

PATULIN

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:	Patulin
CAS number:	149-29-1
Catalog number:	DRE-C15896000
Lot #:	S18434P
Certificate version:	1
Expiry date:	25.10.2023
Physical description of RM:	White crystals of Patulin
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 Diagnostic GmbH Technopark 5, 3430 Tulln, Austria www.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 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

Patulin		
Compound	Purity	
	Certified value ^a	Uncertainty ^b
Patulin	97.0 %	± 3.0 %

^a The certified value is based upon the results by HPLC-DAD 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-DAD and UV-spectrophotometry showed a purity with an estimated mass concentration of total 94.8 % of the investigated sample. Based on these findings, maximum impurity level in solid Patulin can be estimated with 5.1 %.

To cover this range, an approach with an estimated purity of 97.0 % with a symmetrical uncertainty of ± 3.0 % was used according to a procedure which has 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 2.6 % divided by √ 3 resulting in an uncertainty (u_c) of 1.5 %. Following the Guide to the Expression of Uncertainty in Measurement (GUM) [1] the expanded uncertainty of the Patulin purity level is obtained by multiplication with a coverage factor k for which 2 is usually chosen to obtain a confidence level of approx. 95 %. Using this procedure a theoretical value for the purity of the crystalline Patulin sample of 97.0 % and its respective expanded uncertainty of ± 3.0 % can be calculated.

4. Discussion of traceability

The certified value (purity of Patulin) is based on the result of HPLC-DAD 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. Purity assessment of Patulin

5.1. UV-spectrophotometry

The wavelength scale accuracy in both UV and visible regions of the applied spectrophotometer was controlled with holmium oxide in dilute perchloric acid [6]. The absorbance scale and the linearity of the apparatus were validated with potassium dichromate in dilute sulfuric acid [7]. All measurements were performed at 22 ± 3 °C.

The UV absorption spectrum of Patulin was consistent with literature data and showed no detectable impurities.

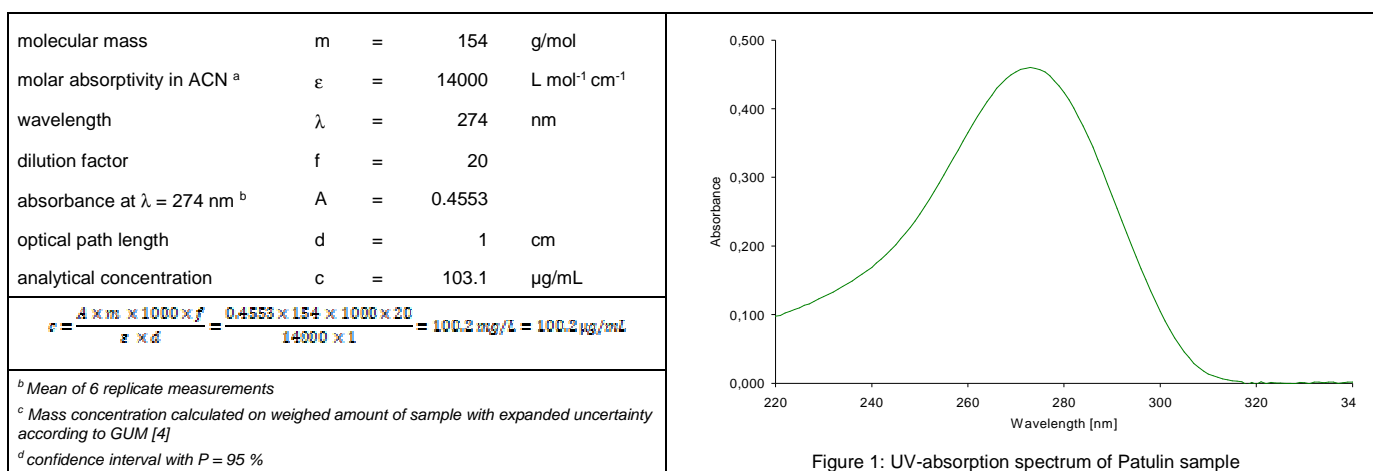


Figure 1: UV-absorption spectrum of Patulin sample

