

REFERENCE MATERIAL CERTIFICATE

SECALONIC ACID D IN CHLOROFORM

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:	Secalonic acid D in chloroform
CAS number:	67-66-3
Catalog number:	DRE-A16929000CH-50
Lot #:	1000002174
Certificate version:	2
Expiry date:	17.07.2021
Starting material:	Secalonic acid D, Lot # S10105B
Physical description of RM:	Solution of Secalonic acid D in chloroform
Packaging and amount of RM:	Amber Certan® glass ampoules, solution of 1.2 mL
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 100 µL. 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.

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).

Hazardous Ingredients
Chloroform

Pictograms



Signal word
Warning

Hazard statement(s)
H302, H315, H351, H373

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3. Certified values and their uncertainties

Secalonic acid D in chloroform		
Compound	Mass concentration ^a	
	Certified value ^b	Uncertainty ^c
Secalonic acid D	50.6 µg/mL	± 0.7 µg/mL

^a Values are based on preparation data and confirmed experimentally by UV-spectrophotometry
^b Mass concentration based on weighed amount, purity and dilution step
^c Expanded uncertainty U (k = 2) of the value u_c according to GUM [4]

3.1 Calculation of uncertainty

The uncertainty of the calibrant solution was calculated on the basis of preparation [5].

Uncertainty components	Description	Standard uncertainty (u)	
Purity (P) of solid Secalonic acid D	P = 99 % ± 1 %	u (P) = 0.6 %	a
Weighing procedure weighted sample: m _{ws} = 5.114 mg	U(m) = 0.0026 mg + 9.51 * 10 ⁻⁶ * m _{Toxin} u(m) = U(m)/2	u (m) = 0.0013 mg	b
Dilution procedure volumetric flask: V _f = 100 mL	calibration: 100 mL ± 0.1 mL repeatability: 0.04 mL volume expansion solvent	u (cal) = 0.04 mL u (rep) = 0.04 mL u (Vol. exp.) = 0.24 mL u (V) = 0.3 mL	c d e f

^a Maximum tolerance of purity (rectangular distribution) was divided by $\sqrt{3}$

^b Calculation of this u-value is based upon the uncertainty formula for the weighed amount as given in the calibration report from annual balance calibration

^c A triangular distribution (division by $\sqrt{6}$) was chosen for the calculation of u (cal)

^d Based on a series of ten fill and weigh experiments on a typical 100 mL flask; the value was used directly as a standard deviation

^e Based on the density of 1.48 g/cm³ at temperature T = 20°C and a maximum temperature variation of ± 3°C, of volume expansion, relative volume expansion coefficient of chloroform is 1210 * 10⁻⁶/°C [6], volume expansion term (rectangular distribution) was divided by $\sqrt{3}$

^f The three contributions are combined to give the u (V) = $\sqrt{u(\text{cal})^2 + u(\text{rep})^2 + u(\text{Vol. exp.})^2}$

Calculation of the combined uncertainty u_c and the expanded standard uncertainty U

$$c_{\text{Toxin}} = \frac{10 \times m_{\text{ws}} \times P}{V_f} = \frac{10 \times 5.116 \times 99.0}{100} = 50.6 \text{ mg/L}$$

$$\frac{u_c(c_{\text{Toxin}})}{c_{\text{Toxin}}} = \sqrt{\left[\frac{u(P)}{P}\right]^2 + \left[\frac{u(m)}{m_{\text{ws}}}\right]^2 + \left[\frac{u(V)}{V_f}\right]^2} = \sqrt{\left[\frac{0.6}{99.0}\right]^2 + \left[\frac{0.0013}{5.116}\right]^2 + \left[\frac{0.3}{100}\right]^2} = 0.007$$

$$u_c(c_{\text{Toxin}}) = c_{\text{Toxin}} \times 0.007 = 50.6 \times 0.007 = 0.35 \text{ mg/L}$$

Calculation of expanded standard uncertainty U using a coverage factor k = 2

$$U(c_{\text{Toxin}}) = u_c(c_{\text{Toxin}}) \times 2 = 0.35 \times 2 = 0.7 \text{ mg/L} = 0.7 \text{ µg/mL}$$

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4. Discussion of traceability

This calibrant is certified on the basis of gravimetric preparation [5]. Thus the certified value (mass concentration of Secalonic acid D) is based on the weighed amount of the starting material and is therefore traceable to the stated purity of the solid raw material. High purity material represents a practical realization of concentration units, through conversion of mass to molar quantity.

5. Confirmation of certified value by 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 [7]. The absorbance scale and the linearity of the apparatus were validated with potassium dichromate in dilute sulfuric acid [8].

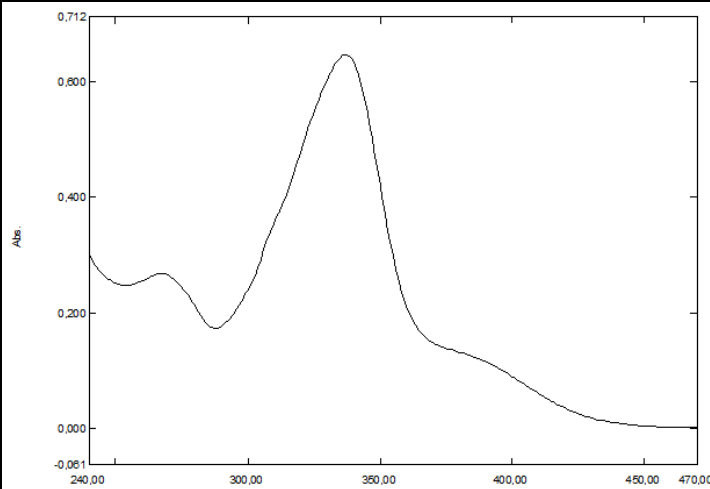
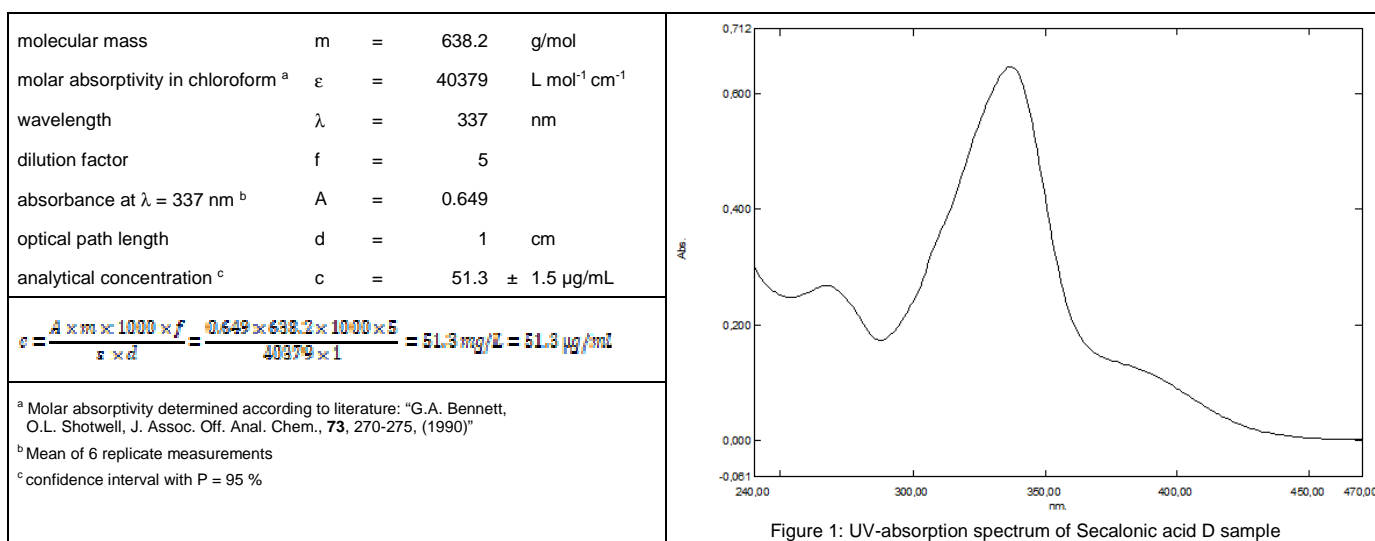


Figure 1: UV-absorption spectrum of Secalonic acid D sample

6. Further information

The purchaser must determine the suitability of this product for its particular use. LGC Standards GmbH makes no warranty of any kind, express or implied, other than its products meet all quality control standards set by LGC Standards GmbH. We do not guarantee that the product can be used for a special application.

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

date: 07.04.2020

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**REFERENCE MATERIAL CERTIFICATE****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), (2008), "Guide to the expression of uncertainty in measurement", (GUM 1995 with minor corrections) 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] E.W. Flick, (1998), "Industrial Solvents Handbook", 5th Ed., Noyes Data Corp. Westwood NJ
- [7] J.C. Travis, J.C. Zwinkels, F. Mercader, A. Ruiz, E.A. Early, M.V. Smith, M. Noel, M. Maley, G.W. Kramer, K.L. Eckerle, D.L. Duewer, Anal Chem., **74**, 3408-3415, (2002) "An International Evaluation of Holmium Oxide Solution Reference Materials for Wavelength Calibration in Molecular Absorption Spectrophotometry"
- [8] Official Methods of Analysis, **970.44**, 1185-1186, (1990) 16th Ed., "Preparation of Standards for Mycotoxins", AOAC Int. Arlington VA