

Certified Reference Material - Certificate of Analysis

Cannabinoid Mixture (Acids) - 6 Component

Catalog Number: C-218S-1ML
Solution Lot: FE02082205
Retest Date: December 2024
Solvent: 1% DIPEA and 0.05% Ascorbic acid in Acetonitrile
Volume per Ampoule: Not less than 1 mL
Storage: Store unopened and upright in sub-freezer (-60 °C to -80 °C).
Shipping: Ship cold. See Stability Section.
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.

Instructions for 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.

Regulatory: USDEA Exempt | Canadian CTK # 007-043
Country of Origin: United States of America
Safety: Danger. See Safety Data Sheet

- ♦ Retest Date - stability studies ongoing. Certificate of Analysis will be updated upon completion of retest.
- ♦ Ampoules are overfilled to ensure a minimum 1 mL volume can be transferred when using a 1 mL Class A volumetric pipette.
- ♦ For quantitative applications, the minimum sample size for intended use is 1 µL.

Component	Chromatographic Purity	Concentration (µg/mL)
Cannabichromenic acid (CBCA)	99.8%	500 ± 10
Cannabidiolic acid (CBDA)	99.3%	500 ± 10
Cannabidivarinic acid (CBDVA)	99.3%	500 ± 10
Cannabigerolic acid (CBGA)	99.3%	500 ± 10
Δ ⁹ -Tetrahydrocannabinolic acid A (THCA-A)	99.1%	500 ± 10
Tetrahydrocannabivarinic acid (THCVA)	97.9%	500 ± 10

- ♦ Uncertainty of the concentration is expressed as an expanded uncertainty in accordance with ISO 17025 and ISO 17034 at the approximate 95% confidence interval using a coverage factor of $k = 2$ and has been calculated by statistical analysis of our production system and incorporates uncertainty of the purity factor, material density, and balance and weighing technique.
- ♦ This standard is prepared gravimetrically and mass results are reported on the conventional basis for weighing in air. Concentration is calculated based on: the actual measured mass; Purity Factor of the analyte(s); measured mass of the solution; and the density of the pure diluent at 20°C.
- ♦ Concentration is corrected for chromatographic purity, residual water, residual solvents and residual inorganics.

Cerilliant certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration/retest date when stored unopened as recommended. Product should be used shortly after opening to avoid concentration changes due to evaporation. Warranty does not apply to ampoules stored after opening.



A handwritten signature in black ink, appearing to read "Dell".

Darron Ellsworth, Quality Assurance Manager

May 09, 2023

Date

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Sigma-Aldrich Production GmbH is a subsidiary of Merck KGaA, Darmstadt, Germany.



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.
- ◆ Concentration is verified against an independently prepared calibration curve gravimetrically prepared using balances calibrated to NIST.
- ◆ In addition, each neat material utilized has been identified and thoroughly characterized through the use of multiple analytical techniques. Spectral data is provided on subsequent pages of the COA.

Standard Solution Assay Parameters

Analysis Method: HPLC/UV
Column: Ascentis Express RP-Amide,
2.7 μ 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
8.0	90	10
10.0	90	10
10.1	65	35

Flow Rate: 1.75 mL/min
Wavelength: 228 nm

Calibration Curve

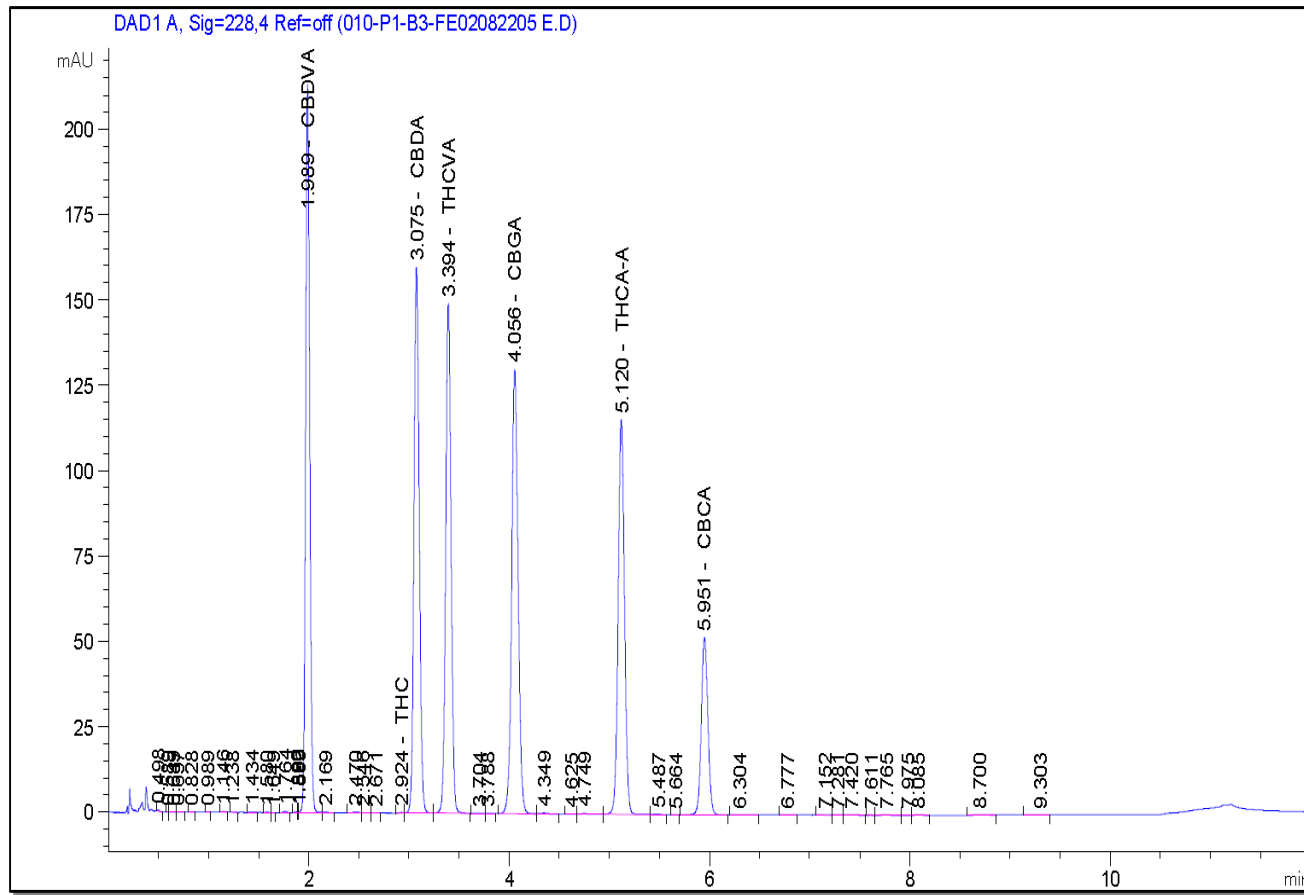
Calibration Curve: Linear Regression
Number of Points: 1

Solution Standard Verification and Homogeneity

Compound	Verified Concentration (μ g/mL)		%RSD - Homogeneity	
	Actual Results	Acceptance Criteria	Actual Results	Acceptance Criteria
Cannabichromenic acid (CBCA)	503	$\pm 5\%$	0.6	$\leq 3\%$
Cannabidiolic acid (CBDA)	503	$\pm 5\%$	0.6	$\leq 3\%$
Cannabidivarinic acid (CBDVA)	512	$\pm 5\%$	0.6	$\leq 3\%$
Cannabigerolic acid (CBGA)	502	$\pm 5\%$	0.6	$\leq 3\%$
Δ^9 -Tetrahydrocannabinolic acid A (THCA-A)	502	$\pm 5\%$	0.6	$\leq 3\%$
Tetrahydrocannabivarinic acid (THCVA)	502	$\pm 5\%$	0.6	$\leq 3\%$

- ◆ Concentration is verified through multiple analyses and is calculated as the average of multiple analyses compared to an independently prepared calibration curve.
- ◆ Homogeneity is ensured through rigorous production process controls statistically analyzed to evaluate risk and verified by analysis. The %RSD of samples pulled from across the lot demonstrate homogeneity.
- ◆ Product is compared to prior lot and meets acceptance criteria of $\pm 5\%$.

Solution Standard Analysis



Neat Material Data

Compound	Lot Number	CAS Number	Chemical Formula	Molecular Weight	Identity Confirmed By
Cannabichromenic acid (CBCA)	FN06172101	185505-15-1	C ₂₂ H ₃₀ O ₄	358.47	LC/MS
Cannabidiolic acid (CBDA)	FN11112009	1244-58-2	C ₂₂ H ₃₀ O ₄	358.47	LC/MS
Cannabidivarinic acid (CBDVA)	FC05222001	31932-13-5	C ₂₀ H ₂₆ O ₄	330.42	LC/MS
Cannabigerolic acid (CBGA)	FC05182001	25555-57-1	C ₂₂ H ₃₂ O ₄	360.49	LC/MS
Δ ⁹ -Tetrahydrocannabinolic acid A (THCA-A)	FC10012003	23978-85-0	C ₂₂ H ₃₀ O ₄	358.47	LC/MS
Tetrahydrocannabivarinic acid (THCVA)	FC07082109	39986-26-0	C ₂₀ H ₂₆ O ₄	330.42	LC/MS

Compound	Chromatographic Purity	Residual Solvent ¹	Residual Water ¹	Residual Inorganics	Mass Balance Purity Factor
CBCA	99.8% ³	0.70%	Not Detected	NA	99.11%
CBDA	99.3% ⁴	0.20%	Not Detected	BQL ²	99.12%
CBDVA	99.3% ⁵	1.34%	BQL ²	< 0.2%	98.00%
CBGA	99.3%	0.38%	BQL ²	0.20%	98.69%
THCA-A	99.1%	0.03%	Not Detected	BQL ²	99.08%
THCVA	97.9% ⁶	1.67%	BQL ²	NA	96.26%

- ♦ 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. A secondary purity method is used as a control but is not reported on the Certificate of Analysis.
- ♦ 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.

¹ Validated analytical method

² Below Quantitation Limit

³ 0.04% Cannabichromene (CBC) and 0.05% Cannabicyclic Acid (CBLA) detected by HPLC/UV analysis.

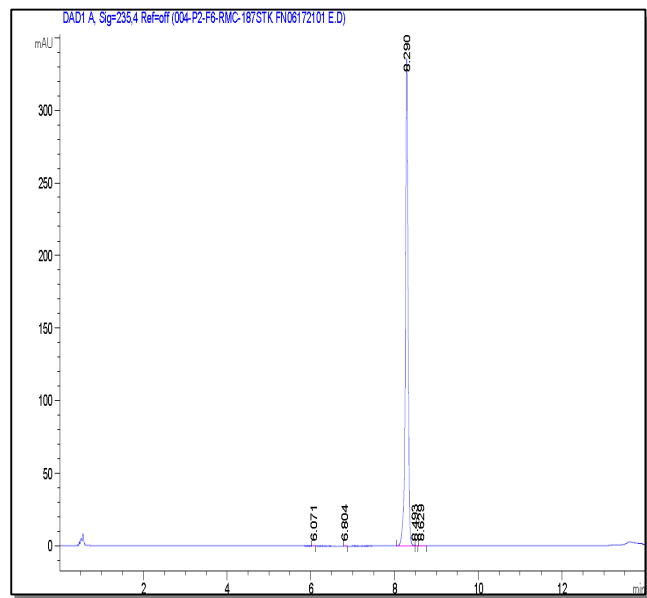
⁴ 0.10% Cannabidiol (CBD) detected by HPLC/UV analysis.

⁵ 0.11% Cannabidivarin (CBDV) detected by HPLC/UV analysis.

⁶ 0.08% Tetrahydrocannabivarin (THCV) detected by HPLC/UV analysis.

Cannabichromenic acid (CBCA)

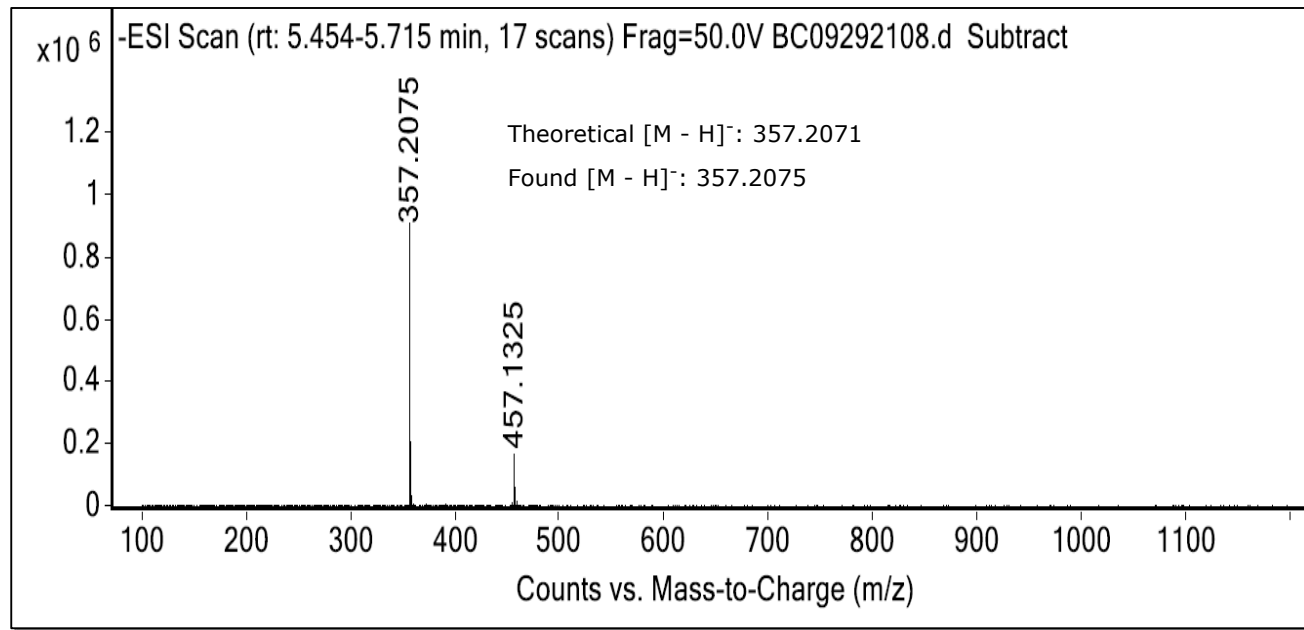
Material Purity by HPLC/UV



Peak #	Ret Time	Area %
1	6.07	0.05
2	6.80	0.04
3	8.29	99.80
4	8.49	0.05
5	8.63	0.07

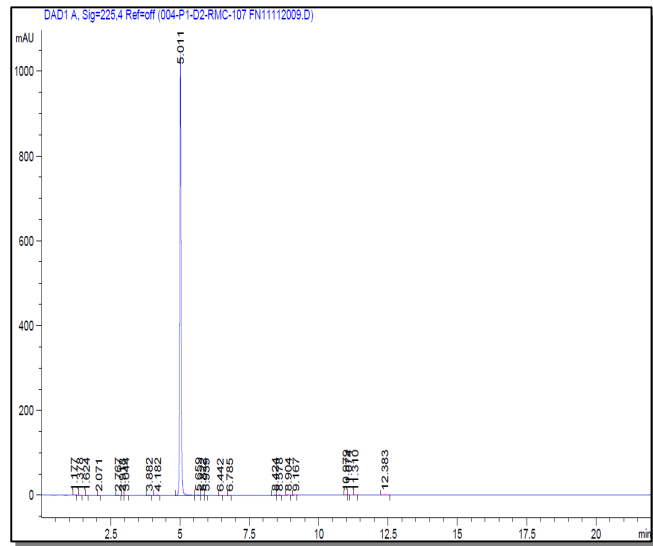
Peak 2 is identified as CBC
Peak 4 is identified as CBLA

Material Identity by LC/MS



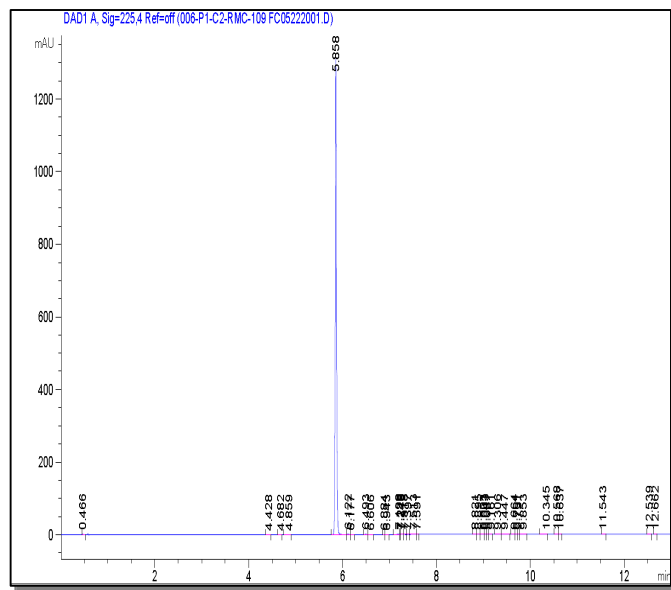
Cannabidiolic acid (CBDA)

Material Purity by HPLC/UV



Cannabidivarinic acid (CBDVA)

Material Purity by HPLC

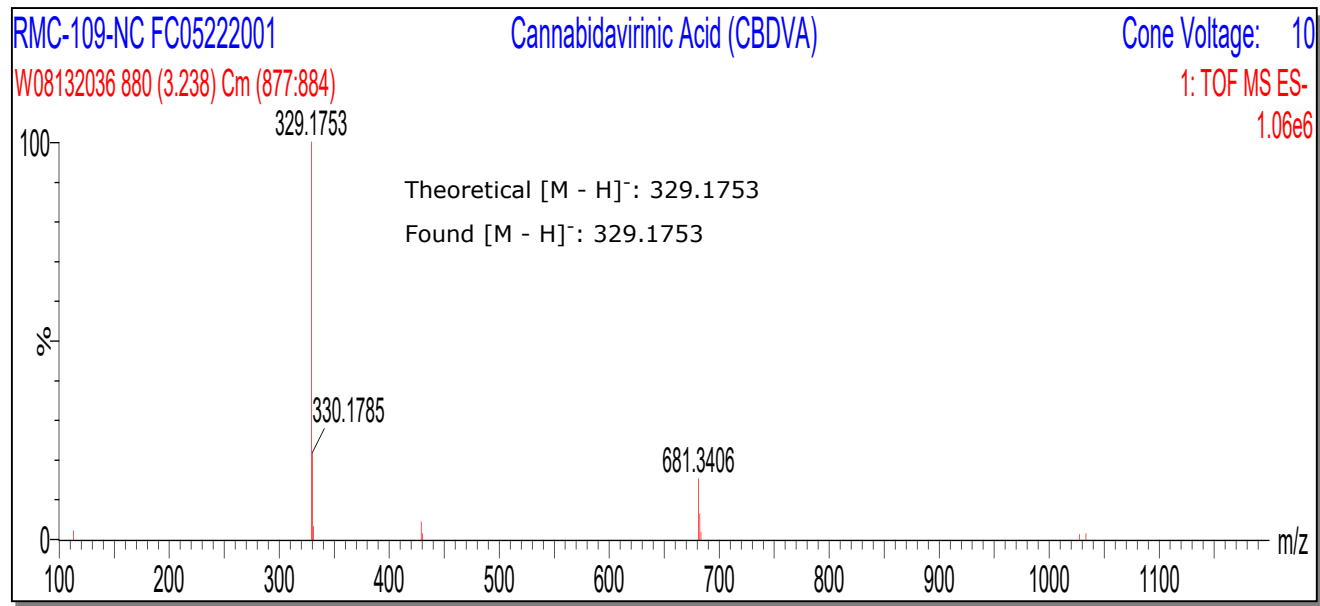


Peak #	Ret Time	Area %
1	0.47	0.06
2	4.43	0.01
3	4.68	0.01
4	4.86	0.01
5	5.86	99.35
6	6.12	0.11
7	6.18	0.01
8	6.49	0.05
9	6.61	0.01
10	6.88	0.00
11	6.94	0.01
12	7.20	0.03
13	7.23	0.01
14	7.25	0.01
15	7.32	0.00
16	7.40	0.01
17	7.51	0.06
18	7.59	0.00
19	8.82	0.01
20	8.90	0.01
21	9.00	0.02
22	9.04	0.02
23	9.08	0.01
24	9.16	0.01
25	9.31	0.05
26	9.45	0.02
27	9.66	0.01
28	9.70	0.01
29	9.75	0.00
30	9.85	0.04
31	10.35	0.01
32	10.57	0.01
33	10.64	0.01
34	11.54	0.01
35	12.54	0.01
36	12.66	0.00

Peak 6 is identified as CBDV

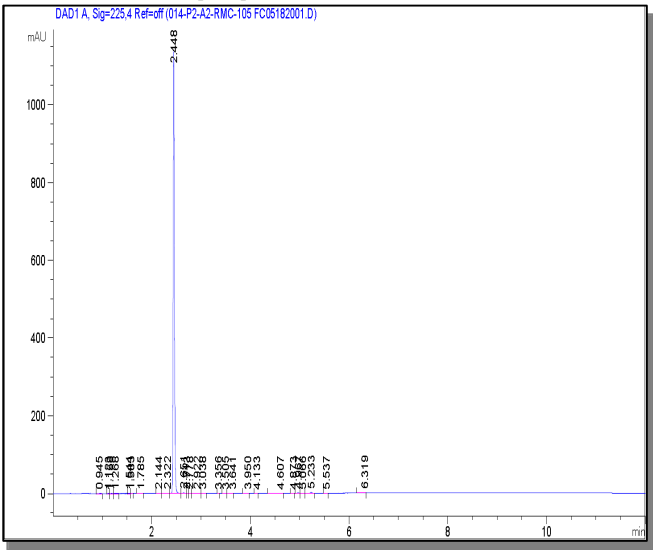
Cannabidivarinic acid (CBDVA) (cont.)

Material Identity by LC/MS



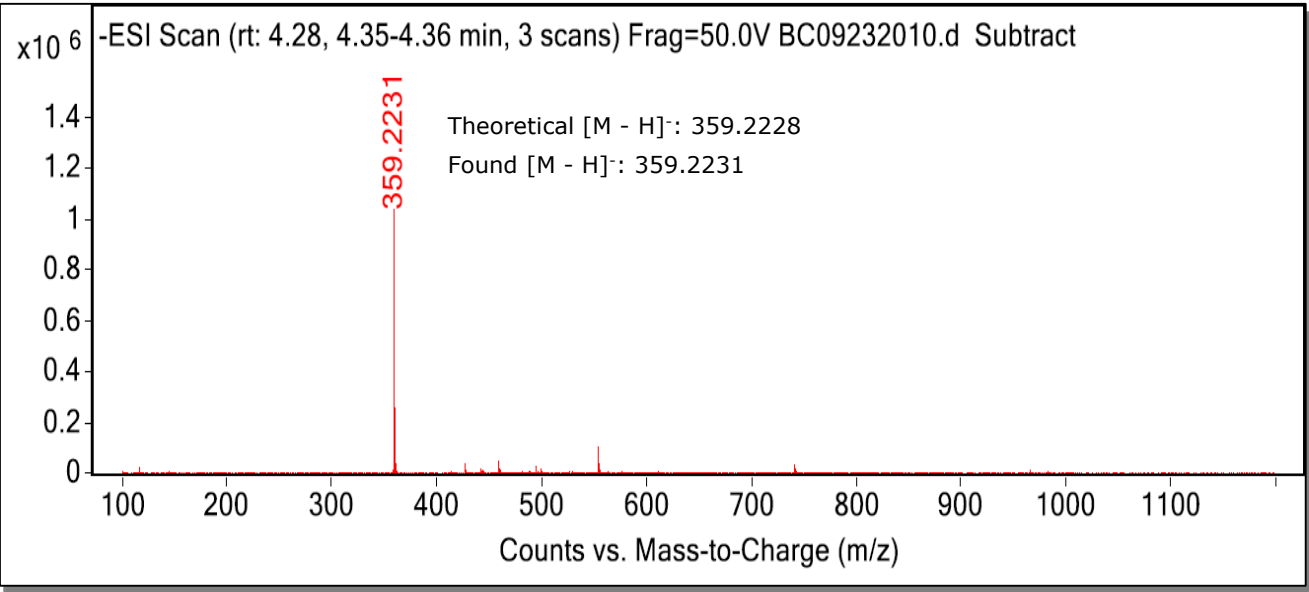
Cannabigerolic acid (CBGA)

Material Purity by HPLC/UV



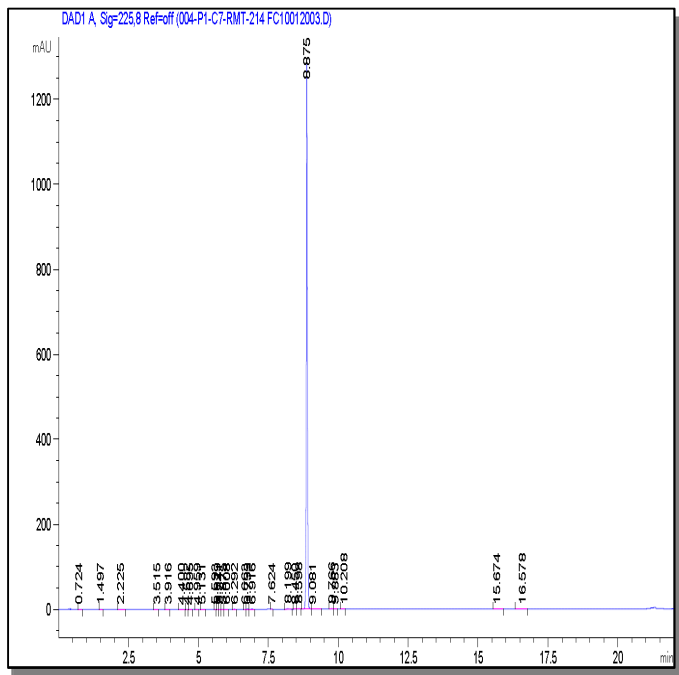
Peak #	Ret Time	Area %
1	0.95	0.01
2	1.12	0.01
3	1.16	0.01
4	1.27	0.03
5	1.54	0.01
6	1.58	0.00
7	1.79	0.01
8	2.14	0.07
9	2.32	0.02
10	2.45	99.12
11	2.65	0.18
12	2.71	0.03
13	2.78	0.03
14	2.92	0.11
15	3.04	0.02
16	3.36	0.01
17	3.51	0.01
18	3.64	0.01
19	3.95	0.02
20	4.13	0.01
21	4.61	0.04
22	4.87	0.01
23	4.97	0.07
24	5.07	0.02
25	5.23	0.15
26	5.54	0.01
27	6.32	0.02

Material Identity by LC/MS



Δ^9 -Tetrahydrocannabinolic acid A (THCA-A)

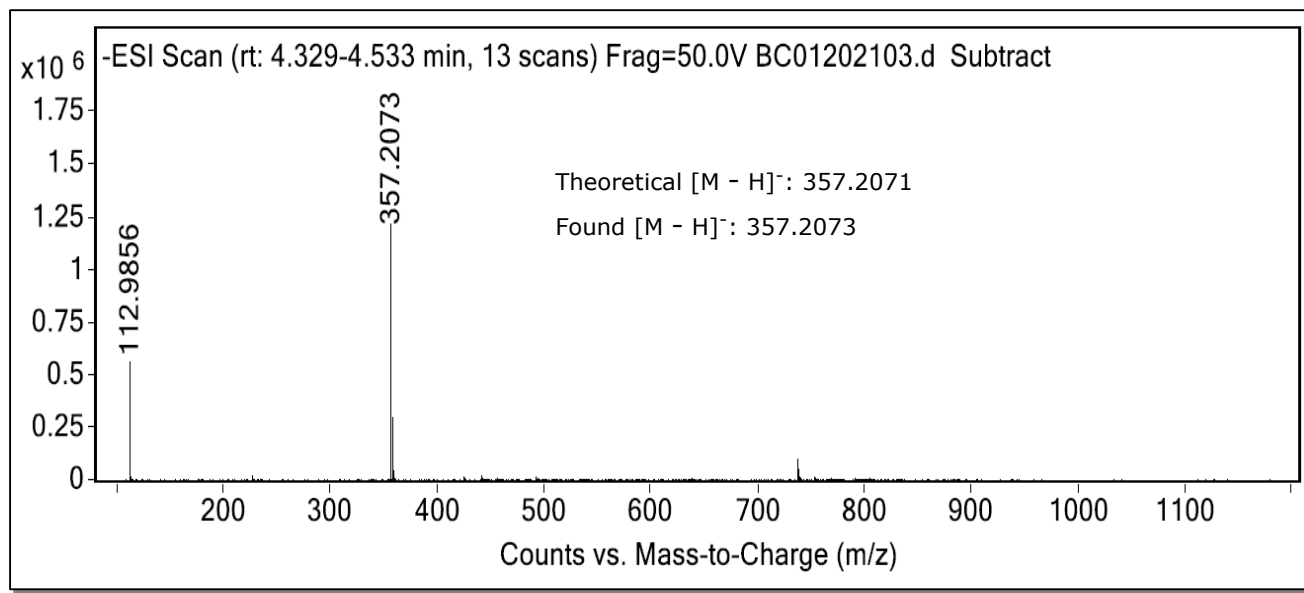
Material Purity by HPLC/UV



Peak #	Ret Time	Area %
1	0.72	0.01
2	1.50	0.00
3	2.23	0.01
4	3.52	0.01
5	3.92	0.01
6	4.40	0.01
7	4.58	0.00
8	4.70	0.00
9	4.96	0.01
10	5.13	0.02
11	5.59	0.00
12	5.68	0.01
13	5.74	0.01
14	5.88	0.01
15	6.01	0.02
16	6.29	0.02
17	6.66	0.00
18	6.76	0.04
19	6.92	0.04
20	7.62	0.01
21	8.20	0.15
22	8.45	0.00
23	8.60	0.11
24	8.88	99.15
25	9.08	0.13
26	9.77	0.11
27	9.88	0.06
28	10.21	0.01
29	15.67	0.02
30	16.58	0.03

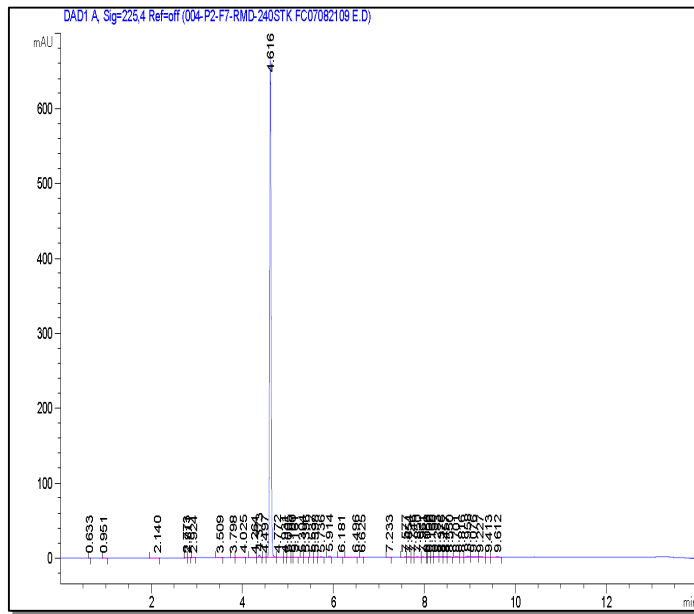
Δ^9 -Tetrahydrocannabinolic acid A (THCA-A) (cont.)

Material Identity by LC/MS



Tetrahydrocannabivarinic acid (THCVA)

Material Purity by HPLC/UV

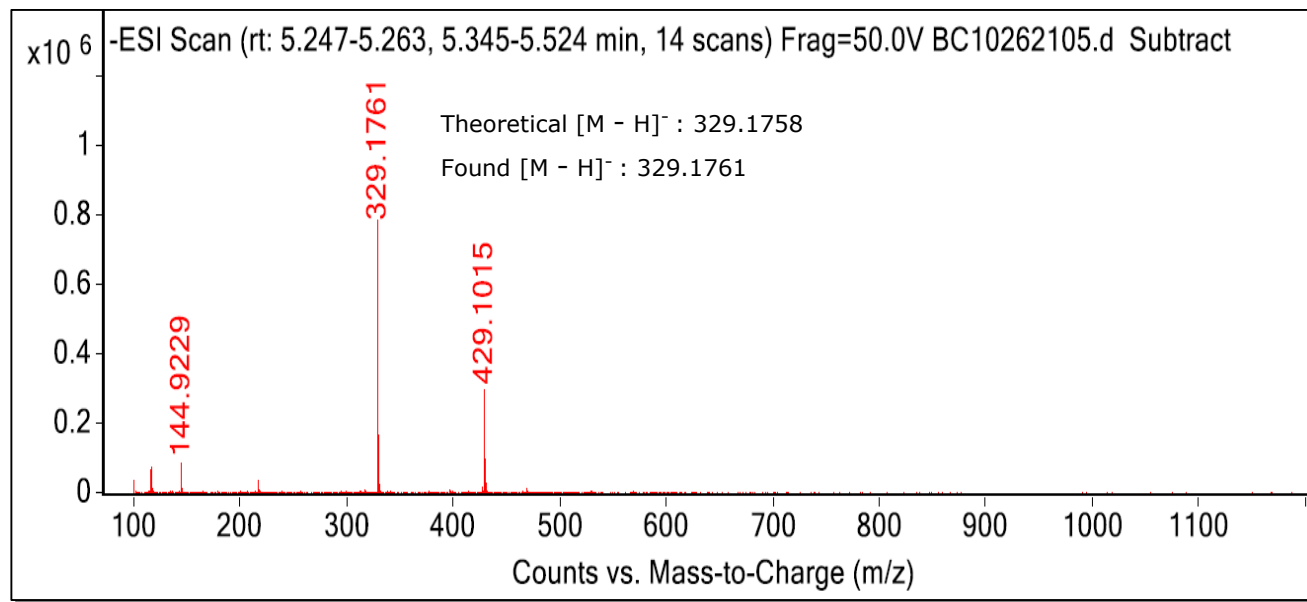


Peak #	Ret Time	Area %
1	0.63	0.01
2	0.95	0.01
3	2.14	0.02
4	2.77	0.06
5	2.81	0.06
6	2.92	0.01
7	3.51	0.02
8	3.80	0.02
9	4.03	0.08
10	4.26	0.02
11	4.37	0.45
12	4.50	0.01
13	4.62	97.87
14	4.77	0.14
15	4.93	0.02
16	5.01	0.03
17	5.10	0.00
18	5.16	0.01
19	5.30	0.01
20	5.40	0.01
21	5.54	0.01
22	5.60	0.03
23	5.74	0.14
24	5.91	0.20
25	6.18	0.01
26	6.50	0.02
27	6.63	0.01
28	7.23	0.01
29	7.58	0.02
30	7.65	0.02
31	7.75	0.01
32	7.85	0.03
33	7.96	0.01
34	8.06	0.00
35	8.10	0.01
36	8.17	0.01
37	8.28	0.03
38	8.38	0.05
39	8.46	0.05
40	8.55	0.04
41	8.70	0.03
42	8.82	0.01
43	8.96	0.18
44	9.08	0.12
45	9.23	0.05
46	9.41	0.01
47	9.61	0.04

Peak 9 is identified as THCVA

Tetrahydrocannabivarinic acid (THCVA) (cont.)

Material Identity by LC/MS



Stability

Short term stability studies have been performed under accelerated conditions for a period of up to four weeks. Short term data is utilized to predict long term stability and 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 accelerated stability findings for this product is listed below.

Storage Condition	Mean Kinetic Temperature (MKT)	Time Period/Result
Sub-Freezer	-70°C	No decrease in purity was noted after four weeks.
Freezer	-15°C	
Refrigerator	4°C	
Room Temperature	21°C	
40°C	40°C	1.26% decrease in purity was noted after four weeks.

Transport/Shipping: Ship cold.

Short Term Storage: Stability data supports short term storage for 12 months at Freezer conditions.

Long Term Stability: Long term stability has been assessed for Sub-Freezer storage (-60 °C to -80 °C) conditions. Stability of a minimum of 19 months has been established through real-time stability studies.

COA Revision History

Revision No.	Date	Reason for Revision
00	June 29, 2022	Initial version.
01	May 09, 2023	Added CTK # to the Regulatory Section.

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