

Bundesanstalt für Materialforschung und -prüfung (BAM)

# Certified Reference Material BAM-S014

Li-NMC 111 Cathode Material

Element	Mass fraction <sup>1)</sup> in %	Uncertainty <sup>2)</sup> in %			
Li	7.62	0.16			
Ni	19.76	0.13			
Mn	18.22	0.14			
Со	19.80	0.12			
0	34.0	1.0			
	in mg/kg	in mg/kg			
Al	14.1	1.9			
С	600	50			
Cr	6.3	0.8			
S	1421	70			
Na	512	12			
Fe	26	4			
1)					

# **Certified Values**

<sup>1)</sup> Unweighted mean value of the means of accepted sets of data (consisting of at least 2 single results), 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 for a period of 36 months beginning with the dispatch of the reference material from BAM.

Date of dispatch:

### Sample Description

The Reference Material is available in the form of powder. It is supplied in glass bottles containing 25 g.

### Transport and Storage

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

# Informative Values

Element	Mass fraction <sup>1)</sup> in mg/kg	Uncertainty <sup>2)</sup> in mg/kg				
Р	12.2	2.2				
Si	< 100					
Ti	< 1					
V	< 2					

<sup>1)</sup> Values were not certified, but given for information, when the number of accepted data sets was considered to be too low (< 5) or when the uncertainty from the inter-laboratory certification was considerably larger than the expected range or in case there were hints that the material was not homogeneous enough.

<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).

Property		Property value	Uncertainty*		
Specific surface	ce area (BET) <sup>1)</sup>	0.487 m²/g	0,028 m²/g		
Isotopic ratio	<sup>6</sup> Li/ <sup>7</sup> Li	12.35 0.02			
Particle size	<b>d</b> 10	6.2 µm	0.5 µm		
	<b>d</b> 50	11.2 μm	0.5 µm		
	<b>d</b> 90	19.1 µm	1.2 μm		
*Estimated expand of laboratory mea	ded uncertainty <i>U</i> wi	th a coverage factor of $k = 2$ , calculated leviation within the laboratory, $n = 7$ )	from the standard deviation		

<sup>1)</sup> The specific surface area was determined using the gas adsorption method with krypton at 77 K to measure the adsorption branch up to  $p/p_0 0.5$ .

# Recommended Use

The reference material is intended for development, calibration, validation and quality control of analytical methods for the determination of elements in samples of similar matrix composition. The minimum sample size for chemical analysis is 0.1 g.

If the reference material is used to determine the specific surface area sample preparation is necessary prior to measurement. Outgassing must be carried out in vacuum with a final pressure of below 10 Pa. The sample has to be heated in vacuum with a rate of 5 K/min to 473.15 K with subsequent holding at 473.15 K for at least five hours. Afterwards, the sample has to be cooled slowly to ambient temperature.

### Instructions for Use

Before taking a sample, homogenization by manually shaking the closed bottle for 20 seconds is strongly recommended.

### Metrological Traceability

To ensure traceability of the certified mass fractions to the SI (Système International d'Unités) calibration was performed using standard solutions prepared from pure metals or stoichiometric compounds or with traceable commercial calibration solutions.



#### Means of Accepted Data Sets

	Certified values Mass fraction in %						in mg/kg						
Line No.	Li	Ni	Mn	Со	0	AI	С	Cr	Fe	Na	S		
1	7.529		17.81	19.47	32.5	11.0	501	5.38	19.5		1270		
2	7.547	19.47	17.91	19.62	33.1	11.8	520	5.92	23.6		1296		
3	7.555	19.53	17.95	19.64	33.7	12.0	584	6.12	24.9	492	1302		
4	7.567	19.58	17.97	19.68	34.1	14.6	585	6.62	27.3	501	1314		
5	7.570	19.65	18.18	19.73	34.2	14.7	590	7.60	28.4	507	1331		
6	7.625	19.67	18.19	19.73	34.2	15.2	604		29.5	512	1336		
7	7.630	19.70	18.25	19.84	34.2	15.8	616		32.1	516	1383		
8	7.635	19.76	18.27	19.86	34.8	17.6	672			516	1383		
9	7.658	19.77	18.28	19.92	35.5		729			538	1454		
10	7.661	19.80	18.32	20.02							1513		
11	7.689	19.97	18.36	20.03							1558		
12	7.702	20.08	18.36	20.12							1560		
13	7.716	20.12	18.54								1572		
14			18.67								1626		
М	7.622	19.76	18.22	19.80	34.0	14.1	600	6.33	26.5	512	1421		
S <sub>M</sub>	0.063	0.21	0.25	0.20	0.9	2.3	70	0.84	4.2	15	123		

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. A data set consists of at least 2 single values of one laboratory.

M : mean of laboratory means

 $s_M$ : standard deviation of laboratory means

Mass fraction in mg/kg							Particle size in µm				
Line No.	Ρ	Si	Ti	v		²Li∕⁰Li	<b>d</b> 10	d₅₀	<b>d</b> 90		
1	10.9	9.0	0.622	0.04		12.33	5.81	10.91	18.07		
2	11.4	18.1	0.717	1.3		12.33	5.88	11.05	18.60		
3	14.4	64.3				12.34	6.21	11.16	18.91		
4		65.2				12.35	6.28	11.35	19.34		
5		76.5					6.35	11.36	19.40		
6		88.5					6.36	11.42	19.50		
7							6.39	11.47	19.57		
М	12.2					12.34	6.18	11.24	19.06		
S <sub>M</sub>	1.9					0.01	0.24	0.21	0.56		

#### Values for information Mass fraction in ma/ka

# Participating Laboratories

- BMW AG, München (Germany)
- Bruker AXS GmbH, Karlsruhe (Germany)
- Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin (Germany)
- Dorfner Analysenzentrum und Anlagenplanungsgesellschaft mbH (ANZAPLAN), Hirschau (Germany)
- Elementar Analysensysteme GmbH, Langenselbold (Germany)
- Evonik Operations GmbH, Hanau-Wolfgang (Germany)
- Forschungsinstitut für Glas Keramik GmbH, Höhr-Grenzhausen (Germany)
- Horn & Co. Analytics GmbH, Wenden-Hünsborn, (Germany)
- Institut für Materialprüfung Glörfeld GmbH, Willich, (Germany)
- Karlsruher Institut für Technologie, Eggenstein-Leopoldshafen (Germany)
- LECO European Application and Technology Center, Berlin (Germany)
- MEET Battery Research Center, Münster (Germany)
- revierlabor, Essen (Germany)
- SPECTRO Analytical Instruments GmbH, Kleve (Germany)
- TU Clausthal, Institut f
  ür Aufbereitung, Deponietechnik und Geomechanik, Clausthal-Zellerfeld (Germany)
- VOLKSWAGEN Aktiengesellschaft, Salzgitter (Germany)

#### **Technical Report**

A detailed technical report describing the analysis procedures and the treatment of the analytical data used to certify BAM-S014 is available on request or can be downloaded from BAM website (www.bam.de).

### Accepted as BAM-CRM on 2023-03-13

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



Dr. S. Richter Committee for Certification Dr. S. Recknagel Project Coordinator

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