Nederlandse norm

NEN 6603

(en)

Milieu en voedingsmiddelen - Eerstelijnscontrole met controlekaarten voor chemische en microbiologische analyses

Environment and food – Internal quality control by the use of control charts with chemical and microbiological analyses

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Foreword

This standard describes quality control of chemical and microbiological analyses within laboratories by using control charts.

The standard applies to chemical and microbiological analyses in all types of samples from the environmental, food and feed application areas.

The standard replaces NPR 6603:1988, *Water en slib – Richtlijnen voor interne kwaliteitsbeheersing met controlekaarten bij chemische analyses.*

Changes relative to NPR 6603:1988:

a) the document has been converted from a Dutch Practical Guideline (NPR) into a standard (NEN);

b) the scope has been expanded to include microbiological analyses in addition to chemical analyses;

c) the standard is now suitable for application in all environment-related matrices, food and animal feed;

d) the procedure for control charts with fixed limits has been added;

e) a procedure for multicomponent analysis based on the mean Z-score has been added;

f) the verification of the mean of the full chart has been added;

g) guidelines for dealing with out-of-control have been added;

h) the statistical assessment of the percentage of random exceedances has been added;

i) the text has been updated thoroughly.

This standard was developed under the responsibility of standards committee 3900 20 'Milieukwaliteit ' by standards subcommittee 390 02007 'Statistische toepassingen'.

Copies of this standard can be obtained, for a fee, from NEN, PO Box 5059, 2600 GB Delft, The Netherlands.

Environment and food – Internal quality control with control charts for chemical and microbiological analyses

1 Scope

This standard describes quality control of chemical and microbiological analyses within laboratories by using control charts.

The standard applies when performing chemical and microbiological analyses on all types of samples from the environmental, food and feed application areas. The method can be used when assessing the validity of laboratory tests as described in NEN-EN-ISO/IEC 17025.

NOTE 1 The procedure described in this standard does not necessarily offer guarantees with regard to the correctness and/or uncertainty of the measured result of a random sample from practice.

NOTE 2 The usability of the procedure described in this standard may cover other areas of application. These, however, have not been investigated.

2 Normative references

The following document is referenced in this document and is indispensable for its application. For undated references, the latest edition of the referenced document (including any amendments) applies.

NEN-EN-ISO/IEC 17025*, General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
action criterion
rule to identify an out-of-control process

3.2
control chart
chart on which a statistical measurand of a series of samples is recorded in a specific sequence to control the process for this measurand and to control and reduce variation

[SOURCE: ISO 3534-2]

Note 1 to entry: The determined sequence is often based on time or the sequence of the sample number.

3.3
control standard
independently prepared standard with the same matrix as the calibration standard

3.4
certified reference material
CRM
reference material characterized by a (metrologically) valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty and, if possible, a statement of metrological traceability

3.5
average run length
ARL
average number of times that a process will have been sampled and assessed before a shift in the level is identified

Note 1 to entry: For an analytic process, this is the average number of control measurements that are required before out-of-control is identified. The reciprocal value indicates the probability of occurrence.

Note 2 to entry: This definition is based on ISO 3534-2.

3.6
out-of-control
condition where the control results can no longer be ascribed to the assumed distribution of probability

4 Objective of quality control of analyses with control charts

Repeated execution of a chemical and microbiological determination in the same sample leads to a variation in the results. This may be caused by randomness, but also because of systematic deviations.

The objective of quality control with control charts (3.2) is:

— to keep the variation in results within the set limits;

— to identify the exceedance of these limits and the presence of trends as quickly as possible;

— to prevent the release of deviating analytical results as much as possible;

— to determine the cause of deviations and to prevent their future occurrence.

5 Position of the standard within the whole of quality control

Quality control of chemical and microbiological (quantitative) analyses can be subdivided as follows:

a) Internal quality control:

— By technician: first-line quality control by those who perform analyses, in particular, by analysing control materials and assessing the results through control charts.

— By laboratory: second-line quality control within the organisation of the laboratory that is as independent as possible from those who perform the analyses, e.g. by providing samples with a known content of the component to be determined.

b) External quality control:

— By Proficiency Testing: third-line, independent external control, mainly through interlaboratory comparison.

This standard is limited to the aforementioned first-line control with control charts and is in particular limited to:

— the choice of control materials and measurands to be controlled;

— setting up a control chart;

— action criteria;

— the registration of results;

— the assessment of results.

6 Principle

Analysis execution is controlled by regular investigation of control materials. The aim is that the control results reflect the quality of the sample analysis as much as possible. For the control measurement, this means that the samples, the execution and the conditions under which the execution takes place shall be as similar as possible to those of the sample analysis.

The control results are shown in a control chart (3.2) with the analysis results on the vertical axis and the sequence in which the results were obtained on the horizontal axis. Furthermore, the central value and the limits at a distance of once, twice and three times the standard deviation[1)] from the central value (1*s*, 2*s* and 3*s* limits) are indicated on the control chart.

The central value is often the mean control result over a longer period, but it can also be the conventional value of the control material. In the latter case, apart from the possibility of monitoring random deviations, also the systematic deviation (correctness) of the control material can be monitored. The applied standard deviation *s* is either the experimentally determined standard deviation of control results from the past or a fixed imposed standard deviation.

The action criteria (3.1) are based on exceedances of the 1*s* and 3*s* limits. If an action criterion is exceeded, the quality is considered to be out-of-control (3.6); the analysis results of samples will not be released and an investigation will be started into the cause of the exceedance and a decision will be taken about rejecting or not (a part of) the results of the relevant measurement run.

The results of the control investigations will be periodically assessed.

1) The standard deviation based on a random sample (i.e. with $n - 1$ degrees of freedom) is meant.

7 Control materials

7.1 General

The conditions that are set with regard to control material are the following:

— homogeneity: the contribution to the variation of analysis results as a consequence of non-homogeneity is negligibly small;

— stability: the contribution to the variation of analysis results as a consequence of instability is secondary;

— representativeness: the variation of analysis results for the control material matches (or is higher than) the expected mean variation for samples from practice that have been prepared in a similar manner as the control material to meet the criteria for homogeneity and stability.

If possible and useful, select a value for the measurand that is well above the detection limit. The *coefficient of variation* (*relative standard deviation)* for this subrange is often virtually constant and one control material will be sufficient for the measuring range.

The *standard deviation* is virtually constant for some analysis methods. In that case, one control material for the measuring range will also be sufficient. Consider applying more than one control material for measuring methods for which the value of measuring subranges differs considerably.

NOTE 1 If the validation test should show that the standard deviation or the coefficient of variation is different for various measuring subranges, it will not automatically mean that a control is required for more than one measuring subrange. If, for example, the cause for the variation for the measuring subrange is the same, but the magnitude is different, the control on one level will protect other levels, too.

NOTE 2 It is not always possible or practical to cover all steps of the analytical process with one control material. The quality of the homogenising process, for example, is not considered with regard to the analysis of non-homogeneous samples from practice. Representativeness in these cases refers to the covered part of the analytic process. Subsequently, quality control of missing parts, such as the residual non-homogeneity of samples from practice after pre-treatment, should be covered differently.

7.2 Control sample from one or more samples from practice

A control sample is a control material that consists of one or more samples from practice. The representativeness for the matrix is often assured for a stable mixture of randomly selected production samples. If the analysis quality is critical for part of the samples (submatrix) and the aim is (also) to monitor the analysis quality of these samples with the control, take a control sample from this part.

Correctness verification is often impossible with a control sample because the control sample does not have an (independent) assigned value.

NOTE Correctness verification is, however, possible if a reference value has been assigned to the control sample based on the analysis results of other laboratories where these results were obtained in accordance with the rules of a soundly organised ring test. If these rules are not met, the test is indicative.

7.3 Sample for the recovery from addition to one or more samples from practice

In this form of control measurement, samples are analysed together with the same samples to which a known quantity of measured component has been added. The recovery is calculated from the

difference in measured results, which is usually a relative value (fraction or percentage). The recovery is included in the control chart. Applying the recovery ensures that correctness verification is possible but it is limited to matrix effects; interference effects are not accounted for. For the time being, this procedure is not applied in microbiology.

The addition shall always be performed on the same (stable and representative) sample from practice or to a random sample from practice from the sample run. The last variant is very effective if matrix effects are involved that differ significantly from sample to sample. Representativeness in the medium term will then, by definition, be assured even if shifts occur in the composition of the matrix over time. A disadvantage is that the exceedance of the action criterion (3.1) does not have to be the result of an out-of-control measuring process only, but that it can also be the result of the (random) composition of the sample matrix.

If possible, add to the sample from practice a quantity that at least leads to a fourfold increase of the value of the measured quantity. Prevent a dilution of the analysis sample by more than 10 %.

NOTE 1 If the increase due to addition is fourfold or more, the deviation for a sample as calculated from the differences is almost equal to the reproducibility deviation for a singular analysis of the sample with addition. If addition at this level is not possible, for example, due to (the risk of) exceeding the measuring range, the deviation of results will be larger than the reproducibility deviation.

NOTE 2 Quality control by means of recovery is especially suitable for the analysis of liquid samples where the quantitatively added component has the same analytical behaviour as the component originally present in the sample.

NOTE 3 For chemical analyses, the recovery of additions to random samples from practice is very suitable to quantify the measurement uncertainty in one go when the influence of storage, ageing, pre-treatment and interference are negligible (see NEN 7779).

7.4 Synthetically prepared control sample

A synthetically prepared control sample is a sample of which the value of the measurand(s) is known within narrow limits based on the preparation procedure. A prerequisite for the application is that the matrix is representative for the matrix of the samples from practice. This mainly applies to those aspects that have an effect on the measured result, such as the influence of humic acids on the clean-up.

With a synthetic chemical control sample, in addition to the verification in relation to random deviations, the verification with regard to systematic deviations of the conventional value is possible. If the matrix of the samples from practice is very variable, the control of unwanted systematic effects will only be effective if the synthetic control sample is representative of the deviating samples of this matrix.

In microbiology, synthetic control samples are usually prepared by freezing or drying microorganisms. As a result, some of the added microorganisms will die and therefore correctness will no longer be possible. This type of control materials are often called RM or SRM ((secondary) reference material).

NOTE 1 Often a control standard (3.3) cannot be used as control material for quality control of the measuring process. It is, however, legitimate to use a control standard as quality control of the calibration process.

NOTE 2 NVN 6419 describes the preparation of synthetic control samples for organic compounds in groundwater and surface water. ASTM D 5905 describes the preparation of household waste water.

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