EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate F - Health, Consumers & Reference Materials (Geel)
Reference Materials Unit

Geel, 22.6.2018

Call for participants for the validation of methods for the determination of organic parameters in eluates and construction products

CEN/TC351 is currently developing a series of methods for the determination of organic and inorganic substances in construction products (CEN/TS16337-1, CEN/TS16337-2 and CEN/TS16337-3). These methods describe the generation of eluates by leaching and the subsequent determination of organic and inorganic substances. In addition, CEN/TS351024 describes the preparation of extracts from construction products.

We are looking for laboratories interested to participate in the validation studies of these technical specifications for organic substances to convert them into full standards.

The interlaboratory validation for organic substances will start in autumn 2018. Laboratories have the choice to participate in the validation study for

- horizontal dynamic surface leaching according to CEN/TS16337-2
- horizontal up-flow percolation test according to CEN/TS16337-3
- determination of organic substances in eluates according to CEN/TS351026
- determination of content of organic substances according to TS351024

The materials selected for the study are render, sealant, asphalt aggregate and recycled mixed aggregate.

Laboratories have the choice to participate in one or several of these validation studies and can even choose the material they want to participate for. In addition, tests can be performed according to EN 14405 (percolation test for waste material). Detailed validation plans are attached in a separate file.

Participation in these studies is on a free-of-charge basis. We will of course provide personal copies of TS not yet released at the time of the study.

Participants will receive a certificate of participation together with the final validation report and a comparison of their results with the mean and standard deviation of all results after completion of the study. However, as this is a method validation study and not a PT scheme, no z-scores and evaluations like "compliant/non-compliant" can be given.

If you are interested in participation, please return the attached registration form to me (thomas.linsinger@ec.europa.eu or my colleague Silvia Garcia Ruiz silvia.garcia-ruiz@ec.europa.eu or fax: +32 14 590 406)

Please do not hesitate to contact us for any further information

Sincerely yours

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Validation plan for CEN/TS 16637-2 and 16637-3 (horizontal dynamic surface leaching test and horizontal up-flow percolation test) and CEN/TS 351024 and TS 351026 (content and eluate analysis) for organic substances

1 GOAL AND SCOPE

The goal of this validation is the determination of repeatability and reproducibility of CEN/TS 16637-2 and 16637-3 and CEN/TS 351024 and TS 351026 for organic substances. Issues like linearity, robustness etc. have been assessed already and are not part of this study.

2 MATERIALS

The following materials will be used for the validation of CEN/TS 16637-2 and 16637-3

	CEN/TS 16637-2		**CEN/TS 16637-3**	
	Material	**Analytes**	**Material**	**Analytes**
Material 1	Render	Diuron, terbutryn, MIT, BIT, OIT, CMIT	Asphalt aggregate	PAH, mineral oil
Material 2	Sealant	Phtalates	Recycled mixed aggregate	PCB, PAH
Quality control material	Multi substance solution			
Amount per material	3 samples each of material 1-3; 1 bottle of the quality control material			

The following materials ill be used for the validation of CEN/TS 351024 and TS 351026

	CEN/TS 351024		**CEN/TS 351026**	
	Material	**Analytes**	**Leachate of**	**Analytes**
Material 1	Render	Diuron, terbutryn, MIT, BIT, OIT, CMIT	Render	Diuron, terbutryn, MIT, BIT, OIT, CMIT
Material 2	Sealant	Phtalates	Sealant	phtalates
Material 3	Asphalt aggregate	PAH, mineral oil	Asphalt aggregate	PAH, mineral oil
Material 4	Recycled mixed aggregate	PCB, PAH	Recycled mixed aggregate	PCB, PAH
Quality control material	Multi substance solution			
Amount per material	3 samples each of material 1-3; 1 bottle of the quality control material			

3 LABORATORIES

The goal is to include 12 laboratories each for the validation of each standard/TS. Laboratories can but do not have to participate in the validation of all four methods. The requirements for participation are:

- Experience in the application of the respective TS
- Implementation of a quality management system that fulfils the requirements of ISO 17025
  Note: Accreditation itself is not required, but the quality system shall ensure proper training of staff, maintenance and calibration of instruments as well as record keeping. For not-accredited laboratories, self-declaration is sufficient.

Laboratories that outsource either the leaching or the final quantification of the leachate/percolate need to inform JRC-Geel on beforehand about their chosen collaborator. This information will be kept confidential, but will be used to ensure that not the same laboratory performs a number of leachings/quantifications.

Note: The chosen number of laboratories, together with the chosen number of replications (3, see below) should allow the estimation of the repeatability and reproducibility standard deviations with uncertainties between 30 and 45 % (repeatability) and 40-60 % (reproducibility).

4 STUDY SETUP

4.1 CEN/TS16637-2 (horizontal dynamic surface leaching test)

The three samples per material shall be leached according to CEN/TS16637-2 with measurements after 6 h, 24 h, 2 d 6 h, 4 d, 9 d, 36 d and 64 d.

After each leaching step, the leachates shall be analysed under repeatability conditions together with a sample of the quality control material.

It is important that the leaching of the three samples of each material is done simultaneously, i.e. under repeatability conditions. The leaching of samples of the other materials can, but does not have to be performed at the same time. In the same way, quantification of the leachates of each time point of the three samples of each material shall be done in one run. The leachates of the other materials can, but do not have to be analysed at the same time.

The analytes to be quantified are listed in the table in section 2.

4.2 CEN/TS16637-3 (horizontal up-flow percolation test)

The three samples per material shall be leached according to CEN/TS16637-3 with eluates collected after cumulative liquid/solid (L/S) ratios of 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 l/kg.

The various fractions shall be analysed under repeatability conditions together with a sample of the quality control materials. Fractions that are collected within 24 h can be analysed together.

It is important that the leaching of the three samples of each material is done simultaneously, i.e. under repeatability conditions. The leaching of samples of the other materials can, but does not have to be performed at the same time. In the same way, quantification of the fractions of each L/S ratio of the three samples of each material shall be done in one run. The leachates of the other materials can, but do not have to be analysed at the same time.

The analytes to be quantified are listed in the table in section 2.

4.3 CEN/TS 351024 (content analysis)

Three test samples shall be prepared from each material and a test portion shall be taken from each test sample. These test portions shall be extracted and analysed according to CEN/TS 351024 together with a sample of the quality control material.

The measurements shall be performed under repeatability conditions.

It is important that the extraction and quantification of the three test portions of each material is done simultaneously, i.e. under repeatability conditions. The digestion and quantification of samples of the other materials can, but does not have to be performed at the same time. The analytes to be quantified are listed in the table in section 2.

4.4 CEN/TS 351026 (eluate analysis)

The four leachates/eluates shall be analysed under repeatability conditions according to CEN/TS 351026 together with a sample of the quality control material.

It is important that the three replicates of each material are measured simultaneously, i.e. under repeatability conditions. The analyses of the leachates of the other materials can, but does not have to be performed at the same time.

The analytes to be quantified are listed in the table in section 2.

5 REPORTING

The JRC will provide reporting sheet that shall be sent electronically, but also as signed hardcopy (or as pdf-file of a signed hardcopy) to the JRC.

Each participating laboratory will receive a certificate of participation in the study.

6 EVALUATION

The data will be evaluated according to ISO 5725-2, i.e.

a) Checking the data for completeness and technical errors
b) Checking for outlying means and variances using the Grubbs and Cochran procedure as well as Mandel's h and k and outlying data will be eliminated.
c) Calculation of the repeatability (s_r) and reproducibility standard deviation (s_R) using one-way analysis of variance (ANOVA) for each element and time point or faction
d) Whenever possible, consolidation the results of c) into as few numbers as possible; in the ideal case, one single value for s_r and s_R valid for all elements and time points or fractions would be applicable.

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Registration for the method validation studies CEN/TS 16637-2, CEN/TS 16637-3, TS 351024 and TS 351026

We are interested in participating in the interlaboratory validation studies of the following CEN/TS (multiple selection is possible), analysing the materials indicated below.

☐ CEN/TS 16637-2 ☐ Render ☐ Sealant	☐ CEN/TS 16637-3 ☐ Asphalt aggregate ☐ Recycled mixed aggregate
☐ CEN/TS 351026 (all 4 eluates/leachates are to be analysed)	☐ CEN/TS 351024 ☐ Render ☐ Sealant ☐ Asphalt aggregate ☐ Recycled mixed aggregate
☐ I would also like to perform tests according to EN 14405 if the amounts of sample permits this	

Laboratory

Laboratory name:
Contact person:
Contact address:
Address for sample delivery:
Telephone number:
Fax number:
Email:

Experience and quality control

We perform approximately tests per year according to CEN/TS 16637-2, 16637-3, TS 351024 or TS 351026.

☐ We are accredited according to ISO/IEC 17025 under the accreditation number .

☐ We are not accredited according to ISO 17025 and have completed the laboratory QA questionnaire overleaf.

Outsourcing

☐ We do not outsource any part of the measurement process.
☐ We outsource to the following laboratory:

Place, date, name _____

LABORATORY QUALITY ASSURANCE QUESTIONNAIRE

(IGNORE IF ACCREDITED)

Quality System (delete as applicable)

Does your company operate a quality system?	YES/NO
Are your working procedures documented and authorised?	YES/NO
Are analytical methods validated?	YES/NO
Do you have a programme in place to ensure appropriate qualification and training of staff?	YES/NO
Do you have a system for document control in place?	YES/NO
Do you have an instrument maintenance plan in place?	YES/NO
Do you check your working conditions for appropriateness?	YES/NO
Are orders reviewed for acceptability?	YES/NO
Do you have a formal system for dealing with complaints?	YES/NO
Do you carry out internal audits?	YES/NO
Would you allow IRMM to audit your system if required?	YES/NO

Other relevant information: .....