

CERTIFIED REFERENCE MATERIAL**CERTIFICATE OF ANALYSIS**

BCR No 141			
Trace Elements in a Calcareous Loam Soil			
Element	Mass fraction (based on dry mass)		Number of accepted sets of results p
	Certified value ⁽¹⁾ expressed as $\mu\text{g.g}^{-1}$	95 % confidence interval ⁽²⁾ expressed as $\mu\text{g.g}^{-1}$	
Cd	0.36	± 0.10	8
Cu	32.6	± 1.4	15
Hg	56.8×10^{-3}	$\pm 4.3 \times 10^{-3}$	9
Pb	29.4	± 2.6	15
Zn	81.3	± 3.7	14

⁽¹⁾ This value is the unweighted mean of p accepted sets of results.
⁽²⁾ The 95 % confidence interval is a measure of the uncertainty and is applicable when the reference material is used for calibration purposes.
When the reference material is used to assess the performance of a method, the user should refer to the recommendations laid down in the last chapter (instructions for use) of the certification report.

DESCRIPTION OF THE SAMPLE

The material consists of a homogeneous powder (particles have passed a sieve with apertures smaller than 90 μm). The material contains the following major and minor elements (not certified) expressed as their oxides (cg.g^{-1}):

Loss at 900 °C: 20.65

SiO₂: 42.58 MgO: 1.19 TiO₂: 0.47 P₂O₅: 0.16 Na₂O: 0.43

CaO: 17.98 Al₂O₃: 10.56 Fe₂O₃: 3.74 K₂O: 1.56

Additional information is presented on the attached sheet.

The RM is available in units of 50 g.

The matrix stability has been checked over a period of approximately 5 years. A change in matrix composition could not be detected over that period.

INSTRUCTIONS FOR USE

The moisture content can be determined by drying an aliquot of the sample for 24 h over phosphorus pentoxide. The sample for analysis should be taken as it is.

The bottle should be stored preferably in a dark and cool place.

Once the bottle has been opened, the material is susceptible to contamination (e.g. laboratory dust or vapours) or losses.

The recommended minimum sample intake is 100 mg.

As the material may segregate partly upon storage, remixing of the bottle contents prior to taking a sample is necessary. A poly tetrafluoro ethene ball is added for that purpose. Shaking during 2 - 4 minutes is usually sufficient.

PARTICIPATING LABORATORIES

- Joint Research Centre, Ispra (Italy)
- Gesellschaft für Strahlen- und Umweltforschung, Neuherberg (F.R. Germany)
- Centre d'Etude de l'Energie Nucléaire CEN/SCK, Mol (Belgium)
- Centre de Recherches Péetrographiques et Géochimiques, Vandœuvre-lès-Nancy (France)
- Centro di Radiochimica e Analisi per Attivazione del CNR, Pavia (Italy)
- ECN, Netherlands Energy Research Foundation — Research Centre, Petten (The Netherlands)
- INRA, Station d'Agronomie «La Grande Ferrade», Pont-de-la-Mayo (France)
- Institut Fresenius, Taunusstein (F.R. Germany)
- Instituut voor Bodemvruchtbaarheid, Haren (The Netherlands)
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- Produits Chimiques Ugine Kuhlmann, Courbevoie (France)
- Risø National Laboratory, Isotope Division, Roskilde (Denmark)
- Service Central d'Analyse CNRS, Vernaison (France)
- Staatliche Landwirtschaftliche Untersuchungs- und Forschungsanstalt, Augustenberg, Karlsruhe (F.R. Germany)
- The Agricultural Institute, Johnstown Castle Research Centre, Wexford (Ireland)
- The Macaulay Institute for Soil Research, Aberdeen (United Kingdom)
- TNO, Technology for Society Division, Delft (The Netherlands)
- Universitaire Instellingen, Antwerpen (Belgium)
- Universität Ulm (F.R. Germany)
- Water Research Centre, Stevenage (United Kingdom)
- Community Bureau of Reference, Brussels (Belgium)

METHODS USED

A wide range of sample pretreatment methods was applied if necessary: among others wet digestion like treatment with nitric, hydrochloric and hydrofluoric acid at low temperatures or in a pressurised bomb, treatment with sulphuric and perchloric acid followed by evaporation with hydrofluoric acid, repeated treatment with hydrofluoric acid followed by a wet oxidative attack or special destruction techniques.

Methods of final determination were:

Instrumental Neutron Activation (Cu, Zn)

(Hydride, Flame or Graphite Furnace) Atomic Absorption Spectrometry (Cd, Cu, Hg, Ni, Pb, Zn)

Inductively Coupled Plasma Spectrometry (Cd, Cu, Ni, Pb, Zn)

Neutron Activation with Radiochemical Separation (Cd, Hg)

Differential Pulse Anodic Stripping Voltammetry (Cd, Cu, Ni, Pb)

Isotope Dilution Mass Spectrometry (Cd, Pb)

Arc Emission Spectrometry (Cd, Cu, Ni, Pb, Zn)

Spectrophotometry (Cu, Ni)

Atomic Fluorescence Spectrometry (Hg)

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NOTE

A detailed technical report on the analysis procedure and the treatment of the analytical data is supplied with each sample.

INFORMATION SHEET ATTACHED TO THE CERTIFICATE OF BCR N° 141

Additional information (not certified) on various contents is given here. The data presented here are average values of sets of results obtained by various techniques in various laboratories.

The aqua regia digestion technique is described in detail in the certification report.

Element	Mass fraction expressed as: $\mu\text{g.g}^{-1}$		Number of Individual sets
	Content	Standard Deviation	
Aqua regia soluble Cd	0.30	0.13	11
Aqua regia soluble Cr	53	9	11
Aqua regia soluble Cu	31.2	2.3	11
Aqua regia soluble Mn	512	63	6
Aqua regia soluble Ni	28.0	4.9	12
Aqua regia soluble Pb	26.3	5.8	11
Aqua regia soluble Zn	70	11	10
Total Co	9.2	1.1	7
Total Cr	75.0	10.4	15
Total Mn	547	32	6
Total Ni	30.9	3.2	13
Total Se	0.160	0.025	5