

where  $P_{11}$  is the probability, that the inspector detects exactly this  $q$  falsifications.

The probability  $P(n, m, q)$  can easily be evaluated by using

the hypergeometric distribution:

$$P_{11}(n, m, q) = \frac{\binom{m}{q} \cdot \binom{n-m}{q-q}}{\binom{n}{q}} = \frac{\binom{m}{q}}{\binom{n}{q}} = \frac{m! (n-q)!}{n! (m-q)!} \quad (19)$$

The upper limit of this function is:

$$P_{11}(n, m, q) = m/n \quad \text{for } q = 1 \quad (20)$$

and this gives:

$$R_{11} \geq 1 - \frac{m}{n} \quad \text{or exactly } R_{11} = 1 - \frac{m! (n-q)!}{n! (m-q)!} \quad (21)$$

Equ. (19) shows the higher the probability of detection, the lower is the reliance which the inspector places on the operator's data.

- (ii) Second model: Under the same assumption about  $q$  as shown in (i), the reliance may be defined:

$$R_{12} = 1 - P_{12}(n, m, q) \quad (22)$$

where  $P_{12}(n, m, q)$  is the probability, that the inspector detects at least one of this falsifications  $q$ .

By using once more the hypergeometric distribution, one has:

$$\begin{aligned} P_{12} &= \sum_{i=1}^q \frac{\binom{n}{q_i} \cdot \binom{n-m}{q-q_i}}{\binom{n}{q}} = 1 - \frac{\binom{m}{0} \cdot \binom{n-m}{q-0}}{\binom{n}{q}} \\ &= 1 - \frac{(n-m)! (n-q)!}{(n-m-q)! \cdot n!} \end{aligned} \quad (23)$$

$$P_{12} = 1 - \frac{(n-m)(n-m-1)(n-m-2) \dots (n-m-q+1)}{n(n-1)(n-2) \dots (n-q+1)}$$

and therefore:

$$R_{12} = 1 - P_{12} = \frac{(n-m)(n-m-1) \dots (n-m-q+1)}{n(n-1)(n-2) \dots (n-q+1)} \quad (24)$$

In table II the different values of  $R_{11}$  and  $R_{12}$  are shown as a function of  $n$  for  $n = 15$ ,  $q = 1$  and  $q = 2$ . For  $q = 1$  the two models are equivalent, but for  $q > 1$  the second model needs a significantly lower degree of reliance.

There is however a possibility, that the inspector has to reject the

operator's data because of a significant deviation from his own results. In case of such a rejection the inspector has the possibility of analysing all the  $n$  samples to establish his own material balance and then he does not use the operator's data.

### 3.1.2 Reliance of the second type

The reliance of the first type does not give a criterion on the basis of which the operator's data can be rejected. A suitable criterion for this rejection can be given by performing a well known test of significance  $\sqrt{2}$  which is discussed below. The level of significance of such a test may be called as the reliance of the second type. Such a test can be carried out for the reference case with the assumption that the relative error  $\delta$  of analyses for both the operator and the inspector are the same. The idea in performing such a test is, that before the inspector can make a statement about a falsification, he must have a certain degree of confidence  $S$  in the measured difference between his data and the operator's data. This means that the inspector would accept the data of the operator even if he finds a difference between his and the operator's data. This difference is a function of the confidence  $S$  and the variances of the two measurements. This test can be performed in the following stages:

- (i) Each from this  $m$  samples controlled has to be analysed twice and the results may be  $c_{11}$  and  $c_{12}$ .
- (ii) The corresponding values of the operator's analyses may be  $c_{21}$  and  $c_{22}$ .
- (iii) The inspector calculates the sample means and sample variances:

$$\begin{aligned}\bar{X}_1 &= \frac{1}{2} (c_{11} + c_{12}) \\ S_1^2 &= \frac{1}{2} (c_{11}^2 + c_{12}^2) - \bar{X}_1^2\end{aligned}\quad (25)$$

and

$$\begin{aligned}\bar{X}_2 &= \frac{1}{2} (c_{21} + c_{22}) \\ S_2^2 &= \frac{1}{2} (c_{21}^2 + c_{22}^2) - \bar{X}_2^2\end{aligned}\quad (26)$$



(iv) Normally the real variances of the analyses  $\sigma_1^2$  and  $\sigma_2^2$  will be known by prior knowledge, that one has to test the correspondence between these variances  $\sigma_1^2$  and  $\sigma_2^2$  and the sample variances  $S_1^2$  and  $S_2^2$  obtained by the actual measurements. Therefore one performs firstly a  $\chi^2$ -test. The quantities  $\frac{2 S_1^2}{\sigma_1^2}$  and  $\frac{2 S_2^2}{\sigma_2^2}$  are distributed

according to a  $\chi^2$ -distribution with 1 degree of freedom. The correspondence can be accepted if the following conditions are fulfilled:

$$\frac{1}{2} \sigma_1^2 \cdot \chi_u^2(1) \leq S_1^2 \leq \frac{1}{2} \sigma_1^2 \cdot \chi_o^2(1) \quad (27)$$

and

$$\frac{1}{2} \sigma_2^2 \cdot \chi_u^2(1) \leq S_2^2 \leq \frac{1}{2} \sigma_2^2 \cdot \chi_o^2(1) \quad (28)$$

where

$\chi_u^2(1)$  is the value of the  $\chi^2$ -distribution at the lower-S-  
(e.g. 95 %) level with one degree of freedom

$\chi_o^2(1)$  is the value of the  $\chi^2$ -distribution at the upper  
significance level (e.g. 95 %)

If these conditions are fulfilled, then one can calculate with  $\sigma_1^2$  and  $\sigma_2^2$  respectively and one can perform the next stage for comparison of the mean-values  $\bar{X}_1$  and  $\bar{X}_2$ :

If:

$$(\bar{X}_1 - \bar{X}_2) / \sigma_d \leq U(S) \quad \text{or} \quad (29)$$

$$(\bar{X}_1 - \bar{X}_2) \leq U(S) \cdot \sigma_d$$

where

$$\sigma_d^2 = \frac{\sigma_1^2}{2} + \frac{\sigma_2^2}{2} \quad (30)$$

is the combined variance and

$U(S)$  the value of the normal distribution at the level of significance  $S$ .

If condition (29) is fulfilled, then it can be assumed, that the corresponding measurements do not differ significantly and the operator's data can be accepted.

The results of such a test expressed as significant deviation in kg Pu/day as a function of relative error  $\delta$ , for reliance of the first type  $R_{11}=R_{12}=0\%$  are presented in table III.

The numerical values presented in table III show for example that before the controller can state with a probability of 99.9 % that the data presented by the operator have been falsified, the operator can falsify his data up to a value of 0.79 kg Pu/day with a relative error of = 1 %.

One can consider the reliance of the second type to be proportional with the statistical confidence S.

It can easily be shown that for a given statistical confidence S the amount Q given in table III for the case of  $R_{1i}=0$  is indirectly proportional to  $1-R_{1i}$  ( $i=1$  or  $2$ ) so that the factor  $Q(S) / 1-R_{1i}$  is the amount by which the operator can falsify his data before the controller can detect it with the statistical confidence S. The different values of  $Q(S) / 1-R_{1i}$  are given in table IV for a relative standard deviation of 1 % and for both the models with  $q = 1$  for the first model or the second model and with  $q > 1$  for the second model only.

It can be seen that by reducing the number of samples M the effort spent by the controller on analyses will be reduced. However, with decreasing number of samples the amount which can be falsified by the operator before the controller can detect it with a chosen statistical confidence is increased. It can be seen, too, that the second model for the single sampling model is more efficient or equal to the first model if the operator performs  $q > 1$  falsifications.

### 3.2 Control of operator's data with the composite sampling method

In case the two conditions required for carrying out this method are fulfilled the composite sampling method can also be used but it presents a slightly different situation. As has been shown in chapter 2.2, in case the controller does not use operator's data he has to take two mixtures and

analyse them to test for homogeneity. In case the controller uses the operator's data only one mixture has to be prepared as the controller can test the results with the data obtained from the operator. Since all the fillings are controlled the probability  $P$  is always 100 % so that no reliance of the first type and only that of the second type has to be considered. The corresponding data as shown in table IV are summarized and given in table V for this method. In this case it should however be noted that two different relative standard deviations, one for the operator  $\delta_B$  and one for the controller  $\delta_K$  have been considered for the measurement accuracy.

### 3.3 Conclusion

On the basis of the results shown in table V the following conclusions can be drawn.

The operator's data can be utilized by the controller for establishing his own material balance provided the following three conditions are fulfilled:

- (a) The data of the operator have to be controlled by the reliance of the first and the second type.
- (b) The required standard deviation for the test has to be guaranteed.
- (c) It has to be clarified what type of measure should be taken in case the data from the operator deviate significantly from those of the controller.

In case these conditions are fulfilled the utilization of operator's data offers the following advantages over the case in which completely independent measurements are carried out by the controller for the establishment of the material balance.

- (i) The effort required for carrying out chemical analyses is considerably less.
- (ii) In case no significant deviation exists between the operator's and the controller's data, the use of the former improves the absolute standard deviation in kg Pu/day of the controller's measurement compared to an independent material balance by the

single sampling method. This improvement is about 5 % in the case of the composite sampling method and about 30 % in the case of the single sampling method if the operator's data are utilized. But the analytical effort required for the composite sampling method using the operator's data will be only 6.7 % of the effort required for an independent measurement. For a single sampling method the effort will be the same.

- (iii) The composite sampling method shows some disadvantages if used by the controller for the establishment of an independent material balance. This method can however be utilized in combination with operator's data effectively because the values of possible falsification in the operator's data can be regulated by a proper choice of the statistical confidence.

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TABLE I. EFFORT AND ABSOLUTE STANDARD DEVIATIONS  $\sigma/\bar{x}$  kg Pu/day  $\bar{x}$   
FOR INDEPENDENT MEASUREMENT

|   | Single sampling method<br>Test of homogeneity |       | Composite sampling method<br>Test of homogeneity |      |
|---|---|-------|--|------|
|   | without                                       | with  | without  | with |
| Effort in<br>chem. analyses                         | > 30  | > 60  | > 2  | > 4  |
| $\sigma$ $\bar{x}$ kg $\bar{x}$<br>$\delta$ = 1 %   | 0.181   | 0.155 | 0.7  | 0.6  |
| $\sigma$ $\bar{x}$ kg $\bar{x}$<br>$\delta$ = 2 %   | 0.362   | 0.310 | 1.4  | 1.2  |
| $\sigma$ $\bar{x}$ kg $\bar{x}$<br>$\delta$ = 0.5 % | 0.091   | 0.077 | 0.35   | 0.3  |

TABLE II. RELIANCE OF THE FIRST TYPE  $R_{11}$  AND  $R_{12}$  FOR SINGLE SAMPLING  
METHOD USING OPERATOR'S DATA

|                                |     | n = 15 |    | q = 1 |    |    |
|--------------------------------|-----|--------|----|-------|----|----|
| m                              | 0   | 3      | 6  | 9     | 12 | 15 |
| $R_{11}$ $\bar{x}$ % $\bar{x}$ | 100 | 80     | 60 | 40    | 20 | 0  |
| $R_{12}$ $\bar{x}$ % $\bar{x}$ | 100 | 80     | 60 | 40    | 20 | 0  |
|                                |     | n = 15 |    | q = 2 |    |    |
| m                              | 0   | 3      | 6  | 9     | 12 | 15 |
| $R_{11}$ $\bar{x}$ % $\bar{x}$ | 100 | 97     | 86 | 66    | 37 | 0  |
| $R_{12}$ $\bar{x}$ % $\bar{x}$ | 100 | 63     | 34 | 14    | 3  | 0  |

TABLE III. SIGNIFICANT AMOUNTS Q (S) IN KG PU /DAY (For R = 0 %) FOR THE SINGLE SAMPLING METHOD USING OPERATOR'S DATA

| Statistical confidence S             |       | 99.9 % | 95 %  | 90 % | 80%  |
|--------------------------------------|-------|--------|-------|------|------|
| Relative standard deviation $\delta$ | 1 %   | 0.79   | 0.425 | 0.33 | 0.21 |
|                                      | 0.5 % | 0.39   | 0.21  | 0.16 | 0.11 |
|                                      | 2 %   | 1.58   | 0.84  | 0.65 | 0.43 |

TABLE IV. SIGNIFICANT AMOUNTS Q ( $S_1, R_1$ ) IN KG PU/DAY FOR THE SINGLE SAMPLING METHOD FOR BOTH THE MODELS ( $R_{11}, R_{12}$ )

| Statistical confidence S               |                    |         |                             |      |      |      |
|--|--------------------|---------|-----------------------------|------|------|------|
| "reliance type 2" $\underline{[ \% ]}$ | 99.9               | 95      | 90                          | 80   |      |      |
| Reliance Type 1                        | Effort in analyses |         |                             |      |      |      |
| $R_{11}$ and $R_{12}$                  | Model 1            | Model 2 | or for                      |      |      |      |
|  | Model 2            |         |                             |      |      |      |
|  | q = 1              | q > 1   |                             |      |      |      |
| 100 %                                  | 0                  | 0       | each falsification possible |      |      |      |
| 80 %                                   | 6                  | ≤ 4     | 3.95                        | 2.1  | 1.7  | 1.1  |
| 60 %                                   | 12                 | ≤ 8     | 1.97                        | 1.1  | 0.83 | 0.53 |
| 40 %                                   | 28                 | ≤ 12    | 1.31                        | 0.7  | 0.55 | 0.35 |
| 20 %                                   | 24                 | ≤ 16    | 0.98                        | 0.53 | 0.41 | 0.26 |
| 0 %                                    | 30                 | 30      | 0.79                        | 0.42 | 0.33 | 0.21 |

TABLE V. SIGNIFICANT AMOUNTS Q (S) IN KG PU/DAY FOR THE  
COMPOSITE SAMPLING METHOD

| Statistical<br>confidence [ % ]<br>"reliance type 2" | 99.9 | 95   | 90   | 80   |
|--|------|------|------|------|
| $\delta_B = \delta_K = 1 \%$                         | 2.23 | 1.18 | 0.93 | 0.61 |
| $\delta_B = 1 \%$                                    | 1.22 | 0.64 | 0.5  | 0.33 |
| $\delta_K = 0.5 \%$                                  |      |      |      |      |
| $\delta_B = 1 \%$                                    | 4.36 | 2.31 | 1.81 | 1.2  |
| $\delta_K = 2 \%$                                    |      |      |      |      |
| $\delta_B = \delta_K = 0.5 \%$                       | 1.1  | 0.59 | 0.46 | 0.3  |
| $\delta_B = 1 \%$                                    | 0.8  | 0.42 | 0.33 | 0.22 |
| $\delta_K = 0.25 \%$                                 |      |      |      |      |

Effort in chemical analyses :  $A \geq 2$

TABLE VI. VALUES OF  $(m'-1)$  AS A FUNCTION OF  $m$  AND THE RELIANCE OF THE SECOND TYPE

| m | 3      | 6      | 9      | 12       | 15         |
|---|--------|--------|--------|----------|------------|
|   | 2/87.5 | 4/90.  | 6/91.  | 8/93.0   | 10/94.0    |
|   |        | 5/98.0 | 7/98.0 | 9/98.0   | 11/98.2    |
|   |        |        | 8/99.8 | 10/99.7  | 12/99.6    |
|   |        |        |        | 11/99.95 | 13/99.92   |
|   |        |        |        |          | 14/99.9999 |

where: 2/87.5 means  $m'-1 = 2$  and reliance = 87.5 %



## Appendix.

### Additional considerations for the use of operator's data.

It was indicated in 3.1 that the inspector can use the operator's data and improve his own results controlling  $m$  from  $n$  tank fillings. He has to assign a certain degree of reliance of the first and the second type to the operator's data. In table IV of the text, the maximum amounts have been presented which the operator can falsify, before his data are rejected on account of the reliance of the second type.

In reality, the possibility for the operator to falsify is much less because of the fact that these maximum amounts which can be falsified, can be obtained only if all the operator's data have lower values than those of the inspectors. This event has a very low probability under the condition that no falsification has taken place. Therefore, an additional test can be constructed on account of this very low probability of such events, to reduce the amounts which the operator can falsify.

The probability  $W$ , that from  $m$  samples controlled,  $m' < m$  or more samples have a negative difference (e.g. the inspector's measurement is higher than that of the operator) under the condition that no falsification has taken place, can be calculated with the help of the binominal distribution:

$$W(m', m, p) = \sum_{j=m'}^m \binom{m}{j} \cdot p^j (1-p)^{m-j} \quad (1)$$

The probability  $p$  for the occurrence of one negative difference is  $p = 1/2$  for normal distributions considered here.

Thus:

$$W(m', m, 1/2) = \left(\frac{1}{2}\right)^m \cdot \sum_{j=m'}^m \binom{m}{j}$$

For example:

The probability, that from  $m = 15$  samples controlled, more than or equal  $m' = 11$  samples have a negative difference, is  $W = 6\%$ .

On the basis of this relationship the following test can be performed by the inspector:

- i. A level of significance  $S = 1 - \alpha$  has to be defined.
- ii. If an event occurs whose probability was less than  $\alpha$  the operator's data are rejected.

Following the definition of the reliance of the second type one can extend this definition for the special case considered here. The reliance of the second type was defined in the text to be proportional to the level of significance  $S$  of a significance test. But the test mentioned above is a significance test too and therefore, by the same reliance of the second type the level of significance in the test mentioned above can also be established.

For example:

If the inspector controls  $m = 12$  from  $n = 15$  tank fillings and if he assigns to the operator's data a reliance of the second type, say 93 %, then he firstly has to test each of the  $m$  samples at the significance level of 93 %. Under the assumption that the operator's data are not rejected during this first test the inspector still can reject the operator's data, in carrying out the second test, described above, if for example the event occurs that in  $m' \geq 9$  cases the values of the measurements of the operator are less than the inspector's values, because of the fact, that the probability of such events is  $W \leq 1 - 0.93 = 7\%$ .

For different values of  $m$ , the limits of rejection of the operator's data for this second test, are presented in table VI as a function of the reliance of the second type.

Table VI shows for example that if from  $m = 9$  samples controlled, for up till 6 samples a negativ difference is obtained, a reliance of 91 % is necessary. That means, if the reliance for this test has been taken to be 91 % and if  $m' = 7$  or more, negative differences are obtained, the inspector has to reject the operator's data and cannot use these data for his material balance.

These considerations can also be applied to the composite sampling method, but the inspector has then to wait a number of  $m$  days before he can perform such a test.