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JRC TECHNICAL REPORT

Precision of test methods to assess the release of organic substances from construction products

Horizontal dynamic surface leaching test CEN/TS 16637-2 horizontal up-flow percolation test CEN/TS 16637-3 content of organic substances CEN/TS 17331 and analysis of organic substances in eluates CEN/TS 17332

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Executive summary

The precision of the methods developed by the European Committee for Standardization (CEN) to assess the release of dangerous organic substances from construction products was evaluated as part of the validation of the methods aiming to convert them in EU standards. This evaluation of precision was done by an interlaboratory comparison organised by the Joint Research Centre (JRC) of the European Commission, in support of the Regulation 305/2011/EU (Construction Products Regulation). The present study focused on organic substances since inorganic substances were studied in previous work.

The objective of this work was to evaluate for organic substances the precision (repeatability and reproducibility) of the methods developed by CEN Technical Committee 351 on dynamic surface leaching and up-flow percolation procedures from construction products, analysis of leachates/eluates and content analysis. This was done by comparing the results obtained by different laboratories when they analysed samples obtained from the same materials using the CEN methods under validation.

The materials and analytes were proposed by CEN Technical Committee 351 to cover a reasonable range of products and substances at measurable levels. Validation plans were drawn up by the JRC in agreement with CEN/TC351. The tested substances were biocides, phthalates, mineral oil, polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs); and the selected construction products were render, sealant, asphalt aggregate and recycled aggregate. The methods assessed were horizontal dynamic surface leaching test CEN/TS 16637-2; horizontal up-flow percolation test CEN/TS 16637-3; content of organic substances – methods for extraction and analysis CEN/TS 17331; and analysis of organic substances in eluates CEN/TS 17332. Specific methods were used for the determination of biocides (WI 351035) and PAHs (WI 351034). Raw materials were obtained from industrial providers and processed to obtain laboratory samples at the JRC premises in Geel, Belgium.

This interlaboratory test was open to expert laboratories from all Member States. Despite all efforts to recruit a higher number of participants, eventually 12 laboratories registered to the study and reported results; between 5 and 7 laboratories analysed each of the product following the CEN methods mentioned above.

Precision of the reported results was evaluated according to ISO 5725-2. The obtained values for repeatability and reproducibility are shown in the annexes of this JRC report, together with the content levels, for each construction product and analyte within this study. For the dynamic surface leaching test CEN/TS 16637-2, relative repeatability standard deviation (RSD_r) was 6 % and relative reproducibility standard deviations (RSD_R) was 54 % (median values). Because of limited stability of biocides in water, it is recommended to assess their release at leaching times shorter than 64 days and to store the leachates in dark glass bottles at 4ºC. For the up-flow percolation test CEN/TS 16637-3, RSD_r of 20 % and RSD_R of 70 % were obtained as median values. For the analysis of eluates, RSD_r were 2-32 % and RSD_R were 23-51 %. And for content analysis, the values for RSD_r are 6-9 % and for RSD_R 27-63%.

Due to the limited number of participants, any conclusion or recommendation must be made with the utmost care. However, despite the limited number of participants which might affect the robustness of this study, the results obtained for organic substances are consistent with the ones obtained for inorganic substances. Therefore it seems reasonable to incorporate the RSD values for organic substances to the standard methods.

1 Introduction

The precision of the methods developed by the European Committee for Standardization (CEN) to assess the release of dangerous organic substances from construction products was evaluated as part of the validation of the methods aiming to convert them in EU standards. This evaluation of precision was done by an interlaboratory comparison organised by the Joint Research Centre (JRC) of the European Commission. This work was done in support of the Regulation 305/2011/EU (Construction Products Regulation), under the Administrative Arrangement nº 34205 between DG GROW and JRC. This study focused on organic substances since inorganic substances were previously studied in previous work ([1]).

The CEN Technical Committee 351 (TC) Construction Products - Assessment of release of dangerous substances has the mandate to develop leaching procedures and procedures to analyse the leachates for organic and inorganic substances taking into account the intended conditions of use of the product. It is split into several working groups (WGs); among them, WG1 focuses on "Release from construction products into soil, ground water and surface water" and it has developed horizontal dynamic surface leaching and up-flow percolation procedures; and WG5 focuses on "Content and Eluate analysis" and it has developed horizontal standardized procedures for the analysis of leachates/eluates and content analysis.

([1]) García-Ruiz, S., Linsinger, T., Cordeiro, F., Conneely, P., Emteborg, H., Held, A., *Interlaboratory comparison to evaluate the precision of measurement methods for the assessment of the release of inorganic substances from construction products*, EUR 30071 EN, Publications Office of the European Union, Luxembourg, 2020, ISBN 978-92-76-10226-7, doi:10.2760/288988, JRC119719

2 Scope

The present study aims to evaluate the precision (repeatability and reproducibility) of methods developed by CEN/TC 351 on leaching, percolation, content and eluate analysis to assess the release of organic hazardous substances from construction products. Issues like limits of detection, linearity, robustness etc. were already assessed ([2-4]) and are not part of this study.

The methods assessed are those developed by CEN/TC 351 Working Group (WG) 1 "Release from construction products into soil, ground water and surface water":

— CEN/TS 16637-2:2014 Horizontal dynamic surface leaching test([5])

— CEN/TS 16637-3:2016 Horizontal up-flow percolation test ([6])

and the methods developed by CEN/TC351 WG5 "Content and eluate analysis in construction products":

— CEN/TS 17331:2019 Content of organic substances – Methods for extraction and analysis([7])

— CEN/TS 17332:2019 Analysis of organic substances in eluates ([8])

— WI 351034:2019Determination of the content of polycyclic aromatic hydrocarbons (PAHs) and of benzene, toluene, ethylbenzene and xylene (BTEX) — Gas-chromatographic method with mass spectrometric detection ([9])

— WI 351035:2019Determination of biocides using LC-MS/MS ([10])

To encourage laboratories to participate in the study, in the cases of CEN/TS 16637-2 and 16637-3 methods reporting was limited to the cumulative released quantities after 64 days (CEN/TS 16637-2) and liquid to solid ratios 2 and 10 (CEN/TS 16637-3), respectively, since those are the regulatory points.

The construction products and organic substances were selected by CEN/TC 351 based on previous validation work (4). The aim was to have leachates with measurable concentration levels and to cover a range of different products and analytes. The tested substances were biocides, phthalates, mineral oil, PAHs and PCBs; and the selected construction products were render, sealant, asphalt aggregate and recycled aggregate (Table 1).

Repeatability and reproducibility of the methods were evaluated according to ISO 5725-2 ([11]).

([2]) *Robustness validation of TS-2 and TS-3 developed by CEN/TC351/WG1 to assess release from products to soil, surface water and groundwater. Final report*, March 2013

([3]) *Additional robustness testing on TS-3 (CEN/TC351/WG1). Final report*, July 2014 (Rev. 2015)

([4]) H. Van De Weghe, M. Van Deun, D. Bertels, J. Lievens, M. Schroeven, G. Vanermen, *CEN/TC 351/WG 5 – Construction products Robustness validation of draft methods for eluate and content analysis of organic substances,* March 2018

([5]) CEN/TS 16637-2:2014 *Construction products – Assessment of release of dangerous substances – Part 2: Horizontal dynamic surface leaching test*

([6]) CEN/TS 16637-3:2016 *Construction products – Assessment of release of dangerous substances – Part 3: Horizontal up-flow percolation test*

([7]) CEN/TS 17331:2019 *Construction products: Assessment of release of dangerous substances - Content of organic substances – Methods for extraction and analysis*

([8]) CEN/TS 17332:2019 *Construction products – Assessment of release of dangerous substances – Analysis of organic substances in eluates*

([9]) WI 351034:2019*Construction products: Assessment of release of dangerous substances* - Determination of the content of polycyclic aromatic hydrocarbons (PAH) and of benzene, toluene, ethylbenzene and xylene (BTEX) — Gas-chromatographic method with mass spectrometric detection *(ICP-OES)*

([10]) WI 351035:2019*Construction products: Assessment of release of dangerous substances* - Determination of biocides using LC-MS/MS

([11]) ISO 5725-2:1994 *Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

Table 1. Organic substances and materials selected for the interlaboratory comparison.

Biocides	Analytes	Diuron, terbutryn, methylisothiazolinone(MIT), benzisothiazolinone(BIT), octylisothiazolinone(OIT), carbendazim
	Material (s)	Organic render
Phthalates	Analytes	Dimethylphthalate (DMP), diethylphthalate (DEP), di-(2-methylpropyl)phthalate (DIBP), dibutylphthalate (DBP), butylbenzylphthalate (BBP), di-(2-ethylhexyl)phthalate(DEHP), dicyclohexylphthalate (DCHP), dioctylphthalate (DOP), di-isononylphthalate (DiNP)
	Material (s)	Sealant
Mineral oil	Analytes	Sum of n-alkanes C10-C40
	Material (s)	Asphalt aggregate
Polycyclic aromatic hydrocarbons (PAHs)	Analytes	Naphthalene,acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3,c,d)pyrene, dibenzo(a,h)anthracene, benzo(g,h,i)perylene
	Material (s)	Asphalt aggregate, recycled aggregate
Polychlorinated biphenyls (PCBs)	Analytes[(1)]	PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153 and PCB-180
	Material (s)	Recycled aggregate

([1]) PCBs are a substance class of 209 different substances. For simplicity, they have been assigned numbers and are generally referred by their number rather than by their chemical name

3 Set up of the study

3.1 Time frame

Validation plans, materials and analytes were distributed to the members WG1 and WG5 of CEN/TC351 on 4 May 2018 and were agreed at the meetings of WG1 and WG5 of CEN/TC 351 on 19/20 June 2018.

The materials were supplied by between June 2018 and January 2019. The processing of the materials (sieving, sub-sampling, preparation of eluates) was finished by April 2019.

Registration was open from June 2018 to March 2019 in an effort to ensure a sufficient number of registrations.

Samples were dispatched on 5 April 2019. The deadline for reporting results was originally set to 15 July 2019 but results were accepted until December 2019 in order to have as many datasets as possible for the evaluation of results.

A preliminary evaluation was sent to all laboratories on 3 December 2019 with the request for comment/correction.

The draft report was released on 2 March 2020 and discussed during CEN/TC 351/WG1 and WG5 meeting on 18 March 2020.

3.2 Validation plan

The validation plan (Annex 1) was developed and discussed at the meetings of working groups 1 and 5 of CEN/TC 351.

The evaluation of the precision of the methods to assess the release of dangerous organic substances from construction products was done by an interlaboratory comparison (also known as ring trial, round robin test, etc.) according to ISO 5725-2 ([11]). This comprises the organization, performance and evaluation of measurements or tests on the same or similar items by two or more laboratories in accordance with predetermined conditions ([12]). The test items (laboratory samples) were obtained from the same field sample and were sent to the participant laboratories to be tested following strictly the methods under validation.

The construction products and analytes to be included in the interlaboratory comparison were selected considering:

- Analytes should be leachable from the products in a reasonable concentration range to be able to evaluate the repeatability and reproducibility.
- Different kinds of products should be tested

Finding products releasing significant levels of organic substances in deionised water (leachant prescribed in CEN/TS 16637-2 and CEN/TS 16637-3 methods) posed a considerable challenge due to the relatively low levels of these substances in construction products and low solubility of organic substances in water. The selected analytes and construction products for the different methods under study are listed in Table 1 and Table 2.

Participant laboratories were asked to perform the analyses under repeatability conditions, i.e. the leaching of three samples from each material was done simultaneously and the quantification of the leachates of each time point of the three samples of each material was performed in one run. Multi-substance standard solutions were also distributed for quality control.

Laboratories were also offered to perform tests according to EN 14405 (up-flow percolation test for the characterisation of waste) to compare with results obtained by CEN/TS 16637-3. Although some participants expressed their interest, eventually no results were reported.

[12] ISO/IEC 17043:2010*Conformity assessment – General requirements for proficiency testing*

Table 2. Construction products and substances selected for the methods under study.

CEN/TS 16637-2 (dynamic surface leaching)	CEN/TS 16637-3 (up-flow percolation)	CEN/TS 17331 ([3]) (content analysis)	CEN/TS 17332 (eluate analysis)
Biocides in render ([1])			Biocides in render
Phthalates in sealant			Phthalates in sealant
	PAHs ([2]) and mineral oil in asphalt aggregate	PAHs ([2]), mineral oil in asphalt aggregate	PAHs ([2]), mineral oil in asphalt aggregate
	PAHs ([2]) and PCBs in recycled aggregate	PAHs ([2]) and PCBs in recycled aggregate	PAHs ([2]) and PCBs in recycled aggregate

([1]) WI 351035 for determination of biocides
([2]) WI 351034 for determination of polycyclic aromatic hydrocarbons (PAHs)
([3]) Analysis of biocides in render and phthalates in render was not performed as the methods were still under discussion at the time of the study

3.3 Participants

This interlaboratory test was open to expert laboratories from all Member States on voluntary and free basis. The requirements for participation were:

- Experience in the application of the respective methods
- Implementation of a quality management system fulfilling the requirements of ISO/IEC 17025[13].

Laboratories had the choice to participate in one or several methods; for each method they had also the choice to analyse one or several materials. The information provided by the participants and the link between them and their results is treated as confidential.

A registration form (Annex 2) was drawn up to allow laboratories to express their interest in participating in the study. The form was distributed on 15 July 2018 by the CEN/TC 351 expert members to their various contacts. Additionally the group of Notified Bodies on Dangerous Substances and Sustainability was invited to participate in June 2018. On 20 July 2018, the JRC-Geel sent the invitation all laboratories that had collaborated in the production of certified reference materials for the analytes concerned. As the number of registrations was low, CEN/TC351 published a reminder to WG1 and WG5 on 17. August 2018 and JRC Geel invited again all laboratories that had participated in the production of reference materials for similar analytes on 18 September 2019.

Despite all the efforts, the aim of at least 12 laboratories for each method and material was not reached. Therefore the workload was reduced to encourage laboratories to participate in the study. In the cases of CEN/TS 16637-2 and 16637-3 methods reporting was limited to the cumulative released quantities after 64 days (CEN/TS 16637-2) and liquid to solid ratios 2 and 10 (CEN/TS 16637-3), respectively, since those are the regulatory points. In addition, participants were offered a financial compensation if they were interested to also perform a variant of the study (addition of stabilisers as used in the USA).

Invitations were sent again to the above mentioned groups in November 2018 and the start of the study was postponed until a minimum number of 6 laboratories for each method was reached. This situation is not ideal since the small number of participants might complicate the extraction of conclusions from the obtained results. Eventually the total number of registered laboratories was 12, but for several of the materials fewer than 6 laboratories registered. As the efforts made so far had not led to more participants, it was regarded as unlikely that any action would result in a significantly higher number of participants. This left the choice between cancelling the study altogether or to proceed with the limited number of participants. It was decided to proceed with the study, being well aware that the low number of participants would limit the conclusions that could be drawn from the results.

[13]ISO/IEC 17025:2017 *General requirements for the competence of testing and calibration laboratories*

Table 3. Number of participants in the interlaboratory comparison per method and material.

Method ([1]) ([2])	Analytes / Material	No. participants
CEN/TS 16637-2(dynamic surface leaching)	Biocides in render	7
	Phthalates in sealant	4
CEN/TS 16637-3 (up-flow percolation)	PAHs and mineral oil in asphalt aggregate	6
	PAHs and PCBs in recycled aggregate	6
CEN/TS 17331 (content analysis)	PAHs, mineral oil in asphalt aggregate	7
	PAHs and PCBs in recycled aggregate	6
CEN/TS 17332 (eluate analysis)	Biocides in render	3
	Phthalates in sealant	1
	PAHs and mineral oil in asphalt aggregate	5
	PAHs and PCBs in recycled aggregate	5

([1]) WI 351035 for determination of biocides
([2]) WI 351034 for determination of polycyclic aromatic hydrocarbons (PAHs)

3.4 Instructions to participants

Each participant received the following materials:

For the horizontal dynamic surface leaching test according to CEN/TS 16637-2

— 3 blocks of extruded polystyrene(XPS) coated with organic render (Material 1)

— 3 tiles coated with sealant (Material 2)

For the horizontal up-flow percolation test according to CEN TS 16637-3

— 1-3 drums filled with asphalt aggregate (Material 3)

— 1-3 drums filled with recycled aggregate (Material 4)

For the content analysis according to CEN/TS 17331

— 1 jar containing approximately 50 g ground asphalt aggregate (Material 5)

— 1 jar containing approximately 50 g ground recycled aggregate (Material 6)

For the eluate analysis according to CEN/TS 17332

— 3 bottles of approximately 25 ml each of water eluate of render (Material 7)

— 3 bottles of approximately 1 l each of water eluate of sealant (Material 8)

— 3 bottles of approximately 1 l each of water eluate of asphalt aggregate (Material 9)

— 3 bottles of approximately 1 l each of water eluate of recycled aggregate (Material 10)

Quality control materials

- 1 vial of diuron, terbutryn and MIT in methanol, concentration approx. 250 mg/l to be used as quality control material (QC biocides)
- 1 vial of phthalate mix in methanol, concentration approx. 1-10 g/l to be used as quality control material (QC phthalates)
- 1 vial of polycyclic aromatic hydrocarbons in toluene, mass fractions around 3 mg/kg to be used as quality control material (QC PAHs)
- 1 vial of mineral oil in heptane, concentration approx. 10 g/l hydrocarbons, to be used as quality control material (QC mineral oil)
- 1 vial of polychlorinated biphenyls in isooctane, concentrations 5-25 mg/kg, to be used as quality control material (QC PCBs).

Detailed instructions were given to participants in the "Sample accompanying letter" (Annex 4).

To encourage laboratories to participate by reducing the costs and workload of the testing programme, it was proposed to report only regulatory points in the following cases:

- cumulative amount after 64 days for the horizontal dynamic surface leaching test (CEN/TS 16637-2)
- cumulative amounts for liquid to solid ratios of 2 l/kg and 10 l/kg for the horizontal up-flow percolation test (CEN/TS 16637-2).

To achieve this, fractions of leachates / eluates were combined and analysed following detailed instructions.

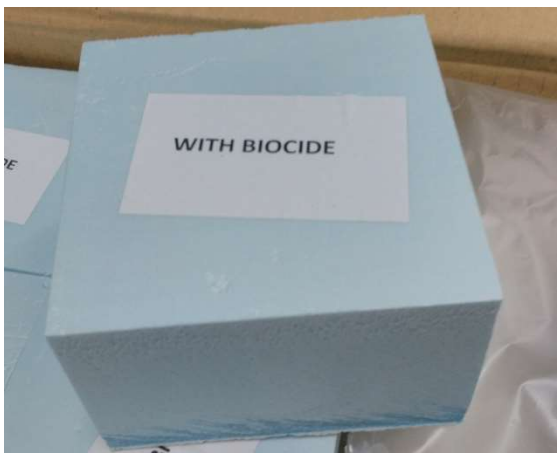
Reporting was done electronically using provided Excel sheets and signed pdf copy to comply with the requirements of ensuring the integrity of electronic data.

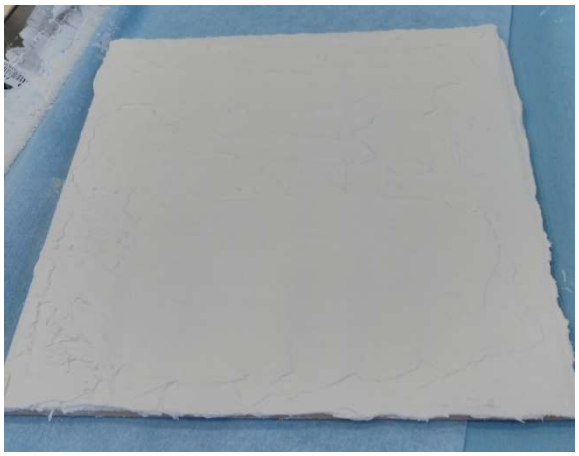
4 Test items

4.1 Preparation of test items

Raw materials were obtained from industrial providers and further processed (if necessary) at the JRC premises in Geel. These raw materials are shown in Figure 1.

Figure 1. Starting materials for the methods CEN/TS 16637-2 and 16637-3. From top to down, and from left to right: (a) organic render, (b) sealant, (c) asphalt aggregate and (d) recycled aggregate





4.1.1 Materials for CEN/TS 16637-2

(a) Organic render

The render test items were prepared by Dr. Robert-Murjahn Institute GmbH (Ober-Ramstadt, Germany). They consisted in extruded polystyrene blocks (10 x 10 cm), of which one surface was coated with an organic render with a known amount of biocides (approx. 730 mg/m^2). Tests at the producer gave an uncertainty on the mass of 8 %, which is sufficient for the interlaboratory test. The test items were stored at 4 ºC.

(b) Sealant

12 cartridges of acrylate sealant containing phthalates, MIT, BIT, CIT were provided by Henkel AG & Co. KGaA (Düsseldorf, Germany).

To prepare the test items, the acrylate sealant was spread on ceramic tiles (20 x 20 cm) covering the complete surface on one side with a thickness of approximately 0.5 mm.

4.1.2 Materials for CEN/TS 16637-3

(c) Asphalt aggregate

Thestarting material was picked up by the JRC at the premises of the Recycling Kombinatie REKO B.V. (Rotterdam, The Netherlands)as a granular mix with particles of different sizes (see image in Figure 1; card added for size comparison).The material was sieved at16mm and fraction > 16 mm discarded. Then the bulk material was manually mixed using a shovel. Sample division was done by increment division according to ISO 11648-2 ([14]). The material was spread on a flat plate in the form of a rectangle of 4-5 cm thickness and arranged in 20 equal parts; each part was collected in a separate polyethylene plastic barrel using a shovel; this was repeated as necessary to fill each barrel in up to 4 kg. The test items (plastic barrels containing 4 kg of aggregate with particles < 16 mm) were stored at room temperature.

(a) Recycled aggregate

The material was picked up by the JRC at the premises of Theo Pouw bv (Utrecht, The Netherlands) as granular solid mixture (see image in Figure 1; pen added for size comparison). The material was also sieved at 16 mm (fraction > 16 mm discarded) and manually mixed using a shovel. Sample division was done by increment division according to ISO 11648-2 ([14]). The material was spread on a flat plate in the form of a rectangle of 4-5 cm thickness and arranged in 20 equal parts; each part was collected in a separate polyethylene plastic barrel using a shovel; this was repeated as necessary to fill each barrel in up to 4 kg. The test items (plastic barrels containing 4 kg of aggregate with particles < 16 mm) were stored at room temperature.

4.1.3 Ground materials for CEN/TS 17331

The asphalt and recycled aggregates prepared for CEN/TS 16637-3 were further sieved at 1 mm using stainless steel sieves and the fraction < 1 mm was collected. The powder was stored in glass bottles at 4 ºC.

4.1.4 Eluates for CEN/TS 17332

Eluates from render,sealant, asphalt aggregate and recycled aggregatewere prepared at the JRC-Geel by surface leaching from XPS blocks, sealant-coated tiles and solid aggregates. Glass or stainless steel containers were used as leaching tanks and deionized water as leachant (in a liquid to surface ratio of 80 l/m2 for render and sealant; and in a liquid to solid ratio of 2 for the asphalt and recycled aggregates) for a single leaching step of 2 days from the start of the test. The water was stirred regularly (mechanical stirring). The solid fraction was discarded or decanted and portions of liquid fractions between 30 ml and 1l of leachates and eluates were bottled in dark glass bottles with PTFE coated screw caps. The bottles were rinsed with detergent and GC-grade hexane prior to their use and plastic material was avoided. Eluates were kept at 4 ºC.

4.2 Determination of moisture content

The moisture content of materials was provided to the participant laboratories. The determination consisted of drying the sample in the oven at (105 ± 3) °C until stable weight. For the crushed materials 1 kg of solid was used and for the ground materials 1 g, respectively.

([14]) ISO 11648-2:2001 *Statistical aspects of sampling from bulk materials – Part 2: Sampling of particulate materials*

5 Evaluation of results

The precision of the collaborative interlaboratory tests under repeatability and reproducibility conditions was evaluated according to ISO 5725-2 ([11]). Repeatability conditions are defined ([15]) as those where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time. Reproducibility conditions are those where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment ([15]). The trueness of the methods was not evaluated as it was not in the scope of this study.

Data treatment was performed using ProLab software (QuoData GmbH, Dresden, Germany). Values reported as lower than limit of detection (LOD) were not included in the data treatment. Statistical outliers were identified by Cochran test (excessive variance within laboratory) and Grubbs test (laboratory mean different from overall mean) and excluded from the data set. The reported results were represented in graphs and cross-checked with participant laboratories to identify potential technical errors. However outlier identification was difficult due to the small number of participants. The graphical consistency technique described in ISO 5725-2 (Mandel's h and Mandel's k graphs) were applied to identify consistent deviations or consistently high standard deviation of a laboratory over all analytes of a given material. Mean and standard deviation under repeatability/ reproducibility conditions were calculated by performing one-way analysis of variance (ANOVA test) for each analyte and fraction, using all retained values (three replicate measurements of each laboratory in most cases). Tables in Annex 7 show the obtained results (mean, standard deviation and relative standard deviation under repeatability/reproducibility conditions) for each method, material, and analyte.

Although the number of laboratories was 6-7 for the dynamic surface leaching test, up-flow percolation test and content analysis, in some cases the number of participants is lower (see tables of Annex 7). This is because some laboratories reported values lower than limit of detection (LOD). Values lower than LOD were not considered in the data set. The number of datasets was further reduced by the exclusion of outliers and consistently deviating results identified using the graphic consistency technique. The number of values in the dataset has to be considered when extrapolating or extracting conclusions from the results.

For the sake of simplicity, in the following discussion the relative repeatability standard deviation (RSD_r) and relative reproducibility standard deviation (RSD_R) will be referred as repeatability (RSD_r) and reproducibility (RSD_R).

5.1 CEN/TS 16637-2 Horizontal dynamic surface leaching test

This leaching procedure is applied to monolithic, plate- or sheet-like construction products and it consists of eight steps/fractions with different duration before the renewal of the leachant (6 hours, 1 day, 2 days and 6 hours, 4 days, 9 days, 16 days, 36 days and 64 days from the start of the test). In the present interlaboratory comparison for organic substances, only cumulative results for the regulatory point after 64 days were assessed.

The selected construction products and analytes to assess the precision of CEN/TS 16637-2 method were biocides in organic render and phthalates in sealant.

5.1.1 Biocides in organic render

The stability of biocides (particularly isothiazolinones) in water is limited, and degradation might be an issue to perform CEN/TS 16637-2 test at the regulatory point after 64 days. Regarding the stability of isothiazolinones in water, it is reported in the literature ([16]) that biodegradation is not so predominant in tap water as in surface water and isothiazolinones in tap water samples stored at 4 ºC are stable for at least 3 weeks. Additionally carbendazim is stable in water at 4 ºC for 28 days ([17]) and its half-life is two months ([18]).

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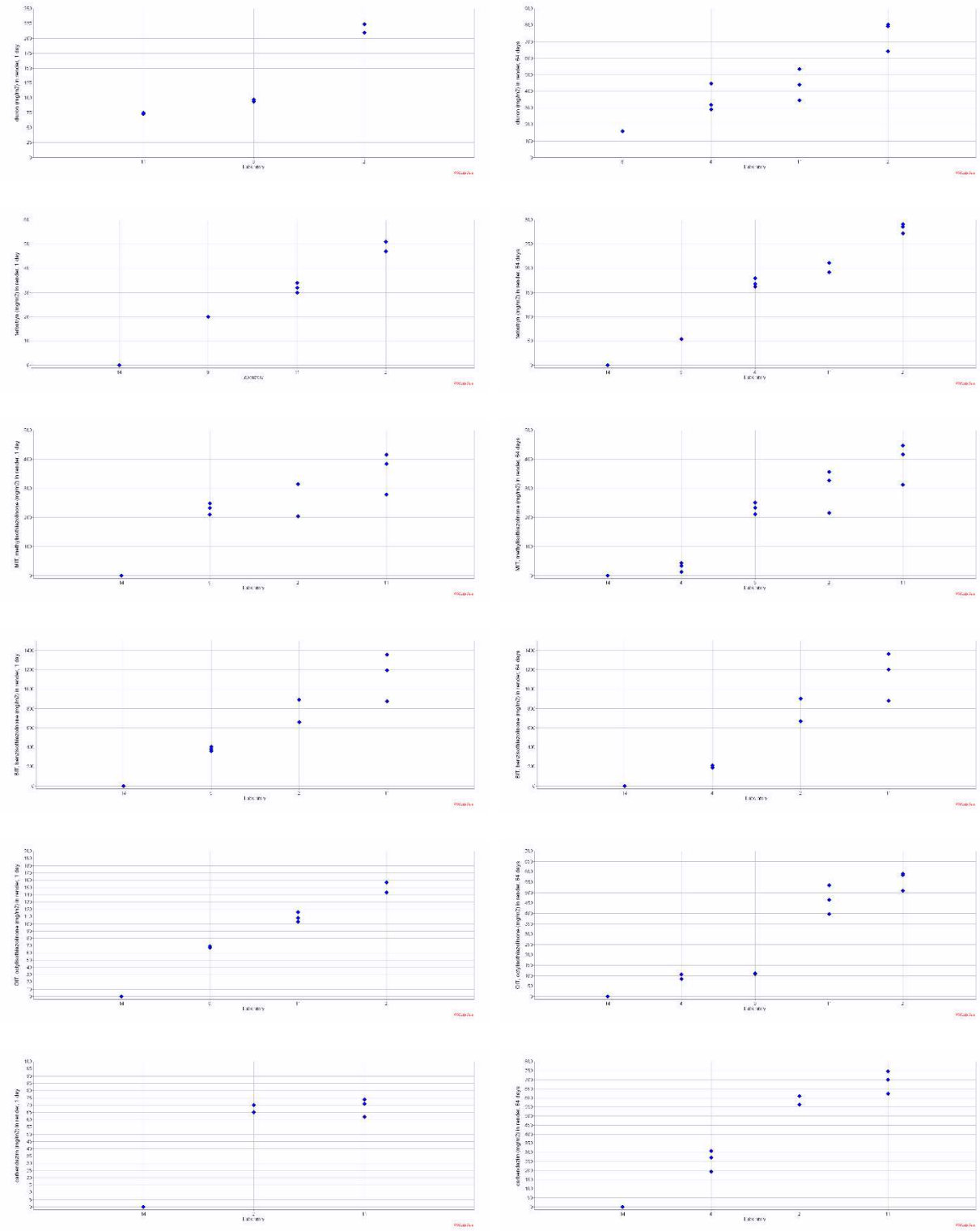
[15] ISO 5725-1:1994 *Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions*

[16] Rafoth, A., Gabriel, S., Sacher, F., Brauch, H.-J., Analysis of isothiazolinones in environmental waters by gas chromatography–mass spectrometry, Journal of Chromatography A, Vol. 1164, 2007, pp 74-81

[17] U.S. Geological Survey:https://pubs.usgs.gov/tm/05/b11/appendix/pdf/s11_esipos_all_pest_deg_18Dec.pdf

[18] *Degradation Study of Benomyl and Carbendazim in Water by Liquid Chromatography and Multivariate Curve Resolution Methods*, Mallat. E., Barceló, D., Tauler, R., Chromatographia Vol.46, No.7/8, October 1997

Figure 2. Reported results from different laboratories for cumulative released quantities of biocides from render after 1 day and 64 daysusing CEN/TS 16637-2 and WI 351035 methods. From top to down: diuron, terbutryn, MIT, BIT, OIT and carbendazim; results after 1 day on the left and after 64 days on the right



Two possibilities were considered to overcome the issue of limited stability: (i) shorter leaching times, and (ii) stabilisation of the leachates.

Regarding leaching times, laboratories were requested to performe their tests at 1 day in addition to the regulatory point of 64 days. Those substances with low stability should not be evaluated in this study at 64 days but at shorter times.

Regarding the leachates, it was recommended to store them at 4 ºC; the addition of preservatives to the leachates was also considered. However, the aim of the study was to test the performance of the technical specifications as they are written without giving additional instructions that could create a bias in comparison with any routine testing laboratory using the same method (otherwise clear instructions regarding stabilisation of the leachates should be given in the procedures to allow comparison of results from different laboratories). The technical specifications currently do not prescribe the use of preservatives, so no recommendation for preservatives was made to the participant laboratories.

Reported results for cumulative released quantities of biocides from render after 1 day and 64 days using CEN/TS 16637-2 method are shown in Figure 2. Seven laboratories reported results; however results from L8 are missing in these charts –but not in the evaluation- due to late reporting. Indeed it was observed that some of the biocides (MIT, BIT and possibly OIT) were not stable in the leachates over 64 days. Although no instruction on the use of preservatives was given, laboratory #2 (L2) used sodium azide 0,1 % as preservative and their results can be compared with others not using any preservative. Values from L2 seem higher than the ones from other laboratories suggesting the effect of the preservative used, but even there MIT and BIT values dropped significantly at 64 days.

No statistical outlier was found after Cochran and Grubbs tests. However visual examination of results in Figure 2 shows suspect values from L14 which are systematically low for all biocides in this test item; in addition this laboratory did not report results for the quality control material that would give more information on their performance. Considering that the lower the number of values, the higher the dispersion of those values and the more difficult for statistical tests to identify outliers, the evaluation of results was done both: (a) considering results after elimination of statistical outliers only; and (b) considering results after elimination of statistiscal outliers and suspect values.

The obtained repeatability (RSD_r) and reproducibility (RSD_R) are shown in Annex 7 and summarized in the graphs below (**Error! Not a valid bookmark self-reference.**3). The obtained values for the released quantities after 1 day are between 5-25 % for RSD_r and 70-85 % for RSD_R. Considering results after elimination of L14 there is an improvement for reproducibility, in the range 35-75 % for RSD_R. Results at 64 days for MIT, BIT, OIT and carbendazim were not included in the evaluation because of the concerns on their stability. Table 4 shows the median values.

Figure 3. Repeatability (RSD_r, left) and reproducibility (RSD_R, right) for cumulative biocide released quantities from render using CEN/TS 16637-2and WI 351035methods. Green: Cumulative release after 1 day after removal of statistical outliers. Light blue: Cumulative release after 64 d after removal of statistical outliers. Red: Cumulative release after 1 d after removal of statistical outliers and L14. Dark Blue: Green: Cumulative release after 64 d after removal of statistical outliers and L14. No data are shown for MIT, BIT, OIT and carbendazim for 64 d because of the instability of the substances.

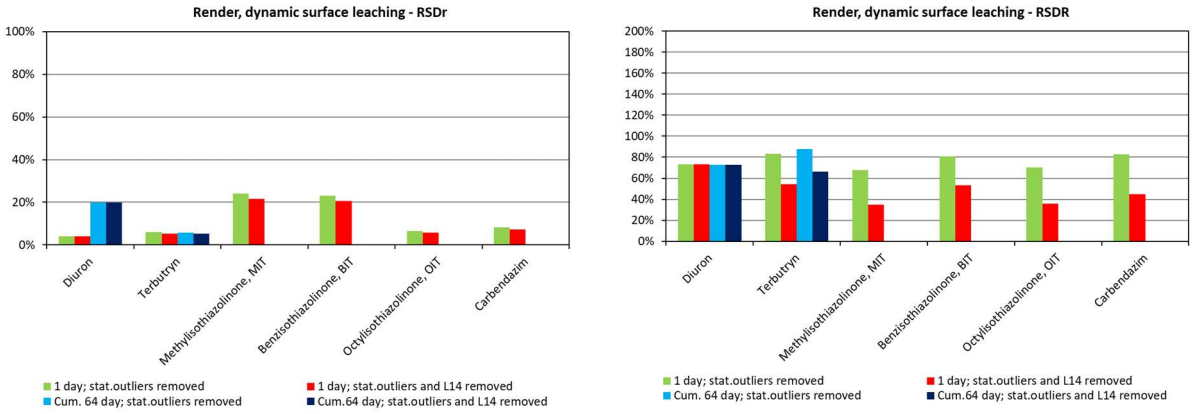


Table 4. Relative repeatability standard deviation RSDr and relative reproducibility standard deviation RSD_R for cumulative biocide released quantities from render using CEN/TS 16637-2 and WI 351035 methods (median values).

	Cumulative 1 day	Cumulative 64 days
RSD_r	6 %	13 %
RSD_R	49 %	70 %

From the results obtained and questions received from participant laboratories, two main recommendationsare extracted:

1) shorter leaching times should be established to assess the release of biocides with limited stability

2) A clear recommendation should be made in the method for biocides on how to preserve the leachates (and the use or not of preservatives) to ensure that all laboratories follow the same procedure and their results are comparable.

Following discussion with CEN/TC 351/WG5, the following recommendations were agreed:

- Limit the duration of the test for biocides to 16 days for reasons of limited stability of biocides
- Use light-protected vessels for the DSLT
- Eluates should be analysed directly after water change or otherwise stored in darkness and kept cool (≤ 4 °C) prior to analysis.The use of NaN_3 as option to prevent microbiological transformation of the biocides is discouraged due to worker safety issues
- Recommendations given in ISO 5667-3 ([19]) may be considered.

5.1.2 Phthalates in sealant

It was not possible to assess the precision of results from released quantities of phthalates from sealant using CEN/TS 16637-2 method because from the 4 laboratories that reported results, only 2 reported values higher than their LOD. While this shows that the sealant is effective in generating an impenetrable surface to water, it precludes an evaluation of results.

[19] *ISO 5667-3:2012 Water quality – Sampling – Part 3: Preservation and handling of water samples*

5.2 CEN/TS 16637-3 Horizontal up-flow percolation test

This percolation procedure is applied to granular construction products and it consists of seven steps/fractions with eluates collected at different liquid to solid ratios (L/S = 0.1, 0.1, 0.3, 0.5, 1, 3 and 5). In this interlaboratory comparison for organic substances, only cumulative results for the regulatory points L/S = 2 and 10 were evaluated.

5.2.1 Mineral oil in asphalt aggregate

It was not possible to assess the precision of results from released quantities of mineral oil from asphalt aggregate using CEN/TS 16637-2 method because of the four laboratories that reported results, only two of them reported values higher than their LOD.

5.2.2 PAHs in asphalt and recycled aggregates

Six laboratories reported results for each asphalt and recycled aggregate. Similarly to Section 5.1.1, after identification of statistical outliers by Cochran and Grubbs tests, there were still suspect values from L12 (asphalt aggregate) and L3+L14 (recycled aggregate). Therefore two scenarios were evaluated: (a) considering results after elimination of statistical outliers only; and (b) considering results after elimination of statistiscal outliers and suspect values. In addition the quantities in the eluates were compared to the LODs to discard the values that were below the limit of quantification (LOQ) and might bias the dispersion of the results.

Figure 4 shows the results from the second scenario.The obtained values for repeatability $RSD_r$for asphalt aggregate were between 16 % - 91 % (L/S =2) and 6 % - 18 % (L/S = 10); for recycled aggregate were 12 %-45 % (L/S =2) and 6 % - 56 % (L/S = 10). Values for reproducibility$RSD_R$for asphalt aggregate were between 42 % - 132 % (L/S =2) and 23 % - 110 % (L/S = 10); for recycled aggregate were 39 %-102 % (L/S =2) and 25 % - 102 % (L/S = 10). Table 5 shows a summary of median values.

Figure 2. Repeatability (RSD_r, left) and reproducibility (RSD_R, right) for cumulative PAHs released quantities from asphalt and recycled aggregate using CEN/TS 16637-3and WI 351034 methods. Green: asphalt aggregate, cumulative L/S =2. Light blue: asphalt aggregate, cumulative L/S =10. Red: recycled aggregate, cumulative L/S =2. Dark blue: recycled aggregate, cumulative L/S =10.

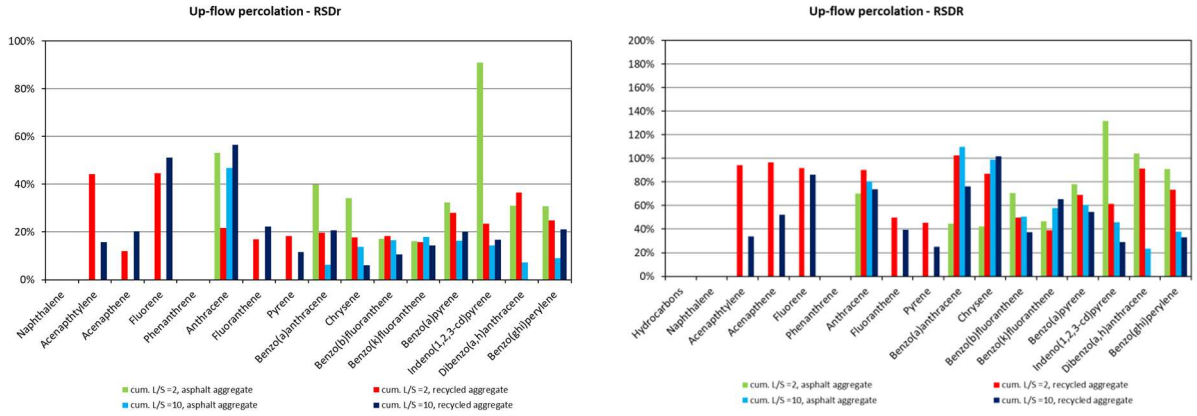


Table 5. Relative repeatability standard deviation RSD_r and relative reproducibility standard deviation RSD_R for cumulative PAH released quantities from aggregates using CEN/TS 16637-3 and WI 351034 methods (median values).

	Asphalt aggregate		Recycled aggregate		Up-flow percolation	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
RSD_r	32 %	14 %	21 %	20 %	25 %	16 %
RSD_R	70 %	58 %	80 %	52 %	73 %	53 %

5.2.3 PCBs in recycled aggregate

It was not possible to assess the precision of results from released quantities of PCBs from recycled aggregate using CEN/TS 16637-3method because offrom the four laboratories that reported results, only two reported values higher than their LOD.

5.3 CEN/TS 17331Content of organic substances

The obtained repeatability (RSD_r) and reproducibility (RSD_R) are shown in Annex 7 and summarised in Figure 5 and Table 6 below. Seven and six laboratories reported results for asphalt and recycled aggregate, respectively. Contents of mineral oil, PAHs and PCBs were higher than the released quantities in the leaching and percolation tests and for that reason it was possible to assess the precision of results not only for PAHs but also for mineral oil and PCBs.

Figure 3. Repeatability RSD_r(left) and reproducibility RSD_R(right) of reported results cumulative released quantities of mineral oil, PAHs and PCBs from asphalt and recycled aggregates using CEN/TS 17331 method (and WI 351034 for PAHs). Red: asphalt aggregate. Blue: recycled aggregate

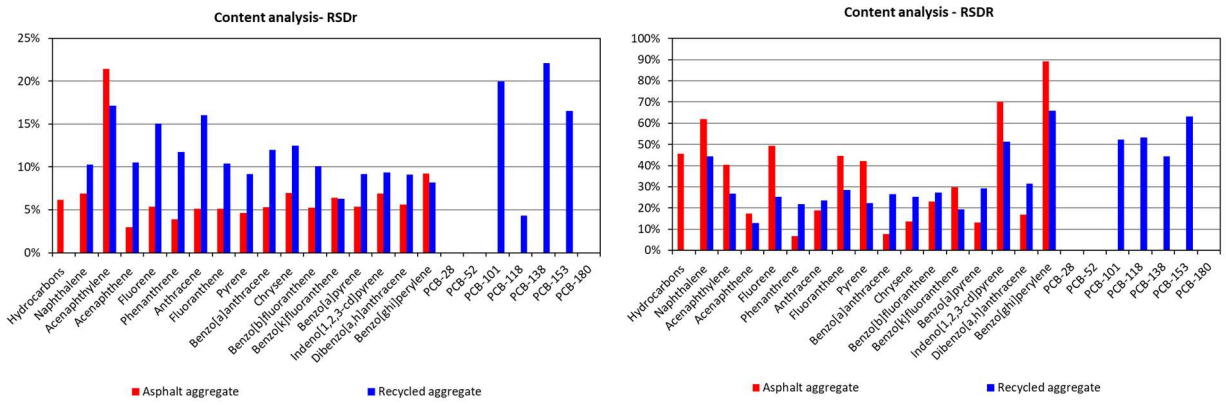


Table 6. Relative repeatability standard deviation RSD_r and relative reproducibility standard deviation RSD_R for cumulative released quantities from asphalt and recycled aggregate using CEN/TS 17331 (median values).

	Hydrocarbons	PAHs (1)	PCBs
RSD_r	6 %	9 %	18 %
RSD_R	46 %	27 %	53 %

(1) PAHs by WI 351035

5.4 CEN/TS 17332Analysis of organic substances in eluates

The obtained repeatability (RSD_r) and reproducibility (RSD_R) for biocides, mineral oil and PAHs in eluates are shown in Annex 7 and summarised in Figure 6 and Table 7 below. Released quantites of PCBs in the eluates were below the LODs.Five laboratories reported results for eluates from asphalt and recycled aggregate, three laboratories for leachates from render and only one laboratory reported results for leachates from sealant (therefore it was not possible to assess the precision of results for sealant).

Figure 6. Repeatability RSD_r(left) and reproducibility RSD_R(right) of reported results cumulative released quantities of biocides, mineral oil and PAHs from render, asphalt and recycled aggregates using CEN/TS 17332 method. Black: render. Red: asphalt aggregate. Blue: recycled aggregate

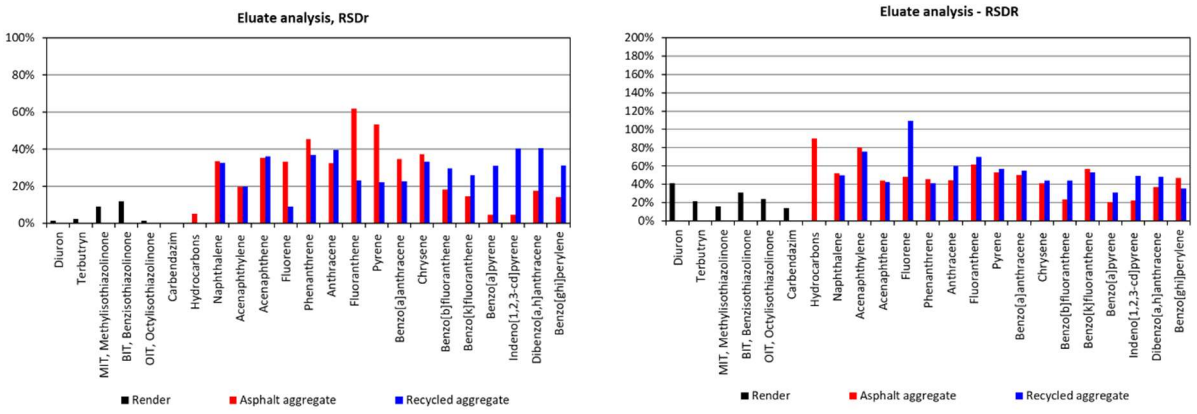


Table 7. Relative repeatability standard deviation RSD_r and relative reproducibility standard deviation RSD_R for cumulative released quantities of biocides, mineral oil and PAHs from render, asphalt and recycled aggregate using CEN/TS 17332 method (median values).

	Biocides (1)	Hydrocarbons	PAHs (2)
RSD_r	2 %	5 %	32 %
RSD_R	23 %	90 %	48 %

(1) Biocides by WI 351034
(2) PAHs by WI 351035

RSD_r and RSD_R for eluate analysis are in a similar range than the ones observed for leaching and percolation test. This is consistent with the fact that the eluates were preparedat JRC from the same sample test materials (render, asphalt and recycled aggregate) as the ones used by the laboratories to prepare their own leachates/eluates. However the reproducibility RSD_R is slightly better for eluate analysis than for leaching and percolation test. This can be explained by the simpler procedure for eluate analysis, which reduces the risk of losses or contamination during the procedure.

RSD_R value for mineral oil is higher than the others; however this value should be taken with precaution. It should be noted than, even starting from the same test materials, many laboratories were not able to report releases higher than their LOD (only 2 of 6 participants) and it was not possible to assess the RSDs of the leaching. There is not enough information to explain the reason for this higher value; possible reasons are the limited number of results, low concentration levels or technical issues related to the test method for mineral oil (4 out of 5 laboratories reported results higher than their LOD – values from laboratory#2 were between 3 and 8 times higher than values from the other laboratories but it was not recognised as outlier by statistical tests). Additionally, RSDs for mineral oil is based in just one parameter (sum of n-alkanes C10-C40) while the RSDs for biocides, PAHs and PCBs are based in more parameters (see Table 1) and those values are a priori more robust.

6 Conclusions

The method performance parameters (reproducibility RSD_R and repeatability RSD_r) for the evaluated methods are summarised in Table 8. Median values are given since the median is more representative than the mean when the data is not following a normal distribution.

These values were extracted considering the obtained data set and checking exceptions case by case. Some outliers were excluded whenever it was justified by statistical or technical reasons. Also values close to the limit of detection to avoid unnecessary bias (the latter are below any critical limit and are not relevant for decision making).

Table 8. Method performance (relative repeatability standard deviation RSD_r and relative reproducibility standard deviation RSD_R) for the evaluated methods and organic substances(median values).

		Biocides (1)	Hydrocarbons	PAHs (2)	PCBs
CEN/TS 16637-2 Dynamic surface leaching test	RSD_r	6 %			
	RSD_R	54 %			
CEN/TS 16637-3 Up-flow percolation test	RSD_r			20 %	
	RSD_R			70 %	
CEN/TS 17332 Analysis of eluates	RSD_r	2 %	5 %	32 %	
	RSD_R	23 %	90 %	48 %	
CEN/TS 17331 Content analysis	RSD_r		6 %	9 %	18 %
	RSD_R		46 %	27 %	53 %

(1) Biocides using WI 351034
(2) PAHs using WI 351035

Due to the limited number of results, any conclusion or recommendation must be made with the utmost care. However, despite the limited number of participants which might affect the robustness of this study, the results obtained for organic substances are consistent with the ones obtained for inorganic substances (1). Therefore it seems reasonable to incorporate the RSD values for organic substances to the standard methods.

Acknowledgements

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- Fraunhofer Institute for Building Physics IBP (Valley, Germany)
- Recycling Kombinatie REKO B.V. (Rotterdam, The Netherlands)
- Theo Pouw bv (Utrecht, The Netherlands)

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The twelve laboratories listed below are kindly acknowledged for their participation in this interlaboratorytest.

Laboratory	**Country**
BAM - Federal Institute for Materials Research and Testing, Division 4.3	Germany
Dr. Robert-Murjahn-Institut, Analytical Department	Germany
Eurofins Umwelt Ost GmbH, Niederlassung Freiberg	Germany
Fraunhofer Institute for Building Physics IBP	Germany
ITCZLIN Institut for testing and certification, a.s.	Czech Republic
Kiwa GmbH	Germany
MAPAG GmbH	Austria
PiCA Prüfinstitut Chemische Analytik GmbH	Germany
SGS INTRON	The Netherlands
Swedish geotechnical institute	Sweden
Synlab Analytical Services	The Netherlands
Tecno Piemonte Spa	Italy

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(5) CEN/TS 16637-2:2014 Construction products – Assessment of release of dangerous substances – Part 2: Horizontal dynamic surface leaching test

(6) CEN/TS 16637-3:2016 Construction products – Assessment of release of dangerous substances – Part 3: Horizontal up-flow percolation test

(7) CEN/TS 17331:2019 Construction products: Assessment of release of dangerous substances - Content of organic substances – Methods for extraction and analysis

(8) CEN/TS 17332:2019 Construction products – Assessment of release of dangerous substances – Analysis of organic substances in eluates

(9) WI 351034:2019 Construction products: Assessment of release of dangerous substances - Determination of the content of polycyclic aromatic hydrocarbons (PAH) and of benzene, toluene, ethylbenzene and xylene (BTEX) — Gas-chromatographic method with mass spectrometric detection (ICP-OES)

(10) WI 351035:2019 Construction products: Assessment of release of dangerous substances - Determination of biocides using LC-MS/MS

(11) ISO 5725-2:1994 Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

(12) ISO/IEC 17043:2010 Conformity assessment – General requirements for proficiency testing

(13) ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories

(14) ISO 11648-2:2001 Statistical aspects of sampling from bulk materials – Part 2: Sampling of particulate materials

(15) ISO 5725-1:1994 Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions

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(19) ISO 5667-3:2012 Water quality – Sampling – Part 3: Preservation and handling of water samples

List of abbreviations and definitions

BBP	Butylbenzylphthalate
BIT	Benzisothiazolinone
BTEX	Benzene, toluene, ethylbenzene and xylene
CEN	European Committee for Standardization
CEN/TC 351	CEN Technical Committee 351 "Construction Products - Assessment of release of dangerous substances"
DBP	Dibutylphthalate
DCHP	Dicyclohexylphthalate
DEHP	di-(2-Ethylhexyl)phthalate
DEP	Diethylphthalate
DG GROW	Directorate General for Internal Market, Industry, Enterpreneurship and SMEs
DIBP	di-(2-Methylpropyl)phthalate
DiNP	di-Isononylphthalate
DMP	Dimethylphthalate
DOP	Dioctylphthalate
EN	European Standard
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
JRC	Joint Research Centre of the European Commission
L/S	Liquid to solid ratio of CEN/TS 16637-3
LOD	Limit of detection
LOQ	Limit of quantification
MIT	Methylisothiazolinone
OIT	Octylisothiazolinone
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PTFE	Polytetrafluoroethylene
QC	Quality Control
RSD_r	Relative repeatability standard deviation
RSD_R	Relative reproducibility standard deviation
SD_R	Reproducibility standard deviation
SD_r	Repeatability standard deviation
TC	Technical Committee
TS	Technical Specification
WG	Working Group
XPS	Extruded polystyrene

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Annexes

Annex 1a. Original validation plans

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

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.

Annex 1b. Modified validation plan to reduce the workload.

Changes to the original validation plan are in blue (blue text as in the letter sent to laboratories)

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

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. Laboratories can also add measurements according to EN 14405 (percolation test for waste material), as few data on the performance of this method is available.

2 MATERIALS

The following materials will be used for the validation of CEN/TS 16637-2 and 16637-3. **Laboratories can participate for one or more materials.**

	CEN/TS 16637-2		CEN/TS 16637-3	
	Material	**Analytes**	**Material**	**Analytes**
Material 1	Render	Diuron, terbutryn, MIT, BIT, OIT, CMIT	Asphalt aggregate	PAHs, mineral oil
Material 2	Sealant	Phthalates (DMP, DIPB, DBP, BBP, DEHP, DCHP)	Recycled mixed aggregate	PCBs, PAHs
Quality control	Multi substance solution			
Amount per material	3 samples each of material 1-3; 1 bottle of the quality control material			

The following materials will 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	Phthalates (DMP, DIPB, DBP, BBP, DEHP, DCHP)	Sealant	Phthalates (DMP, DIPB, DBP, BBP, DEHP, DCHP)
Material 3	Asphalt aggregate	PAH, mineral oil	Asphalt aggregate	PAHs, mineral oil
Material 4	Recycled mixed aggregate	PCBs, PAHs	Recycled mixed aggregate	PCBs, PAHs
Quality control	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 participate for one or several materials (e.g. only leaching of the asphalt aggregate according to CEN/TS 16637-3 or only total content analysis of the sealant)

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

The latest draft standards will be provided for all TS that are not published yet. This applies in particular to the determination methods for biocides and PAHs.

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

The three samples per material shall be leached according to CEN/TS16637-2. **Only the cumulative amount after 64 days needs to be determined.** To achieve this, equal amounts of leachates of the various times shall be mixed and the mixture is analysed after 64 days together with the quality control material. Detailed instructions will be sent with the samples.

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.

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. **Only the cumulative L/S fractions 2.0 l/kg and 10.0 l/kg need to be determined**. To achieve this, appropriate fractions of liquid/solid (L/S) ratios of 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 l/kg shall be mixed to achieve one sample representative of the cumulative l/s of 2.0 and one sample representative of the cumulative l/s of 10.0 l/kg . Detailed instructions will be sent with the samples.

It is important that the leaching of the three samples of each material is done simultaneously, i.e. under repeatability conditions.

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 extraction 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.

4.5 Use of polydimethylsiloxane (PDMS) when using CEN/TS16637-2

In addition to leaching according CEN/TS 16637-2, participants have the possibility to apply a variant employing polymethylsiloxane. This procedure is currently being tested by the US EPA and aims at reducing adsorption losses during the leaching by adsorbing the analytes onto the polymer from which they are extracted in a subsequent step.

Detailed instructions will be provided together with the samples.

Note that it is not possible to participate only for this variant – the same material must also be analysed using CEN/TS16637-2.

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.

Annex 2. Registration form

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate F – Health, Consumers and Reference Materials
Reference Materials Unit

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

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 ☐ I also will apply the variant method using PDMS	☐ CEN/TS 16637-3 ☐ Asphalt aggregate ☐ Recycled mixed aggregate ☐ I also will apply the variant method using PDMS
☐ CEN/TS 351026 (all 4 eluates/leachates are to be analysed)	☐ CEN/TS 351024 ☐ 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: .....

Annex 3. Invitation letter for participants

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

Dr Thomas Linsinger

Retiesweg 111, 2440 Geel Tel: +32 14 571 958 email: thomas.linsinger@ec.europa.eu

Annex 4. Sample accompanying letter

EUROPEAN COMMISSION

JOINT RESEARCH CENTRE
Directorate F - Health, Consumers & Reference Materials (Geel/Ispra)
Reference Materials Unit

Instructions for the interlaboratory validation according to CEN/TS 16637, CEN/TS 17331 and CEN/TS 17332 (WI 351034 for PAHs, WI 351035 for biocides)

Sample storage

Samples must be kept at 4 °C

List of materials

For the horizontal dynamic surface leaching test according to CEN/TS 16637-2:

- 3 blocks of EPS coated with organic render (Material 1)
- 3 tiles coated with sealant (Material 2)

For the horizontal up-flow percolation test according to CEN TS 16637-3:

- 1/3 drums filled with asphalt aggregate (Material 3)
- 1/3 drums filled with recycled aggregate (Material 4)

For the content analysis

- 1 jar containing approximately 50 g ground asphalt aggregate (Material 5)
- 1 jar containing approximately 50 g ground recycled aggregate (Material 6)

For the eluate analysis

- 3 bottles of approximately 25 ml each of water eluate of render (Material 7)
- 3 bottles of approximately 1 l each of water eluate of sealant (Material 8)
- 3 bottles of approximately 1 l each of water eluate of asphalt aggregate (Material 9)
- 3 bottles of approximately 1 l each of water eluate of recycled aggregate (Material 10)

Quality control materials

- 1 vial of diuron, terbutryn and MIT in methanol, concentration approx. 250 mg/l to be used as quality control material (QC biocides)
- 1 vial of phthalate mix in methanol, concentration approx. 1-10 g/l to be used as quality control material (QC phthalates)
- 1 vial of polycyclic aromatic hydrocarbons in toluene, mass fractions around 3 mg/kg to be used as quality control material (QC PAHs)
- 1 vial of mineral oil in heptane, concentration approx. 10 g/l hydrocarbons, to be used as quality control material (QC mineral oil)
- 1 vial of polychlorinated biphenyls in isooctane, concentrations 5-25 mg/kg, to be used as quality control material (QC PCBs).

Measurements to be performed

For the horizontal dynamic surface leaching test according to CEN/TS 16637-2

The three samples per material shall be leached according to CEN/TS16637-2. **Only the cumulative amount after 64 days from the start of the tests needs to be determined.** To achieve this, equal amounts of leachates of the various times shall be mixed and the mixture is analysed after 64 days together with the quality control material:

Page 1 of 4

1) At each leaching step, take the same amount of volume and combine these amounts into one composite (pull) sample. Important: use same leaching volume for all leaching steps and take same volume from each portion!

2) Store the leachates in a fridge until the end of the test. Ensure that you keep at least the first two eluates (likely with the highest concentration) until reporting of results.

3) Measure this composite sample and multiply the result with 8 (as there are 8 leaching steps). Important: The total volume used in each fraction/step must be used in the calculation (not the volume extracted for making the composite).

It is important that the leaching of the three samples of the same material is done simultaneously, i.e. under repeatability conditions. The leaching of samples of the other material can, but does not have to be performed at the same time.

In the same way, quantification of the leachates of the three samples of each material shall be performed in one run. The leachates of the other materials can, but do not have to be analysed at the same time.

When analysing the leachates, the quality control sample shall be analysed as well to detect potential measurement bias.

The analytes to be quantified are listed below.

The leaching should start not later than 3 weeks after receipt of the samples.

For the horizontal up-flow percolation test according to CEN/TS 16637-3:

Three samples per material shall be percolated according to CEN/TS16637-3. **Only the cumulative L/S fractions 2.0 l/kg and 10.0 l/kg need to be determined.** To achieve this, appropriate fractions of liquid/solid (L/S) ratios of 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 l/kg shall be mixed to achieve one sample representative of the cumulative L/S of 2.0 and one sample representative of the cumulative L/S of 10.0 l/kg:

- Step 1: Collect eluate until L/S ratio of 2.0 is reached. Measure this eluate.
  U(2.0)=V*c/m, with *V* the volume of the eluate in l, *c* the measured concentration in mg/l and *m* the mass in kg.
- Step 2: In a new vessel, collect eluate until L/S ratio of 8.0 is reached. Measure this eluate.
  U(10.0)=V*c/m +U(2.0), with *V* the volume of the new eluate, *c* the measured concentration and *m* the mass.

The materials must be homogenised before filling the columns. Samples were sieved to 1 cm but the criteria of 45% < 4 mm needs to be checked.

It is important that the percolation of the three samples of the same material is done simultaneously, i.e. under repeatability conditions. The percolation of samples of the other material can but do not have to be performed at the same time.

In the same way, quantification of the eluates of the three samples of each material shall be performed in one run together with a sample of the quality control material.

The analytes to be quantified are listed below.

For the content analysis according to CEN/TS 17331 (PAHs WI 351034, biocides WI 351035)

Three test portions shall be taken from each test sample. These test portions shall be extracted and analysed according to CEN/TS 17331 (WI 351034 for PAHs and WI 351035 for biocides) together with a sample of the quality control material.

The eluates must be centrifuged before measurement. There are two options:
a) 30 min at 20 000 g to 30 000 g using a high speed centrifuge, or
b) 5 h at 2 000 g to 2 500 g in glass bottles using an lower speed centrifuge
The bottle and any remaining solid must be rinsed with solvent.

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 extraction 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 below.

For the eluate analysis according to CEN/TS 17332 (PAHs WI 351034, biocides WI 351035)

The four leachates/eluates shall be analysed under repeatability conditions according to CEN/TS 17332 (WI 351034 for PAHs and WI 351035 for biocides) 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 below.

List of analytes to be quantified

- Biocides in organic render:
  - Diuron
  - Terbutryn
  - Methylisothiazolinone, MIT
  - Benzisothiazolinone, BIT
  - Octylisothiazolinone, OIT
  - Carbendazim
- Phthalates in sealant:
  - Dimethylphthalate, DMP
  - Diethylphthalate, DEP
  - Di-(2-methylpropyl)phthalate, DIBP
  - Dibutylphthalate, DBP
  - Butylbenzylphthalate, BBP
  - Di-(2-ethylhexyl)phthalate, DEHP
  - Dicyclohexylphthalate, DCHP
  - Dioctylphthalate, DOP
  - Di-isononylphthalate, DiNP

- Mineral oil in asphalt aggregate
- Polycyclic aromatic hydrocarbons (PAHs) in asphalt aggregate / recycled aggregate
  - Naphthalene
  - Acenaphthylene
  - Acenaphthene
  - Fluorene
  - Phenanthrene
  - Anthracene
  - Fluoranthene
  - Pyrene
  - Benzo(a)anthracene
  - Chrysene
  - Benzo(b)fluoranthene
  - Benzo(k)fluoranthene
  - Benzo(a)pyrene
  - Indeno(1,2,3,c,d)pyrene
  - Dibenzo(a,h)anthracene
  - Benzo(g,h,i)perylene
- Polychlorinated biphenyls (PCBs) in recycled aggregate:

PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153 and PCB-180

Reporting

Reporting should be done using the electronic reporting sheet provided by email.

Please send the results electronically **by 15July2019** to Silvia.GARCIA-RUIZ@ec.europa.eu

Please also send a pdf-file of a signed copy, as this allows us to comply with the requirements of ensuring the integrity of electronic data of ISO 5727 and ISO 17043.

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

Silvia Garcia-Ruiz and Thomas Linsinger

Annex 5. Reporting Excel sheets

Test report interlaboratory validation CEN TS 16637 - 2
Material 1: Render

Receiver JRC, Retieseweg 111, 2440 Geel, BE
Method used CEN TS 16637-2

	Sample 1 mg/m²	Sample 2 mg/m²	Sample 3 mg/m²	QC Material mg/kg	Extract 1 µg/l	Extract 2 µg/l	Extract 3 µg/l	Procedural blank, µg/l	LOD µg/l
Sample code									
Surface (m²)									
Start date of the leaching test									
Cumulative result after 1 day (combined fractions 1+2)									
Volume of leachant at each step/fraction (l)									
Volume of portion taken at each step/fraction (l)									
Date of measurement									
Diuron									
Terbutryn									
Methylisothiazolinone, MIT									
Benzisothiazolinone, BIT									
Octylisothiazolinone, OIT									
Carbendazim									
Cumulative result after 63 days (combined fractions from 3 to 8)									
Volume of leachant at each step/fraction (l)									
Volume of portion taken at each step/fraction (l)									
Date of measurement									
Diuron									
Terbutryn									
Methylisothiazolinone, MIT									
Benzisothiazolinone, BIT									
Octylisothiazolinone, OIT									
Carbendazim									
Cumulative result after 64 days (calculated)									
Diuron	0	0	0						
Terbutryn	0	0	0						
Methylisothiazolinone, MIT	0	0	0						
Benzisothiazolinone, BIT	0	0	0						
Octylisothiazolinone, OIT	0	0	0						
Carbendazim	0	0	0						

Laboratory
Department
Address
Name of responsible

Date , Signature

Test report interlaboratory validation CEN TS 16637 - 3
Material 3: Asphalt aggregate

Receiver JRC, Retieseweg 111, 2440 Geel, BE
Method used CEN TS 16637-3

	Sample 1 mg/kg dry	Sample 2 mg/kg dry	Sample 3 mg/kg dry	QC Material mg/kg	Extract 1 µg/l	Extract 2 µg/l	Extract 3 µg/l	Procedural blank, µg/l	LOD µg/l
Sample code									
Mass (kg)									
Start date of the percolation test									
Cumulative results L/S = 2 l/kg									
Cummulative volume of eluate (l)									
Date of measurement									
Hydrocarbons (mineral oil)									
Cummulative volume of eluate (l)									
Date of measurement									
Naphthalene									
Acenaphthylene									
Acenaphthene									
Fluorene									
Phenanthrene									
Anthracene									
Fluoranthene									
Pyrene									
Benzo[*a*]anthracene									
Chrysene									
Benzo[*b*]fluoranthene									
Benzo[*k*]fluoranthene									
Benzo[*a*]pyrene									
Indeno[1,2,3-*cd*]pyrene									
Dibenzo[*a,h*]anthracene									
Benzo[*ghi*]perylene									
Cumulative results L/S = 10 l/kg									
Cummulative volume of eluate (l)									
Date of measurement									
Hydrocarbons (mineral oil)									
Cummulative volume of eluate (l)									
Date of measurement									
Naphthalene									
Acenaphthylene									
Acenaphthene									
Fluorene									
Phenanthrene									
Anthracene									
Fluoranthene									
Pyrene									
Benzo[*a*]anthracene									
Chrysene									
Benzo[*b*]fluoranthene									
Benzo[*k*]fluoranthene									
Benzo[*a*]pyrene									
Indeno[1,2,3-*cd*]pyrene									
Dibenzo[*a,h*]anthracene									
Benzo[*ghi*]perylene									

Laboratory
Department
Address
Name of responsible

Date , Signature

Test report interlaboratory validation CEN TS 17331, WI 351034
Material 5: Asphalt aggregate

Receiver JRC, Retieseweg 111, 2440 Geel, BE
Method used CEN TS 17331, WI 351034

	Sample 1 mg/kg dry	Sample 2 mg/kg dry	Sample 3 mg/kg dry	QC Material mg/kg	Extract 1 µg/l	Extract 2 µg/l	Extract 3 µg/l	Procedural blank, µg/l	LOD µg/l
Sample code									
Sample intake (g)									
Date of extraction									
Date of measurement									
Hydrocarbons (mineral oil)									
Sample intake (g)									
Date of extraction									
Date of measurement									
Naphthalene									
Acenaphthylene									
Acenaphthene									
Fluorene									
Phenanthrene									
Anthracene									
Fluoranthene									
Pyrene									
Benzo[*a*]anthracene									
Chrysene									
Benzo[*b*]fluoranthene									
Benzo[*k*]fluoranthene									
Benzo[*a*]pyrene									
Indeno[*1,2,3-cd*]pyrene									
Dibenzo[*a,h*]anthracene									
Benzo[*ghi*]perylene									

Laboratory
Department
Address
Name of responsible

Date , Signature

Test report interlaboratory validation CEN TS 17332, WI 351035
Material 7: Render

Receiver JRC, Retieseweg 111, 2440 Geel, BE
Method used CEN TS 17332, WI 351035

	Sample 1 µg/l	Sample 2 µg/l	Sample 3 µg/l	QC Material mg/kg	Procedural blank, µg/l	LOD µg/l
Sample code						
Sample intake (ml)						
Date of analysis						
Diuron						
Terbutryn						
Methylisothiazolinone, MIT						
Benzisothiazolinone, BIT						
Octylisothiazolinone, OIT						
Carbendazim						

Laboratory
Department
Address
Name of responsible

Date , Signature

Annex 6. Questionnaires

Construction products – Assessment of release of dangerous substances –Organic

Method used: CEN/TS 16637-2 Horizontal dynamic surface leaching test

Receiver: JRC, Retieseweg 111, 2440 Geel, BE

a) General

1) Date of receipt of the laboratory sample:

2) Identification of the laboratory sample, including sample code:

3) Storage conditions of the laboratory and test samples:

4) Dimensions of the test portion and calculation method of the geometric surface area (A):

5) The way in which the test portion is suspended in or affixed to the leaching vessel:

b) Leaching test conditions

6) Date of the test (beginning and end):

7) Type of deionised water used as leachant:

8) Identification of the test equipment and instruments used, including the materials and dimensions of the leaching vessel:

9) Actual leachant renewal times of all periods (t_i to t8):

10) Actual leachant volume for all periods/fractions:

11) Temperature range during the performance of the test:

12) Any deviations, additions to or exclusions from the test specification, and any other relevant information; including any operation not specified in the method or regarded as optional which might have affected the results:

c) Analytical report

13) Turbidity, pH and conductivity of the eluate fractions collected:

14) Centrifugation procedure followed, if any:

15) Any dilutions that were carried out:

16) Methods of preservation of the eluate fractions:

17) Eluate bottles (e.g. material, pre-cleaned):

18) Analytical method applied:

19) Date of measurement

d) Results of the leaching test:

Please note that you should report cumulative released quantities after 1 day and after 64 days from the start of the test.

Laboratory:

Address:

Name of responsible:

Date and signature:

Construction products – Assessment of release of dangerous substances –Organic

Method used: CEN/TS 16637-3 Horizontal up-flow percolation test

Receiver: JRC, Retieseweg 111, 2440 Geel, BE

a) General

1) Date of receipt of the laboratory sample:

2) Identification of the laboratory sample, including sample code:

3) Sample preparation (e.g. method of size-reduction, drying, sub-division) and storage conditions of the laboratory sample and test sample:

4) Percentage of particle < 4 mm:

5) Crushing equipment, if any:

6) Drying temperature:

7) Compaction method (for column packing):

b) Leaching test conditions

8) Date of the test (beginning and end):

9) Type of deionised water used as leachant:

10) Test equipment and instruments used, including the diameter of the column:

11) Actual flow rate during the course of the test:

12) True cumulative L/S-ratios of the eluate fractions collected:

13) Temperature range during the performance of the test:

14) Any deviations, additions to or exclusions from the test specification, and any other relevant information; including any operation not specified in the method or regarded as optional which might have affected the results:

c) Analytical report

15) Turbidity, pH and conductivity of the eluate fractions collected:

16) Centrifugation procedure followed, if any:

17) Any dilutions that were carried out:

18) Methods of preservation of the eluate fractions:

19) Eluate bottles (e.g. material, pre-cleaned):

20) Analytical method applied:

21) Date of measurement

Laboratory:

Address:

Name of responsible:

Date and signature:

Construction products – Assessment of release of dangerous substances

Method used: CEN/TS 17331 Content of organic substances. Methods for extraction and analysis (PAH analysis according to WI 351034)

Receiver: JRC, Retieseweg 111, 2440 Geel, BE

a) General

1) Date of receipt of the laboratory sample:

2) Identification of the laboratory sample, including sample code:

3) Description of test sample treatment and storage, if relevant:

b) Extraction

4) Date of the test sample extraction:

5) Identification of the test specification or description of the method or procedure:

6) Describe any deviation, addition or exclusion from the test specification, and the reason of this deviation:

c) Sample analysis

7) Date of the test sample analysis:

8) Method of analysis and brief description of the experimental measurement procedure:

9) If PAHs were analysed, whether the PAH contents have been corrected for the recovery of the surrogate standard:

10) Any deviations, additions to or exclusions from the test specification, and any other relevant information; including any operation not specified in the method or regarded as optional which might have affected the results:

Laboratory:

Address:

Name of responsible:

Date and signature:

Construction products – Assessment of release of dangerous substances – Elements

Method used: CEN/TS 17332 Analysis of organic substances in eluates (PAH analysis according to WI 351034 and biocide analysis according to 351035)

Receiver: JRC, Retieseweg 111, 2440 Geel, BE

1) Date of receipt of the laboratory sample:

2) Identification of the laboratory sample, including sample code:

3) Description of eluate treatment and storage, if relevant:

4) Date of the sample treatment and analyses:

5) Identification of the test specification or description of the method or procedure:

6) If PAHs were analysed, whether the PAH contents have been corrected for the recovery of the surrogate standard:

7) Any deviations, additions to or exclusions from the test specification, and any other relevant information; including any operation not specified in the method or regarded as optional which might have affected the results:

Laboratory:

Address:

Name of responsible:

Date and signature:

Annex 7. Evaluation of results

Annex 7.1. CEN/TS 16637-2 (horizontal dynamic surface leaching test)

RENDER

Results after elimination of statistical outliers	Diuron		Terbutryn		Methylisothiazolinone, MIT		Benzisothiazolinone, BIT		Octylisothiazolinone, OIT		Carbendazim	
	1 day	64 days	1 day	64 days	1 day	64 days	1 day	64 days	1 day	64 days	1 day	64 days
Mean (mg/m^2)	110	371	23	128	211	189	557	525	83	246	41	327
SD_R	80	270	19	112	143	157	448	476	58	238	34	306
SD_r	4.4	73	1.4	7.4	51	46	129	129	5.4	38	3.4	40
RSD_R	73 %	73 %	83 %	88 %	68 %	83 %	81 %	91 %	70 %	97 %	82 %	94 %
RSD_r	4 %	20 %	6 %	6 %	24 %	25 %	23 %	25 %	6 %	15 %	8 %	12 %
No. laboratories with results > LOD	4	5	5	6	5	6	5	5	5	6	4	5
No. outliers												
No. laboratories after outlier elimination	4	5	5	6	5	6	5	5	5	6	4	5
No. values after outlier elimination	11	14	14	17	15	18	15	15	14	16	12	15

Results after elimination of outliers and L14	Diuron		Terbutryn		Methylisothiazolinone, MIT		Benzisothiazolinone, BIT		Octylisothiazolinone, OIT		Carbendazim	
	1 day	64 days	1 day	64 days	1 day	64 days	1 day	64 days	1 day	64 days	1 day	64 days
Mean (mg/m^2)	110	371	29	155	264	227	696	657	105	303	54	409
SD_R	80	270	16	103	92	142	371	432	37	226	24	283
SD_r	4.4	73	1.5	8.1	57	51	144	144	6.1	42	3.9	45
RSD_R	73 %	73 %	54 %	66 %	35 %	63 %	53 %	66 %	36 %	75 %	45 %	69 %
RSD_r	4 %	20 %	5 %	5 %	22 %	22 %	21 %	22 %	6 %	14 %	7 %	11 %
No. laboratories with results > LOD	4	5	5	6	5	6	5	5	5	6	4	5
No. outliers			1	1	1	1	1	1	1	1	1	1
No. laboratories after outlier elimination	4	5	4	5	4	5	4	4	4	5	3	4
No. values after outlier elimination	11	14	14	17	15	18	15	15	14	16	12	15

MIT, BIT and OIT not stable in the 64 days fraction (in grey); stability of carbendazim in water is also limited (half-life of two months)

Annex 7.2. CEN/TS 16637-3 (horizontal up-flow percolation test)

ASPHALT AGGREGATE

Results after elimination of statistical outliers	Hidrocarbons		Naphthalene		Acenapthtylene		Acenapthene		Fluorene		Phenanthrene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	0.31	0.84	2.9E-05	9.8E-05	1.1E-04	2.5E-05	2.2E-05	5.9E-03	1.7E-04	4.0E-04	5.0E-05	1.7E-04
SD_R	0.18	0.51	2.0E-05	4.0E-05	1.6E-04	3.2E-05	2.6E-05	8.3E-03	2.3E-04	3.8E-04	5.4E-05	8.4E-05
SD_r	0.18	0.18	1.2E-06	1.5E-05	3.2E-05	4.9E-06	2.0E-05	1.1E-03	3.6E-05	9.9E-05	4.6E-05	4.5E-05
RSD_R	59 %	60 %	71%	41%	146%	127%	117%	141%	141%	94%	87%	51%
RSD_r	59 %	22 %	4%	15%	29%	19%	91%	19%	22%	24%	86%	27%
No. laboratories with results > LOD	2	2	2	2	6	4	4	2	6	3	2	2
No. outliers					1	2	1		1			
No. laboratories after outlier elimination	2	2	2	2	5	2	3	2	5	3	3	2
No. values > LOD after outlier elimination	6	6	5	6	13	5	6	6	10	8	6	6

In grey results from less than 3 laboratories or released quantities below the limit of quantification

Results after elimination of outliers and L12	Hidrocarbons		Naphthalene		Acenapthtylene		Acenapthene		Fluorene		Phenanthrene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	0.31	0.84	2.9E-05	9.8E-05	1.1E-04	2.5E-05	2.2E-05	5.9E-03	1.7E-04	2.9E-04	5.0E-05	1.7E-04
SD_R	0.18	0.51	2.0E-05	4.0E-05	1.6E-04	3.2E-05	2.6E-05	8.3E-03	2.5E-04	3.7E-04	5.4E-05	8.4E-05
SD_r	0.18	0.18	1.2E-06	1.5E-05	3.2E-05	4.9E-06	2.0E-05	1.1E-03	3.6E-05	4.1E-05	4.6E-05	4.5E-05
RSD_R	59 %	60 %	71%	41%	146%	127%	117%	141%	151%	129%	108%	51%
RSD_r	59 %	22 %	4%	15%	29%	19%	91%	19%	22%	14%	91%	27%
No. laboratories with results > LOD	2	2	2	2	6	4	4	2	6	3	3	2
No. outliers					1	2	1		2	1	1	
No. laboratories after outlier elimination	2	2	2	2	5	2	3	2	4	2	2	2
No. values > LOD after outlier elimination	6	6	5	6	13	5	6	6	9	6	5	6

In grey results from less than 3 laboratories or released quantities below the limit of quantification

Results after elimination of statistical outliers	Anthracene		Fluoranthene		Pyrene		Benzo(a)anthracene		Chrysene		Benzo(b)fluoranthene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	3.0E-05	7.7E-05	1.0E-04	4.5E-04	3.0E-04	3.2E-04	8.5E-05	5.4E-04	6.6E-05	5.0E-04	2.0E-04	6.7E-04
SD_R	2.1E-05	6.2E-05	1.1E-04	6.5E-04	3.2E-04	2.8E-04	3.8E-05	5.9E-04	2.8E-05	5.0E-04	1.4E-04	3.4E-04
SD_r	1.6E-05	3.6E-05	1.1E-04	1.1E-04	6.0E-05	5.8E-05	3.4E-05	3.4E-05	2.3E-05	6.9E-05	3.5E-05	1.4E-04
RSD_R	70%	81%	106%	146%	105%	87%	45%	110%	42%	99%	70%	52%
RSD_r	53%	47%	106%	25%	20%	18%	40%	6%	34%	14%	17%	21%
No. laboratories with results > LOD	5	5	3	3	2	3	5	5	5	4	6	6
No. outliers	2	2					2	1	2	1	1	
No. laboratories after outlier elimination	3	3	3	3	2	3	3	4	3	3	5	6
No. values > LOD after outlier elimination	6	6	7	7	5	7	6	10	6	8	11	15

In grey results from less than 3 laboratories or released quantities below the limit of quantification

Results after elimination of outliers and L12	Anthracene		Fluoranthene		Pyrene		Benzo(a)anthracene		Chrysene		Benzo(b)fluoranthene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)			1.0E-04	4.5E-04	3.0E-04	3.2E-04	8.5E-05	5.4E-04	6.6E-05	5.0E-04	2.0E-04	5.7E-04
SD_R	2.1E-05	6.2E-05	1.1E-04	6.5E-04	3.2E-04	2.8E-04	3.8E-05	5.9E-04	2.8E-05	5.0E-04	1.4E-04	2.9E-04
SD_r	1.6E-05	3.6E-05	1.1E-04	1.1E-04	6.0E-05	5.8E-05	3.4E-05	3.4E-05	2.3E-05	6.9E-05	3.5E-05	9.4E-05
RSD_R			106%	146%	105%	87%	45%	110%	42%	99%	70%	51%
RSD_r			106%	25%	20%	18%	40%	6%	34%	14%	17%	17%
No. laboratories with results > LOD	5	5	3	3	2	3	5	5	5	4	6	6
No. outliers	2	2					2	1	2	1	1	1
No. laboratories after outlier elimination	3	3	3	3	2	3	3	4	3	3	5	5
No. values > LOD after outlier elimination	6	6	7	7	5	7	6	10	6	8	11	12

In grey results from less than 3 laboratories or released quantities below the limit of quantification

Results after elimination of statistical outliers	Benzo(k)fluoranthene		Benzo(a)pyrene		Indeno(1,2,3-cd)pyrene		Dibenzo(a,h)anthracene		Benzo(ghi)perylene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	2.0E-04	4.5E-04	1.7E-04	4.8E-04	1.3E-04	4.1E-04	7.3E-05	2.3E-04	9.1E-05	5.1E-04
SD_R	8.7E-05	2.5E-04	1.3E-04	2.6E-04	1.7E-04	2.2E-04	7.6E-05	2.1E-04	8.3E-05	2.1E-04
SD_r	2.9E-05	8.7E-05	5.5E-05	7.6E-05	1.2E-04	5.2E-05	2.3E-05	2.8E-05	2.8E-05	8.2E-05
RSD_R	43%	55%	78%	53%	132%	54%	104%	89%	91%	41%
RSD_r	14%	19%	32%	16%	91%	13%	31%	12%	31%	16%
No. laboratories with results > LOD	4	6	6	6	6	5	5	5	6	5
No. outliers	1		1		1		1		2	
No. laboratories after outlier elimination	3	6	5	6	5	5	4	5	4	5
No. values > LOD after outlier elimination	8	15	13	15	13	13	9	11	10	13

Results after elimination of outliers and L12	Benzo(k)fluoranthene		Benzo(a)pyrene		Indeno(1,2,3-cd)pyrene		Dibenzo(a,h)anthracene		Benzo(ghi)perylene	
	2.0E-04	4.4E-04	1.7E-04	4.3E-04	1.3E-04	3.2E-04	7.3E-05	2.3E-04	9.1E-05	4.4E-04
Mean (mg/kg dry)	8.7E-05	2.8E-04	1.3E-04	2.6E-04	1.7E-04	1.5E-04	7.6E-05	2.4E-04	8.3E-05	1.6E-04
SD_R	2.9E-05	7.7E-05	5.5E-05	7.0E-05	1.2E-04	4.7E-05	2.3E-05	1.1E-05	2.8E-05	3.9E-05
SD_r	43%	64%	78%	61%	132%	46%	104%	105%	91%	38%
RSD_R	14%	18%	32%	16%	91%	14%	31%	5%	31%	9%
RSD_r	4	6	6	6	6	5	5	5	5	5
No. laboratories with results > LOD	1	1	1	1	1	1	1	1	2	1
No. outliers	3	5	5	5	5	4	4	4	4	4
No. laboratories after outlier elimination	8	12	13	12	13	10	9	9	10	10
No. values > LOD after outlier elimination	2.0E-04	4.4E-04	1.7E-04	4.3E-04	1.3E-04	3.2E-04	7.3E-05	2.3E-04	9.1E-05	4.4E-04

RECYCLED AGGREGATE

Results after elimination of statistical outliers	Naphthalene		Acenapthtylene		Acenapthene		Fluorene		Phenanthrene		Anthracene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	4.1E-04	1.7E-02	5.8E-04	2.6E-03	1.1E-02	9.5E-02	1.0E-02	2.0E-02	1.3E-04	9.4E-04	4.1E-03	1.4E-02
SD_R	4.4E-04	2.3E-02	7.9E-04	3.0E-03	1.2E-02	7.0E-02	1.1E-02	2.0E-02	1.4E-04	1.2E-03	3.5E-03	1.2E-02
SD_r	1.7E-04	2.1E-02	1.2E-04	2.2E-03	1.4E-03	9.7E-03	3.1E-03	1.1E-02	1.2E-04	1.2E-03	8.7E-04	1.1E-02
RSD_R	108%	133%	136%	115%	115%	74%	103%	102%	104%	125%	87%	85%
RSD_r	40%	125%	20%	84%	13%	10%	30%	56%	89%	125%	21%	79%
No. laboratories with results > LOD	3	5	6	6	6	6	6	6	5	5	6	6
No. outliers	1				1	2		1	2	2		
No. laboratories after outlier elimination	2	5	6	6	5	4	6	5	3	3	6	6
No. values > LOD after outlier elimination	5	12	13	14	11	9	15	12	7	9	15	15

In grey results from less than 3 laboratories or released quantities below the limit of quantification

Results after elimination of outliers,L3 and L14	Naphthalene		Acenapthtylene		Acenapthene		Fluorene		Phenanthrene		Anthracene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	6.5E-04	2.6E-02	2.4E-04	2.1E-03	1.3E-02	1.2E-01	7.6E-03	2.3E-02	1.9E-04	8.4E-04	3.4E-03	1.4E-02
SD_R		2.8E-02	2.2E-04	2.1E-03	1.3E-02	4.5E-02	7.0E-03	2.0E-02	1.7E-04	8.8E-04	3.0E-03	1.0E-02
SD_r	2.0E-04	2.8E-02	1.0E-04	1.2E-03	1.6E-03	1.1E-02	3.4E-03	1.2E-02	1.7E-04	8.8E-04	7.3E-04	7.9E-03
RSD_R		106%	94%	101%	97%	37%	92%	86%	88%	105%	90%	74%
RSD_r	30%	106%	44%	57%	12%	9%	45%	51%	88%	104%	22%	56%
No. laboratories with results > LOD	3	5	6	6	6	6	6	6	5	5	6	6
No. outliers	2	2	2	2	2	3	2	2	3	3	2	2
No. laboratories after outlier elimination	1	3	4	4	4	3	4	4	2	2	4	4
No. values > LOD after outlier elimination	3	7	8	9	9	7	10	10	4	6	10	10

In grey results from less than 3 laboratories or released quantities below the limit of quantification

Results after elimination of statistical outliers	Fluoranthene		Pyrene		Benzo(a)anthracene		Chrysene		Benzo(b)fluoranthene		Benzo(k)fluoranthene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	1.3E-02	5.7E-02	1.1E-02	4.2E-02	1.6E-03	1.6E-03	1.2E-03	1.2E-03	7.9E-04	1.5E-03	4.5E-04	9.2E-04
SD_R	9.2E-03	4.5E-02	6.3E-03	3.0E-02	1.5E-03	1.4E-03	1.1E-03	1.3E-03	4.3E-04	1.0E-03	2.7E-04	7.9E-04
SD_r	1.9E-03	1.3E-02	1.6E-03	4.6E-03	3.1E-04	3.7E-04	2.3E-04	1.1E-04	1.9E-04	2.2E-04	1.2E-04	1.4E-04
RSD_R	70%	80%	59%	71%	94%	86%	91%	107%	55%	66%	58%	86%
RSD_r	14%	23%	15%	11%	20%	22%	19%	10%	24%	15%	25%	15%
No. laboratories with results > LOD	6	6	6	6	6	6	6	6	6	6	6	6
No. outliers						1		2				
No. laboratories after outlier elimination	6	6	6	6	6	5	6	4	6	6	6	6
No. values > LOD after outlier elimination	15	15	15	15	15	12	15	9	15	15	15	15

Results after elimination of outliers,L3 and L14	Fluoranthene		Pyrene		Benzo(a)anthracene		Chrysene		Benzo(b)fluoranthene		Benzo(k)fluoranthene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	1.2E-02	4.7E-02	1.0E-02	3.6E-02	1.2E-03	1.9E-03	8.8E-04	1.4E-03	8.5E-04	1.3E-03	3.8E-04	7.0E-04
SD_R	5.9E-03	1.8E-02	4.7E-03	9.0E-03	1.2E-03	1.4E-03	7.7E-04	1.4E-03	4.2E-04	5.0E-04	1.5E-04	4.6E-04
SD_r	2.0E-03	1.0E-02	1.9E-03	4.2E-03	2.3E-04	3.9E-04	1.6E-04	8.4E-05	1.6E-04	1.4E-04	6.0E-05	1.0E-04
RSD_R	50%	39%	45%	25%	102%	76%	87%	102%	50%	37%	39%	65%
RSD_r	17%	22%	18%	12%	20%	21%	18%	6%	18%	11%	16%	14%
No. laboratories with results > LOD	6	6	6	6	6	6	6	6	6	6	6	6
No. outliers	2	2	2	2	2	2	2	3	2	2	2	2
No. laboratories after outlier elimination	4	4	4	4	4	4	4	3	4	4	4	4
No. values > LOD after outlier elimination	10	10	10	10	10	10	10	7	10	10	10	10

Results after elimination of statistical outliers	Benzo(a)pyrene		Indeno(1,2,3-cd)pyrene		Dibenzo(a,h)anthracene		Benzo(ghi)perylene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	6.0E-04	1.0E-03	2.4E-04	4.2E-04	1.5E-04	3.8E-04	3.0E-04	5.9E-04
SD_R	4.5E-04	1.0E-03	1.5E-04	1.7E-04	1.7E-04	4.4E-04	2.3E-04	4.2E-04
SD_r	1.6E-04	2.0E-04	5.6E-05	7.4E-05	3.6E-05	6.0E-05	6.0E-05	1.1E-04
RSD_R	75%	97%	63%	41%	114%	114%	77%	72%
RSD_r	27%	20%	24%	18%	24%	16%	20%	19%
No. laboratories with results > LOD	6	6	6	5	6	4	6	4
No. outliers								
No. laboratories after outlier elimination	6	6	6	5	6	4	6	4
No. values > LOD after outlier elimination	15	15	14	13	14	8	14	12

Results after elimination of outliers,L3 and L14	Benzo(a)pyrene		Indeno(1,2,3-cd)pyrene		Dibenzo(a,h)anthracene		Benzo(ghi)perylene	
	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10	L/S = 2	L/S = 10
Mean (mg/kg dry)	5.2E-04	7.2E-04	2.6E-04	4.7E-04	8.3E-05	1.0E-04	2.4E-04	3.9E-04
SD_R	3.6E-04	3.9E-04	1.6E-04	1.4E-04	7.6E-05	6.4E-05	1.8E-04	1.3E-04
SD_r	1.5E-04	1.4E-04	6.2E-05	7.9E-05	3.0E-05	5.6E-06	5.9E-05	8.3E-05
RSD_R	69%	55%	61%	29%	91%	62%	73%	33%
RSD_r	28%	20%	24%	17%	37%	5%	25%	21%
No. laboratories with results > LOD	6	6	6	5	6	4	6	4
No. outliers	2	2	2	2	2	1	2	1
No. laboratories after outlier elimination	4	4	4	3	4	3	4	3
No. values > LOD after outlier elimination	10	10	10	9	10	5	10	9

Annex 7.3. WI 17731 (content of organic substances)

ASPHALT AGGREGATE

Results after elimination of statistical outliers	Hidrocarbons	Naphthalene	Acenapthtylene	Acenapthene	Fluorene	Phenanthrene	Anthracene	Fluoranthene	Pyrene
Mean (mg/kg dry)	1879	0.57	0.50	5.1	4.7	21	6.0	70	49
SD_R	856	0.35	0.20	0.88	2.3	1.4	1.1	31	20
SD_r	116	0.039	0.11	0.15	0.25	0.82	0.31	3.6	2.3
RSD_R	46 %	62 %	40 %	17 %	49 %	7 %	19 %	45 %	42 %
RSD_r	6 %	7 %	21 %	3 %	5 %	4 %	5 %	5 %	5 %
No. laboratories with results > LOD	7	4	5	6	6	6	6	6	6
No. outliers			1	1		2	1		
No. laboratories after outlier elimination	7	4	4	5	6	4	5	6	6
No. values > LOD after outlier elimination	18	10	10	14	17	11	13	17	17

Results after elimination of statistical outliers	Benzo(a)anthracene	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene	Dibenzo(a,h)anthracene	Benzo(ghi)perylene
Mean (mg/kg dry)	17	15	17	8.8	12	9.3	2.0	8.8
SD_R	1.3	2.0	3.8	2.6	1.6	6.5	0.33	7.9
SD_r	0.89	1.0	0.87	0.56	0.66	0.65	0.11	0.81
RSD_R	8 %	14 %	23 %	30 %	13 %	70 %	17 %	89 %
RSD_r	5 %	7 %	5 %	6 %	5 %	7 %	6 %	9 %
No. laboratories with results > LOD	6	6	6	6	6	6	6	6
No. outliers	1	1	1	1	1		1	
No. laboratories after outlier elimination	5	5	5	5	5	6	5	6
No. values >LOD after outlier elimination	14	14	14	14	14	17	13	17

RECYCLED AGGREGATE

Results after elimination of statistical outliers	Naphthalene	Acenapthtylene	Acenapthene	Fluorene	Phenanthrene	Anthracene	Fluoranthene	Pyrene	Benzo(a)anthracene
Mean (mg/kg dry)	1.08	0.16	10.7	11	65	13	126	79	41
SD_R	0.48	0.043	1.4	2.8	14.1	3.1	36	18	10.9
SD_r	0.11	0.027	1.1	1.7	7.6	2.1	13	7.2	4.9
RSD_R	44 %	27 %	13 %	25 %	22 %	24 %	28 %	22 %	27 %
RSD_r	10 %	17 %	11 %	15 %	12 %	16 %	10 %	9 %	12 %
No. laboratories with results > LOD	5	5	6	6	6	6	6	6	6
No. outliers	1								
No. laboratories after outlier elimination	4	5	6	6	6	6	6	6	6
No. values > LOD after outlier elimination	10	13	17	17	17	17	17	17	17

Results after elimination of statistical outliers	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene	Dibenzo(a,h)anthracene	Benzo(ghi)perylene
Mean (mg/kg dry)	37	37	17	28	15	3.7	10.0
SD_R	9.2	10.0	3.3	8.3	7.7	1.2	6.6
SD_r	4.6	3.7	1.1	2.6	1.4	0.3	0.82
RSD_R	25 %	27 %	19 %	29 %	51 %	31 %	66 %
RSD_r	13 %	10 %	6 %	9 %	9 %	9 %	8 %
No. laboratories with results > LOD	6	6	6	6	6	6	6
No. outliers			1			1	
No. laboratories after outlier elimination	6	6	5	6	6	5	6
No. values > LOD after outlier elimination	17	17	14	17	17	13	17

Results after elimination of statistical outliers	PCB-28	PCB-52	PCB-101	PCB-118	PCB-138	PCB-153	PCB-180
Mean (mg/kg dry)	5.5E-03	3.5E-03	1.0E-02	6.1E-03	1.3E-02	1.3E-02	8.0E-03
SD_R	8.1E-03	5.2E-03	5.4E-03	3.3E-03	5.8E-03	8.4E-03	6.7E-03
SD_r	1.0E-04	0.0E+00	2.1E-03	2.6E-04	2.9E-03	2.2E-03	4.6E-04
RSD_R	148%	146%	52%	53%	44%	63%	84%
RSD_r	2%		20%	4%	22%	17%	6%
No. laboratories with results > LOD	2	2	3	3	3	3	2
No. outliers							
No. laboratories after outlier elimination	2	2	3	3	3	3	2
No. values>LOD after outlier elimination	4	4	7	5	7	7	4

In grey results from less than 3 laboratories or quantities below the limit of quantification

Annex 7.4. WI 17732 (analysis of organic substances in eluates)

RENDER

Results after elimination of statistical outliers	Diuron	Terbutryn	Methylisothiazolinone, MIT	Benzisothiazolinone, BIT	Octylisothiazolinone, OIT	Carbendazim
Mean (µg/l)	2708	772	10358	15289	3105	1398
SD_R	1119	169	1666	4715	749	202
SD_r	41	17	928	1814	43	14
RSD_R	41 %	22 %	16 %	31 %	24 %	14 %
RSD_r	1.5 %	2.2 %	9.0 %	11.9 %	1.4 %	1.0 %
No. laboratories with results > LOD	3	3	3	3	3	2
No. outliers						
No. laboratories after outlier elimination	3	3	3	3	3	2
No. values > LOD after outlier elimination	8	9	9	9	9	6

In grey results from less than 3 laboratories or quantities below the limit of quantification

ASPHALT AGGREGATE

Results after elimination of statistical outliers	Hidrocarbons	Naphthalene	Acenapthtylene	Acenapthene	Fluorene	Phenanthrene	Anthracene	Fluoranthene	Pyrene
Mean (µg/l)	398	0.015	0.16	0.048	0.046	0.24	0.13	1.22	0.96
SD_R	359	0.008	0.13	0.021	0.022	0.11	0.058	0.76	0.51
SD_r	21	0.005	0.031	0.017	0.015	0.11	0.042	0.76	0.51
RSD_R	90%	52%	80%	44%	48%	46%	45%	62%	53%
RSD_r	5%	34%	19%	35%	33%	46%	32%	62%	53%
No. laboratories with results > LOD	4	4	5	4	5	5	5	5	5
No. outliers		1			1		1		
No. laboratories after outlier elimination	4	3	5	4	4	4	4	5	5
No. values > LOD after outlier elimination	9	9	15	12	14	12	12	15	15

In grey results from less than 3 laboratories or quantities below the limit of quantification

Results after elimination of statistical outliers	Benzo(a)anthracene	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene	Dibenzo(a,h)anthracene	Benzo(ghi)perylene
Mean (µg/l)	1.09	1.10	2.07	1.12	1.95	1.23	0.33	1.12
SD_R	0.55	0.45	0.48	0.64	0.40	0.28	0.12	0.53
SD_r	0.38	0.41	0.38	0.16	0.091	0.057	0.058	0.16
RSD_R	50%	41%	23%	57%	21%	22%	37%	47%
RSD_r	35%	37%	18%	14%	5%	5%	18%	14%
No. laboratories with results > LOD	5	5	5	5	5	5	5	5
No. outliers			1	1	2	2	1	1
No. laboratories after outlier elimination	5	5	4	4	3	3	4	4
No. values >LOD after outlier elimination	15	15	12	12	9	9	12	12

RECYCLED AGGREGATE

Results after elimination of statistical outliers	Naphthalene	Acenapthtylene	Acenapthene	Fluorene	Phenanthrene	Anthracene	Fluoranthene	Pyrene	Benzo(a)anthracene
Mean (µg/l)	0.043	0.061	0.62	2.6	3.5	2.2	20	17	4.5
SD_R	0.022	0.046	0.26	2.9	1.4	1.3	14	9.5	2.5
SD_r	0.014	0.012	0.22	0.24	1.3	0.85	4.7	3.7	1.0
RSD_R	50 %	75 %	42 %	109 %	41 %	60 %	70 %	56 %	55 %
RSD_r	33 %	20 %	36 %	9 %	37 %	40 %	23 %	22 %	23 %
No. laboratories with results > LOD	4	5	5	5	5	4	5	5	5
No. outliers				1					1
No. laboratories after outlier elimination	4	5	5	4	5	4	5	5	4
No. values after > LOD outlier elimination	11	15	15	12	15	12	15	15	12

Results after elimination of statistical outliers	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene	Dibenzo(a,h)anthracene	Benzo(ghi)perylene
Mean (µg/l)	5.7	5.1	3.1	4.3	2.2	0.85	1.9
SD_R	2.5	2.3	1.6	1.3	1.1	0.41	0.68
SD_r	1.9	1.5	0.80	1.3	0.91	0.34	0.60
RSD_R	44 %	44 %	53 %	31 %	49 %	48 %	36 %
RSD_r	33 %	30 %	26 %	31 %	40 %	41 %	31 %
No. laboratories with results > LOD	5	5	5	5	5	5	5
No. outliers							
No. laboratories after outlier elimination	5	5	5	5	5	5	5
No. values > LOD after outlier elimination	15	15	15	15	15	15	15

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