

Certificate of the Reference Material

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Name of the Material : Elements in Soil
Material Code : UME EnvCRM 03
Issue Date : 22.03.2019
Revision Date : 11.10.2021 (Revision history can be found on the last page)
Validity Period of the Certificate : 12 months from the sales date
Certified Values :

| Element | Mass Fraction (mg/kg) | | Element | Mass Fraction (mg/kg) | |
|-------------------|--------------------------------|------------------------------|-------------------|--------------------------------|------------------------------|
| | Certified Value ^[1] | Uncertainty ^[1,2] | | Certified Value ^[1] | Uncertainty ^[1,2] |
| As ^[3] | 79.9 | 5.9 | Mn ^[5] | 674 | 70 |
| Cd ^[4] | 1.29 | 0.08 | Ni ^[5] | 51.7 | 7.1 |
| Co ^[5] | 42.0 | 4.5 | Pb ^[7] | 64.1 | 2.1 |
| Cr ^[6] | 115.6 | 16.9 | Sb ^[3] | 1.81 | 0.19 |
| Cu ^[5] | 63.5 | 8.2 | V ^[5] | 76.2 | 10.1 |
| Fe ^[6] | 26748 | 1761 | Zn ^[5] | 150.6 | 26.7 |
| Hg ^[6] | 0.315 | 0.047 | | | |

[1] The certified values and the uncertainties are traceable to the International System of Units (SI). Certified value is corrected for dry mass. Moisture content is determined at (103 ± 2) °C until constant weight.

[2] The expanded uncertainty of the certified value includes characterization, homogeneity, stability components and is stated as the standard uncertainty of measurement multiplied by the coverage factor $k = 2$, which for a normal distribution corresponds to a coverage probability of approximately 95 %. The standard uncertainty of measurement has been determined in accordance with GUM "Guide to the Expression of Uncertainty in Measurement".

[3] Calculated from the arithmetic mean of the dataset obtained by single reference k_0 -INAA method.

[4] Calculated from the arithmetic mean of the two reference ID ICP-MS method.

[5] Calculated from the median of the accepted results submitted by laboratories using different methods.

[6] Calculated from the arithmetic mean of the reference ID ICP-MS and k_0 -INAA methods.

[7] Calculated from the arithmetic mean of the reference ID TIMS and ID ICP-MS methods.

TÜBİTAK UME, as a reference material producer, has been accredited by TÜRKAK according to TS EN ISO 17034 with the accreditation number AB-0001-RM.

Sales Date


Dr. Mustafa ÇETİNTAŞ
Director

The following pages are an integral part of the certificate. The use of current certificate is customers' responsibility.

Most recent certificate can be downloaded from www.ume.tubitak.gov.tr.

Informative Values

| Parameter ^[1] | Mass Fraction ^[2] (g/100g) | |
|--------------------------|---------------------------------------|--------------------|
| | Assigned Value | Standard Deviation |
| Moisture Content | 4.15 | 0.18 |

[1] Moisture content is determined at (103 ± 2) °C until constant weight.

[2] Calculated from means of 4 laboratory results.

Extractable Element Content According to ISO 11466 and/or ISO 12914 Methods

| Element | Mass Fraction (mg/kg) | | Element | Mass Fraction (mg/kg) | |
|-------------------|-------------------------------|--------------------|-------------------|-------------------------------|--------------------|
| | Assigned Value ^[1] | Standard Deviation | | Assigned Value ^[1] | Standard Deviation |
| As ^[2] | 70.3 | 2.1 | Mn ^[6] | 520 | 7 |
| Cd ^[3] | 1.29 | 0.10 | Ni ^[4] | 48.9 | 8.8 |
| Co ^[4] | 41.7 | 3.2 | Pb ^[5] | 63.1 | 11.6 |
| Cr ^[4] | 56.4 | 17.6 | Sb ^[8] | 1.38 | 0.28 |
| Cu ^[5] | 67.3 | 7.9 | V ^[2] | 69.5 | 2.3 |
| Fe ^[6] | 19189 | 227 | Zn ^[5] | 128.5 | 11.7 |
| Hg ^[7] | 0.310 | 0.009 | | | |

[1] Assigned value is corrected for dry mass. Moisture content is determined at (103 ± 2) °C until constant weight.

[2] Calculated from the ICP-MS and ICP-OES means obtained by ISO 12914 method.

[3] Calculated from the ICP-MS and ASV by ISO 12914 method and ICP-MS means by ISO 11466 method.

[4] Calculated from ICP-MS and ICP-OES by ISO 12914 method and ICP-MS means by ISO 11466 method.

[5] Calculated from ICP-MS, ICP-OES, ASV by ISO 12914 method and ICP-MS means by ISO 11466 method.

[6] Calculated from ICP-MS means by ISO 11466 method.

[7] Calculated from ICP-MS means obtained by ISO 12914 method.

[8] Calculated from ISO 12914 method and ICP-MS means by ISO 11466 method.

Additional Elemental Information Determined by Single *k_o*-INAA method

| Element | Mass Fraction (mg/kg) | | Element | Mass Fraction (mg/kg) | |
|-------------------|-------------------------------|----------------------------|-------------------|-------------------------------|------------------------------|
| | Assigned Value ^[1] | Uncertainty ^[2] | | Assigned Value ^[1] | Uncertainty ^[1,2] |
| Al ^[3] | 58864 | 4166 | Mg ^[3] | 39008 | 2840 |
| Ba | 289 | 20 | Na | 10130 | 712 |
| Br | 7.89 | 0.56 | Rb | 65.9 | 4.6 |
| Ca | 88024 | 5304 | Sr | 461 | 38 |
| Cs | 7.14 | 0.50 | Ta | 0.807 | 0.056 |
| Hf | 3.97 | 0.28 | Ti ^[3] | 3854 | 340 |
| K | 15774 | 1114 | Zr | 176 | 14 |

[1] Calculated from the mean of the dataset obtained by single reference *k_o*-INAA method (long and short irradiation). Results are dry mass corrected. Moisture content is determined at (103 ± 2) °C until constant weight.

[2] The expanded uncertainty of assigned value includes characterization component and is stated as the standard uncertainty of measurement multiplied by the coverage factor *k*=2, which for a normal distribution corresponds to a coverage probability of approximately 95 %. The standard uncertainty of measurement has been determined in accordance with GUM "Guide to the Expression of Uncertainty in Measurement".

[3] Results were obtained by *k_o*-INAA via short irradiation.

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|------------|---|--------------------------------|

Description

The material is filled in amber glass bottles containing about 25 g of soil each. The bottles and their content were sterilized by γ -irradiation at a minimum dose of 25 kGy. Additional information is presented in the certification report.

Intended Use

This material is intended to be used for method validation of the determination of As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, V and Zn mass fractions in soils and quality control purposes.

Instructions for Use

Before opening and taking a sample, the bottle should be shaken to re-homogenize the content.

Minimum sample intake is 0.2 g for certified elements and 1 g for moisture content determination. After use, the bottle should be immediately and tightly recapped. Moisture content should be determined on separate samples at $(102 \pm 3) ^\circ\text{C}$ until constant weight.

Storage Conditions

The material should be stored at $(18 \pm 4) ^\circ\text{C}$ in dark and clean environment. This material can be safely dispatched under conditions where the temperatures do not exceed $60 ^\circ\text{C}$ for up to 4 weeks, i.e. at ambient temperature without applying any cooling elements.

TÜBİTAK UME cannot be held responsible for changes that might happen to the material at customer's premises due to noncompliance with the instructions for use, and the storage conditions given.

Safety Information

The usual laboratory safety measures apply as in the case of similar powders.

It is strongly recommended that the material must be handled and disposed according to the safety guidelines where applicable.

It is recommended to avoid inhalation of powder material and work under appropriate ventilation conditions. Please refer to the Safety datasheet (SDS) before any use of the material.

Participants

Information about the laboratories participated in the characterization study are given in the table below.

| Laboratory | Address |
|-------------|--|
| BAM | Bundesanstalt für Materialforschung und -prüfung, Berlin, GERMANY |
| DMDM | Directorate of Measures and Precious Metals, Belgrade, SERBIA |
| IMBIH | Institute of Metrology of Bosnia and Herzegovina, Sarajevo, BOSNIA and HERZEGOVINA |
| IJS | Jožef Stefan Institute, Ljubljana, SLOVENIA |
| NTUA | National Technical University of Athens, Athens, GREECE |
| TÜBİTAK UME | National Metrology Institute, Gebze - Kocaeli, TURKEY |
| UW | University of Warsaw, Warsaw, POLAND |

Methods and/or Techniques Used for the Determination of the Certified Values

Techniques used in the characterisation studies:

| Method/Technique | Parameter |
|---|---|
| Atomic Absorption Spectroscopy (AAS) | Fe |
| Cold Vapor AAS (CV-AAS) | Hg |
| Inductively Coupled Plasma Mass Spectrometry (ICP-MS) | As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, V, Zn |
| Isotope Dilution Inductively Coupled Plasma Mass Spectrometry (ID ICP-MS) | Cd, Cr, Fe, Hg, Ni, Pb, Zn |
| Isotope Dilution Thermal Ionization Mass Spectrometry (ID TIMS) | Cu, Pb |
| Microwave Plasma Atomic Emission Spectrometry (MP-AES) | Co, Cr, Cu, Fe, Mn, Ni, Pb, V, Zn |
| k_0 -Instrumental Neutron Activation Analysis (k_0 -INAA) | As, Co, Cr, Fe, Hg, Mn, Sb, V, Zn |

Commutability

UME EnvCRM 03 was produced by blending the original processed soil with 8 elements spiked soil (additional information is given in the certification report). The analytical behaviour is assumed to be similar for an anthropogenic contaminated soil. It should be noted that the extractability of the 8 spiked elements (Cd, Co, Cu, Hg, Ni, Pb, Sb, Zn) from this CRM can be different to the extractability from an unspiked soil tested by the user's laboratory due to the possibility that these elements might exist in different chemical forms.

Revision History

| Date | Remarks |
|------------|---|
| 22.03.2019 | First issue. |
| 11.10.2021 | Certificate is updated due to changes in the format of certificate for reference materials. |