

Environmental Metrology Unit: The SIT Center n.211

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ISPRA
***Istituto Superiore per la Protezione e la Ricerca
Ambientale***

RMs for environmental monitoring

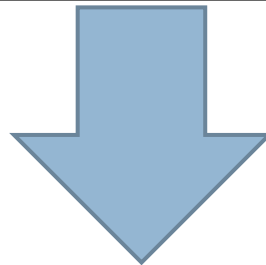
- **Are used for:**
 - **internal/external QC procedures**
 - **methods validation and collaborative studies**
 - **calibration of instruments**

- **Customers of ISPRA RMs are the Italian regional laboratories (ARPA/APPA)**



ISPRA Reference Materials

Availability of RMs with reliable property values is a key factor for reliable test data



**ISO /IEC 17025 and ISO
Guide 34 Accreditation**





ISPRA Accreditation Scope

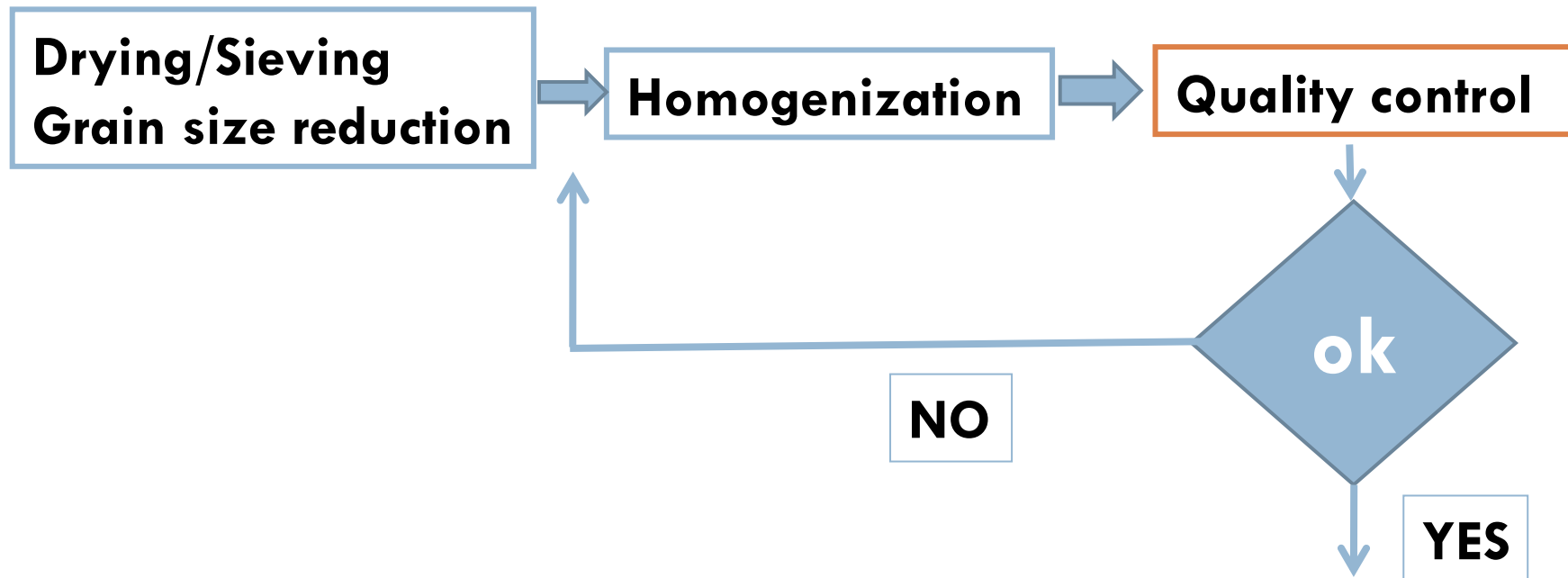
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Measurand	Accreditation range (µg/g)	Expanded Uncertainty (µg/g)
As in soil/sediment	10.0 ÷ 65.0	0.7 ÷ 4.6
Cd in soil/sediment	0.20 ÷ 30.0	0.02 ÷ 2.1
Ni in soil/sediment	10.0 ÷ 300	0.7 ÷ 21
Pb in soil/sediment	20.0 ÷ 200	1.4 ÷ 14
Cu in soil/sediment	10.0 ÷ 200	0.7 ÷ 14
Co in soil/sediment	10.0 ÷ 200	0.7 ÷ 14
Mn in soil/sediment	10.0 ÷ 450	0.7 ÷ 32



Planning

- Intended use of the RM
- Need of a feasibility study
- Planning of all production phases
- Worker safety procedures

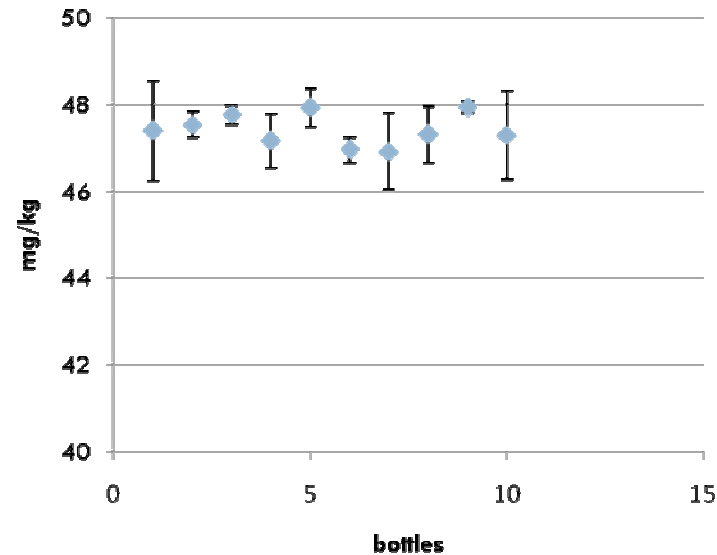


Homogeneity study

Method
(ISO Guide 35:2006)

- **10 bottles**
- **3 replicates for each bottle**
- **Measurements in repeatability conditions**
- **ANOVA to calculate**
 - **Method uncertainty**
 - **Between bottle heterogeneity**

APAT-RM014 – As mass fractions



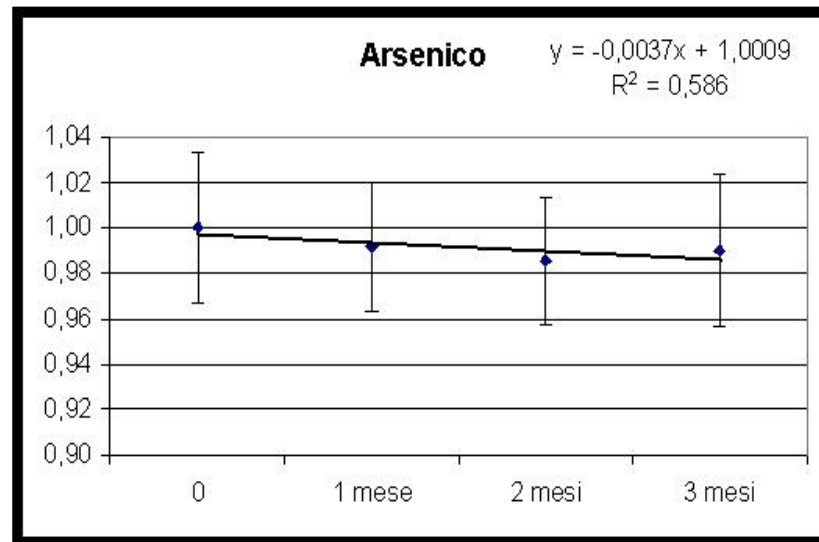
$$u_{bb} = 0.287 \text{ mg/kg}$$

Stability study

Method (ISO Guide 35:2006)

- **Isochronous method**
- **2 temperatures (40 and 20 °C)**
- **Reference temperature (-18 °C)**
- **5 bottles for each period**
- **Measurements in repeatability conditions**
- **ANOVA**
- **Linear regression on normalized data**

APAT-RM014 – As mass fractions



$u_{st} = 0.276 \text{ mg/kg}$



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QC & Homogeneity/Stability Study Measurement Methods

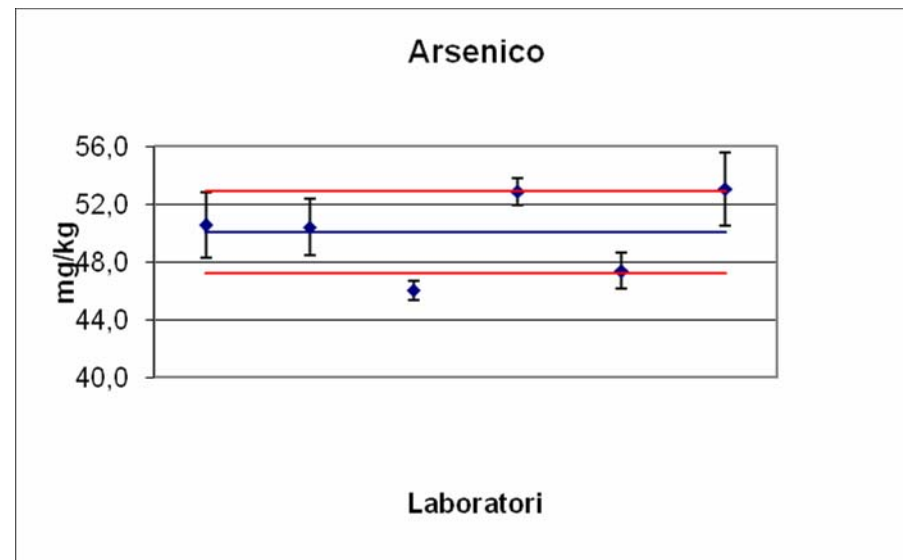
ISO/IEC 17025 requirements	Method characteristics
Selection of methods	Fast measurement methods with high repeatability (CHN, EDXRF)
Method validation	Selectivity & calibration. Repeatability is calculated during each study
Estimation of measurement uncertainty	Measurement uncertainty is not used in ANOVA & regression
Results traceability	It is not needed in relative measurements



Method
(ISO Guide 35:2006)

- 1 lab using 1 method
- 1 lab using different methods
- N labs using 1 method
- N labs using different methods
- Metrological traceability
- Measurements in repeatability conditions
- **Assignment of property values**

APAT-RM014 – As mass fractions



$$u_{car} = 1.83 \text{ mg/kg}$$



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Measurement Method for Characterization in ISPRA Lab

ISO/IEC 17025 requirements	Measurement method
Selection of methods	EN ISO 13656:2002 using external calibration of ICP-MS
Method validation	Full validation (selectivity, linearity, LOD, bias, roughness)
Estimation of measurement uncertainty	Uncertainty calculation as required by GUM
Results traceability	Metrological traceability





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Uncertainty budget & RM assigned value

Quantity x_i	Estimate x_i	Standard uncertainty $u(x_i)$	Sensitivity coefficient c_i	Contribution to the standard uncertainty $u_i(y)$
c_{car}	50.123 mg/kg	1.832 mg/kg	1	1.832 mg/kg
δc_{bb}	0.0 mg/kg	0.287 mg/kg	1	0.287 mg/kg
δc_{msts}	0.0 mg/kg	0.276 mg/kg	1	0.276 mg/kg
c_{RM}	50.123 mg/kg			1.874 mg/kg

$$U = 2 u_{RM}$$



$$c_{RM} = 50.1 \pm 3.7 \text{ mg/kg}$$





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Future....

- Ions in water solutions
- Cr, Hg in soil and sediments
- Organic substances in soil and sediments
- RMs Certification Committee

Collaborations with other RMs producers





Thank you for your attention



Short movie

"Reference Materials:
Looking Forward"

*Directed by:
Marco Pisapia (ISPRA)*



Short movie

Produced by ISPRA:

- *DIPARTIMENTO PER LE ATTIVITA' BIBLIOTECARIE, DOCUMENTALI E PER L'INFORMAZIONE – Servizio Portale Web*
- *DIPARTIMENTO STATO AMBIENTE E METROLOGIA AMBIENTALE – Servizio Metrologia Ambientale*

