
Karakterizacija blata - Protokol za metode validacije fizikalnih lastnosti blata

Characterization of sludges - Protocol for validating methods for physical properties of sludges

Charakterisierung von Schlämmen - Protokoll für Validierungsmethoden von physikalischen Schlammeigenschaften

Caractérisation des boues - Protocole de validation des méthodes de détermination des propriétés physiques des boues

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ICS:

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English Version

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This Technical Report was approved by CEN on 7 February 2006. It has been drawn up by the Technical Committee CEN/TC 308.

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Foreword

This Technical Report (CEN/TR 15252:2006) has been prepared by Technical Committee CEN/TC 308 "Characterization of sludges", the secretariat of which is held by AFNOR.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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Introduction

To be published as a “*standard*”, all the methods defined by CEN must be validated through interlaboratory trials. The *validation* procedure is carried out after the 6-months enquiry and before the formal vote, in order to incorporate comments received during enquiry into the final version to be officially approved.

Validation usually consists in analyzing reference samples circulated to many laboratories in different countries and in evaluating the “*repeatability/reproducibility*” of measurements, where:

- *Repeatability* is defined as the ability of a method to reproduce a measurement while being tested under an unchanging set of conditions. This does not imply that the obtained value is correct, but rather that it is the same every time;
- *Reproducibility* is the same as repeatability, but at a different set of conditions. It is therefore a more realistic indication of a method to reproduce a measurement, whenever a predefined set of conditions is recreated.

However, for physical parameters, with the exception of calorific value, samples of “*dried sludge*” cannot be used for analysis, but only “*fresh sludge*” samples, with the consequence that many problems arise because:

- the sludge physical characteristics change with time of storage, also considered that any preservation practice (e.g. freezing) makes things worse;
- the sludge physical characteristics are strongly affected by transport and handling;
- fresh sludge requires particular precautions and authorization for transport by ordinary delivering systems.

This involves that “*fresh sludge*” samples need to be examined by laboratories close to wastewater or waterworks plants and analysed as soon as possible, minimising their manipulation, so the circulation of fresh sludge samples to laboratories in several places and in different countries requires well defined procedures to guarantee the reliability of results.

A valid alternative could consist in the examination of “*synthetic sludge*” samples to be on-site prepared on the base of a defined recipe and ingredients.

Another possibility is carrying out validation tests through “*circulation of analysts*”, and not “*circulation of samples*”, thus allowing analysts from the participating laboratories to meet in a laboratory close to the place where samples are collected.

Finally, if any of the above options cannot be followed, validation could be limited to a “*confidence level evaluation*” through the statistical evaluation of sludge samples taken in different works and examined in laboratories located close to the respective works.

Therefore, one of the three procedures, which are described in the following, should be followed:

- the first by examining “*real or synthetic reference*” samples in different laboratories (*conventional round robin tests*);
- the second in examining “*reference*” samples in one laboratory, close to the place where samples are collected, where analysts from the participating laboratories will meet and work with their own apparatuses (*modified round robin tests*);
- the third including the statistical evaluation of sludge samples taken in different works and examined in laboratories located close to the respective works (*confidence level evaluation*).

In all cases, preliminary planning and arrangements are required.

1 Scope

This Technical Report specifies procedures for validating characterization methods for physical properties of sludge.

This Technical Report is applicable to sludge and sludge suspensions from:

- storm water handling;
- urban wastewater collecting systems;
- urban wastewater treatment plants;
- treating industrial wastewater similar to urban wastewater (as defined in Directive 91/271/EEC);
- water supply treatment plants.

This method is also applicable to sludge suspensions from other origin.

2 Normative references

Not applicable

3 Preliminary arrangements

For each material to be tested, the statistical evaluation must be based on the results obtained from at least nine laboratories, of at least three different countries.

National members of interested CEN/TCs will take care to select special laboratories in their countries; these laboratories should preferably have been successfully involved in round robin tests in the past.

In addition, the analysts should have enough experience in statistics; it is favourable for him having a computer based calculation program of the defined statistical method.

Preliminary arrangements involve the definition of:

- the method to be tested;
- the nature of samples;
- methods of statistical calculation;
- a list of all participants to testing, including address, contact person, telephone, fax and e-mail;
- costs and agreement about the coverage of the expenses.

4 Conventional round robin tests (C-RRT)

4.1 General

In this case, the “organizer”:

- a) prepares *real sludge* samples, and circulates them to the participating laboratories; or

b) prepares the necessary ingredients to produce *synthetic sludge samples*, and circulate them to the participating laboratories, together with the relevant recipe.

NOTE Synthetic sludge must be obtained by suspending in distilled water various ingredients which could be “inorganic”, e. g. kaolin, quartz sand etc., and “organic”, e. g. alginate, yeast etc.

4.2 Procedure

4.2.1 Preparation of samples by the organizer. The amount of circulated samples has to be defined depending on the type of the parameter to be investigated. In all cases an additional 20 % of samples shall be prepared and held by the organizer; these can be used if any problems are encountered during the exercise.

NOTE To have enough statistical material, a RRT should include at least 3 samples at different concentrations of the investigated parameter.

4.2.2 The sample container should tolerate a slight increase of the inner pressure; possible alternatives are wide neck bottles made from polyethylene or polypropylene (only for inorganic determinations) and metal bottles (when organics are the targets).

NOTE In any case, sample bottles should be half-filled to favour maintaining aerobic conditions.

4.2.3 In case of biodegradable sludge, the dispatcher cools down the samples at + 4 °C, and organizes a sufficient cooling for the transport; this can be done by cooling bags or cooling batteries.

NOTE It has to be decided, case by case, if synthetic sludge has to be used; for biodegradable sludge, proper cooling systems must be adopted: sludge freezing must absolutely be avoided.

4.2.4 The dispatcher organizes the transport by using common international parcel services, and fixes the time of arrival (day and hour); a travel time of 24 h is possible.

4.2.5 The dispatcher sends the following materials and papers to the participants:

- method in the version to be validated;
- homogenized samples;
- modalities of storage before analysis;
- form for results (with remarks);
- period of measurements (not more than 1 week, starting within 24 h after samples received by laboratories);
- deadline for sending back the results.

4.2.6 The dispatcher informs all participants (by phone or e-mail) about the time of arrival.

NOTE It is very important to contact all participants and to organize that all samples will be received from authorized persons.

4.2.7 All analyses will be made in the determined period. Deviations from the method are strictly forbidden. If necessary, deviations have to be reported in the result form, and the results sent back to the organizer before the deadline.

4.3 Statistical calculations

4.3.1 An initial check should be made to confirm a normal distribution of results. If the distribution is not normal then appropriate robust statistics should be employed.

4.3.2 Assuming a normal distribution, the statistical calculations should be carried out according to ISO 5725.

4.3.3 Rejection of outliers should also follow ISO 5725. All Type B (reproducibility) outliers should be eliminated.

4.3.4 All Type C outliers from the mean of one lab (repeatability) should be included.

4.3.5 In addition the elimination of “obvious” outliers by the statistical organizer (Type D) shall be allowed as long as they are fully documented with the reason for elimination.

4.3.6 Each sample shall be analysed in replicate (x times) on y separate days (x and y to be agreed for each individual exercise).

4.3.7 The operator summarizes the statistical work by including:

- number of participants;
- mean value for every tested parameter;
- reproducibility (absolute and relative);
- repeatability (absolute and relative);
- number of outliers.

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5 Modified round robin tests (M-RRT)

5.1 General

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If circulation of real or synthetic sludge samples is not possible due to difficulties associated to their handling, transportation and costs, then round robin tests could be carried out through “*circulation of analysts*”, i. e. the analysts coming from the laboratories participating to the exercise will meet in a common laboratory, close to the place where samples are collected, and will work there with their own apparatuses. The exercise can be repeated at other locations for examination of different sludge samples.

5.2 Procedure

5.2.1 Decision by the organizer of location of the round robin tests exercise, and mailing of the following information to the participants:

- method in the version to be validated;
- list of apparatuses and materials to be provided by each participant, or available at site;
- place, date and duration of measurements (not more than 1 week, starting within 24 h after samples collection or preparation).

5.2.2 Preparation of samples by the organizer (as from C-RRT). The amount of samples has to be defined depending on the type of the parameter to be investigated. In all cases an additional 20 % of samples shall be prepared and held by the organizer; these can be used if any problems are encountered during the exercise.

NOTE To have enough statistical material, a RRT should include at least 3 samples at different concentrations of the investigated parameter.