

Certificate of Analysis^(Ver.1.0)

U-[¹³C₁₄]-Alternariol,U-[¹³C₁₅]-Alternariol monomethyl ether,U-[¹³C₂₂]-Tentoxin,U-[¹³C₁₀]-Tenuazonic acid in Methanol

1. General information

This document is designed and the certified value(s) and uncertainty(ies) are determined in accordance with ISO Guide 31[1].

2. Description of the Reference Material (RM)

Name: U-[¹³C₁₄]-Alternariol,U-[¹³C₁₅]-Alternariol monomethyl ether,
U-[¹³C₂₂]-Tentoxin,U-[¹³C₁₀]-Tenuazonic acid in Methanol

Catalog number: STD#7059U

CAS number: U-[¹³C₁₄]-Alternariol:641-38-3(unlabeled);
U-[¹³C₁₅]-Alternariol monomethyl ether:26894-49-5(unlabeled);
U-[¹³C₂₂]-Tentoxin:28540-82-1(unlabeled);
U-[¹³C₁₀]-Tenuazonic acid:610-88-8(unlabeled);

Formula: U-[¹³C₁₄]-Alternariol:¹³C₁₄H₁₀O₅ ;
U-[¹³C₁₅]-Alternariol monomethyl ether:¹³C₁₅H₁₂O₅ ;
U-[¹³C₂₂]-Tentoxin:¹³C₂₂H₃₀N₄O₄;
U-[¹³C₁₀]-Tenuazonic acid:¹³C₁₀H₁₅NO₃;

Formula weight: U-[¹³C₁₄]-Alternariol:272.23 ;
U-[¹³C₁₅]-Alternariol monomethyl ether:287.26 ;
U-[¹³C₂₂]-Tentoxin:436.50;
U-[¹³C₁₀]-Tenuazonic acid:207.23;

Lot #: **2A01A27**

Starting material : U-[¹³C₁₄]-Alternariol : lot#J19216P,Pribolab Pte.Ltd.
U-[¹³C₁₅]-Alternariol monomethyl ether : lot#J19216P,Pribolab Pte.Ltd.
U-[¹³C₂₂]-Tentoxin,lot#I19018P,Pribolab Pte.Ltd.
U-[¹³C₁₀]-Tenuazonic acid,lot#T18112J,Pribolab Pte.Ltd.

Solvent: Methanol,LiChrosolv[®],Merck

Amount: 1.2mL

Production date: **29/Nov/2021**

Expiry date: **28/Nov/2022**

Name of the supplier: Pribolab Pte.Ltd.

2.1 Intended use of the RM

- for laboratory use only
- internal standard[2]

2.2 Instruction for the correct use of the RM

The compound should be stored at 2-8°C in a dark place. Before usage of the RM, the compound should be allowed to warm to temperature (20±3°C). 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.

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.

| Hazardous Ingredients | Concentration in% | Pictograms | Signal word | Hazard statement(s) |
|-----------------------|-------------------|---|-------------|------------------------------|
| Methanol | >99.9 |  | Danger | H225, H301, H311, H331, H370 |

3. Certified values and their uncertainties

| U-[¹³ C ₁₄]-Alternariol, U-[¹³ C ₁₅]-Alternariol monomethyl ether, U-[¹³ C ₂₂]-Tentoxin, U-[¹³ C ₁₀]-Tenuazonic acid in Methanol | | |
|--|---------------------------------|--------------------------|
| Compound | Mass concentration ^a | |
| | Certified value ^b | Uncertainty ^c |
| U-[¹³ C ₁₄]-Alternariol, 90.63 atom% ¹³ C | 1.00 µg/mL | ±0.02 µg/mL |
| U-[¹³ C ₁₅]-Alternariol monomethyl ether, 98.36 atom% ¹³ C | 1.01 µg/mL | ±0.01 µg/mL |
| U-[¹³ C ₂₂]-Tentoxin, 96.99 atom% ¹³ C | 1.00 µg/mL | ±0.02 µg/mL |
| U-[¹³ C ₁₀]-Tenuazonic acid, 97.53 atom% ¹³ C | 10.04 µg/mL | ±0.18 µg/mL |

a Values are based on preparation data and confirmed experimentally by HPLC-DAD
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[3]

4. Isotopic enrichment and isotope pattern

| Isotope pattern ^a | | | |
|--|-----------------------|---|-----------------------|
| Compound | Isotopic distribution | Compound | Isotopic distribution |
| U-[¹³ C ₁₄]-Alternariol | 53.96% | U-[¹³ C ₂₂]-Tentoxin | 52.09% |
| U-[¹³ C ₁₃]-Alternariol | 30.77% | U-[¹³ C ₂₁]-Tentoxin | 29.58% |
| U-[¹³ C ₁₂]-Alternariol | 9.45% | U-[¹³ C ₂₀]-Tentoxin | 18.33% |
| U-[¹³ C ₁₅]-Alternariol monomethyl ether | 83.33% | U-[¹³ C ₁₀]-Tenuazonic acid | 82.30% |
| U-[¹³ C ₁₄]-Alternariol monomethyl ether | 8.75% | U-[¹³ C ₉]-Tenuazonic acid | 10.70% |
| U-[¹³ C ₁₃]-Alternariol monomethyl ether | 7.92% | U-[¹³ C ₈]-Tenuazonic acid | 7.00% |

Calculated isotopic enrichment level ^a: U-[¹³C₁₄]-Alternariol 90.63 atom% ¹³C; U-[¹³C₁₅]-Alternariol monomethyl ether 98.36 atom% ¹³C; U-[¹³C₂₂]-Tentoxin 96.99 atom% ¹³C; U-[¹³C₁₀]-Tenuazonic acid 97.53 atom% ¹³C

^a Approximation based on LC-MS/MS data

5. Discussion of traceability

This calibrant is certified on the basis of gravimetric preparation [4]. Thus the certified value (mass concentration of U-[¹³C₁₄]-Alternariol, U-[¹³C₁₅]-Alternariol monomethyl ether, U-[¹³C₂₂]-Tentoxin, U-[¹³C₁₀]-Tenuazonic acid) 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.

6. Confirmation of certified value by HPLC-DAD

The certified concentration of the gravimetric prepared solution was confirmed by HPLC-DAD against an independently prepared reference batch of unlabeled Alternariol, Alternariol monomethyl ether, Tentoxin, Tenuazonic acid.

7. Further information

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

Inspected by



Quality System Specialist

References:

- [1] ISO Guide 31:2015 - 1-18, "Reference materials – contents of certificates, labels and accompanying documentation"
- [2] G. Häubl, F. Berthiller, R. Krska, R. Schuhmacher, "Suitability of a fully ^{13}C isotope labelled internal standard for the determination of the mycotoxin deoxynivalenol by LC-MS/MS without clean-up", *Anal. Bioanal. Chem.* 384 (3), (2006), 692-696
- [3] International Organization for Standardization (ISO), (2008), "Guide to the expression of uncertainty in measurement", (GUM 1995 with minor corrections) 1st Ed. Geneva, Switzerland
- [4] E.W. Flick, (1998), "Industrial Solvents Handbook", 5th Ed., Noyes Data Corp. Westwood NJ