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Analysis of Vitamin E and Vitamin E Acetate in Hemp Vaping Oil Products

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Introduction

Vitamin E and vitamin E acetate are sometimes used in the production of eCigarettes and cannabinoid vaping oils. By December 2019, more than 2400 hospitalizations occurred in the U.S. for Electronic-cigarette, or Vaping, product use–Associated Lung Injury (EVALI) with an interstate study indicating 94% of the EVALI cases were positive for vitamin E acetate compared to 0/99 "healthy comparator" controls [1]. To support these studies, manufacturers and regulatory agencies need a quick, simple and accurate method for additionally testing relevant vaping products for vitamin E and vitamin E acetate. Herein, we adapted a published cannabinoid method for hemp analysis [2] to simultaneously identify and quantify vitamin E acetate and vitamin E.

Experimental

Five samples of commercially-available vaping oil were diluted 1000-fold and analyzed using an Agilent LC/MSD iQ system with an ESI source and OpenLab CDS 2.4 Software. Chromatographic conditions were optimized by adapting a published methodology [3] of a 16 cannabinoid mixture to improve analysis speed while maintaining separation (Figure 1). For identification and quantification of the vitamin E compounds, *m/z* 431.1 and 473. 2, were monitored in addition to the cannabinoid compounds.

16 cannabinoid UV method

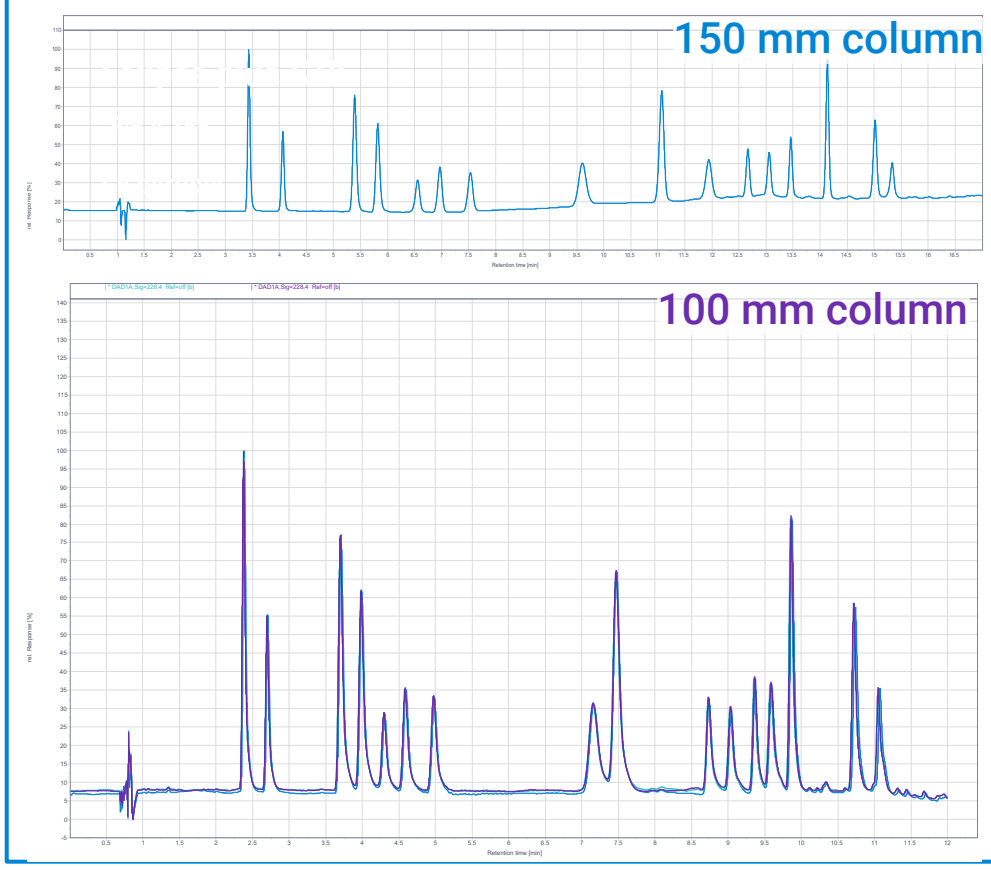


Figure 1: The upper UV chromatogram utilized the published [3] 150 mm column for an elution period of 15.5 minutes; the lower UV chromatogram utilized a 100 mm column for an 11 minute elution period.

Experimental

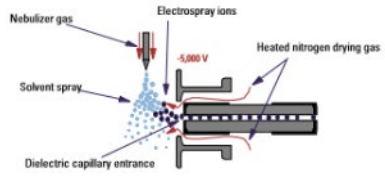
Analytical Method

Parameter	Value			
Column	Agilent Poroshell 120 EC-C18, 3.0 × 100 mm, 1.9 µm @ 30.0 °C			
Flow rate	0.500 mL/min			
Solvent A	0.1% Formic Acid in H_2O			
Solvent B	100% ACN			
Solvent C	100% MeOH			
Solvent D	10 mM NH_4HCO_2 in H_2O			
Gradient	%A	%B	%C	%D
Time: 0.0	29	70	0	1
3.20	29	70	0	1
7.20	12	0	87	1
10.00	0	0	95	5
Post Time	5 minutes			
UV Signal	228 nm			

MS Parameter	Value
Mode	Positive Ion
Gas Temp.	325 °C
Gas Flow	13 L/min
Nebulizer Pressure	55 psi
Capillary Voltage	3500 V
Acquisition	SIM/Scan

MS Signals	Value	
Scan	200-700 m/z, 89 ms, Frag=110V 300-700 m/z, 71 ms, Frag=110V	
SIM (m/z)	Vit. E. Acetate: 495.4	CBG: 317.2
Time = 15 ms	Vitamin E: 473.4	CBD, THC (ISO): 316.5
Frag = 135V	CBGA: 361.2	THC CBD CBL CBC: 315.2
	CBCA THCA CBDA: 359.2	CBN: 311.2
	CBNA: 355.1	CBDV, THCV: 287.2
	CBDVA THCVA: 331.2	

Analytical Configuration



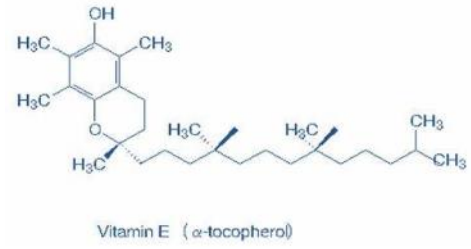


Figure 2: Analytical Configuration: Agilent 1260 HPLC with mass detection using the LC/MSD iQ

Calibration Curve - Vitamin E Acetate.

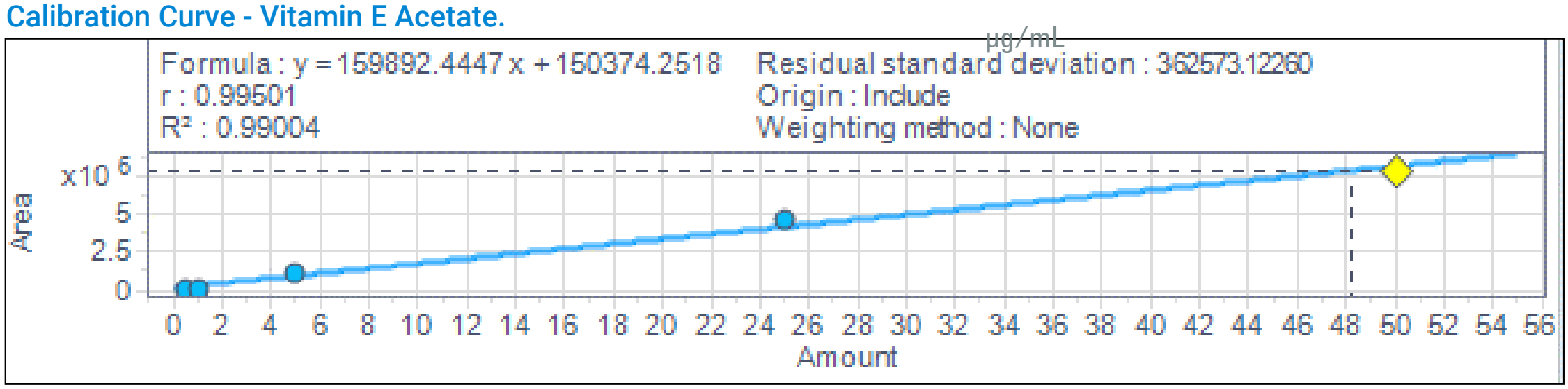
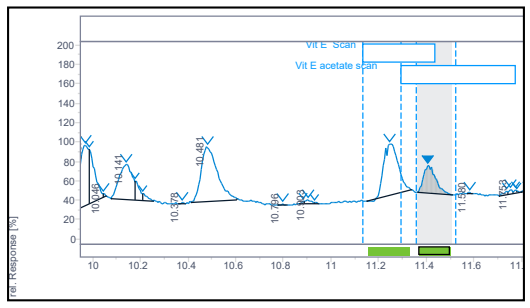


Figure 3: Calibration Curve – Vitamin E Acetate

Spectral library confirmation: Spectral matching and purity results

Peaks | Summary

#	Name	Signal description	RT (min)	MS Conf. Matcl	MS Purity
74	Vit E acetate scan	MS1 +TIC SCAN ESI Frag=110V Gain...	11.409	1000	100.00
73	Vit E Scan	MS1 +TIC SCAN ESI Frag=110V Gain...	11.248	1000	100.00



Spectral matching is compared to a known reference spectra. Scan data was used for the library search. 1000 == 100% match compared to the library. Unknown spectra can be exported and searched against the library.

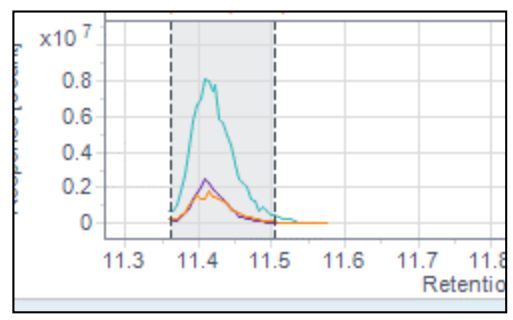


Figure 4: Spectral Library Matching of Vitamin E and Vitamin E Acetate

LOD and LOQ based on SIM data.

Analyte	LOD	LOQ
Vitamin E	0.010 µg/mL	0.025 µg/mL
Vit. E acetate	0.010 µg/mL	0.025 µg/mL

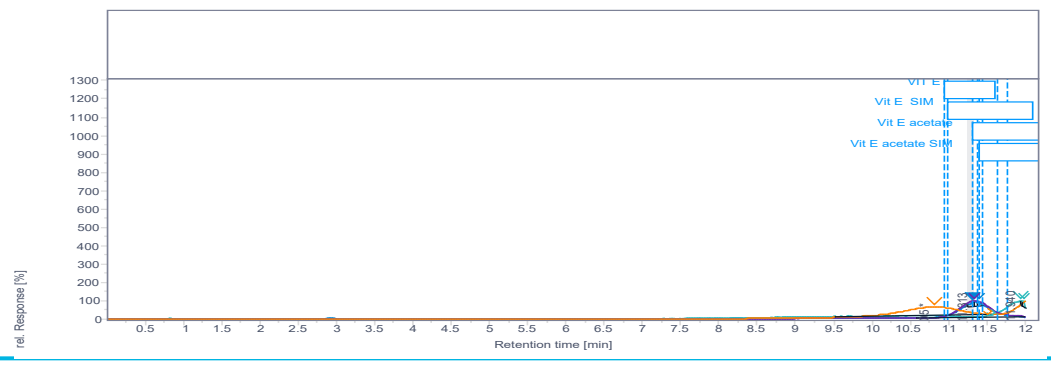


Figure 5: Limits of Detection and Limits of Quantitation for Vitamin E and Vitamin E Acetate by LC/MSD iQ

Results and Discussion

SIM ions of spiked chromatogram

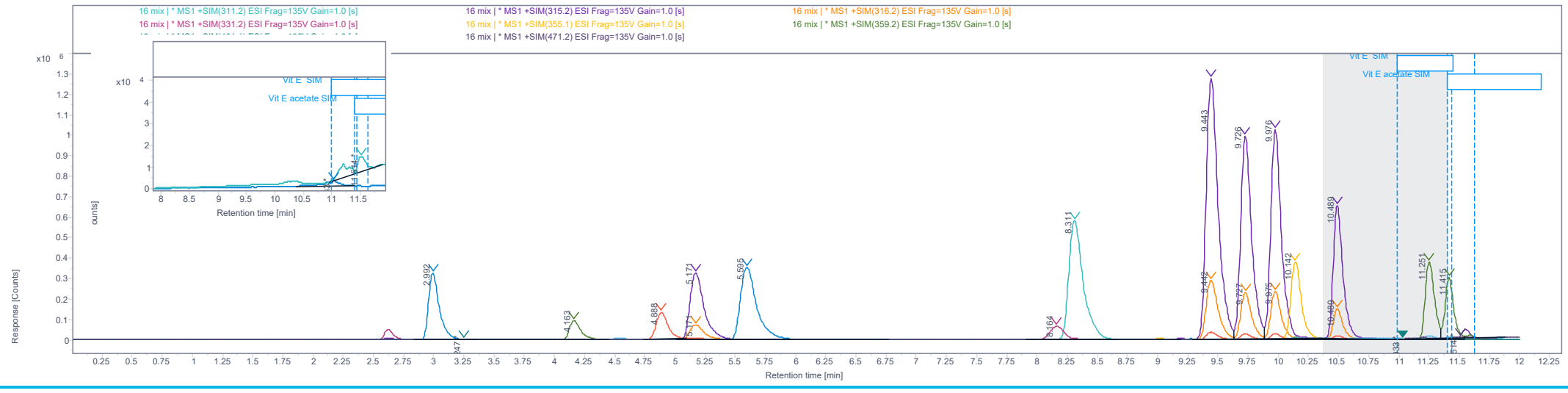
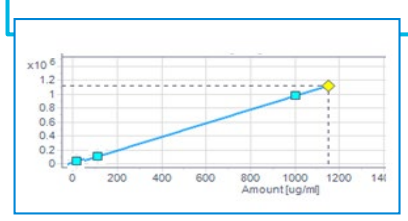


Figure 6: Overlay of all SIM ions in spike of vitamin E and vitamin E acetate into 16 cannabinoid mix in hemp seed oil

Analytical Results (n.d. = not detected)

Sample #	Vitamin E	Vitamin E acetate
1	n.d	0.06 ug/ml
2	n.d.	0.04 ug/ml
3	n.d.	n.d.
4	n.d	n.d
5	0.09 ug/ml	0.02 ug/ml
6	n.d.	0.09 ug/ml
7	n.d.	0.05 ug/ml
8	0.04 ug/ml	0.07 ug/ml
9	n.d	0.05 ug/ml
10	n.d	0.02 ug/ml
1 ug/ml spike	0.95 ug/ml	1.03 ug/ml
Commercial oil		1125 ug/ml



Larger calibration curve was created for a commercial sample of vitamin E acetate oil.

Conclusions

In this study, vitamin E acetate and vitamin E was appended to a previously published method for the quantitation of cannabinoids in hemp seed oil. Low PPM LOD and LOQ values were established in this matrix.

The results determined that, without changes to the published method, vitamin E and vitamin E acetate can be appended for identification and quantification in vaping oil samples. Further, the full scan data of the unknown samples were successfully used with a known library to identify vitamin E and vitamin E acetate in the samples.

References

[1] Blount BC, et al. (2020) Vitamin E Acetate in Bronchoalveolar-Lavage Fluid Associated with EVALI. N Engl J Med. 382(8):697-705.

[2] D'Antonio S, et al. (2020) Quantitation of Phytocannabinoid Oils Using the Agilent Infinity II 1260 Prime/InfinityLab LC/MSD iQ LC/MS System. Agilent Application Note 5994-1706EN, Agilent Technologies, Inc.

[3] Kowalski, D. Laine, Improved Routine Cannabinoids Analysis with Liquid Chromatography-Diode Array Ultraviolet Detection for the Current Cannabis Market, Oral presentation, AOAC International Conference, August 26- August 29, Toronto, Ontario, Canada, 2018.

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