

Bundesanstalt für Materialforschung und -prüfung (BAM)

in cooperation with the Unterausschuss Glasanalyse within  
Deutsche Glastechnische Gesellschaft e.V. (DGG)

## Certified Reference Material

### BAM-S051

Iron in Flat Glass

#### Certified Values

Parameter	Mass fraction <sup>1)</sup> in %	Uncertainty <sup>2)</sup> in %
Fe(II)	0.0155	0.0013
Fe(total)	0.0481	0.0017

<sup>1)</sup> Unweighted mean value of the means of 13 accepted sets of data, each set being obtained by a different laboratory and/or a different method of measurement.

<sup>2)</sup> Estimated expanded uncertainty  $U$  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 until 08/2048.

#### Values for information

Element	Mass fraction <sup>1)</sup> in %	Uncertainty <sup>2)</sup> in %
Fe(III), calculated	0.0326	0.0021
Fe(II), calculated as Fe <sub>2</sub> O <sub>3</sub>	0.0226	0.0022

<sup>1)</sup> Values were not certified, but given for information, because they are not based on measurements but only on calculations using the certified values.

<sup>2)</sup> Estimated expanded uncertainty  $U$  with a coverage factor of  $k = 2$ , corresponding to a level of confidence of approx. 95 %, calculated from the uncertainties of the certified values.

#### Sample Description

The Reference Material is available in the form of slides (approx. 100 mm x 50 mm, 5.9 mm thickness).

#### Recommended Use

The CRM is intended for quality control. The minimum sample size for wet chemical analysis is 0.2 g.

#### Transport and Storage

The material should be stored in the dark in a dry and clean environment at room temperature. Transport under normal ambient conditions.

## Means of Accepted Data Sets

Certified values  
Mass fraction in %

Values for information

Line No.	Fe(II)	Fe(total)	Fe(III)
1	---	0.0448	
2	0.0119	0.0457	
3	0.0132	0.0465	
4	0.0139	0.0469	
5	0.0141	0.0480	
6	0.0145	0.0480	
7	0.0152	0.0483	
8	0.0155	0.0485	
9	0.0156	0.0485	
10	0.0157	0.0486	
11	0.0162	0.0501	
12	0.0162	0.0529	
13	0.0195	---	
14	0.0195	---	
<i>M</i>	0.0155	0.0481	0.0323
<i>s<sub>M</sub></i>	0.0022	0.0021	
$\bar{s}_i$	0.0010	0.0009	

The laboratory mean values have been examined statistically to eliminate outlying values. Where a " --- " appears in the table it indicates that an outlying value has been omitted (technical or statistical). A data set consists of 1 to 6 single values of one laboratory.

*M* : mean of laboratory means

*s<sub>M</sub>* : standard deviation of laboratory means

$\bar{s}_i$  : averaged repeatability standard deviation (square root of the mean of laboratory variances)

## Participating Laboratories

Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany

Cetim Grand Est, Schiltigheim, France

Dorfner Anzaplan GmbH, Hirschau, Germany

Fraunhofer-Institut für Silicatforschung, Würzburg, Germany

IGR Institut für Glas- und Rohstofftechnologie GmbH, Göttingen, Germany

NSG, Lathom, United Kingdom

RISE – Section for Glass”, Växjö, Sweden

Schott AG., Mainz, Germany

St. Gobain Recherche, Aubervilliers, France

St. Gobain R+D Centre, Herzogenrath, Germany

T. Şişe ve Cam Fab. A.Ş. Science and Technology Center, Gebze Kocaeli, Turkey

TU Bergakademie Freiberg, Institut für Keramik, Glas- und Baustofftechnik, Freiberg, Germany

Zentrum für Glas- und Umweltanalytik GmbH, Ilmenau, Germany

## Analytical Method used for Certification

Element	Line Number	Method
Fe(II)	2	Volumetric determination with Cerium with ortho-phenanthroline after HF/H <sub>2</sub> SO <sub>4</sub> decomposition under inert atmosphere
	3	XANES
	4, 5, 6, 8, 13	Spectrophotometry according to DIN ISO 14719:2011, with cover gas
	7	Volumetric determination by Ce(SO <sub>4</sub> ) <sub>2</sub> with ferroin after HF/H <sub>2</sub> SO <sub>4</sub> decomposition under inert atmosphere
	9, 11	Spectrophotometry with 1-10 phenanthroline after dissolution with HF/H <sub>2</sub> SO <sub>4</sub> under N <sub>2</sub> -atmosphere
	10	Photometric determination of transmission at 1000 nm, calculation of Fe(II)-content
	12	Spectrophotometry according to DIN ISO 14719:2011, without cover gas
	14	Spectrophotometry after dissolution in HF/ H <sub>2</sub> SO <sub>4</sub> and H <sub>3</sub> BO <sub>3</sub> with cover gas
Fe(total)	1, 12	ICP-OES, according to DIN ISO DIN 51086-2
	2	Spectrophotometry with 1-10 phenanthroline after reduction
	3, 7	Spectrophotometry according to DIN ISO 14719:2011,
	4	XRF on pressed powder samples with ascorbic acid
	5	Spectrophotometry after dissolution in HF/ H <sub>2</sub> SO <sub>4</sub> and H <sub>3</sub> BO <sub>3</sub> with cover gas
	6, 9	XRF
	8	Spectrophotometry according to BS 2649-2:1957
	10	ICP-OES after dissolution with HNO <sub>3</sub> /HF
	11	XRF after melting with Li <sub>2</sub> B <sub>4</sub> O <sub>7</sub> with cover gas

**Abbreviations:** ICP-OES – Inductively coupled plasma - optical emission spectrometry  
XANES – X-ray absorption near edge structure  
XRF – X-ray fluorescence spectrometry

## Instructions for Use

Before use, the surface of the material should be cleaned carefully without scratching.

## Metrological Traceability

To ensure traceability of the certified mass fractions to the SI (Système International d'Unités) calibration was performed using certified standard solutions or pure metals or pure substances of known stoichiometry.

## Technical Report

A detailed technical report describing the analysis procedures and the treatment of the analytical data used to certify BAM-S051 is available on request or can be downloaded from BAM website ([www.bam.de](http://www.bam.de)).

## Absorption measurement

One participating laboratory measured the absorption of BAM-S051 directly on the solid glass specimens, see Fig. 1.

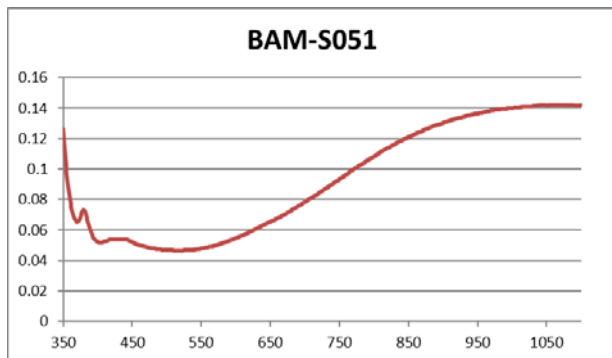


Fig. 1: Absorption spectrum of BAM-S051 (wavelength range: 300 - 1100 nm)

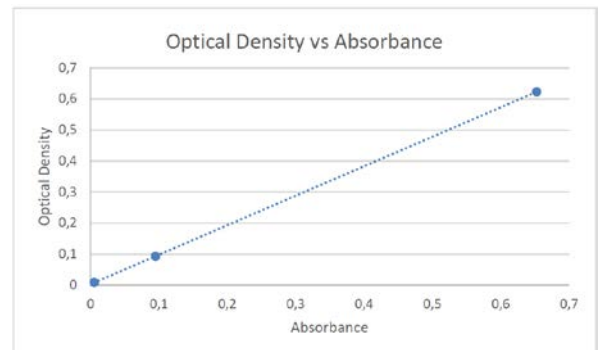


Fig. 2: The optical density (concentration x thickness) plotted versus measured absorbance

Fig. 2 shows the correlation between the optical density, calculated from Fe(II)-content of BAM-S050, BAM-S051 and BAM-S052 obtained by chemical analysis and thickness of the slides and the directly measured absorbance. The good correlation between the chemical method and the physical method confirms the certified values for Fe(II) in BAM-S050, BAM-S051 and BAM-S052.

**Accepted as BAM-CRM on**

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

Dr. S. Richter  
Committee for Certification

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Project Coordinator

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