

3-ACETYLDEOXYNIVALENOL

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:	3-Acetyldeoxynivalenol
CAS number:	50722-38-8
Catalog number:	DRE-C10233000-10MG
Lot #:	S17481A
Certificate version:	1
Expiry date:	27.11.2022
Physical description of RM:	White crystals of 3-Acetyldeoxynivalenol
Packaging and amount of RM:	Amber glass ampoules fitted with teflon faced butyl septa and PP screw caps, quantities of 10 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 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

3-Acetyldeoxynivalenol		
Compound	Purity	
	Certified value ^a	Uncertainty ^b
3-Acetyldeoxynivalenol	99.4 %	± 0.6 %

^a The certified value is based upon the results from several analytical techniques
^b Expanded uncertainty U ($k=2$) of the value u_c according to GUM [4]

4. Discussion of traceability

The purity was confirmed by HPLC-UV and UV-Spectrophotometry. All weighting and dilution steps for preparation were done using calibrated equipment (microbalances, pipettes). The gravimetric preparation furthermore was performed in a standardized and certified class A flask with stated uncertainty as well as using traceable thermometers for temperature controlled preparation. The whole preparation process is therefore traceable to SI units and metrological traceability is given.

5. Purity assessment of 3-Acetyldeoxynivalenol

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 [5]. The absorbance scale and the linearity of the apparatus was validated with potassium dichromate in dilute sulfuric acid [6]. All measurements were performed at 25°C.

The UV absorption spectrum of 3-Acetyldeoxynivalenol was consistent with literature data [7] and showed no detectable impurities.

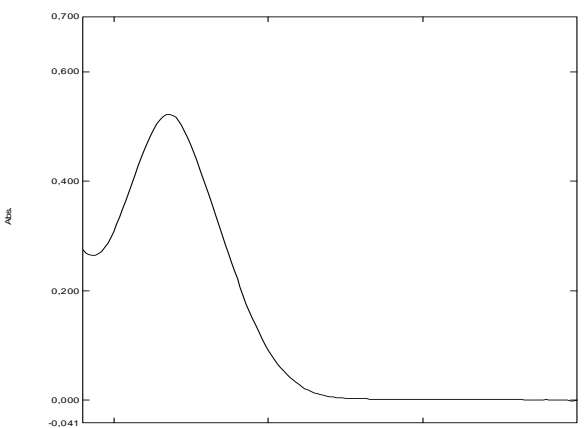
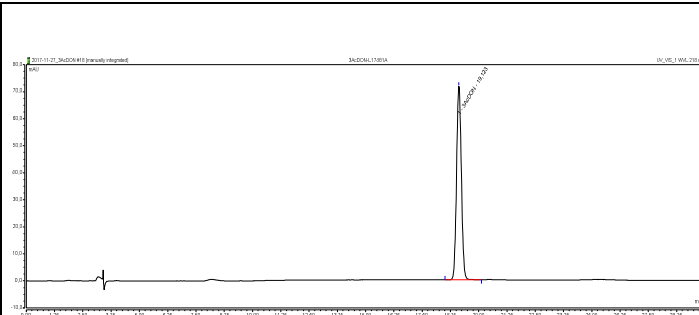
molecular mass	m	=	338.4	g/mol	
molar absorptivity in AcN ^a	ϵ	=	6800	L mol ⁻¹ cm ⁻¹	
wavelength	λ	=	218	nm	
dilution factor	f	=	4		
absorbance at $\lambda = 218$ nm ^b	A	=	0.5183		
optical path length	d	=	1	cm	
mass concentration ^c	c_m	=	102.8 ± 1.2	µg/mL	
analytical concentration ^d	c	=	103.2 ± 3.0	µg/mL	
$c = \frac{A \times m \times 1000 \times f}{\epsilon \times d} = \frac{0.5183 \times 338.4 \times 1000 \times 4}{6800 \times 1} = 103.2 \text{ mg/L} =$					
^a Molar absorptivity determined according to literature: "G.A. Bennett, O.L. Shotwell, J. Assoc. Off. Anal. Chem., 73 , 270-275, (1990)" ^b Mean of 6 replicate measurements ^c Mass concentration calculated on weighed amount of sample with expanded uncertainty according to GUM [4] ^d confidence interval with P = 95 %					

Figure 4: UV absorption spectrum of 3-Acetyldeoxynivalenol sample

REFERENCE MATERIAL CERTIFICATE

5.2. HPLC-DAD:

The purity check using gradient LC-DAD of the 3-Acetyldeoxynivalenol sample showed one main peak and one minor impurity peak. The peak purity of the main signal was examined by diode array spectra of the 3-Acetyldeoxynivalenol peak and led to the conclusion that this peak consists only of 3-Acetyldeoxynivalenol. The peak area of the single minor signal was 0.3 % (8.4 min) compared to the main peak.

<table style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 15%;">column</td> <td colspan="2">Phenomenex Luna C18(2), 3 x 100 mm, 5µm</td> </tr> <tr> <td>injection volume</td> <td colspan="2">100 µL sample</td> </tr> <tr> <td>solvent A</td> <td colspan="2">Acetonitrile / water (10 / 90)</td> </tr> <tr> <td>solvent B</td> <td colspan="2">acetonitrile</td> </tr> <tr> <td>flow rate</td> <td colspan="2">0.5 mL / min</td> </tr> <tr> <td>gradient</td> <td style="text-align: center;">time in minutes (min)</td> <td style="text-align: center;">% solvent B</td> </tr> <tr> <td></td> <td style="text-align: center;">0 – 3</td> <td style="text-align: center;">0</td> </tr> <tr> <td></td> <td style="text-align: center;">3 – 6</td> <td style="text-align: center;">0 - 11</td> </tr> <tr> <td></td> <td style="text-align: center;">6 – 20</td> <td style="text-align: center;">11</td> </tr> <tr> <td></td> <td style="text-align: center;">20 – 21</td> <td style="text-align: center;">11 - 0</td> </tr> <tr> <td></td> <td style="text-align: center;">21 – 30</td> <td style="text-align: center;">0</td> </tr> <tr> <td>DAD settings</td> <td colspan="2">218 nm</td> </tr> <tr> <td>sample dilution</td> <td colspan="2">1:20 with water</td> </tr> </table>	column	Phenomenex Luna C18(2), 3 x 100 mm, 5µm		injection volume	100 µL sample		solvent A	Acetonitrile / water (10 / 90)		solvent B	acetonitrile		flow rate	0.5 mL / min		gradient	time in minutes (min)	% solvent B		0 – 3	0		3 – 6	0 - 11		6 – 20	11		20 – 21	11 - 0		21 – 30	0	DAD settings	218 nm		sample dilution	1:20 with water		 <p style="text-align: center;">HPLC-DAD separation of 3-Acetyldeoxynivalenol sample</p> <table style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th></th> <th style="text-align: center;">time</th> <th style="text-align: center;">area</th> <th style="text-align: center;">height</th> </tr> </thead> <tbody> <tr> <td>3-Acetyldeoxynivalenol</td> <td style="text-align: center;">19.12 min</td> <td style="text-align: center;">17.64</td> <td style="text-align: center;">71.73</td> </tr> </tbody> </table>		time	area	height	3-Acetyldeoxynivalenol	19.12 min	17.64	71.73
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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: 27.11.2017

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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] 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"
- [6] Official Methods of Analysis, **970.44**, 1185-1186, (1990) 16th Ed., "Preparation of Standards for Mycotoxins", AOAC Int. Arlington VA
- [7] E.W. Sydenham, P.G. Thiel, R. Vleggaar, J. AOAC Int., **79**, 1365-1379, (1996), "Physicochemical Data for Some Selected Fusarium Toxins"