

Bundesanstalt für Materialforschung und -prüfung (BAM)

# Certified Reference Material BAM-U117

Total Cyanide in Soil

# **Certified Value**

Measurand	Mass fraction <sup>1)</sup> in mg/kg	Uncertainty <sup>2)</sup> in mg/kg		
Total cyanide	11.0	0.7		
<sup>1)</sup> Unweighted mean value of the means of accepted sets of data (consisting of 4 single results corrected to the dry mass content of the material at $(105 \pm 2)$ °C), each set being obtained by a different laboratory and/or a different method of measurement.				
<sup>2)</sup> Estimated expanded uncertainty <i>U</i> with a coverage factor of $k = 2$ , corresponding to a level of confidence of approx. 95 %, as defined in the Guide to the expression of uncertainty in measurement, (GUM, ISO/IEC Guide 98-3:2008).				

This certificate is valid for a period of 12 months beginning with the dispatch of the reference material from BAM.

Date of dispatch:

Sample-No.:

# Additional material information

CRM BAM-U117 is available as a powder with particle sizes below 125  $\mu$ m and is supplied in 100 mL amber glass bottles containing (77 ± 1) g.

The dry mass content of the bottled soil material at the time of certification was (99.64  $\pm$  0.27) %, corresponding to a drying temperature of (105  $\pm$  2) °C. The indicated uncertainty represents the standard deviation of the mean of 15 laboratory results. The indicated value of the dry mass content should be regarded as being indicative.

#### Recommended Use

The intended purpose of CRM BAM-U117 is the verification of analytical results obtained for the mass fraction of total cyanide in soils and soil-like materials applying the standardized procedures DIN ISO 11262:2012 [1] and DIN EN ISO 17380:2013 [2]. As any reference material, it can also be used for routine performance checks (quality control charts) or validation studies.

### Material Description

The CRM BAM-U117 represents a mixture of a sandy soil collected from a contaminated former gasworks area in the Berlin region (Germany) and an unpolluted sandy soil from Nalaikh region (Mongolia). The raw materials were dried at ambient air to constant mass, and afterwards the fractions passing a 2 mm screen were ground to particle sizes below 125  $\mu$ m. Before bottling, the two fractions were mixed and homogenized.

For the main matrix constituents of the bottled material the following non-certified results were obtained by X-ray fluorescence analysis (WD-XRF):

Element	Si	Al	К	Na	Ca	Fe
Mass fraction in %	35.6	6.9	2.9	2.5	0.8	0.8

Mass fractions of other elements detectable with WD-XRF were less than 0.2 %.

Further informative analytical results obtained during sample characterization from original material BAM-U116/CGL306:

Parameter	Mass fraction (in %)	Analytical method
Loss on ignition at 550 °C	0.9	EN 15935 [3]
Total carbon (TC)	0.2	ISO 10694 [4]

#### Instructions for Use

Before withdrawing a sub-sample, the bottle should be allowed to reach room temperature. Thereafter, the bottle should be closed tightly and stored at  $(4 \pm 2)$  °C. The stability of the reference material is not affected by short periods of handling at ambient temperature during transport and use.

The minimum sample size for a determination is 5 g.

When determining the content of total cyanide, the analytical protocol prescribed by DIN ISO 11262:2012 or DIN EN ISO 17380:2013 must strictly be followed. All analytical results have to be corrected for dry mass content of the material which should be determined according to ISO 11465:1993 [5] using a separate sub-sample. The value given in the table below (99.64 %) should be regarded as being indicative only.

#### Participating Laboratories

AWV- Dr. Busse GmbH, Plauen AZBA Analytisches Zentrum Berlin-Adlershof GmbH, Berlin Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin GEOTAIX Umwelttechnologie GmbH, Würselen ICA - Institut für Chemische Analytik GmbH, Leipzig IFU GmbH Gewerbliches Institut für Fragen des Umweltschutzes, Heitersheim IHU Geologie und Analytik, Gesellschaft für Ingenieur- Hydro- und Umweltgeologie mbH, Stendal Institut Dr. Lörcher und Partner mbB Handelschemiker, Ludwigsburg Institut Dr. Nowak GmbH & Co. KG, Ottersberg Laboratorien Dr. Döring GmbH, Bremen Terrachem-Essen GmbH, Essen Thüringer Umweltinstitut Henterich GmbH, Krauthausen UCL Umwelt Control Labor GmbH, Lünen Wartig Chemieberatung GmbH, Marburg WESSLING GmbH, Weiterstadt

### Storage

CRM BAM-U117 can be shipped at ambient temperature. Upon receipt the material has to be stored at a temperature of  $(4 \pm 2)$  °C in its original tightly closed bottle.

#### Metrological Traceability

The certified mass fraction of total cyanide in CRM BAM-U117 is operationally-defined referring to the analytical protocols prescribed by DIN ISO 11262:2011 and DIN EN ISO 17380:2013. The photometric determination of the liberated cyanide is traceable to the International System of Units (SI) via calibration using substances with certified analyte content.

C M	ertified value ass fraction		Value for information
Line No.	Total cyanide* mg/kg	Method	Dry matter %
1	9.6	DIN EN ISO 17380	99.05
2	9.7	DIN EN ISO 17380	99.3
3	9.8	DIN EN ISO 17380	99.33
4	10.1	DIN ISO 11262	99.5
5	10.4	DIN ISO 11262	99.5
6	10.6	DIN EN ISO 17380	99.6
7	11.0	DIN ISO 11262	99.6
8	11.0	DIN ISO 11262	99.6
9	11.2	DIN EN ISO 17380	99.80
10	11.3	DIN EN ISO 17380	99.8
11	11.6	DIN EN ISO 17380	99.8
12	11.7	DIN ISO 11262	99.8
13	12.1	DIN ISO 11262	99.85
14	12.1	DIN ISO 11262	100
15	13.4	DIN EN ISO 17380	100
М	11.0		99.64
S <sub>M</sub>	1.1		0.27

# **Means of Accepted Data Sets**

\* corrected to the dry mass content of the material at (105  $\pm$  2) °C

The laboratory mean values have been examined statistically to eliminate outlying values. A data set consists of 4 single values of one laboratory.

- M : mean of laboratory means
- $s_{M}$ : standard deviation of laboratory means

#### **Technical Report**

A detailed technical report describing the analysis procedures and the treatment of the analytical data used to certify BAM-U117 is available on request or can be downloaded from BAM website (www.bam.de).

#### References

- [1] DIN ISO 11262:2012: Soil quality Determination of total cyanide (Bodenbeschaffenheit - Bestimmung von Gesamtcyanid)
- [2] DIN EN ISO 17380:2013: Soil quality Determination of total cyanide and easily liberatable cyanide - Continuous-flow analysis method (Bodenbeschaffenheit -Bestimmung des Gehalts an Gesamtcyanid und leicht freisetzbarem Cyanid -Verfahren mittels kontinuierlicher Durchflussanalyse)
- [3] EN 15935:2012: Sludge, treated biowaste, soil and waste Determination of loss on ignition
- [4] ISO 10694:1995: Soil quality Determination of organic and total carbon after dry combustion (elementary analysis)
- [5] ISO 11465:1993: Soil quality Determination of dry matter and water content on a mass basis. Gravimetric method

#### Accepted as BAM-CRM on 2023-11-09

#### Bundesanstalt für Materialforschung und -prüfung (BAM)

Dr. S. Richter Committee for Certification Dr. S. Recknagel Project Coordinator

BAM holds an accreditation as a reference material producer according to ISO/IEC 17034. This accreditation is valid only for the scope as specified in the certificate D-RM-11075-01-00. DAkkS is a signatory of the multilateral agreement (MLA) between EA, ILAC and IAF for mutual acceptance.



This Reference Material is offered by:

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