

Worklist: 4899

REVIEWED
By Brittany Wylie at 2:48 pm, Apr 12, 2021

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>
C2021-0605	1	UCK	AM 3 Urine Carboxy-THC



GA

AM 3: Carboxy-THC Urine Extraction

Extraction Date: 4/2/21

Analyst: Anne Nord

Negative Urine Lot: 2121

GC/MS ID: 65198

Hexane Lot: fisher 42712

Ethyl Acetate Lot: fisher 020419

Silylating Agent Lot: Cerilliant FN08181601

Pre-Analytic:

- 1. *Positive Control Working Solution Preparation Instructions:*
Add 180uL of 100ug/mL 11-nor-9-carboxy- Δ 9-THC Stock Solution to 9.82mL Methanol. Other volumes may be prepared. Solution is stable for 1-year or the expiration of the stock reference material (whichever is sooner). Store under refrigeration
- 2. Verify Tune and Tune evaluation completed within the previous 7 days. Tune and Tune evaluation reports initialed and filed.
- 3. Create GCMS sequence to include controls, case blanks and case samples.

Analytic:

- 1. Remove working solutions, external control, negative urine and case samples from cold storage.
- 2. To each labeled round bottomed tube add 3mL sample, using negative urine sample for both negative and positive control. Positive control: added working solution to equal approximately 60 ng/ml Carboxy-THC
- 3. Add 500uL 1N KOH to all tubes. Check pH. (*If pH <12, add additional 500ul 1N KOH*). (*Note: put a mark on the tube or separate the tubes that have a pH <12 as you will need to know this in step 5*).
- 4. Place tubes in 40C water bath for ~15 min. Remove and allow to cool.
- 5. If original pH was >12, add 1.5mL pH 1.8 Saturated Phosphate Buffer and 3mL Hexane/Ethyl Acetate (87:13)
If original pH was <12, add 3mL pH 1.8 Saturated Phosphate Buffer and 4mL Hexane/Ethyl Acetate (87:13)
- 6. Rock at ambient temp for ~ 10 minutes.
- 7. Centrifuge for ~ 10 min at ~3500rpm.
- 8. Transfer solvent to tapered bottom tube and evaporate to dryness under nitrogen @ 37C.
- 9. Add 50uL Ethyl Acetate and 50uL silylating reagent, cap and vortex. Heat @ 95C for 15min, then allow to cool.
- 10. Transfer sample to labeled ALS vial with insert.
- 11. Place ALS Vials in appropriate location on GCMS rack and run using appropriate GCMS method.

Post-Analytic

- 1. Complete Data analysis on all samples and corresponding sample blanks
- 2. Did positive and negative control samples provide intended response? yes
- 3. **Criteria for ID:** RT +/1 0.1 min., Ion Ratio of 347:473 & 371:488 within +/- 20%.
Sample response greater than Min Corrected Area, Diluted samples
- 4. Central File Packet to include: LIMS Worklist, Method Checklist, Working solution prep sheet, and Control sample GCMS data printouts

Comments: See deviation spiked control made at 600ng/ml.

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Toxicology AM method 3 spiked 60ng/ml (actual concentration of this preparation 600ng/ml)

Stock solution 18 ul (100 ug/ml) C-THC in 2.482 mls neg urine lot 2121

Ppd 4/2/21 (used on 4/2/21) by amn

Drug	lot	expiration
C-THC	FE01061702	3/1/2022

~~A~~

Toxicology AM method 3 commercial control

Pinpoint THC quantitation plate (IDP-108-2) lot 201206 exp 06/06/21

added 3mls negative urine (2121) to CAL 5 (concentration 25ng/ml THC-COOH)



Run Date: 4/2/2021

Worklist #: 4899

Laboratory Case #: Negative control

Case Calculations:

EIC Ion 371 Corr. Area: 0

EIC Ion 473 Corr. Area: 0

EIC Ion 488 Corr. Area: 0

473:371 Ratio: **#DIV/0!**

488:371 Ratio: **#DIV/0!**

Quality Control Calculations:

	1st Control	2nd Control
EIC Ion 371 Corr. Area:	26708513	28974520
EIC Ion 473 Corr. Area:	12657525	13732313
EIC Ion 488 Corr. Area:	7949794	8703921
Ratio of 473:371:	0.474	0.474
		Average: 0.474
Ratio of 488:371:	0.298	0.300
		Average: 0.299

Acceptable Retention Time Range: 4.396 to 4.596

Approximate Minimum Corrected Area of 371 Ion: 534170

Acceptable 473:371 Ratio Range: 0.379 to 0.569

Acceptable 488:371 Ratio Range: 0.239 to 0.359



Data Path : D:\DATA\2021\am 2\040221 am2 am 3\
 Data File : 00501010.D
 Acq On : 02 Apr 2021 14:10
 Operator : Instrument 65198
 Sample : negative control am 3
 Misc : am 3
 ALS Vial : 5 Sample Multiplier: 1

Integration Parameters: autoint1.e
 Integrator: ChemStation

Method : D:\MassHunter\GCMS\1\methods\TOXI-A 10115.M
 Title :

Signal : EIC Ion 371.00 (370.70 to 371.70): 00501010.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
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No peaks were detected using the method integration parameters!

Signal : EIC Ion 473.00 (472.70 to 473.70): 00501010.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
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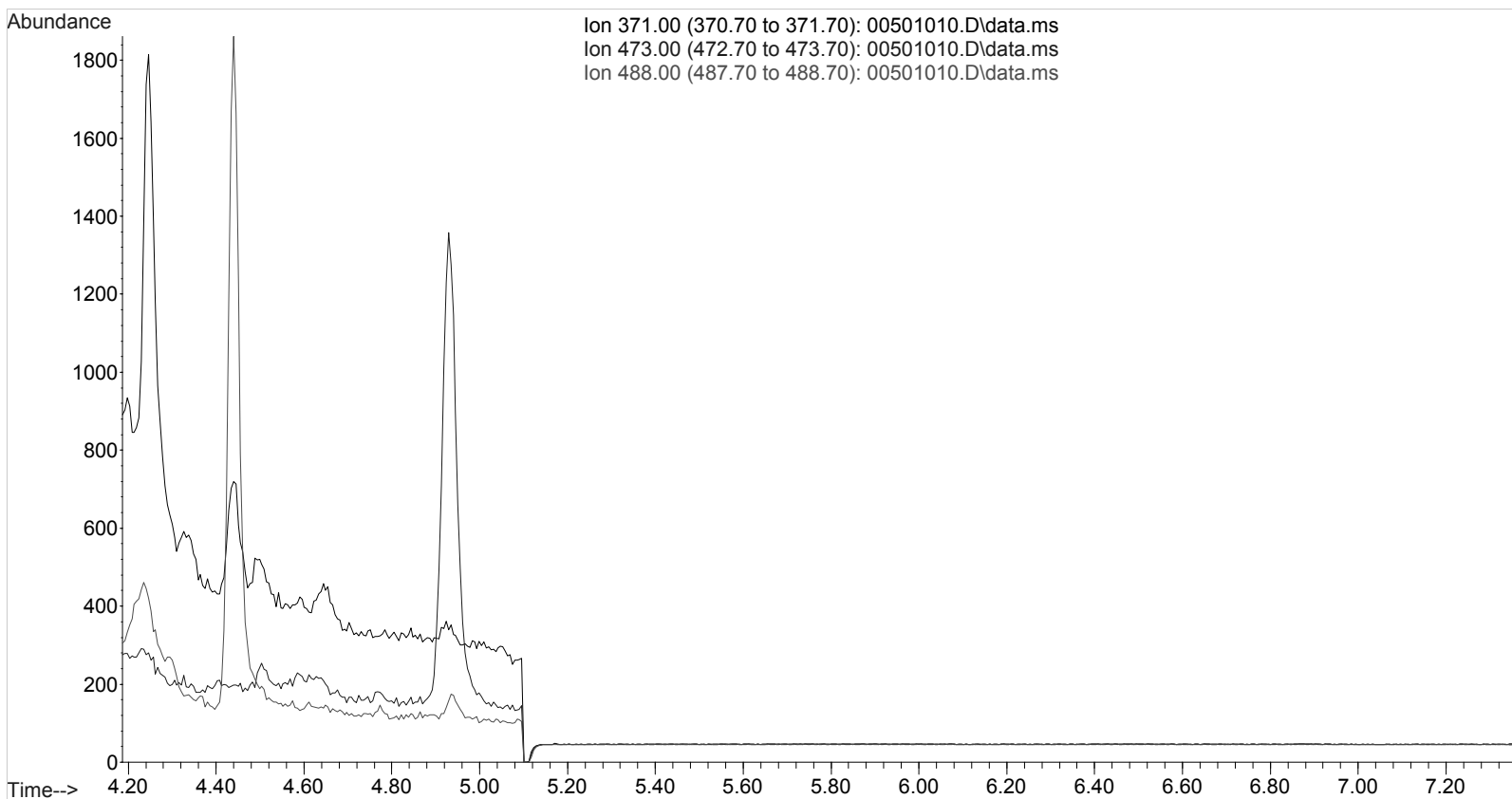
No peaks were detected using the method integration parameters!

Signal : EIC Ion 488.00 (487.70 to 488.70): 00501010.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
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No peaks were detected using the method integration parameters!

TOXI-A 10115.M Fri Apr 02 16:13:57 2021





Run Date: 4/2/2021

Worklist #: 4899

Laboratory Case #: Pinpoint Control

Case Calculations:

EIC Ion 371 Corr. Area: 812124

EIC Ion 473 Corr. Area: 377611

EIC Ion 488 Corr. Area: 218109

473:371 Ratio: **0.465**

488:371 Ratio: **0.269**

Quality Control Calculations:

	1st Control	2nd Control
EIC Ion 371 Corr. Area:	26708513	28974520
EIC Ion 473 Corr. Area:	12657525	13732313
EIC Ion 488 Corr. Area:	7949794	8703921
Ratio of 473:371:	0.474	0.474
		Average: 0.474
Ratio of 488:371:	0.298	0.300
		Average: 0.299

Acceptable Retention Time Range: 4.396 to 4.596

Approximate Minimum Corrected Area of 371 Ion: 534170

Acceptable 473:371 Ratio Range: 0.379 to 0.569

Acceptable 488:371 Ratio Range: 0.239 to 0.359



Data Path : D:\DATA\2021\am 2\040221 am2 am 3\
 Data File : 00801013.D
 Acq On : 02 Apr 2021 14:38
 Operator : Instrument 65198
 Sample : C-THC control pinpoint
 Misc : am 3
 ALS Vial : 8 Sample Multiplier: 1

Integration Parameters: autoint1.e
 Integrator: ChemStation

Method : D:\MassHunter\GCMS\1\methods\TOXI-A 10115.M
 Title :

Signal : EIC Ion 371.00 (370.70 to 371.70): 00801013.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.495	39	58	82	BB	46877	812124	100.00%	100.000%

Sum of corrected areas: 812124
 Signal : EIC Ion 473.00 (472.70 to 473.70): 00801013.D\data.ms

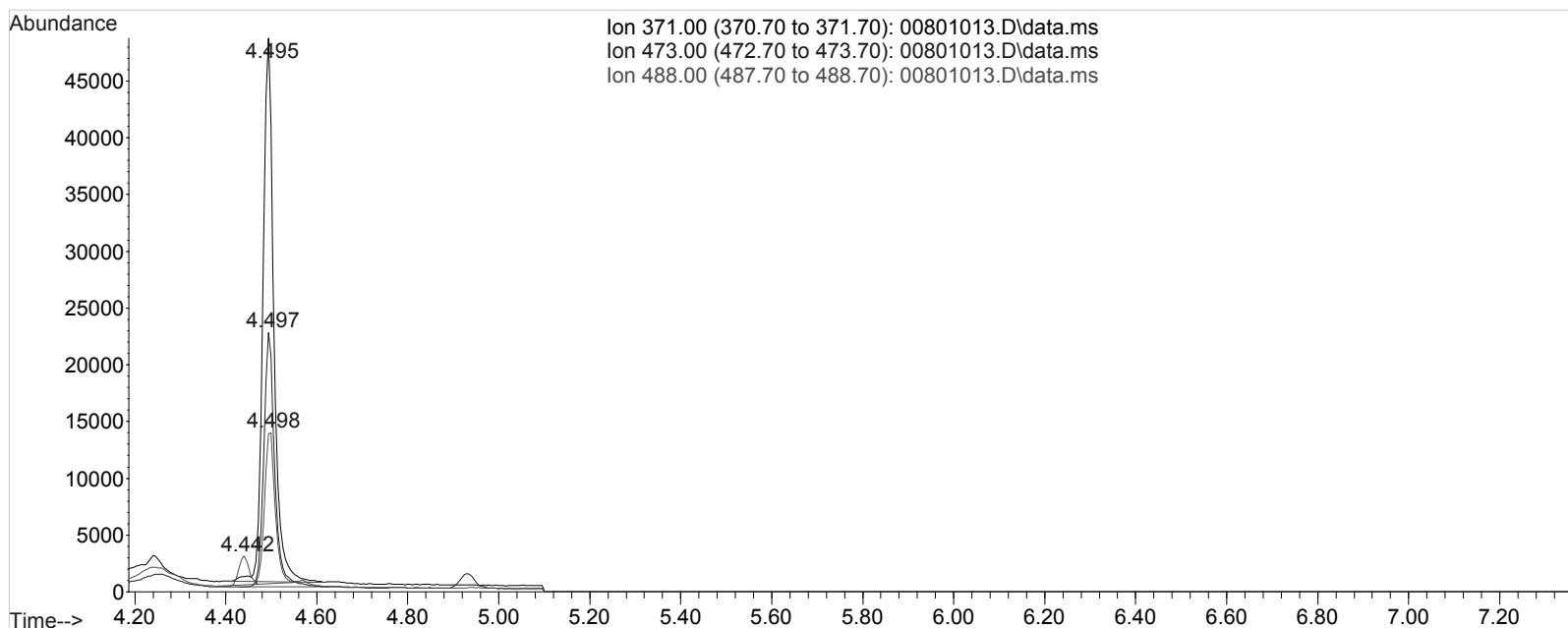
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.497	46	59	83	BB	22036	377611	100.00%	100.000%

Sum of corrected areas: 377611
 Signal : EIC Ion 488.00 (487.70 to 488.70): 00801013.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.442	37	48	53	BV	2479	36679	16.82%	14.396%
2	4.498	53	59	69	VV	13380	218109	100.00%	85.604%

Sum of corrected areas: 254787

TOXI-A 10115.M Fri Apr 02 16:16:47 2021





Data Path : D:\DATA\2021\am 2\040221 am2 am 3\
Data File : 00601009.D
Acq On : 02 Apr 2021 14:00
Operator : Instrument 65198
Sample : 60ng C-THC
Misc : am3
ALS Vial : 6 Sample Multiplier: 1

Integration Parameters: autoint1.e
Integrator: ChemStation

Method : D:\MassHunter\GCMS\1\methods\TOXI-A 10115.M
Title :

Signal : EIC Ion 371.00 (370.70 to 371.70): 00601009.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.216	3	6	23	BV	3903	78342	0.29%	0.292%
2	4.496	45	59	130	VB	1497194	26708513	100.00%	99.708%

Sum of corrected areas: 26786855

Signal : EIC Ion 473.00 (472.70 to 473.70): 00601009.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.498	49	59	106	BV	712987	12657525	100.00%	100.000%

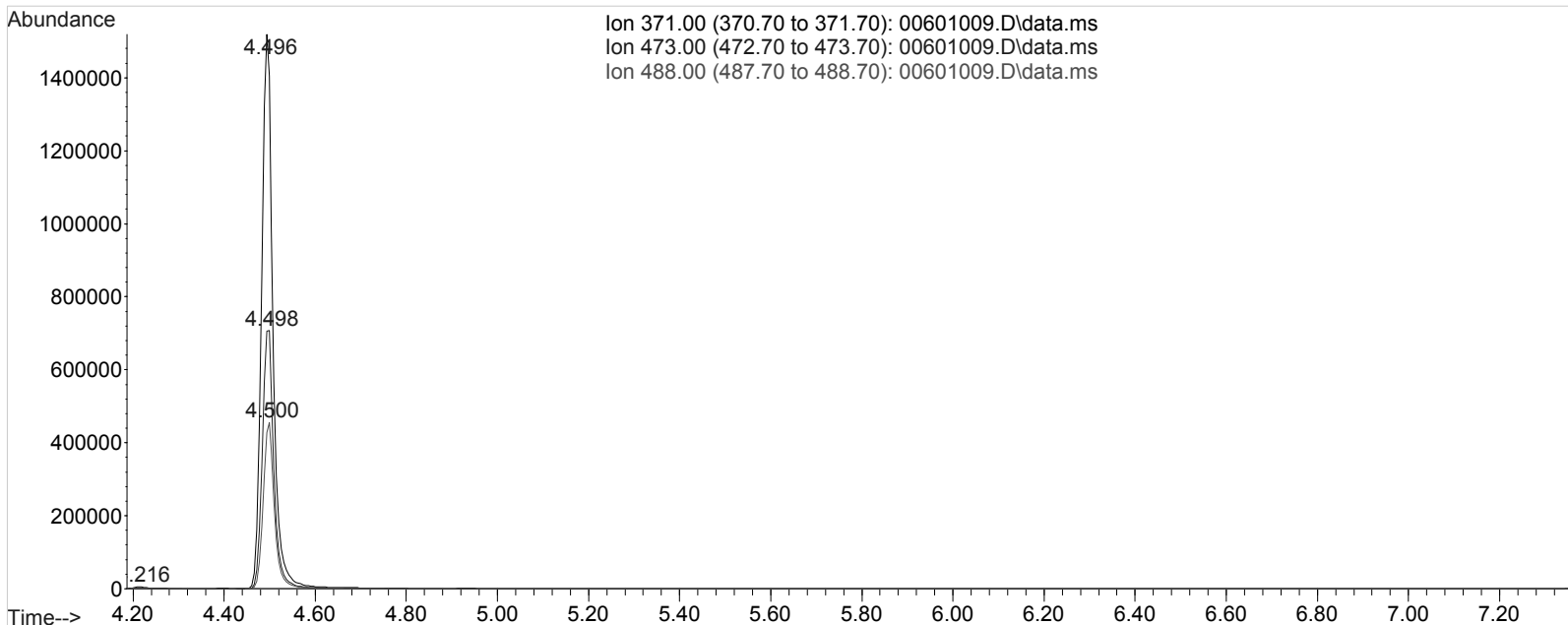
Sum of corrected areas: 12657525

Signal : EIC Ion 488.00 (487.70 to 488.70): 00601009.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.500	51	59	115	VB	450716	7949794	100.00%	100.000%

Sum of corrected areas: 7949794

TOXI-A 10115.M Fri Apr 02 16:11:57 2021





Data Path : D:\DATA\2021\am 2\040221 am2 am 3\
Data File : 00601016.D
Acq On : 02 Apr 2021 15:06
Operator : Instrument 65198
Sample : 60 ng CTHC control
Misc : am 3 end of run control
ALS Vial : 6 Sample Multiplier: 1

Integration Parameters: autoint1.e
Integrator: ChemStation

Method : D:\MassHunter\GCMS\1\methods\TOXI-A 10115.M
Title :

Signal : EIC Ion 371.00 (370.70 to 371.70): 00601016.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.215	3	6	24	BV	3965	76497	0.26%	0.263%
2	4.496	44	58	134	VB	1712280	28974520	100.00%	99.737%

Sum of corrected areas: 29051017
Signal : EIC Ion 473.00 (472.70 to 473.70): 00601016.D\data.ms

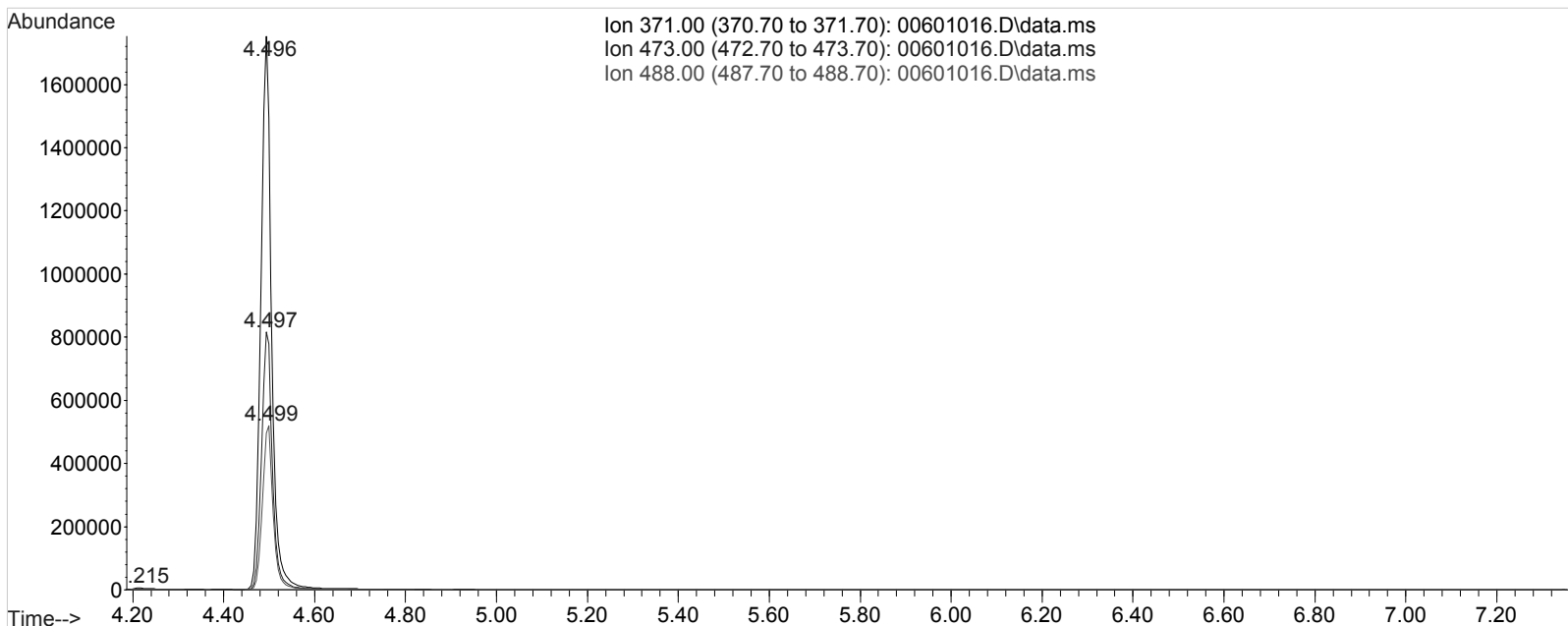
peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.497	49	59	106	BV	809777	13732313	100.00%	100.000%

Sum of corrected areas: 13732313
Signal : EIC Ion 488.00 (487.70 to 488.70): 00601016.D\data.ms

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	4.499	51	59	119	VB	517154	8703921	100.00%	100.000%

Sum of corrected areas: 8703921

TOXI-A 10115.M Fri Apr 02 16:12:55 2021



**Idaho State Police
Forensic Services
Toxicology Discipline**

Request for Departure from an Analytical Method

Date of Request

04-12-2021

Forensic Scientist

Anne Nord

Analytical Method

Toxicology AM #3: Qualitative 11-nor-9-THC-D9-COOH (Carboxy-THC) in Urine

Request

3.4.1.1 A minimum of one spiked 60 ng/mL and one commercial Carboxy-THC containing control must be analyzed in each batch of samples.

3.4.1.2 60 ng/mL Carboxy-THC Spiked Control

4.2.3.2.1.1.2 Assessment of relative strength of case sample to 60 ng/mL control. The response of case samples will be compared to a 60 ng/mL control sample. The analyst will pick either of the responses from the positive control and divide the response of the 371 ion by 5; this will be defined as the approximate minimum response. The approximate minimum response will be documented in the analyst's notes. -The analyst will compare this response to the response for each case sample.

I would like to request the following deviation:

When I prepared the 60 ng spiked control, I prepared it at 600 ng/ml rather than 60. I would like to use this control and divide the total response by 50 rather than 5 to approximate the minimum response. The only case sample that was run in this batch (worklist 4988) had a response greater than the 600 ng/ml prepared control.

Discipline Leader Review

Departure approved

Comments:

Departure Not Approved

Comments:

Celena Shrum
Toxicology Discipline Lead
Date: 04/12/2021