








REVIEWED
By Tamara Salazar at 10:42 am, Aug 29, 2022



Worklist: 6074

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
C2022-1568	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1765	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1767	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
C2022-1805	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1836	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1839	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1860	1	BCK	AM 27 Blood THC Quant by LC-QQQ	



Worklist: 6075

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>
C2022-1817	1	BCK	AM 27 Blood THC Quant by LC-QQQ





AM# 27: Quantitation of THC and Metabolites in Blood and Urine by LC-MS/MS

Extraction Date 8/24/22
Plate lot#: 220309

Analyst: Anne Nord
Plate re-test: 9/9/2022

Mobile phase A: 0.1% Formic Acid in LCMS Water
MTBE LCMS Methanol

Mobile phase B: 0.1% Formic acid in Acetonitrile
Hexane

Blank Blood Lot: 22B52016-1 **Urine Blank:** 7722

Column: UCT Selectra DA 100 x 2.1mm 3um

LCMS-QQQ ID: 69679

Pre-Analytic:

- 1. Check levels of mobile phases and needle wash refill as needed. Ensure waste is not full.
- 2. Ensure correct column is installed and begin mobile phase flow allow to equilibrate ~ 30 minutes.

Analytic:

- 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
- 2. Urine hydrolysis: add 1.5 ml urine to blank plate, add 250 ul 1N KOH mix and incubate at 40 degrees for 15 minutes.
Pipette 1000µL blood (calibrated pipette) Pipette ID: I41142J in wells of analytical (standards) plate.
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 4. Pipette 500µL 0.1% formic acid in water blood sample, 500 ul saturated phosphate buffer in urine in wells of analytical plate.
- 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 6. Transfer 800µL of blood+acid or urine acid mixture to corresponding wells of SLE+ plate.
- 7. Apply positive pressure for approx. 10-15 seconds (or until no liquid remains on top of sorbent).
(Load at 85-100 PSI- Selector to the right) Manifold ID: 66792
- 8. Wait 5 minutes.
- 9. Add 2.25mL MTBE. (Add in 3 increments of 750uL)
- 10. Wait 5 minutes.
- 11. Apply positive pressure for approx. 15 seconds. (10-15 PSI- Selector to the left).
- 12. Add 2.25mL Hexane. (Add in 3 increments of 750uL)
- 13. Wait 5 minutes.
- 14. Apply positive pressure for approx. 15 seconds. (10-15 PSI- Selector to the left).
- 15. Remove plate containing eluate. Place on SPE Dry and evaporate to dryness at approx. 35°C.
SPE Dry ID: 66819
- 16. Reconstitute in 100µL 100% MeOH and heat seal plate with foil. Place in autosampler and run worklist.


Post-Analytic

- 1. Create batch and process data.
- 2. Make any necessary integration changes, Curve weighting of Linear 1/x with r^2 values ≥ 0.98 for each analyte
- 3. RT +/- 3% or 0.100 min, whichever is greater, +/- 20% Accuracy for greater than (+/- 30% for 10ng/ml or less). Ion ratios must be within +/- 20% of the averaged calibrators
- 4. Case sample response for THC 1ng/ml, OH-THC 3ng/mL (quantitative blood), Carboxy-THC: 5 ng/mL (qualitative only) will be reported. Samples with a THC or OH-THC response over 50 ng/mL will be reported out as greater than 50 ng/mL.
- 5. Did all QCs pass for each analyte? (if not is it describe in comments section)
- 6. Enter QCs into control charting.
- 7. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: THC below 3 ng will be reported as < 3ng/ml. THC-OH not evaluated due ratio lowering as concentration increased.

C2022-1765-2 did not inject, I reinjected, I was watching the sample run no response for IS when it should come out, so I stopped the run. I added 50ul methanol and injected the sample again, this sample was evaluated. (I could see liquid in the well I am not sure why the needle could not reach it, most likely it evaporated just enough)

**Idaho State Police
Forensic Services**



Request for Departure from an Analytical Method or Quality Standard

Deviation Number (assigned by QM): TOX-22-02

Date of Request:
03/02/2022

Requestor/Discipline:
Celena Shrum/Toxicology

Analytical Method/Quality Standard, Revision #:
Toxicology AM #25, AM #26, and AM #27, Revision 13

Temporary or Permanent Deviation:
Permanent

Scope of Deviation (record specific information, e.g. affected programs, evidence types, expected end date; etc):

Deviation will remain in place until the change is made in the next method revision.

Deviation Request (Describe detailed instructions of the changes being made; include reference to specific section number(s) in the method manual):

Toxicology AM #25 3.3.1.1 Internal standards are prepared by the ToxBBox plate manufacturer and contained on the 96 well plate. If the run contains urine samples, a positive external urine control must also be run.

Toxicology AM #26 3.3.2 A negative control will be run with each extraction. If the run contains urine samples, a negative urine control and external positive urine control must also be included.

Toxicology AM #27 3.3.2 A negative control will be run with each extraction. If the run contains urine samples, a negative urine control and positive external urine control will also be included in the run.

The deviation is to include the option of using an internal urine control in lieu of an external urine control.



Technical Justification for Analytical Method Deviations:

Internal controls serve the same purpose as external controls but also helps to avoid the possible issues that can occur with using external controls (incorrect spiking, incorrect preparation, evaporation of compounds, etc.). If these errors occur, runs need to be repeated and this wastes time, sample, and supplies.

Technical Review

Departure approved
Comments:

Departure Not Approved
Comments:



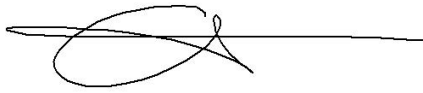
Approver: Rachel Cutler
Title: Lab Manager

Date: 3/2/22

Quality Review

Quality Approver: Jason Crowe
Title: Quality Manager
Date: 3/2/2022





	1	2	3	4	5	6
a	cal 1	Internal urine	1860-1			
b	cal 2	negative blood	1767-2			
c	cal 3	1568-1	negative urine			
d	cal 4	1765-2				
e	Cal 5	1805-1				
f	cal 6	1817-1				
g	cal 7	1836-1				
h	Internal control (blood)	1839-1				

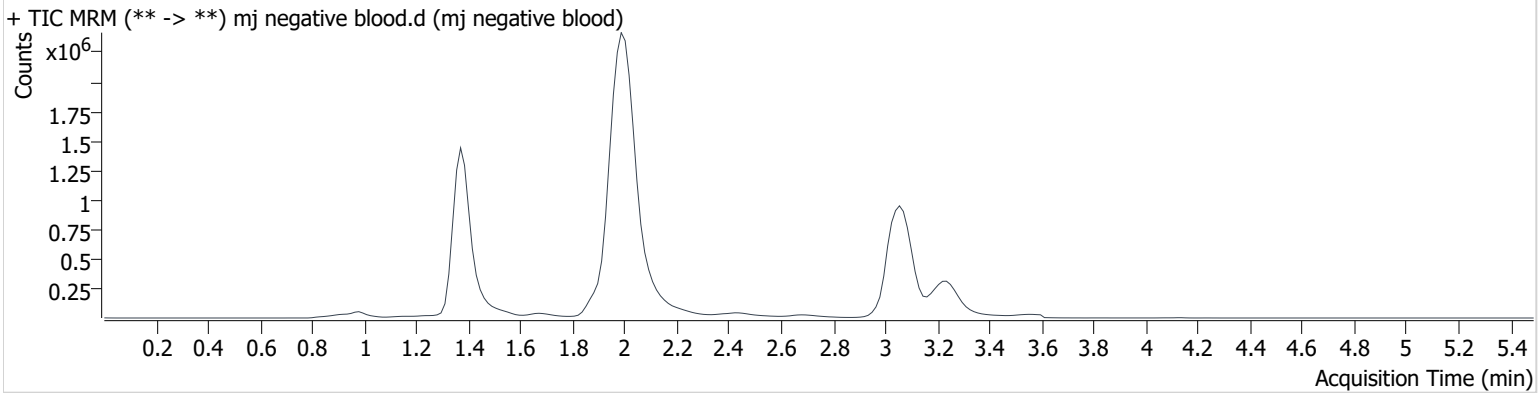
c2022-____-__

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:38:41 PM

Instrument	69679	Data File	mj negative blood.d
Type	Sample	Sample	mj negative blood
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-B2	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 11:15:04 AM		
Sample Info.			

Sample Chromatogram

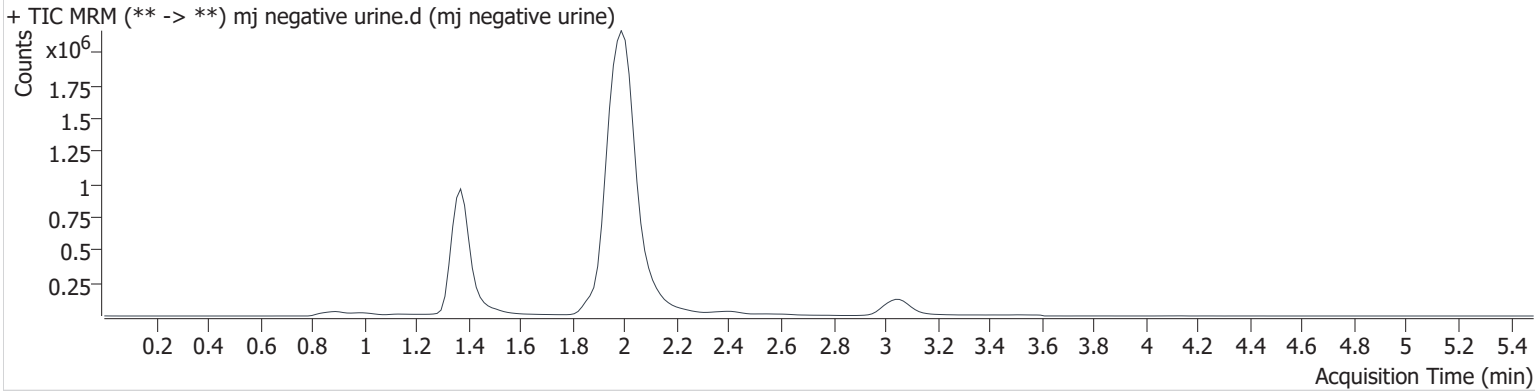


AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj negative urine.d
Type	Sample	Sample	mj negative urine
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-C3	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 1:15:30 PM		
Sample Info.			

Sample Chromatogram

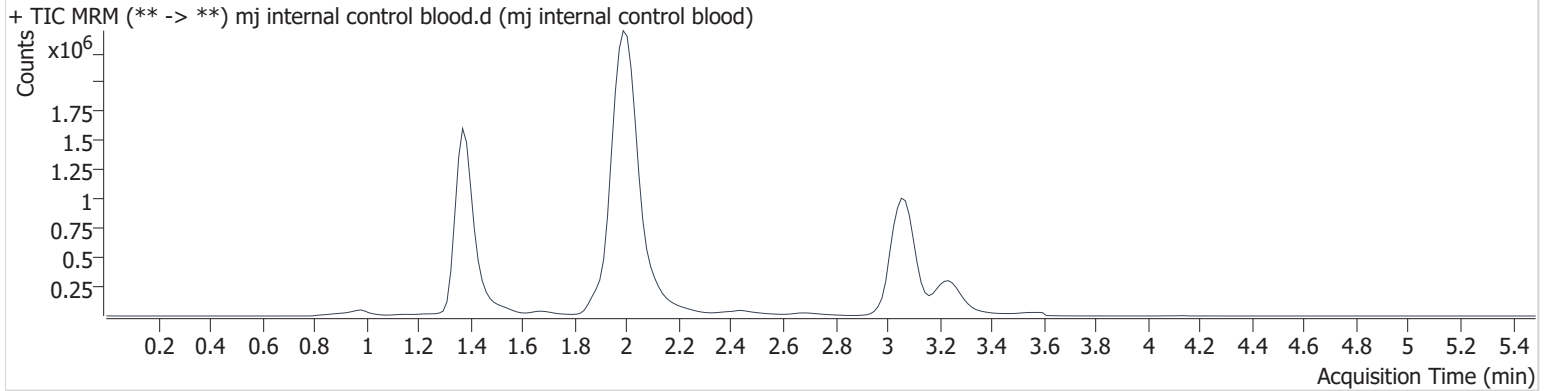


AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj internal control blood.d
Type	QC	Sample	mj internal control blood
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-H1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 11:01:35 AM		
Sample Info.			

Sample Chromatogram



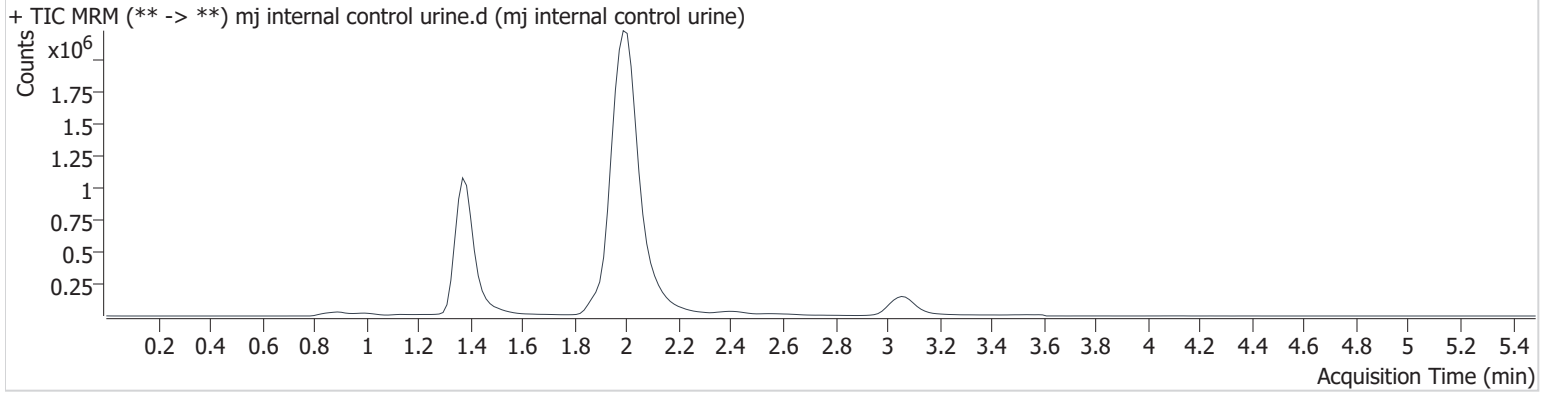
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.403	98645	∞	254.8	∞	1336827	14.896 ng/ml
THC	3.077	590251	94080.6	23.7	3957.0	5134362	4.780 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj internal control urine.d
Type	Sample	Sample	mj internal control urine
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-A2	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 11:08:20 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.403	52919	∞	283.1	1113.3	796039	13.525 ng/ml
THC	3.077	102805	∞	22.7	138.2	961296	4.473 ng/ml

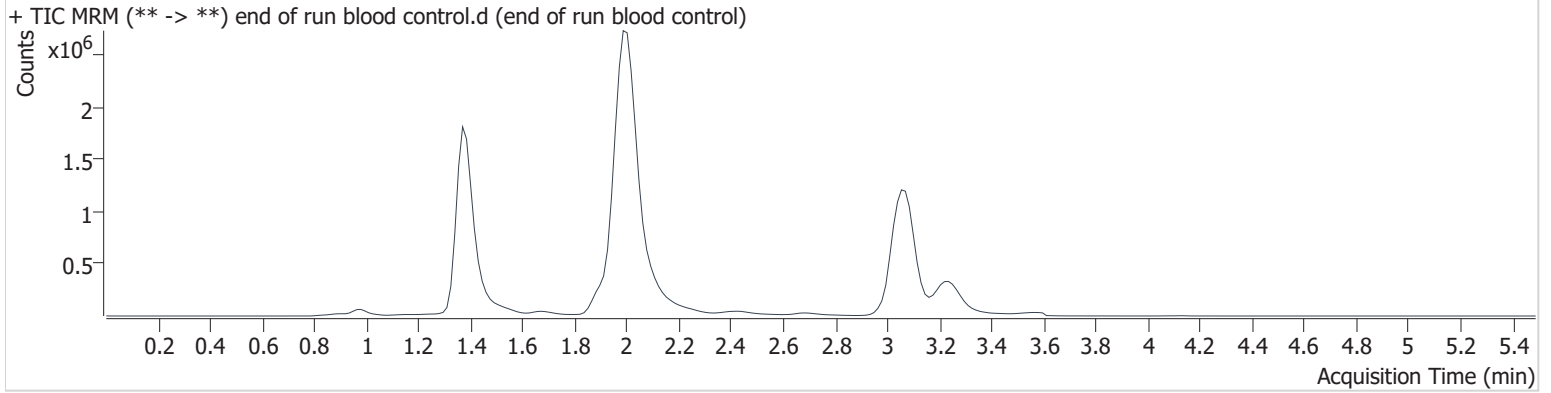
AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	end of run blood control.d
Type	Sample	Sample	end of run blood control
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-H1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 1:47:15 PM		

Sample Info.

Sample Chromatogram



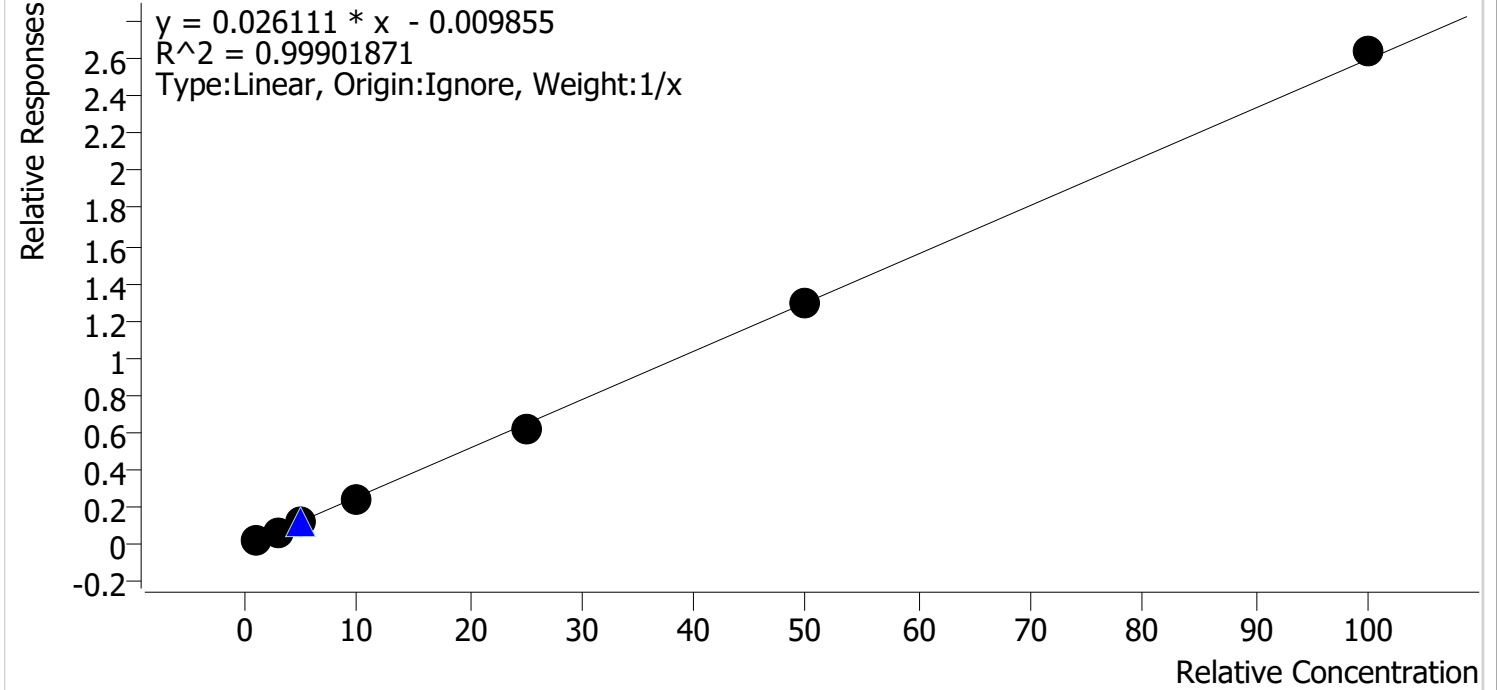
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.403	100938	449.5	265.6	1213.5	1436302	14.237 ng/ml
THC	3.077	701138	5804.0	24.5	1506.1	5994478	4.857 ng/ml

Compound Calibration Report



Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Last Cal. Update 8/24/2022 2:02 PM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-d3

THC - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 1 QCs



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✓	1.0	1.2	120.6
mj cal 2	2	✓	3.0	2.8	92.5
mj cal 3	3	✓	5.0	4.7	94.2
mj cal 4	4	✓	10.0	9.4	93.9
mj cal 5	5	✓	25.0	24.2	96.9
mj cal 6	6	✓	50.0	50.1	100.2
mj cal 7	7	✓	100.0	101.6	101.6

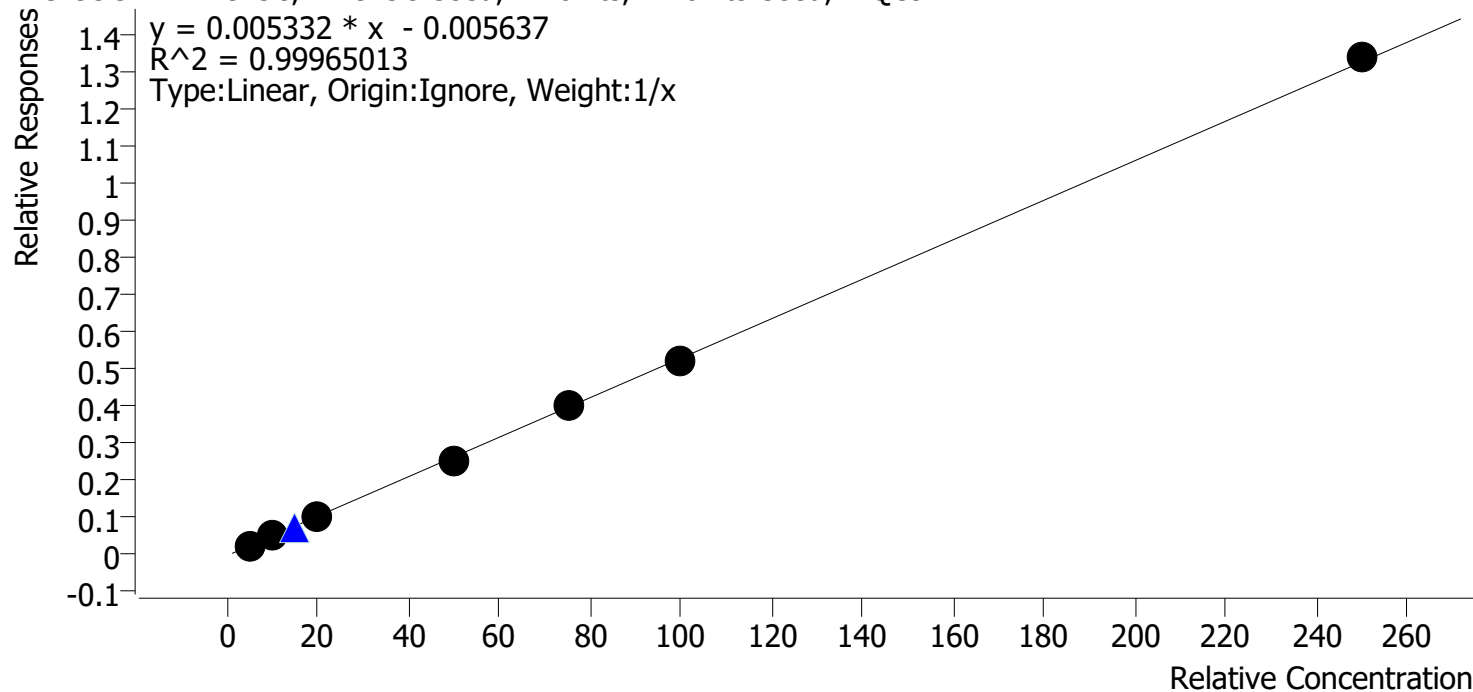
samples below 3ng will be reported as less than 3 ng/ml due to 1ng cal accuracy.

Compound Calibration Report



Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Last Cal. Update 8/24/2022 2:02 PM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-d9

THC-COOH - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 1 QCs



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✓	5.0	5.0	99.3
mj cal 2	2	✓	10.0	10.7	106.6
mj cal 3	3	✓	20.0	19.3	96.7
mj cal 4	4	✓	50.0	48.6	97.2
mj cal 5	5	✓	75.0	75.3	100.4
mj cal 6	6	✓	100.0	99.0	99.0
mj cal 7	7	✓	250.0	252.1	100.9

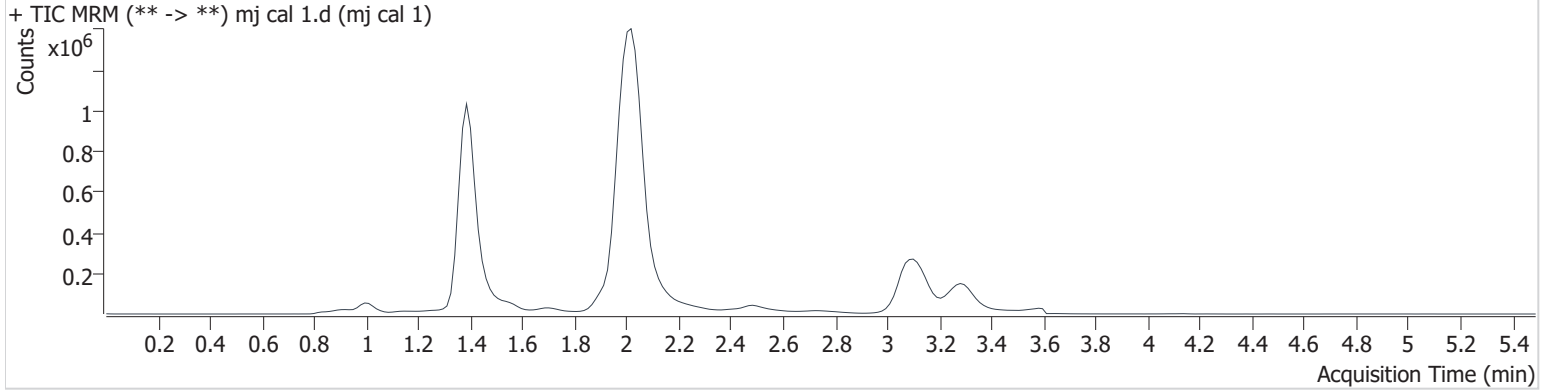
AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj cal 1.d
Type	Cal	Sample	mj cal 1
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-A1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 10:07:46 AM		

Sample Info.

Sample Chromatogram



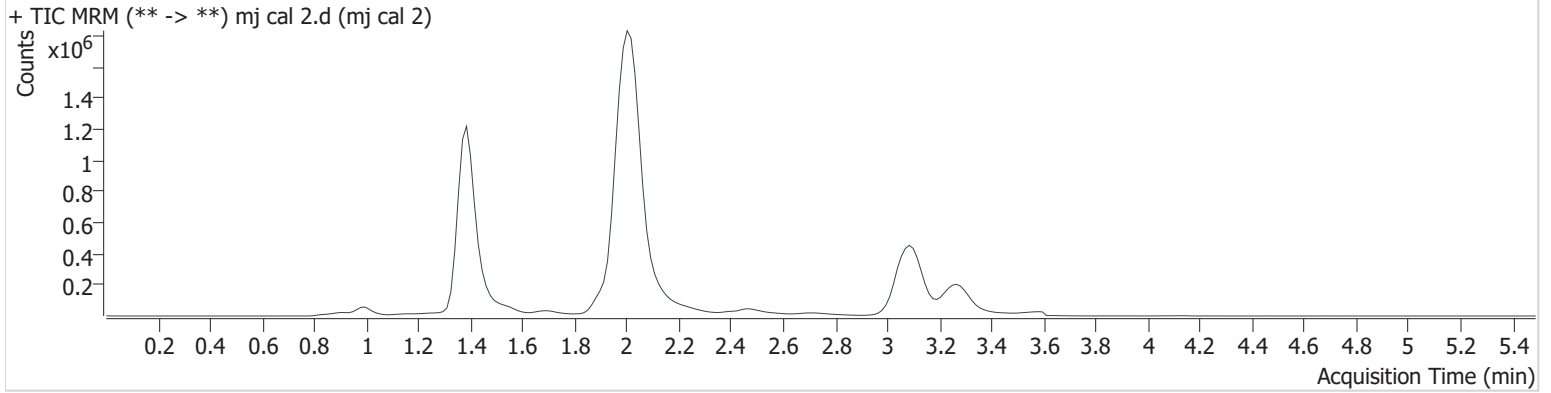
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.418	14349	33.5	286.2	∞	688965	4.963 ng/ml Low
THC	3.137	32696	325.3	29.0	31.9	1510628	1.206 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj cal 2.d
Type	Cal	Sample	mj cal 2
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-B1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 10:14:30 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.418	44732	∞	231.6	∞	873435	10.662 ng/ml
THC	3.107	151210	1864.3	26.7	184.0	2414722	2.776 ng/ml

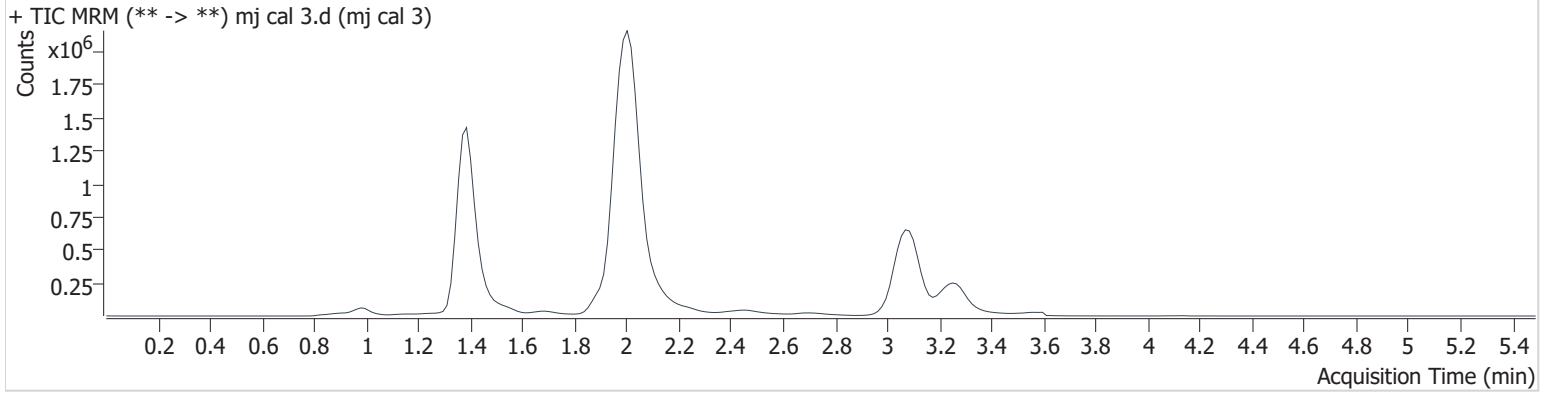
AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj cal 3.d
Type	Cal	Sample	mj cal 3
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-C1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 10:21:15 AM		

Sample Info.

Sample Chromatogram



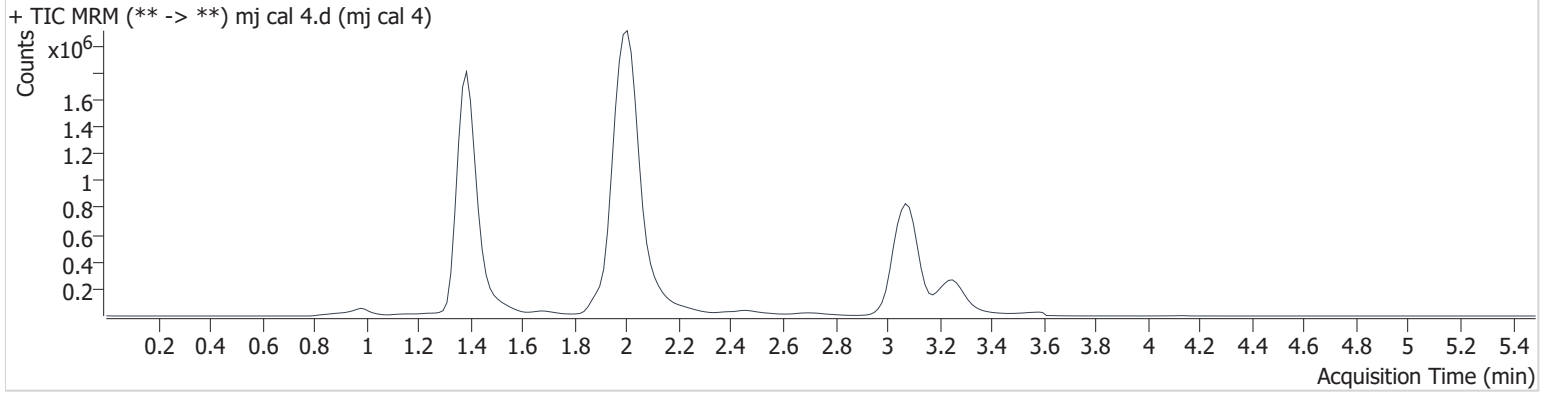
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.403	103494	∞	258.9	∞	1061849	19.337 ng/ml
THC	3.107	380193	4688.7	24.4	1539.1	3359715	4.711 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj cal 4.d
Type	Cal	Sample	mj cal 4
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-D1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 10:27:59 AM		
Sample Info.			

Sample Chromatogram



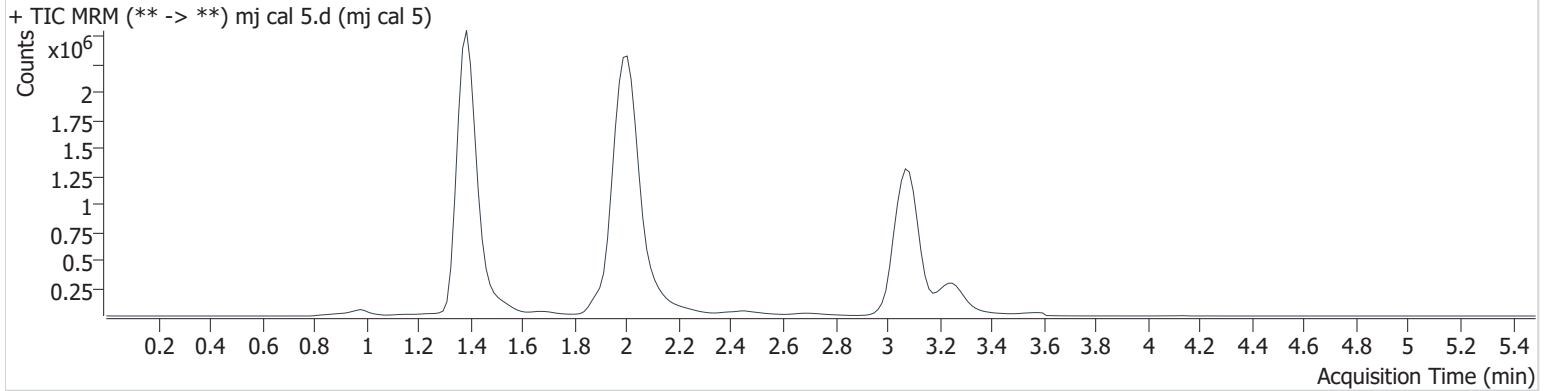
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.403	282712	1525.4	266.4	∞	1115693	48.581 ng/ml
THC	3.092	894468	4475.0	23.5	2203.4	3800322	9.392 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj cal 5.d
Type	Cal	Sample	mj cal 5
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-E1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 10:34:43 AM		
Sample Info.			

Sample Chromatogram



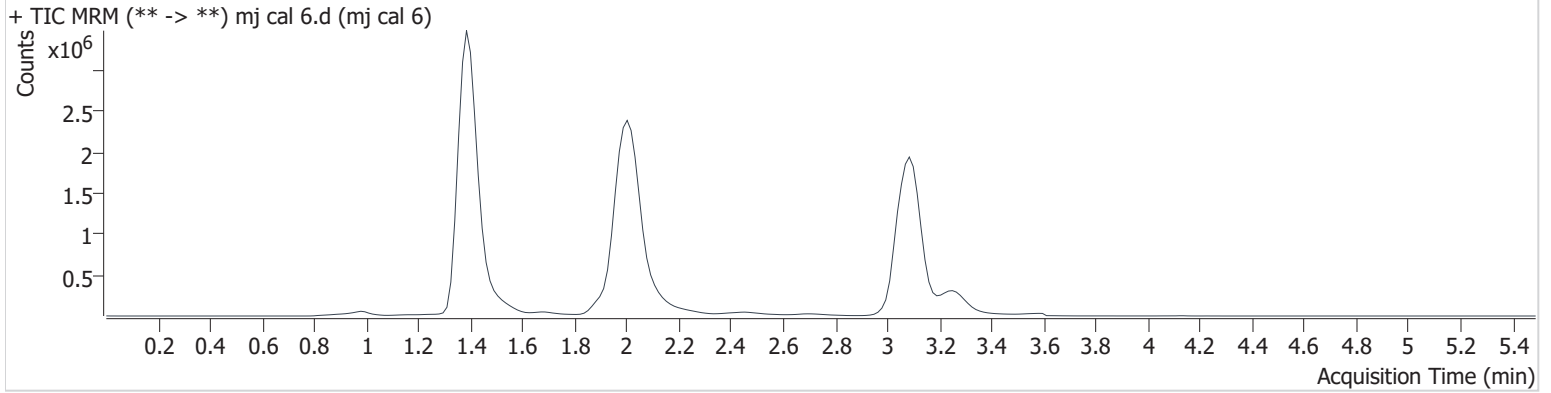
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.403	469498	∞	258.5	3908.0	1186209	75.288 ng/ml
THC	3.092	2787046	44499.6	24.4	10589	4475402	24.228 ng/ml
					123.9		

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj cal 6.d
Type	Cal	Sample	mj cal 6
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-F1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 10:41:27 AM		
Sample Info.			

Sample Chromatogram



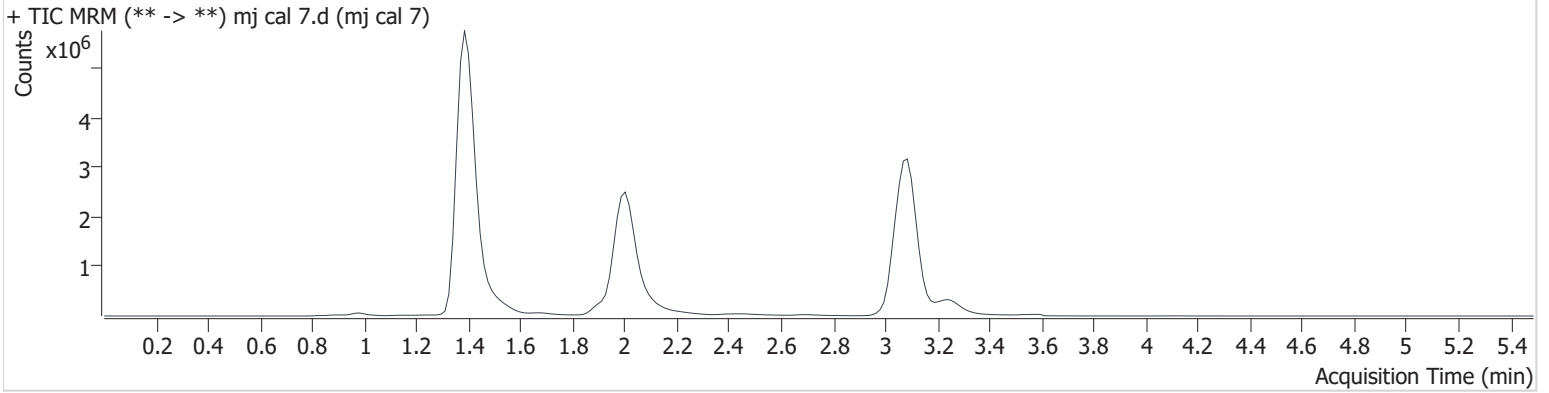
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.418	622977	∞	259.6	∞	1192578	99.028 ng/ml
THC	3.092	5959378	138007.1	25.0	16942.1	4590280	50.099 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\082422\QuantResults\cann.batch.bin
Calibration Last Update 8/24/2022 2:02:32 PM

Instrument	69679	Data File	mj cal 7.d
Type	Cal	Sample	mj cal 7
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-G1	Comment	
Injection Volume	10		
Acq. Date-Time	8/24/2022 10:48:12 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.403	1384485	16572.3	260.4	∞	1034137	252.142 ng/ml
THC	3.092	11307644	∞	25.3	17788.6	4278862	101.588 ng/ml