Validation programme and support by the JRC

Silvia García Ruiz, Thomas Linsinger, Fernando Cordeiro

The European Commission's science and knowledge service
Joint Research Centre

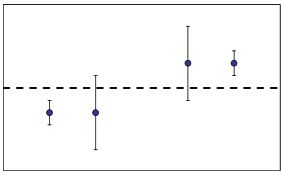
CEN/TC 351 conference, 17 October 2019, Vilnius (LT)

1

Need for validation studies

- CEN/TC 351 "Construction products" – Assessment of release of dangerous substances (WG1 WG5 into soil and waters)

How reliable are test results?



2

2

What is validation?

Validation of a method=
Is it fit for its intended use?

Precision:

- **Repeatability**
  = method, lab, operator, equipment within short time intervals
- **Reproducibility**
  = method,
  ≠ laboratories, operators, equipment

Method performance parameters typically evaluated during validation	
Working range	Single laboratory validation
Linearity	
Limit of Detection (LOD)	
Limit of Quantification	
Selectivity	*"Robustness validation"*
Specificity	
Trueness	
Repeatability	**Inter-laboratory validation**
Intermediate precision	
Reproducibility	

3

3

Challenges of this study

- Horizontal methods -> broad range of materials and analytes
- Test materials: homogeneity, levels of substances, stability…
- Finding (voluntary) participants: extensive plan of analyses

4

4

Test methods and scope

- Scope: analytes, levels, matrices, target uncertainty

Methods for leaching, content and eluate analysis	
CEN/TS 16637-2	Horizontal dynamic surface leaching test (DSLT)
CEN/TS 16637-3	Horizontal up-flow percolation test (column test)
1st stage: inorganic substances	
CEN/TS 17195	Analysis of inorganic substances in eluates
CEN/TS 17196	Digestion by aqua regia
CEN/TS 17201	Methods for analysis of aqua regia digests

5

5

Inorganic substances: Materials tested

CEN/TS 16637-2 (DSLT)		CEN/TS 16637-3 (percolation)	
Monolithic Copper Slag (MCS)		Crushed Copper Slag (CCS)	
Cement Stabilised Coal Fly Ash (CSC)		Recycled concrete (RC)	
Autoclaved Aerated Concrete (AAC)		Crushed Masonry (CM)	

6

6

Inorganic substances: Materials tested

CEN/TS 17195 (eluate analysis)		CEN/TS 17196 (aqua regia)	
Leachates from MCS, CSC, AAC		Ground copper slag, RC and AAC	
Eluates from CCS, RC, CM		Polyethylene (LDPE)	

7 European Commission

7

Inorganic substances : Participants

Method	No. participants
Dynamic surface leaching (DSLT)	**14-18**
Up-flow percolation (column test)	**13**
Eluate analysis	**16**
Aqua regia digestion	**12**

- Geographical distribution: mainly Germany and The Netherlands; also Belgium, Finland, Greece, Slovenia and Sweden

8 European Commission

8

Inorganic substances : Measurements

- **Arsenic, barium, calcium, (chloride,) chromium, fluoride, molibdenum, lead, sulfur, antimony, selenium, sulphate, strontium, vanadium, zinc**
- Triplicate analysis and Quality Control
- All measurements under repeatability conditions

Evaluation of results

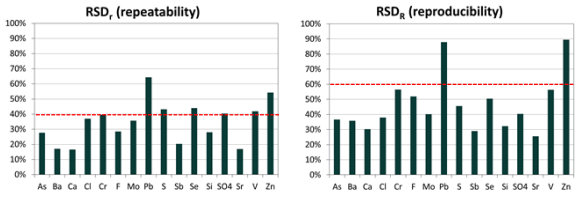
- **ISO 5725-2:** Repeatability and reproducibility of a standard measurement method

9 European Commission

9

CEN/TS 16637-2 Horizontal dynamic surface leaching test

Example: Cement stabilised coal fly ash - cumulative 64 days



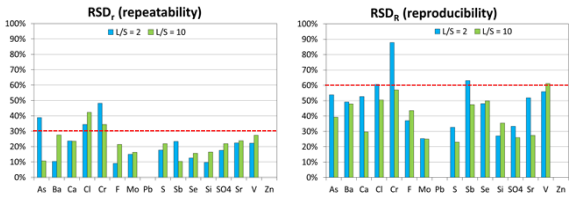
Duration from start of test: 6 hours, 1 day, 2d 6h, 4 d, 9 d, 16 d, 36 d and **64 d**

10 European Commission

10

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

Example: Crushed masonry - cumulative L/S = 2 and 10



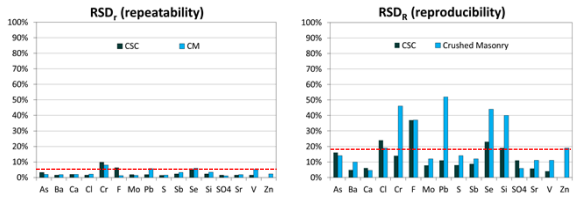
Cumulative liquid to solid ratio (l/kg) 0.1, 0.2, 0.5, 1, **2**, 5 and **10**

11 European Commission

11

17195 Analysis inorganic substances in eluates

Example: Cement stabilised coal fly ash, crushed masonry



12 European Commission

12

Inorganic substances: summary

Typical values*	16637-2 (DSLT)	16637-3 (percolation)
Repeatability standard deviation, RSD_r	20-45 %	15-30 %
Reproducibility standard deviation, RSD_R	40-65 %	30-60 %

* most of median values, rounded

- **Variability** of results covers:
  material homogeneity, sample preparation and measurement

13

13

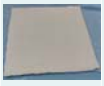
Organic substances: Test methods

Methods for leaching, content and eluate analysis	
CEN/TS 16637-2	Horizontal dynamic surface leaching test (DSLT)
CEN/TS 16637-3	Horizontal up-flow percolation test (column test)
2nd stage: organic substances	
CEN/TS 17332	Analysis of organic substances in eluates
CEN/TS 17331	Content of organic substances
WI 351034	PAHs
WI 351035	Biocides

14

14

Organic substances: Materials tested

CEN/TS 16637-2 (DSLT)		CEN/TS 16637-3 (percolation)	
Material	***Analytes***	***Material***	***Analytes***
Render 	Biocides	Asphalt aggregate 	PAHs, mineral oil
Sealant 	Phthalates	Recycled aggregate 	PCBs, PAHs

15

15

Organic substances: Materials tested

CEN/TS 17331 (extraction)		CEN/TS 17332 (eluate analysis)	
Material	***Analytes***	***Material***	***Analytes***
		Render	Biocides
		Sealant	Phthalates
Asphalt aggregate	PAHs, mineral oil	Asphalt aggregate	PAHs, mineral oil
Recycled aggregate	PCBs, PAHs	Recycled aggregate	PCBs, PAHs

* PAHs: Polycyclic aromatic hydrocarbons
PCBs: Polychlorinated biphenyls

16

16

Organic substances: participants

- **6-7** laboratories per method/material

Next steps

- ONGOING Evaluation of results - Organic substances
- EN standards

17

17

Thanks

- Material providers
- Participant laboratories
- CEN/TC 351

Questions?

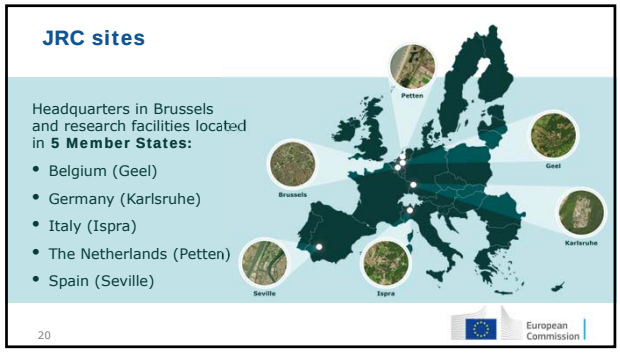
Silvia.GARCIA-RUIZ@ec.europa.eu

18

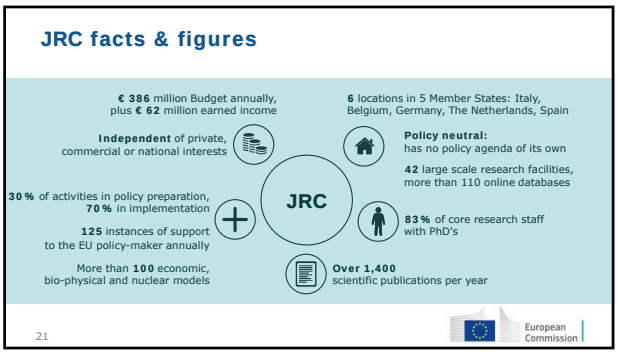
18


JRC's mission
"As the science and knowledge service of the Commission our mission is to support EU policies with independent evidence throughout the whole policy cycle"
19
European Commission

19


JRC sites
Headquarters in Brussels and research facilities located in 5 Member States:
Belgium (Geel)
Germany (Karlsruhe)
Italy (Ispra)
The Netherlands (Petten)
Spain (Seville)
Petten
Geel
Brussels
Karlsruhe
Seville
Ispra
20
European Commission

20


JRC facts & figures
€ 386 million Budget annually, plus € 62 million earned income
6 locations in 5 Member States: Italy, Belgium, Germany, The Netherlands, Spain
Independent of private, commercial or national interests
Policy neutral: has no policy agenda of its own
42 large scale research facilities, more than 110 online databases
30 % of activities in policy preparation, 70 % in implementation
JRC
83 % of core research staff with PhD's
125 instances of support to the EU policy-maker annually
More than 100 economic, bio-physical and nuclear models
Over 1,400 scientific publications per year
21
European Commission

21


Stay in touch
EU Science Hub: ec.europa.eu/jrc
Twitter: @EU_ScienceHub
Facebook: EU Science Hub - Joint Research Centre
LinkedIn: Joint Research Centre
YouTube: EU Science Hub
22
European Commission

22