
Karakterizacija blata – Protokol za organiziranje in vodenje medlaboratorijskih preskušanj metod za kemijske in mikrobiološke analize blata

Characterization of sludges - Protocol for organizing and conducting inter-laboratory tests of methods for chemical and microbiological analysis of sludges

Charakterisierung von Schlämmen - Protokoll zur Durchführung von Ringversuchen für Verfahren zur chemischen, mikrobiologischen und physikalischen Analyse von Schlämmen

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English Version

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conducting inter-laboratory tests of methods for chemical and
microbiological analysis of sludges**

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Durchführung von Ringversuchen für Verfahren zur
chemischen, mikrobiologischen und physikalischen
Analyse von Schlämmen

This Technical Report was approved by CEN on 30 September 2005. It has been drawn up by the Technical Committee CEN/TC 308.

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Foreword

This Technical Report (CEN/TR 15175:2006) has been prepared by Technical Committee CEN/TC 308 "Characterization of sludges", the secretariat of which is held by AFNOR.

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Introduction

For international harmonization it is essential that a fit for purpose interlaboratory test using a range of typical sludges with relevant analyte concentrations be carried out prior to proposing for publication any method as a Technical Report.

At least ten laboratories from three different countries should participate in this test.

Only experienced laboratories routinely carrying out sludge analysis should participate.

The organizer should aim for 12 to 15 laboratories to participate in each test in as many countries as possible.

Efforts are needed to ensure that there is a minimum of intersample variation amongst the circulated samples.

It is recommended that each test includes a range of sludges that is representative of the proposed scope of the method, as a minimum this should include an untreated (raw) sludge, a treated (an aerobically digested) sludge, a presscake and samples of any other sludges relevant to the method being tested. In addition a spiking solution and a calibration check standard will generally be distributed.

If possible, samples should be in the same form as would normally be submitted to a laboratory for analysis.

For sample preparation methods without a final measurement stage (e.g. Aqua regia digestion for metal analysis), see Annex A.

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1 Scope

This Technical Report specifies the requirements for interlaboratory tests to validate chemical and microbiological methods for sludges.

2 Planning protocol

The main test variables to be agreed are:

- a) organising laboratory;
- b) documented method to be tested;
- c) number and nature of the samples;
- d) statistical calculations including rejection of outliers;
- e) deadlines for sending out samples, receipt of results from participants and final test report from the organizer;
- f) spiking protocol to calculate recovery;
- g) analysis of a calibration check standard. It will also allow a check to be made on the comparative accuracy of the calibration standards used by all the participants. This can sometimes explain differences between laboratories).

3 Interlaboratory test protocol issues

3.1 Test organiser and dispatch of samples

It is essential that the bulk material is homogeneous. This should be checked using a suitable low cost parameter (e.g. Cu, Zn).

The organizer will compile a test contact list with:

- a) contact name;
- b) contact full address;
- c) contact phone numbers (named contact and switchboard);
- d) contact fax number;
- e) contact E-mail address.

The organizer will obtain a written statement (see Annex B) from each laboratory agreeing to participate, agreeing to follow the provided method exactly and meet the deadlines for start and completion of analysis and for submission of the results on the appropriate form.

An additional 20 % of samples shall be prepared and held by the organizer at -18°C or less. These can be used if any problems are encountered during the test. For microbiology this may not be appropriate.

3.2 Participating laboratories

Participating laboratories should be laboratories that routinely carry out sludge analysis.

3.3 Statistical calculations

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.

Assuming a normal distribution, the statistical calculations should be carried out according to ISO 5725 by a suitably experienced person for chemical parameters.

Suitable rejection procedures should be used for microbiological parameters.

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

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

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.

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

4 Interlaboratory test procedure

4.1 General

The organizer should procure and prepare the samples. These should contain appropriate concentrations of the analyte.

The minimum sample mass for wet sludges should be 1 kg. For presscakes a minimum mass of 500 g should be sent. For special purposes (e.g. methods that only require a few grams of sample) a smaller amount of sample will be circulated.

All samples will be sent out in polyethylene, polypropylene, stainless steel or aluminium containers that will tolerate a slight increase in pressure. It is not thought necessary, or advisable, to use glass bottles. Each bottle will be clearly labelled with the type of sample, the analysis required and the dates by which the analysis should commenced and completed.

All sample containers will be packed in stout sealed polyethylene / polypropylene insulated containers.

Samples will be sent out at a temperature below [4 °C] with suitable cooling boxes/bags/packs to ensure that the temperature remains below [4°C] for at least 48 h.

4.2 Courier arrangements

The organizer will arrange a suitable courier with a guaranteed 24 h delivery that will accept sewage and other sludge samples.

The sample pick-up time will be agreed with the courier. The delivery time window for each participating laboratory by an authorized person will be agreed between the laboratory and the organising laboratory.

Each participant will be informed of the expected delivery time window and should ensure that suitable sample reception facilities are available.

The courier will be provided with full addresses (including the postcode) and contact details for each participating laboratory.

A simple chain of custody protocol will be implemented for transport and receipt of the samples.

All participating laboratories will upon receipt immediately record the temperature of the samples. They should then store the received samples at a temperature of less than 4 °C until commencement of analysis. This should be within two working days (they have had due warning) of receipt of the samples.

The organizer should ensure that all appropriate sample transport of hazardous biological and chemical material regulations are complied with (e.g. pathogens, dioxin or PCB standard solutions etc.) This will cover within and between country deliveries of the samples.

4.3 Documentation

Documentation to be sent with the samples will be:

- a) another copy of the agreed method;
- b) spiking solution/material if relevant;
- c) instructions for the test;
- d) form for the submission of results;
- e) analysis start date (within four days after dispatch);
- f) analysis completion date (not more than two weeks after the dispatch date);
- g) deadline for return of results (within three weeks of sample dispatch);
- h) full contact details of the organizer to allow for queries and for the return of results.

5 Final report of the test

The organizer should provide a full report of the test to all participants by the agreed deadline. This will include:

- a) a listing of all of the results;
- b) mean value for each tested parameter;
- c) calibration check solution result;
- d) reproducibility (absolute and relative);
- e) repeatability (absolute and relative);
- f) recovery (if relevant);
- g) names and countries of laboratories that returned valid results;
- h) total number of results returned;
- i) temperature of the samples upon receipt at the participating laboratories;
- j) number of outliers from non-rejected laboratories;