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COMMISSION OF THE EUROPEAN COMMUNITIES

COMMUNITY BUREAU OF REFERENCE - BCR

CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCR No 146

Trace Elements in a Sewage Sludge of Mainly Industrial Origin

Element	Mass fraction (based on dry mass)		Number of accepted sets of results p
	Certified value ⁽¹⁾ expressed as $\mu\text{g/g}$	95% confidence interval ⁽²⁾ expressed as $\mu\text{g/g}$	
Cd	77.7	- 2.6	17
Co	11.8	- 0.7	9
Cu	934	- 24	15
Mn	588	- 24	15
Hg	9.49	- 0.76	13
Ni	280	- 18	11
Pb	1270	+ 28	12
Zn	4059	- 90	15

⁽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: 37.7

SiO₂: 22.8 MgO: 3.30 TiO₂: 2.91 P₂O₅: 5.90 Na₂O: 0.30

CaO: 14.2 Al₂O₃: 9.00 Fe₂O₃: 2.65 K₂O: 0.58

Additional information is presented on the attached sheet.

The RM is available in units of 50 g.

The stability of this material has been tested over a 4 year period by determining the contents of Cd, Cr, Pb, Zn, Cu, Ni and Mn using the aqua regia digestion. No evidence of a change in the contents could be found.

INSTRUCTIONS FOR USE

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

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

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

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 polytetrafluoro ethene ball is added for that purpose. Shaking during 2 - 4 minutes is usually sufficient.

WARNING

When working with this material the same health precautions should be applied as when working with real sludges.

PARTICIPATING LABORATORIES

- Joint Research Centre, Ispra (Italy)
- Gesellschaft für Strahlen- und Umweltforschung, Neuherberg (F.R. Germany)
- 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 Maye (France)
- Institut Fresenius, Taunusstein (F.R. Germany)
- Instituut voor Bodemvruchtbaarheid, Haren (The Netherlands)
- Instituut voor Nucleaire Wetenschappen, RUG, Gent (Belgium)
- Istituto Italiano di Idrobiologica CNR, Pallanza (Italy)
- Kernforschungsanlage Jülich, Institut für angewandte und physikalische Chemie, Jülich (F.R. Germany)
- Laboratorium voor Analytische en Agrochemie, RUG, Gent (Belgium)
- Landwirtschaftskammer Westfalen-Lippe, Joseph-König-Institut, Münster (F.R. Germany)
- Max Planck Institut für Metallforschung, Institut für Reinststoffanalyse, Schwäbisch Gmünd (F.R. Germany)
- 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)
- 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, Co, Mn, Zn)

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

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

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° 146

Additional information (not certified) on various contents is presented here. The data are mean values of various 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	76.5	4.9	12
Aqua regia soluble Co	9.9	0.8	5
Aqua regia soluble Cr	769	79	8
Aqua regia soluble Cu	921	47	11
Aqua regia soluble Mn	555	32	9
Aqua regia soluble Ni	269	17	13
Aqua regia soluble Pb	1255	41	10
Aqua regia soluble Zn	4002	296	10
Total Cr	784	37	13
Total Se	1.7	0.4	6