

BAM Bundesanstalt für Materialforschung und -prüfung

in co-operation with the Committee of Chemists of GDMB
Gesellschaft der Metallurgen und Bergleute e.V.

Certified Reference Material

BAM-S011

Niobium pentoxide powder

Certified Value

Parameter	Mass fraction ¹⁾ in mg/kg	Uncertainty ²⁾ in mg/kg
Fluorine	128	13

¹⁾ The certified value is the mean of 8 series of results obtained by different laboratories. 4 different analytical methods were used for the measurement of the parameter. Calibration was carried out with pure substances of definite stoichiometry or with solutions prepared from them thus achieving traceability to SI unit.

²⁾ The certified uncertainty is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurements (GUM) with a coverage factor of $k = 2$.

Indicative values

Parameter	Mass fraction ¹⁾ in mg/kg	Uncertainty ²⁾ in mg/kg
Aluminium	0.29	0.16
Chromium	0.031	0.005
Copper	0.040	0.009
Iron	0.26	0.08
Tantalum	8	6
Molybdenum	<0.05	
Nickel	<0.3	

¹⁾ The indicative values are the means of 3 to 5 series of results (depending on the parameter) obtained by different laboratories. 2 or 3 different analytical methods were used for the measurement of one parameter.

²⁾ The indicated uncertainty is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurements (GUM) with a coverage factor of $k = 2$.

Sample description

The certified reference material BAM-S011 consists of niobium pentoxide powder. The material is supplied in polyethylene bottles containing 50 g each.

Informative Values

One laboratory determined silicon to a content of 2.1 mg/kg.

Parameter		Value
Particle size	D ₁₀	0.87 μm
	D ₅₀	2.2 μm
	D ₉₀	18.1 μm
The particle size distribution (volume) was determined by laser granulometry.		

Recommended Use

The main area of application is checking the trueness of results when the certified parameter fluorine in niobium pentoxide is determined by a laboratory. Based on own results and on the certified value the uncertainty of own measurements can be calculated.

Recommendations for Correct Sampling and Sample Preparation

To ensure a representative sub-sampling for the analysis the bottle containing the CRM should be shaken in different directions for about two minutes before taking the sub-sample. According to the sub-sample mass for homogeneity testing a minimum sub-sample mass of 1000 mg is specified for the analyte Fluorine. The opening duration of the bottle should be as short as possible. The lid of the bottle containing a special sealing gasket should be locked tightly immediately after usage. It is not required to dehydrate the sample before starting measurements.

Means of Accepted Data Sets

Mass fraction in mg/kg
 Certified value

Indicative values

Line-No.	F		Al	Cr	Cu	Fe	Ta	Mo	Ni
1	102		0.09	0.025	0.033	0.162	3.62	< 0.01	< 0.01
2	113		0.24	0.030	0.034	0.197	6.87	< 0.01	0.026
3	121		0.25	0.031	0.041	0.242	12.2	0.012	< 0.3
4	124		0.33	0.037	0.052	0.315		< 0.05	
5	126		0.56	< 0.1		0.365			
6	139								
7	140								
8	160								
<i>M</i>	128		0.29	0.031	0.040	0.256	7.57		
<i>s_M</i>	18		0.18	0.005	0.009	0.084	4.36		

M : Arithmetic mean of the laboratory means

s_M : Standard deviation of the laboratory means

Note: "< - values" were not included into the calculation of *M* and *s_M*

Analytical methods

Element	Line No.	
F	1, 2, 3, 4, 5	ISE
	6	GF-SS-CSMAS
	7	GF-CSMAS
	8	GD-MS
Al	1, 4	GD-MS
	2, 3	ICP-MS
	5	GF-CSAAS
Cr	1, 4	GD-MS
	2, 3	ICP-MS
Cu	1, 3	GD-MS
	2, 5	ICP-MS
	4	GF-CSAAS
Fe	1, 2	GD-MS
	3, 4	ICP-MS
	5	GF-CSAAS
Ta	1, 3	ICP-MS
	2	ICP-OES
Mo	1, 2	ICP-MS
	3, 4	GD-MS
Ni	1, 2	GD-MS
	3	ICP-MS

List of abbreviations

GD-MS	Glow discharge mass spectrometry
GF-CSAAS	Graphite furnace continuum source atomic absorption spectrometry
GF-CSMAS	Graphite furnace continuum source molecular absorption spectrometry
GF-SS-CSMAS	Graphite furnace solid sampling continuum source molecular absorption spectrometry
ICP-OES	Inductively coupled plasma optical emission spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
ISE	Ion sensitive electrode

Transport and Storage

The CRM should be stored in its original container in a dust-free and dry environment.

Safety Guidelines

For detailed information on the safe handling of the handling of the material, please see the Material Safety Data sheet distributed by the producer of the material (available on request).

Stability

Niobium oxide is known to be stable.

Technical Report

A detailed technical report describing the analysis procedures and the treatment of the analytical data used to certify BAM-S011 is available on request.

Participating Laboratories

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