

Certified Reference Material

BAM-U200

(Iron rich sewage sludge ash)

Certified Values
(Mass fractions total content)

Element	Mass fraction ^a in %	Uncertainty U^b in %
Ca	13.2	2.1
Fe	10.0	1.0
P	8.8	1.3
Al	4.9	0.4
Mg	1.51	0.15
K	1.06	0.12
Na	0.51	0.10
Zn	0.270	0.016
Cu	0.131	0.008
	in mg/kg	in mg/kg
Pb	325	26
Cr	146	12
Ni	79	9
Sn	63	4
As	23.5	2.1
Cd	3.6	0.3

^a Unweighted mean value of the means of accepted sets of data, each set being obtained by at least 4 laboratories and/or with different methods of measurement. The values are traceable to the SI (Système International d'Unités) by the use of pure substances of known stoichiometry for calibration.

^b Estimated expanded uncertainty U with a coverage factor of $k = 2$, corresponding to a confidence level of about 95 %, as defined in ISO/IEC Guide 98-3:2008 [Uncertainty of measurement - Part 3: Guide to the expression of uncertainty of measurement (GUM:1995)].

Certified Values

(Aqua regia extractable mass fractions according to DIN EN 16174:2012-11)

Element	Mass fraction ^a in %	Uncertainty <i>U</i> ^b in %
Ca	13.1	2.1
Fe	9.9	0.9
P	7.9	1.4
Al	4.19	0.28
Mg	1.37	0.10
K	0.79	0.08
Na	0.32	0.06
Zn	0.235	0.018
Cu	0.114	0.029
	in mg/kg	in mg/kg
Pb	288	38
Cr	113	13
Ni	69	7
Sn	61	5
As	22.9	1.0
Cd	3.6	0.4

^a Unweighted mean value of the means of accepted sets of data, each set being obtained by at least 4 laboratories and/or with different methods of measurement. The values are traceable to the SI (Système International d'Unités) by the use of pure substances of known stoichiometry for calibration.

^b Estimated expanded uncertainty *U* with a coverage factor of *k* = 2, corresponding to a confidence level of about 95 %, as defined in ISO/IEC Guide 98-3:2008 [Uncertainty of measurement - Part 3: Guide to the expression of uncertainty of measurement (GUM:1995)].

End of Validity

The validity of this certificate ends 5 years after delivery.

Date of dispatch:

Material Description

The homogenized sewage sludge ash BAM-U200 is in powder form. Approx. 37 g of the sewage sludge ash BAM-U200 was filled into a brown 50 mL wide-neck bottle.

Recommended Use

The purpose of CRM BAM-U200 is to verify analytical results for total mass fractions and aqua regia extractable mass fractions of listed elements in sewage sludge ash. Approximately 0.2 g of material should be used for a wet chemical analysis.

Handling

When handling the sample, the bottle should be left open for as short a time as possible. Care should be taken not to absorb moisture after opening the bottle. The material should be used as it comes out of the bottle. However, it is recommended to re-homogenize by manually shaking the closed bottle before taking a partial sample.

Transport and Storage

After receipt, the material should be stored at a temperature below 25 °C in the tightly closed original bottle. BAM cannot be held responsible for changes that occur during storage of the material at the customer's site, especially with opened samples.

Participating Laboratories

Certification took place at various laboratories within BAM.

Means of Accepted Data Sets BAM-U200 total mass fractions

Certified Values

Value in %

Lfd. Nr.	Ca	Fe	P	Al	Mg	K	Na	Zn	Cu
1	9.48	7.43	5.67	3.83	1.18	0.816	0.264	0.214	0.103
2	10.69	8.49	6.35	4.73	1.21	0.938	0.417	0.249	0.118
3	10.97	9.44	9.02	4.77	1.47	1.034	0.461	0.250	0.130
4	14.50	9.99	9.39	4.87	1.57	1.043	0.556	0.252	0.132
5	15.11	10.78	9.71	4.93	1.58	1.128	0.570	0.271	0.134
6	15.16	10.91	9.92	5.46	1.63	1.140	0.579	0.278	0.134
7	16.50	11.27	10.07	5.53	1.71	1.310	0.688	0.278	0.134
8		11.50	10.32		1.72			0.280	0.136
9								0.284	0.144
10								0.303	0.148
Ave	13.20	9.98	8.81	4.88	1.51	1.06	0.51	0.27	0.13
S _{Ave}	2.74	1.44	1.78	0.56	0.21	0.16	0.14	0.03	0.01

Value in mg/kg

Lfd. Nr.	Pb	Cr	Ni	Sn	As	Cd
1	275.0	124.8	62.9	59.0	19.5	3.3
2	285.3	127.4	65.3	62.1	20.2	3.5
3	303.9	133.4	72.8	62.3	22.5	3.6
4	316.8	136.2	79.4	62.6	23.3	3.9
5	337.0	137.2	80.3	65.0	23.7	
6	340.4	142.0	81.0	67.6	23.8	
7	360.0	150.3	82.5		24.0	
8	383.9	164.2	82.6		25.2	
9		171.0	106.0		29.3	
10		176.7				
Ave	325.3	146.3	79.2	63.1	23.5	3.6
S _{Ave}	37.2	18.4	12.5	2.9	2.8	0.3

A measurement series comprises the respective single values of a laboratory (at least 3, normally 6 single values)

Ave: Arithmetic mean of the measurement series mean values

S_{Ave}: Standard deviation of the measurement series mean values)

Analytical Method (BAM-U200 total mass fractions)

Mass fractions	Line No.	Method/Procedure
Ca	1, 3, 4, 5, 7	ICP OES
	2	ICP MS
	6	XRF, Reconstitution
Fe	1, 2	ICP MS
	3, 5, 7, 8	ICP OES
	4	NAA
	6	XRF, Reconstitution
P	1, 2	ICP MS
	3, 4, 6, 7, 8	ICP OES
	5	XRF Reconstitution
Al	1, 4	ICP MS
	2, 3, 5, 7	ICP OES
	6	XRF, Reconstitution
Mg	1, 3	ICP MS
	2, 4, 5, 7, 8	ICP OES
	6	XRF, Reconstitution
K	1, 4	ICP MS
	2, 5, 6, 7	ICP OES
	3	NAA
Na	1, 6	ICP MS
	2, 4, 5, 7	ICP OES
	3	NAA
Zn	1, 4, 10	ICP MS
	2	NAA
	3, 5, 6, 7, 9	ICP OES
	8	XRF, Reconstitution
Cu	1, 2, 7	ICP MS
	3	NAA
	4, 5, 6, 9, 10	ICP OES
Pb	1, 6, 7	ICP MS
	2, 3, 4, 5	ICP OES
	8	XRF, Reconstitution
Cr	1, 2, 8	ICP MS
	3, 4, 5, 6, 9	ICP OES
	7	NAA
	10	XRF, Reconstitution
Ni	1, 3, 6, 8, 9	ICP OES
	2, 4, 7	ICP MS
	5	XRF, Reconstitution
As	1, 3, 6	ICP MS
	2, 5, 7, 8, 9	ICP OES
	4	NAA

Sn	1, 3, 5, 6	ICP OES
	2, 4	ICP MS
Cd	1, 2, 3	ICP MS
	4	ICP OES

ICP OES: Inductively Coupled Plasma Emission Spectrometry

ICP MS: Inductively Coupled Plasma – Mass Spectrometry

NAA: Neutron Activation Analysis

XRF, Reconstitution: Reconstitution with X-Ray fluorescence Analysis

Means of Accepted Data Sets BAM-U200 Aqua Regia extractable mass fractions

Certified Values

Value in %

Lfd. Nr.	Ca	Fe	P	Al	Mg	K	Na	Zn	Cu
1	9.02	7.92	5.38	3.52	1.16	0.619	0.178	0.184	0.097
2	11.42	9.35	6.95	4.10	1.28	0.705	0.301	0.223	0.097
3	11.90	9.61	7.77	4.11	1.33	0.788	0.320	0.225	0.100
4	12.79	9.72	8.19	4.13	1.38	0.811	0.340	0.229	0.112
5	13.91	10.22	8.92	4.35	1.40	0.823	0.346	0.242	0.117
6	15.11	10.78	10.32	4.44	1.47	0.828	0.356	0.248	0.120
7	17.55	11.36		4.72	1.57	0.938	0.417	0.257	0.134
8								0.271	0.134
Ave	13.10	9.85	7.92	4.19	1.37	0.79	0.32	0.23	0.11
S_{Ave}	2.75	1.11	1.69	0.37	0.14	0.10	0.07	0.03	0.02

Value in mg/kg

Lfd. Nr.	Pb	Cr	Ni	Sn	As	Cd
1	211.8	79.4	59.5	54.9	21.4	3.3
2	222.6	100.0	61.1	57.2	21.9	3.5
3	239.8	109.5	62.8	57.3	22.6	3.7
4	303.9	109.8	66.6	59.3	23.2	4.1
5	319.7	111.1	68.7	63.6	23.7	
6	325.9	128.0	69.0	64.6	24.6	
7	337.0	128.8	82.6	67.6		
8	342.1	137.2	84.4			
Ave	287.8	113.0	69.4	60.7	22.9	3.6
S_{Ave}	54.0	18.4	9.4	4.7	1.2	0.3

A measurement series comprises the respective single values of a laboratory (at least 3, normally 6 single values)

Ave: Arithmetic mean of the measurement series mean values

S_{Ave}: Standard deviation of the measurement series mean values)

Analytical Methods (BAM-U200 Aqua Regia extractable mass fractions)

Mass fractions	Line No.	Method/Procedure
Ca	1, 2 3, 4, 5, 6, 7	ICP MS ICP OES
Fe	1, 4 2, 3, 5, 6, 7	ICP MS ICP OES
P	1, 2, 3, 4, 5, 6, 7	ICP MS ICP OES
Al	1, 4 2, 3, 5, 6, 7	ICP MS ICP OES
Mg	1, 2 3, 4, 5, 6, 7	ICP MS ICP OES
K	1, 2 3, 4, 5, 6, 7	ICP MS ICP OES
Na	1, 5 2, 3, 4, 6, 7	ICP MS ICP OES
Zn	1, 2, 4 3, 5, 6, 7, 8	ICP MS ICP OES
Cu	1, 2, 3 4, 5, 6, 7, 8	ICP MS ICP OES
Pb	1, 2, 8 3, 4, 5, 6, 7	ICP MS ICP OES
Cr	1, 3, 4 2, 5, 6, 7, 8	ICP MS ICP OES
Ni	1, 4, 6, 7, 8 2, 3, 5	ICP OES ICP MS
Sn	1, 6 2, 3, 4, 5, 7	ICP OES ICP MS
As	1, 2 3, 4, 5, 6	ICP MS ICP OES
Cd	1, 2, 3 4	ICP MS ICP OES

ICP OES: Inductively Coupled Plasma Emission Spectrometry

ICP MS: Inductively Coupled Plasma – Mass Spectrometry

Metrological Traceability

The total contents of the specified elements in BAM-U200 are metrologically traceable to the kilogram. The method for determining the aqua regia extractable contents for the specified elements in BAM-U200 are operationally defined referring to the analytical protocols prescribed in DIN EN 16174:2012-11.

Literature

A detailed report describing the certification of the reference material BAM-U200 is available on request or can be downloaded from the BAM website (<https://rrr.bam.de>).

DIN EN 16174:2012-11: Sludge, treated biowaste and soil - Digestion of aqua regia soluble fractions of elements; German version EN 16174:2012.

Accepted as a BAM-CRM on Monat TT, JJJJ

Bundesanstalt für Materialforschung und -prüfung (BAM)

(Dienstsiegel/
Dienststempel)

Titel Name
Committee for Certification

Dr. M. Ostermann
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