

Sterigmatocystin

1. General information

This document is designed and the certified value(s) and uncertainty(ies) are determined in accordance with ISO Guide 31 [1] and Eurachem / CITAC Guides [2,3].

2. Description of the Reference Material (RM)

Name:	Sterigmatocystin
CAS number:	10048-13-2
Catalog number:	DRE-C16974700
Lot #:	S13335S
Certificate version:	4
Expiry date:	16.08.2021
Physical description of RM:	White crystals of Sterigmatocystin
Packaging and amount of RM:	Amber glass ampoules fitted with teflon faced butyl septa and PP screw caps, quantity of 5 mg of RM
Name and address of the manufacturer:	Romer Labs Division Holding GmbH Technopark 5 3430 Tulln, Austria www.romerlabs.com, office-europe@romerlabs.com
Name and address of the supplier:	LGC Standards GmbH Mercatorstraße 51, 46485 Wesel, Germany Tel +49(0)2 81 98 87 0, Fax +49(0)2 81/98 87 199 www.lgcstandards.com

2.1 Intended use of the RM

- for laboratory use only
- calibration of analytical instruments

2.2 Instruction for the correct use of the RM

The ampoules should be stored at 2-8°C or below in a dark place. Before usage of the RM, the ampoules should be allowed to warm to room temperature. The recommended minimum sub-sample amount for all kinds of application is 1 mg. The expiry date of this RM is based on the current knowledge and holds only for proper storage conditions in the originally closed flasks/packages. Solutions prepared for calibration purposes should be protected from exposure to light. Discard solutions after use in accordance with appropriate safety regulations for chemical substances.

2.3 Hazardous situation

The normal laboratory safety precautions should be observed when working with this RM. Further details for the handling of this RM are available as safety data sheet (SDS).

3. Certified values and their uncertainties

Sterigmatocystin		
Compound	Purity	
	Certified value ^a	Uncertainty ^b
Sterigmatocystin	99.7 %	± 0.3 %

^a The certified value is based upon the results by LC-MS/MS analysis
^b Expanded uncertainty U (k = 2) of the value u_c according to GUM [4]

3.1 Calculation of the certified value and discussion of uncertainty

The purity check with LC-MS/MS showed 2 minor unknown impurities with an estimated mass concentration of total 0.1 % and 0.4 % of the investigated sample. Based on these findings, maximum impurity level in solid Sterigmatocystin can be estimated with 0.5 %.

To cover this range, an approach with an estimated purity of 99.7 % with a symmetrical uncertainty of ± 0.3 % was used according to a procedure which has recently been accepted by the European Commission within a Standards, Measurements and Testing (SMT) project [5]. The conservative assumption of a rectangular distribution was made and the tolerance of 0.25 % divided by √3 resulting in an uncertainty (u_c) of the presented purity level of 0.14. Following the Guide to the Expression of Uncertainty in Measurement (GUM) [1] the expanded uncertainty of the Sterigmatocystin purity level is obtained by multiplication with a coverage factor k for which 2 is usually chosen to obtain a confidence level of

ca. 95 %. Using this procedure a theoretical value for the purity of the crystalline Sterigmatocystin sample of 99.7 % and its respective expanded uncertainty of ± 0.3 % can be calculated.

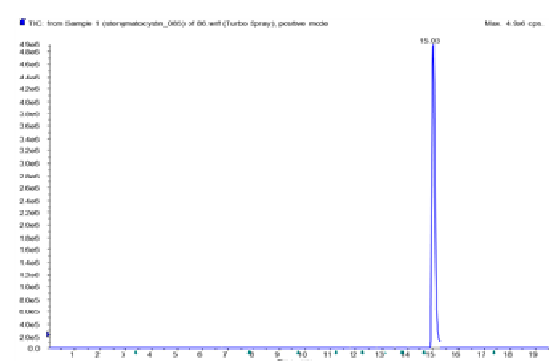
4. Discussion of traceability

The certified value (purity of Sterigmatocystin) is based on the result of LC-MS/MS analysis which was previously used as method for purity assessment of solid mycotoxins [5]. High purity material represents a practical realization of concentration units, through conversion of mass to molar quantity.

5. HPLC-MS/MS analysis of Sterigmatocystin [6]

5.1 Purity assessment

The purity check using gradient LC-MS/MS of the Sterigmatocystin sample showed one main peak and 2 minor unknown impurities after blank subtraction. The purity was calculated according literature [7] (100 % method).

column	Phenomenex Gemini [®] C ₁₈ -column, 150 x 4.6 mm, 5µm		 <p>Figure 1: TIC of Sterigmatocystin sample</p>																
Sample dilution	1:20 with solvent A																		
injection volume	5 µL sample																		
solvent A	methanol/water/acetic acid 10/89/1 with 5mM ammonium acetate																		
solvent B	methanol/water/acetic acid 97/2/1 with 5mM ammonium acetate																		
flow rate	1.0 mL / min																		
gradient	time in minutes (min)	% solvent B																	
	0 – 2	0																	
	2 – 5	0-50																	
	5 – 14	50-100																	
	14 – 18	100																	
	18 – 22	0																	
Source type	ESI, negative mode		<table border="1"> <thead> <tr> <th></th> <th>time [min]</th> <th>height [cps]</th> <th>% of total peak area</th> </tr> </thead> <tbody> <tr> <td>unknown</td> <td>6.17</td> <td>2000</td> <td>0.1</td> </tr> <tr> <td>unknown</td> <td>12.16</td> <td>9000</td> <td>0.4</td> </tr> <tr> <td>Sterigmatocystin</td> <td>15.03</td> <td>2.2E+06</td> <td>99.5</td> </tr> </tbody> </table>		time [min]	height [cps]	% of total peak area	unknown	6.17	2000	0.1	unknown	12.16	9000	0.4	Sterigmatocystin	15.03	2.2E+06	99.5
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REFERENCE MATERIAL CERTIFICATE

6. Further information

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approved for release by: *Laurence Treccani-Chinelli, Global Supply Chain Manager - LGC Standards*

date: 13.01.2020

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References:

- [1] ISO Guide 31:2015 - 1-18, "Reference materials – contents of certificates, labels and accompanying documentation"
- [2] Eurachem / CITAC Guide, 1-37, (2003), "Traceability in Chemical Measurement"
- [3] Eurachem / CITAC Guide CG4, 1-133, (QUAM:2012.P1), "Quantifying Uncertainty in Analytical Measurement", 3rd Ed.
- [4] International Organization for Standardization (ISO), (1995), "Guide to the Expression of Uncertainty in Measurement", 1st Ed. Geneva, Switzerland
- [5] R.D. Josephs, R. Krska, S. MacDonald, P. Wilson, H. Pettersson, J. AOAC Int. **86**, 50-60, (2003), "Preparation of a Calibrant as Certified Reference Material for Determination of the Fusarium Mycotoxin Zearalenone"
- [6] M. Sulyok, R. Krska R. Schuhmacher, Anal. Bioanal. Chem. **389**, 1505-1523, (2007), "A liquid chromatography/tandem mass spectrometric multi-mycotoxin method for the quantification of 87 analytes and its application to semi-quantitative screening of moldy food samples"
- [7] V.R. Meyer, Fallstricke und Fehlerquellen in der HPLC in Bildern, 2. Auflage, 126-127, (1999), Verlag Wiley-VCH, "Quantifizierung im 99 % Bereich"