

Producer: *Geological Survey of Slovak Republic, Geoanalytical Laboratories, Accredited Testing Laboratory, Reference Laboratory of the Ministry of the Environment of Slovak Republic, Markušovská cesta 1, 052 40 Spišská Nová Ves, Slovakia*

CERTIFICATE OF REFERENCE MATERIAL *BENTONIT 1*

In accordance with § 7 of the Slovak Act No. 142/2000, coll.

registration number: 65/13

Type of material: *natural bentonite*

Code:

Certified value: *component name, certified value and uncertainty (expanded uncertainty (U) with a coverage factor (k) equal to 2)*

Component	Content µg/g	U
As	62.1	4.8
Ba	650	34
Be	3.80	0.40
Ce	96.8	7.2
Cs	67.1	5.4
Dy	3.42	0.30
Er	2.11	0.20
Eu	0.643	0.080
Ga	20.42	1.40
Gd	3.63	0.42
Hf	4.28	0.70
Hg	0.370	0.060
Ho	0.694	0.080
La	63.1	5.6
Li	235	24
Lu	0.382	0.062
Nb	50.4	3.4
Nd	25.9	2.8
Pb	32.7	3.6
Pr	8.45	0.88
Rb	62.1	5.0
Sb	18.5	1.4

Component	Content µg/g	U
Sm	4.15	0.46
Sn	4.09	0.70
Sr	168	6
Th	46.3	4.0
Tm	0.350	0.062
U	9.06	1.36
Y	22.01	1.70
Yb	2.40	0.30
Zn	50.3	3.8
Zr	142	8
Al ₂ O ₃ (%)	19.12	0.40
CaO (%)	1.94	0.10
Fe ₂ O ₃ total (%)	2.17	0.06
K ₂ O (%)	1.01	0.04
MgO (%)	3.74	0.14
MnO (%)	0.092	0.004
Na ₂ O (%)	0.747	0.052
P ₂ O ₅ (%)	0.030	0.006
SiO ₂ (%)	62.85	0.60
TiO ₂ (%)	0.142	0.010
LOI (1000°C) (%)	7.36	0.52

Traceability : *Interlaboratory comparative tests according ISO Guide 35: 1989*

Packing: **50g ± 1g of the powdered material with particle size < 0,09 mm**

Minimal weight for analysis: **250 mg**

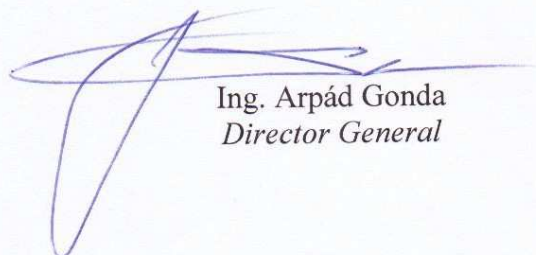
Batch number: **18302**

Valid until: **June 2023**

Personnel responsible for preparation and certification process: **RNDr. Pavol Lučivjanský, Ing. Daniela Mackových, CSc.**

Bratislava,

June 13, 2013



Ing. Arpád Gonda
Director General

Certification conditions:

The interlaboratory comparative tests and one-stage nested design according ISO Guide 35 was used for the certification of reference material. Thirty seven Laboratories from thirteen countries took part in the certification of chemical composition of bentonite. The certified values were calculated as the arithmetic mean of laboratory means after excluding of outlying values. The expanded uncertainty - U was evaluated according to, requirements of Guide to the Expression of Uncertainty in Measurement, ISO 1993.

Noncertified (informative) values:

The contents of elements in CRM – BENTONITE 1

Component	Component content in $\mu\text{g/g}$	U	N	N _p	n	n _p	Analytical Method
H ₂ O at 105°C	10.36 (%)	0.44	30	28	100	92	I,K,N1
H ₂ O at 300°C	3.35 (%)	1.3	10	9	37	33	K,N1
S as SO ₃ T	0.034 (%)	0.024	15	9	57	36	B,H1,H2,K,D
Ag	0.731	*	3	2	8	8	C
B	24.43	*	2	1	8	4	B
Bi	0.462	0.134	4	4	16	16	C
Br	3.28	*	1	1	4	4	H2
Cd	0.089	0.080	11	5	19	17	B,C
Co	1.51	0.60	16	13	59	49	ABC,B,C,H1,H2
Cr	2.89	1.72	22	10	83	40	AAC,ABA,B,C,H2,I
Cu	2.72	0.80	21	18	79	66	AAA,B,C,H2,I
Ge	1.56	1.14	3	3	12	12	C
In	0.019	*	1	1	4	4	C
Mo	0.748	0.328	6	5	24	20	C
Ni	3.90	0.96	22	19	83	71	AAA,AAC,ABC,B,C,H2,I
Sc	2.41	0.24	5	4	20	16	C,I
Se	0.593	*	1	1	4	4	C
Ta	5.22	0.80	6	5	22	18	C
Tb	0.563	0.070	10	7	38	26	C
Te	0.013	*	1	1	4	4	C
Tl	1.53	0.84	5	5	20	20	C
V	6.43	2.18	21	18	79	67	B,C,H2,I
W	0.294	0.066	3	3	12	12	C

* - number of accepted laboratory means < 2

U - expanded uncertainty (k=2)

N - number of all laboratory means

N_p - number of accepted laboratory means

n - number of all laboratory measurements

n_p - number of accepted laboratory measurements

List of Participating Laboratories:

1. Austrian Research Centres Seibersdorf, Chemical Analytics, Wien, Austria
2. Bayerisches Geologisches Landesamt, München, Germany
3. BRGM, Service Analyse et Caractérisation Minerale, Orleans, France

4. British Geological Survey, Nottingham, Great Britainian
5. Centre de Geochimie de la Surface CNRS-UMR 7517, Strasbourg, France
6. Český Geologický Ústav, Analytické Laboratoře, Praha 5, The Czech Republic
7. Department of Earth Sciences Aarhus University, Aarhus, Denmark
8. Department of Earth Sciences, The Open University, Milton Keynes, Great Britainian
9. Ekochem, a.s., Divize laboratoří Stráž pod Ralskem, Stráž pod Ralskem, The Czech Republic
10. EL spol. s r.o., Ekologické a veterinárne laboratóriá, Spišská Nová Ves, Slovakia
11. Estonian Environmental Research Centre, Tallinn, Estonia
12. Federal Institute for Geosciences and Natural resources, Hannover, Germany
13. GEL, s.r.o., odbor Geoanalytické laboratóriá, Chemické laboratórium, Turčianske Teplice, Slovakia
14. GeoForschungsZentrum Potsdam PB4.3, Potsdam, Germany
15. Geological Institute of Hungary, Laboratory, Budapest, Hungary
16. Prírodovedecká fakulta Univerzity Komenského, Geologický inštitút, Bratislava, Slovakia
17. Geological Survey of Estonia, Tallinn, Estonia
18. Geological Survey of Finland , Geolaboratory Espoo, Espoo, Finland
19. Geological Survey of Norway, Laboratory Section, Trondheim, Norway
20. Geologický ústav SAV, Fyz. chemické laboratórium, Bratislava, Slovakia
21. Institute of Geology Tallinn Technical University, Tallinn, Estonia
22. Nová huť, a.s., Výzkumný a zkušební ústav, WS-Spektrometrické laboratoře, Ostrava – Kunčice, The Czech Republic
23. OKD, DPB Paskov, a.s., divize Ekotechnika, Analytické laboratoře, Paskov, The Czech Republic
24. Polish Geological Institute, Central Chemical Laboratory, Warszawa, Poland
25. SAŽP, COHEM, Odbor ekoanalytiky odpadov, Bratislava, Slovakia
26. Slovenské magnezitové závody, a.s., Oddelenie skúšobníctva – DVVK, Jelšava, Slovakia
27. Spectro Analytical Instruments, Kleve, Germany
28. Štátny geologický ústav Dionýza Štúra, Geoanalytické laboratóriá, Spišská Nová Ves, Slovakia
29. U.S. Steel Košice, Labortest, s.r.o., Kvantometrické laboratórium, Košice, Slovakia
30. Vernadsky Institute of Geochemistry and Analytical Chemistry, Central Laboratory of Substance Division, Moscow, Russia
31. VÚSH, a.s., ZL-ATElab, Analytická laboratoř, Brno, The Czech Republic
32. WSB Labor GmbH, Krems, Austria
33. X-ray analytical facilities, University of St. Andrews, Department of geology, St. Andrews, Scotland
34. ZTS-MATEC, a.s., Areál ZTS č. 924, Skúšobné laboratórium analytickej chémie, Dubnica nad Váhom, Slovakia

Analytical technique

Atomic absorption spectrometry – Flame

- | | |
|-----|--|
| AAA | Without background correction using air-acetylene |
| AAC | With deuterium background correction using air-acetylene |

Atomic absorption spectrometry – electrothermal atomisation

ABA	Without background correction, without chemical modifier
ABC	With deuterium background correction, without chemical modifier

Another techniques

B	Atomic emission spectrometry with inductively coupled plasma
C	Inductively coupled plasma mass spectrometry
D	Spectrophotometry UV-VIS
H1	X-ray fluorescence with material melted
H2	X-ray fluorescence with material pressed
I	Optical emission spectrometry
K	Gravimetry
N1	Others

Recertification: Recertification of the CRM was completed on June 13, 2013. Stability tests and certified values revision were accomplished by inter-laboratory verification of the certified elements concentrations in 4 laboratories by different methods. Certificate has been approved by the Certified Reference Materials Committee and re-issued by SMU Bratislava.

Recertification values measured by the inter-laboratory participants were published in a Report on Stability tests CRM of natural bentonite edited by State geological institute Dionýz Štúr, Markušovská cesta 1, 052 40 Špišská Nová Ves in April 2013.

Participants:

- State geological institute Dionýz Štúr, Geoanalytical laboratories, Spišská Nová Ves, Slovak Republic
- EUROFINS BEL/NOVAMANN s.r.o., Testing laboratory, GEL Turčianske Teplice, Slovak Republic
- SPECTRO Analytical Instruments GmbH, Kleve, Germany,
- Czech geological service, Central laboratory, Praha, Czech Republic.

Method used:

- AAS – atomic absorption spectrometry – flame atomisation
- atomic emission spectrometry
- spectrofotometry
- AAS – hydride technique
- AMA – atomic absorption spectrometry
- gravimetry

Storage: Storage of the reference material does not request specific arrangements. The certified reference material is necessary to store at 15 – 25°C in a dark and dry ambient.

Expected application: The reference material is assigned to the validation of analytical methods and for the assessment of the competence of skilled analyst to analyse elements in specific type of matrix material.

Instruction for use: Determination the humidity has to be determined separately by drying at 105°C into constant mass and the analytical results have to be recalculated.

The producer does not guarantee certified values listed in this document in the case of CRM damage caused by the user !

The certificate shall be reproduced only in full and untouched. Without the written approval of the Slovak Institute of Metrology can not be reproduced any part of the certificate.