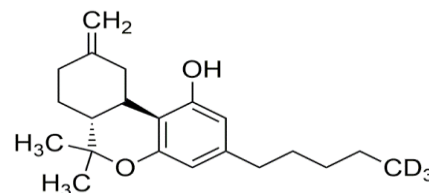


# Certified Reference Material - Certificate of Analysis

## (-)-exo-THC-D<sub>3</sub>, Primary Measurement Standard

(6aR,10aR)-6a,7,8,9,10,10a-hexahydro-6,6-dimethyl-9-methylene-3-(5,5,5-trideutropentyl)-6H-dibenzo[b,d]pyran-1-ol

**Product No.:** T-154-1ML  
**Lot No.:** FE04142123  
**Description of CRM:** (-)-exo-THC-D<sub>3</sub> in Methanol (Solution)  
**Retest Date:** November 2024 See Stability Section  
**Storage:** Store unopened in freezer (-10 °C to -25 °C).  
**Shipping:** Ambient. See Stability Section  
**Chemical formula:** C<sub>21</sub>H<sub>27</sub>D<sub>3</sub>O<sub>2</sub>  
**CAS No.:** NA  
**Regulatory:** USDEA Exempt | Canadian TK # 007-018



### Analyte

**Certified Concentration ± associated uncertainty  $U$ ,  $u = k * u$  ( $k = 2$ )**

**(-)-exo-THC-D<sub>3</sub>**

**100.0 ± 0.6 µg/mL**

**Metrological traceability:** Traceable to the SI and higher order standards from NIST through an unbroken chain of comparisons. See "Details on metrological traceability" on page 3.

**Measurement method:** The certified value is calculated from high precision weighing of thoroughly characterized starting material. See "Details about certification process" on page 3.

**Intended use:** This Certified Reference Material is suitable for the in vitro identification, calibration, and quantification of the analyte(s) in analytical and R&D applications. Not suitable for human or animal consumption.

**Minimum sample size:** 1 µL for quantitative applications

**Instructions for handling and correct use:** Concentration is corrected for chromatographic purity, residual water, residual solvents, and residual inorganics. No adjustment required before use. Users should quantitatively transfer desired volume using established good laboratory practices to spike into matrix or to dilute to the desired concentration. Each ampoule is intended for one-time use. For MS Applications, we advise laboratories not to mix lots during a single sequence.

**Health and safety information:** Danger. Please refer to the Safety Data Sheet for detailed information about the nature of any hazard and appropriate precautions to be taken.

**Accreditation:** Cerilliant Corp. is accredited by the US accreditation authority ANAB as registered reference material producer AR-1353 in accordance with ISO 17034 and registered testing laboratory AT-1352 according to ISO/IEC 17025.



Darron Ellsworth, Quality Assurance Manager

**December 08, 2023**

Issue Date

Cerilliant Corporation, 811 Paloma Drive, Suite A, Round Rock, TX, 78665, USA,  
 Tel: 800-848-7837 / 512-238-9974; www.cerilliant.com  
 Sigma-Aldrich Production GmbH is a subsidiary of Merck KGaA, Darmstadt, Germany.



**Packaging:**

2 mL amber USP Type 1 glass ampoule containing not less than 1 mL of certified solution. Ampoules are overfilled to ensure a minimum of 1 mL volume can be transferred when using a 1mL Class A volumetric pipette.

**Details on starting materials:**

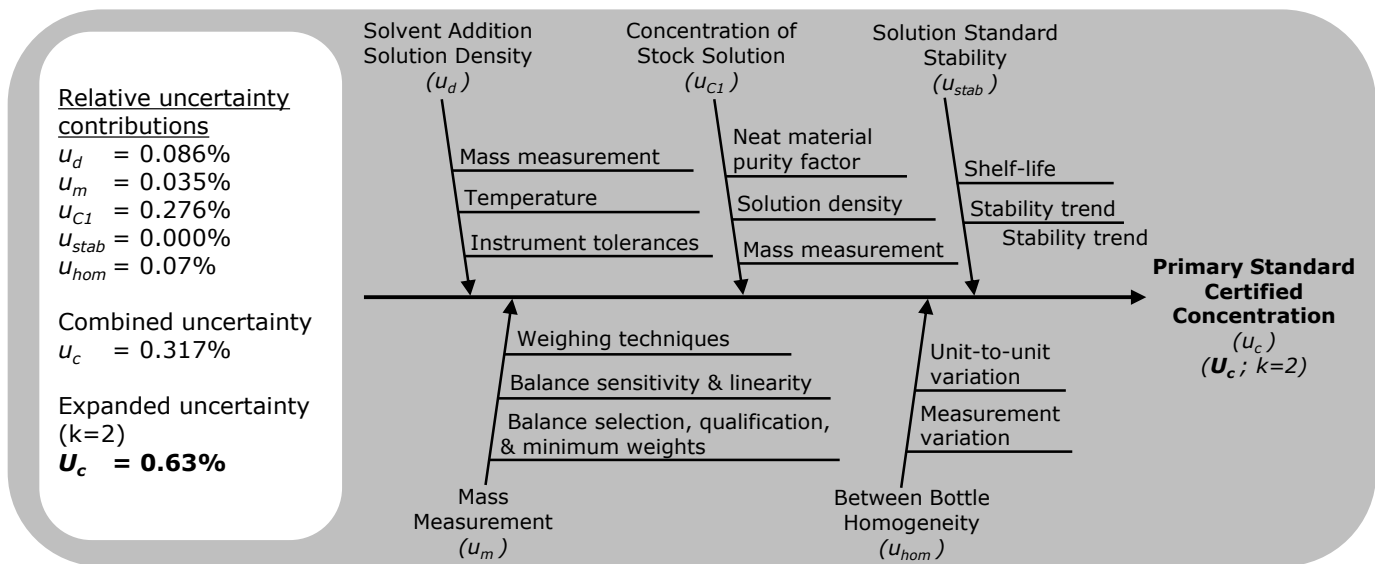
Each raw material utilized has been identified and thoroughly characterized through the use of multiple analytical techniques and is assigned a Mass Balance Purity Factor. Spectral data is provided on subsequent pages of this CoA.

**Certificate of Origin:**

Cerilliant Corporation certifies no material of animal origin (BSE/TSE) was used in the preparation of this product. This material is a product of the USA.

**Associated uncertainty:**

The uncertainty has been calculated by statistical analysis of all aspects of our production system and incorporated uncertainty of the mass balance purity factor, material density, balance, weighing technique, and homogeneity. Uncertainty components of the gravimetrically prepared Primary Standard concentration are shown in the figure below. Uncertainty is expressed as an expanded uncertainty in accordance with ISO 17034 at the approximate 95% confidence interval using a coverage factor of k=2. Uncertainty contribution from neat material homogeneity was established to be negligible through establishment of process controls and verification of the control process. Stability uncertainty was determined to be negligible by regression analysis.



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**Details on metrological traceability:**

- ♦ This standard has been gravimetrically prepared using balances that have been fully qualified and calibrated to ISO 17025 requirements. All calibrations utilize NIST traceable weights which are calibrated externally by a qualified ISO 17025 accredited calibration laboratory to NIST standards. Qualification of each balance includes the assignment of a minimum weighing by a qualified and ISO 17025 accredited calibration vendor taking into consideration the balance and installed environmental conditions to ensure compliance with USP tolerances of NMT 0.10% relative error.
- ♦ Fill volume to predetermined specifications is gravimetrically verified throughout the dispensing process using qualified and calibrated balances.
- ♦ The density and material Mass Balance Purity Factor of each raw material is traceable to the SI and higher order reference materials through mass measurement and instrument qualification and calibrations.

**Details about certification process:**

This standard has been prepared and certified under the ISO 17034, ISO/IEC 17025, and ISO 9001 standards. This standard meets the requirements of a Certified Reference Material and a Primary Standard as defined by ISO and is traceable to the SI and higher order standards through an unbroken chain of comparisons.

- ♦ Nominal concentration is calculated based on: the actual mass; Mass balance purity factor of the analyte(s); measured mass of the solution; and the density of the pure diluent at 20°C.
- ♦ Fill volume is gravimetrically verified throughout the dispensing process using qualified and calibrated balances.
- ♦ Concentration is verified against an independently prepared calibration solution gravimetrically prepared.
- ♦ Additional certification information available upon request.

### Solution Standard Verification

Concentration accuracy and within- and between-bottle homogeneity are analytically verified against an independently prepared calibration solution.

Standard Solution Assay Parameters		Calibration Curve	
<b>Analysis Method:</b>	HPLC/UV	<b>Calibration Curve:</b>	Linear Regression
<b>Column:</b>	Ascentis Express C18, 2.7 µm, 3.0 x 100 mm	<b>Number of Points:</b>	4
<b>Mobile Phase:</b>	Acetonitrile:0.1% Phosphoric acid in Water (85:15)	<b>Linearity (r) :</b>	1.000
<b>Flow Rate:</b>	1.5 mL/min		
<b>Wavelength:</b>	228 nm		
		Verified Concentration (µg/mL)	%RSD - Homogeneity
Standard Solution	Lot Number	Actual Results	Actual Results
New Lot	FE04142123	101.3	0.2
<ul style="list-style-type: none"><li>♦ Concentration is verified through multiple analyses and is calculated as the average of multiple analyses compared to an independently prepared calibration solution.</li><li>♦ Within-sample and between-sample homogeneity of the New Lot is ensured through rigorous production process controls statistically analyzed to evaluate risk and verified by analysis. Multiple samples pulled from across the lot using a random stratified sampling plan were analyzed to verify homogeneity. % RSD results shown above for the New Lot demonstrate ampoule-to-ampoule homogeneity.</li></ul>			

### Analyte Certification - Mass Balance Purity Factor

Each analyte is thoroughly identified and characterized using an orthogonal approach. A mass balance purity factor is assigned incorporating chromatographic purity and residual impurities. The mass balance purity factor is utilized to calculate the weighing adjustment necessary to ensure accuracy of the solution standard concentration.

<b>Material Name:</b>	(-)-exo-THC-D <sub>3</sub>	<b>Chemical Formula:</b>	C <sub>21</sub> H <sub>27</sub> D <sub>3</sub> O <sub>2</sub>
<b>Material Lot:</b>	FC01142014	<b>CAS Number:</b>	NA
		<b>Molecular Weight:</b>	317.48

#### Material Characterization Summary

Analytical Test	Method	Results
Primary Chromatographic Purity by HPLC/UV Analysis	20384348	97.1%
Secondary Chromatographic Purity by GC/FID Analysis	20384346	97.9% <sup>1</sup>
Identity by GC/MS Analysis	20384214	Consistent with Structure <sup>1</sup>
Isotopic Purity and Distribution by GC/MS SIM Analysis	20384214	0.62% D <sub>0</sub> vs D <sub>3</sub>
		0.58% D <sub>0</sub> 4.64% D <sub>2</sub>
		1.02% D <sub>1</sub> 93.75% D <sub>3</sub>
Identity by <sup>1</sup> H-NMR Analysis	20384224	Consistent with Structure
Residual Solvent Analysis by GC/FID Headspace	20397799 <sup>2</sup>	3.20%
Residual Water Analysis by Karl Fischer Coulometry	20398075 <sup>2</sup>	Not Detected
Mass Balance Purity Factor		94.04%

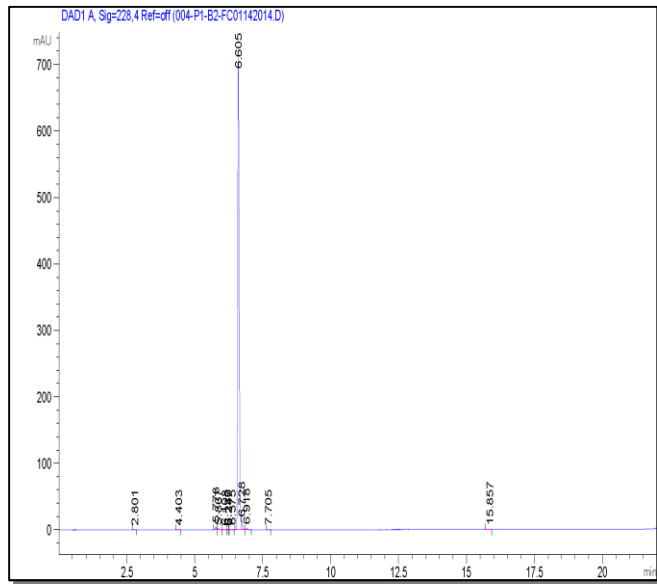
<sup>1</sup> An isobaric impurity was identified by GC/FID and GC/MS analyses.

<sup>2</sup> Validated analytical method

- ♦ The primary chromatographic purity is calculated as the average of two independently performed analyses utilizing two different methods. Acceptance criteria requires the purity values to be within 0.5% of each other.
- ♦ The primary purity method was selected to optimize resolution of impurities while minimizing degradation of the analyte. Secondary purity methods with orthogonal detector capabilities from the primary purity method are used as controls to confirm an accurate purity value.
- ♦ The primary chromatographic purity value is used to calculate the Mass Balance Purity Factor.
- ♦ A secondary chromatographic purity method is utilized as a control.
- ♦ Mass Balance Purity Factor = [(100 - wt% residual solvent - wt% residual water - wt% residual inorganics) x Chromatographic Purity/100].
- ♦ Mass Balance Purity Factor does not include adjustment for chiral and/or isotopic purity.

## Spectral and Physical Data

### HPLC/UV



**Column:** Ascentis Express C18, 2.7  $\mu\text{m}$ ,  
3.0 x 100 mm

**Mobile Phase:** A: Acetonitrile  
B: 0.1% Phosphoric acid in Water

**Gradient:**

Time (min)	% A	% B
0.0	65	35
10.0	95	5
20.0	95	5
20.1	65	35
25.0	65	35

**Flow Rate:** 0.6 mL/min

**Wavelength:** 228 nm

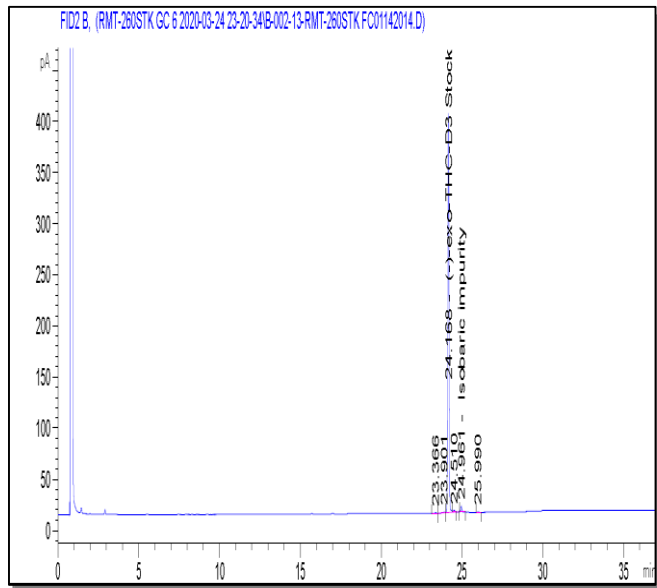
**Sample Name:** FC01142014

**Acquired:** February 01, 2021

Peak #	Ret Time	Area %
1	2.80	0.01
2	4.40	0.05
3	5.78	0.50
4	5.87	0.22
5	6.11	0.05
6	6.23	0.00
7	6.25	0.00
8	6.38	0.07
9	6.61	97.17
10	6.73	1.58
11	6.92	0.27
12	7.71	0.05
13	15.86	0.03

## Spectral and Physical Data (cont.)

### GC/FID



**Column:** DB-35ms, 30 m x 0.53 mm ID,  
1.0 µm film thickness

**Temp Program:** 60°C to 200°C at 10°C/min  
200°C to 280°C at 5°C/min  
hold 7 min

**Injector Temp:** Cool-on-Column

**Detector Temp:** 325°C

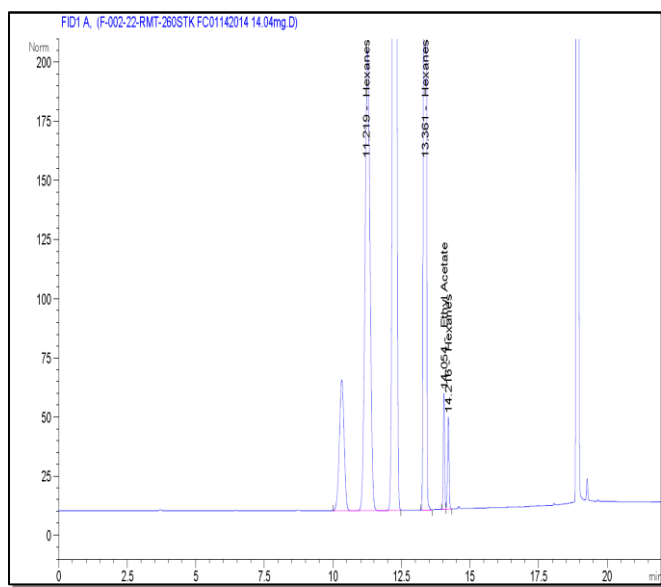
**Sample Name:** FC01142014

**Acquired:** March 25, 2020

Peak #	Ret Time	Area %
1	23.37	0.06
2	23.90	0.18
3	24.17	97.93
4	24.51	0.27
5	24.96	1.41
6	25.99	0.15

Peak 5 has been identified as an isobaric impurity.

### Residual Solvent Analysis by GC/FID Headspace



**Column:** DB-ALC1 30 m x 0.53 mm,  
3 µm film thickness

**Temp Program:** 40°C hold 12 min to 220°C  
at 40°C/min hold 5.5 min

**Carrier Gas:** Helium

**Flow Rate:** 2.0 mL/min

**Detector Heater Temp:** 250°C

**Injector:** Headspace Sampler

**HS Oven Temp:** 60°C

**Vial Equilibration:** 10 minutes

**Sample Name:** FC01142014

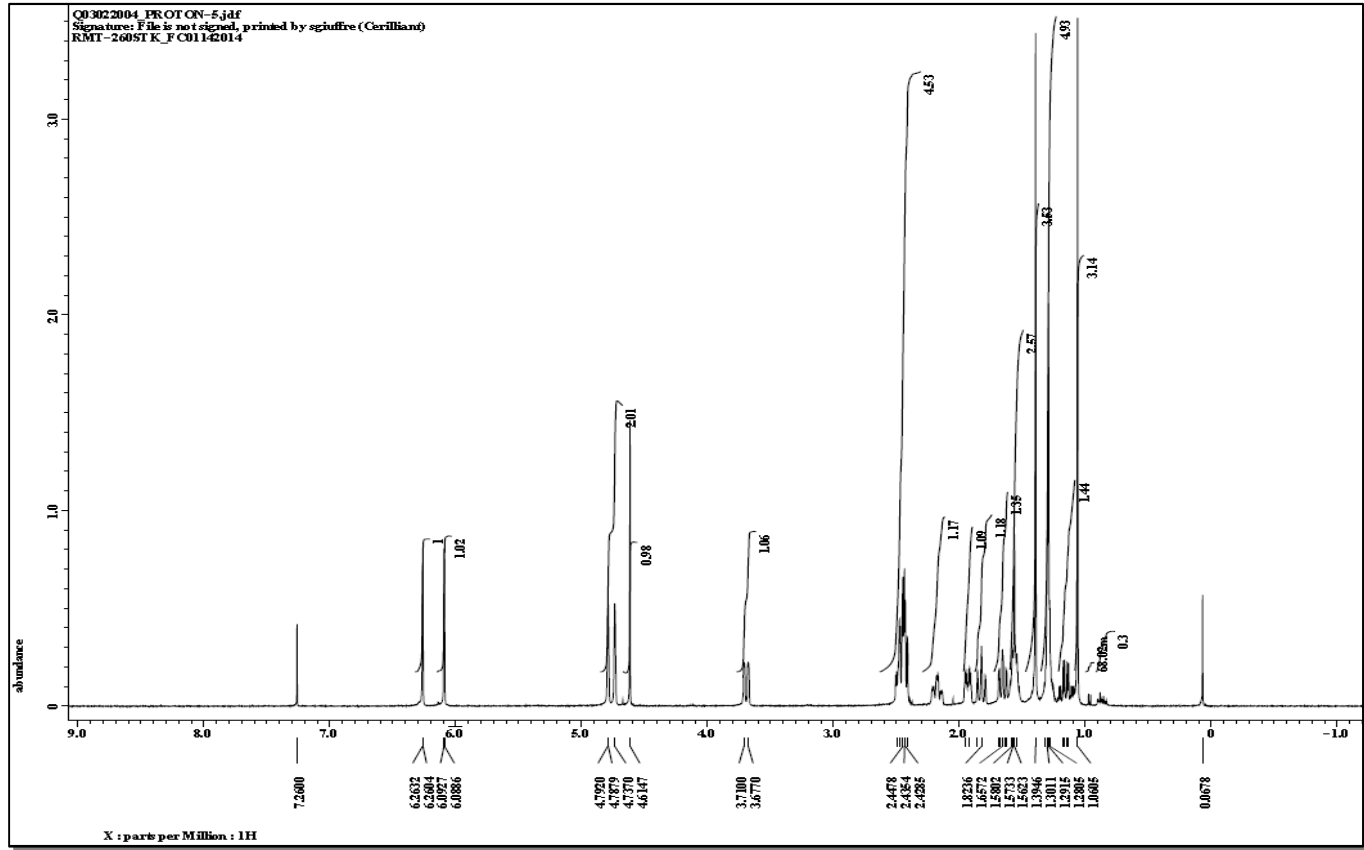
**Acquired:** March 11, 2020

Peak	Compound	Area	Weight %
1	Hexanes	14641.30	2.83
2	Ethyl acetate	164.89	0.37
3	NMP	NA	NA
<b>Total</b>			<b>3.20</b>

Spectral and Physical Data (cont.)

<sup>1</sup>H NMR

Instrument: JEOL ECS 400  
Solvent: Chloroform-D

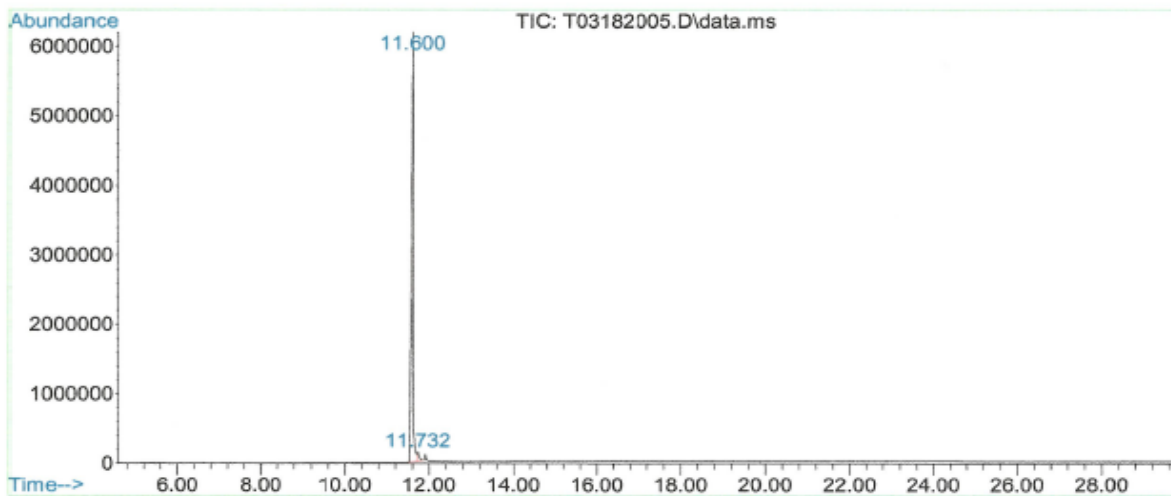


## Spectral and Physical Data (cont.)

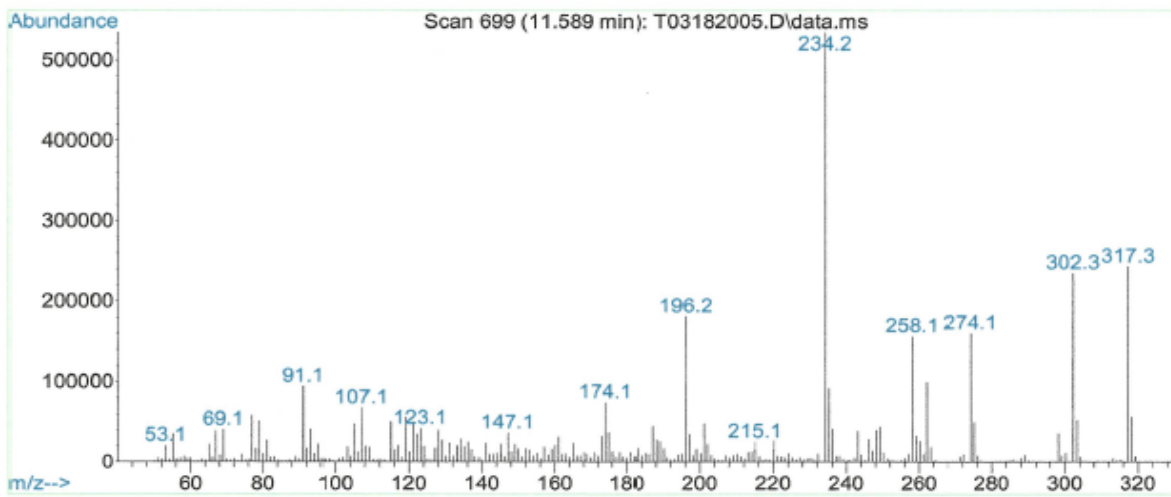
### GC/MS

Compound Name : (-)-exo-THC-D3  
Lot Number : FC01142014  
Instrument : Agilent GCMS  
Operator : ECM(SGIUFFRE)  
Date Reported : Wed Mar 18 12:15:09 2020  
Column Type : DB-5ms, 30m x 0.25mm ID, 0.25um film thickness  
Temp. Program : 50°C to 200°C@40°C/min, 200°C to 300°C@10°C/min, 16min hold  
Injector Temp. : Cool on-column  
Carrier Gas : Helium  
Flow Rate (mL\min) : 0.80 mL/min  
Transfer Line Temp. : 280°C  
Scan Range : 50-500

#### Total Ion Chromatogram



#### Mass Spectrum



## Stability

Short term stability studies have been performed in multiple storage conditions for a period of up to four weeks. Short term data is utilized to support transport conditions and normal laboratory use. Real-time stability studies are performed at the recommended storage conditions over the life of the product.

**Short Term Stability:** A summary of stability findings for a related product (T-033, exo-THC) is listed below.

Storage Condition	Targeted Mean Kinetic Temperature (MKT)	Time Period/Result
Freezer	-20°C	No decrease in purity was noted after four weeks.
Refrigerator	5°C	
Room Temperature	20°C	
40°C	40°C	

**Transport/Shipping:** Stability studies support the transport of this product at ambient conditions.

**Long Term Stability:** Long term stability has been assessed for Freezer storage (-10 °C to -25 °C) conditions. Stability of a minimum of 31 months has been established through real-time stability studies.

## Commutability

This standard is a solution of a pure substance in an organic solvent and is a Primary Standard. This Primary Standard is suitable for use in the preparation of calibrators and/or controls in any biological matrix. This standard is not in a biological matrix and therefore commutability to methods or standards in biological matrices does not apply.

## COA Revision History

Revision No.	Date	Reason for Revision
00	May 25, 2021	Initial version.
01	May 13, 2022	Revised Retest Date from June 2022 to April 2023.
		Added Long Term Stability data.
02	February 22, 2023	Revised Retest Date from April 2023 to February 2024.
03	December 08, 2023	Revised Retest Date from February 2024 to November 2024.

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