

2/15/2022

Worklist: 5600**REVIEWED**

By Brittany Wyllie at 8:05 am, Feb 17, 2022

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
C2022-0089	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-0115	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-0120	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-0168	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
C2022-0176	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-0191	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-0195	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-0206	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-0273		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 2/14/22
Plate lot#: 211018

Analyst: Anne Nord
Plate re-test: 4/18/22

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: 22B52020 **Urine Blank:** 83121 **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.

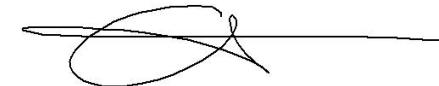
Analytic:

- 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 **1000 μ L blood (calibrated pipette)** Pipette ID: k52558g in wells of analytical (standards) plate.
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 4. Pipette **500 μ L 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 **800 μ L 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 750 μ L)**
- 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 750 μ L)**
- 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 **100 μ L 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 QC's pass for each analyte? (if not is it describe in comments section)
- 6. Enter QC's into control charting.
- 7. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: An extraction was done on 2/11/22 the plate lot was 210609, this lot required external controls to be run after 12/02/21 they were not run this extraction was not evaluated. The samples were re-extracted and run 2/14/22.



	1	2	3	4	5	6
A	cal 1	Internal control	206-1			
B	cal 2	negative blood	273-2			
C	cal 3	089-1	negative urine			
D	cal 4	115-1	168-1			
E	Cal 5	120-1 *	120-1			
F	cal 6	176-1				
G	cal 7	191-1				
H	Internal control	195-1				

C2022-0____

* well clogged another aliquot was taken and extracted.



IDAHO STATE POLICE

MEMORANDUM

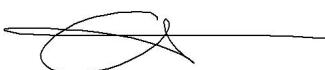
DATE: 3/3/2022

TO: Toxicology Discipline/ Jason Crowe

FROM: Celena Shrum- Toxicology Discipline lead

SUBJECT: Use of internal control in lieu of external control

Toxicology Analytical Methods #25, 26, and 27 specify that if a run contains urine samples, a negative control and **external** urine control must also be included in the run. The purpose of this control is to demonstrate that the extraction worked as intended and to ensure that the results and concentrations obtained are accurate. It was decided in October 2021 that extra QC's would be included on the analytical plates so that they could be used as an internal control for runs with urine cases instead of continuing with including an external control. An internal control serves the same purpose as an external control but is prepared and placed on the analytical plate rather than being prepared in-house and placed on the plate at the time of testing. Utilizing internal controls versus external increases the efficacy of the controls used by ensuring consistent spiking and preparation, eliminating evaporation of compounds, etc. There is no quality issue with any of the cases, since an additional urine control was used that served the same purpose as the external control, but it was a violation of the wording specified in the method.



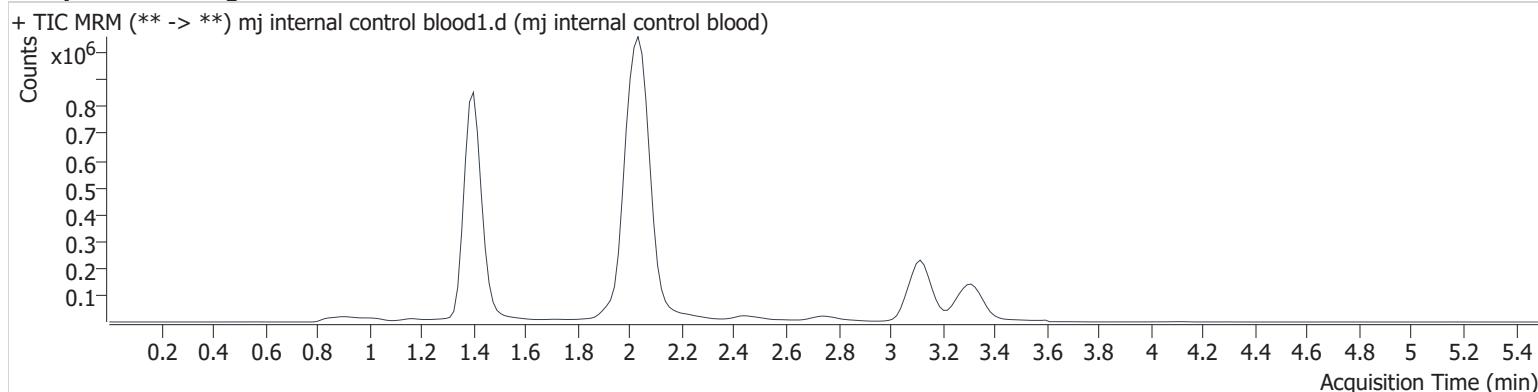
AM #27 Cannabinoids

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Calibration Last Update 2/15/2022 9:54:05 AM

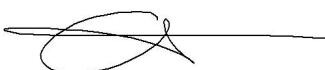
Instrument	69679	Data File	mj internal control blood1.d
Type	QC	Sample	mj internal control blood
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-H1	Comment	
Injection Volume	10		
Acq. Date-Time	2/14/2022 4:50:21 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.394	40641	605.0	798.2	689.5	2433544	5.279 ng/ml
THC-COOH	1.431	160755	205.3	34.5	113.4	728033	15.982 ng/ml
THC	3.137	101496 4761076828 7841.0		26.7	422.1	875921	4.584 ng/ml

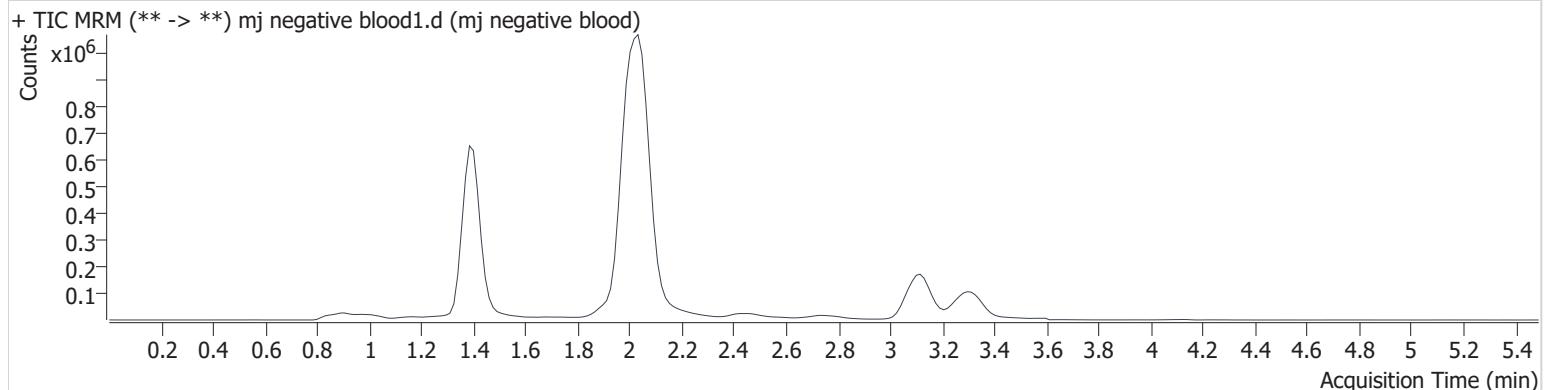


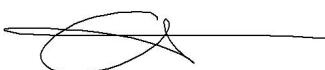
AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\021122\QuantResults\cann.batch.bin
Calibration Last Update 2/15/2022 9:54:05 AM

Instrument	69679	Data File	mj negative blood1.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	2/14/2022 4:57:06 PM		
Sample Info.			

Sample Chromatogram



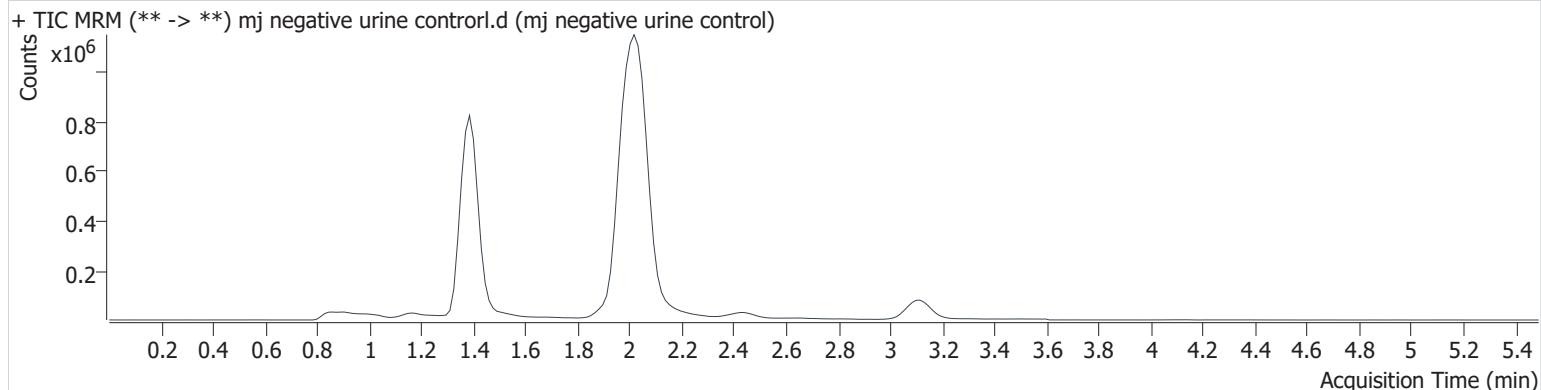


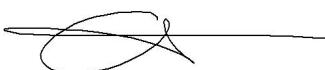
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Instrument	69679	Data File	mj negative urine controrl.d
Type	Sample	Sample	mj negative urine control
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-C3	Comment	
Injection Volume	10		
Acq. Date-Time	2/15/2022 8:49:01 AM		
Sample Info.			

Sample Chromatogram



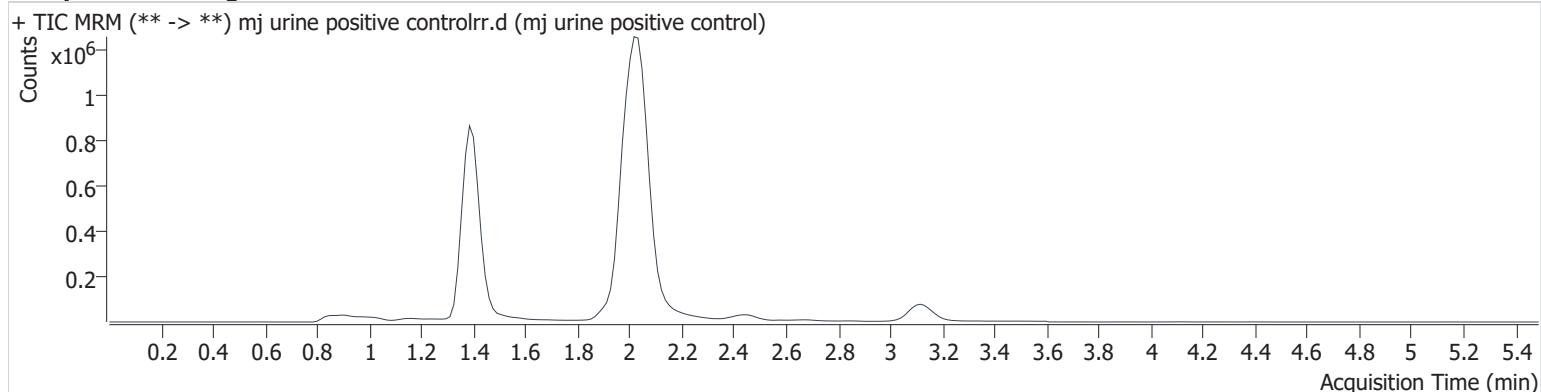


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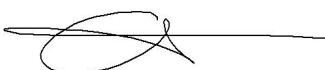
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Instrument	69679	Data File	mj urine positive controlrr.d
Type	Sample	Sample	mj urine positive control
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-A2	Comment	
Injection Volume	10		
Acq. Date-Time	2/15/2022 9:29:07 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.394	49406	∞	809.6	∞	2951215	5.292 ng/ml
THC-COOH	1.416	105713	222.4	33.3	622.2	490168	15.626 ng/ml
THC	3.137	51132	∞	25.1	∞	439096	4.605 ng/ml

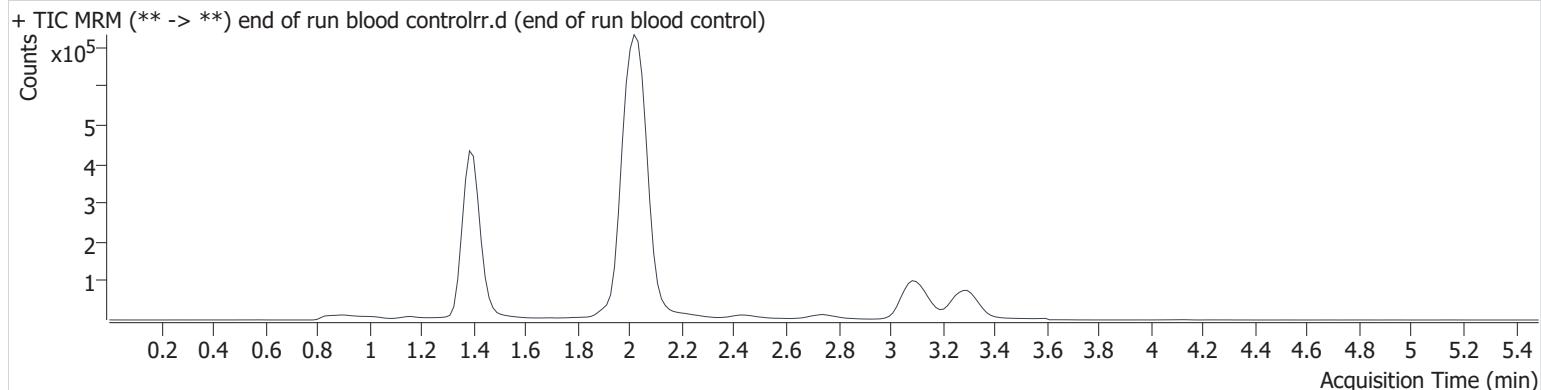


AM #27 Cannabinoids

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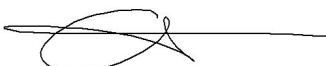
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Type	Sample	Sample	end of run blood control
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-H1	Comment	
Injection Volume	10		
Acq. Date-Time	2/15/2022 9:35:51 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.394	20133	814.5	795.9	∞	1236404	5.149 ng/ml
THC-COOH	1.416	84534	809.4	31.6	155.5	402821	15.225 ng/ml
THC	3.137	42821	∞	29.1	43845 43263 598.8	363626	4.652 ng/ml

Compound Calibration Report

**Batch results**

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Last Cal. Update

2/15/2022 9:54 AM

Analyst Name

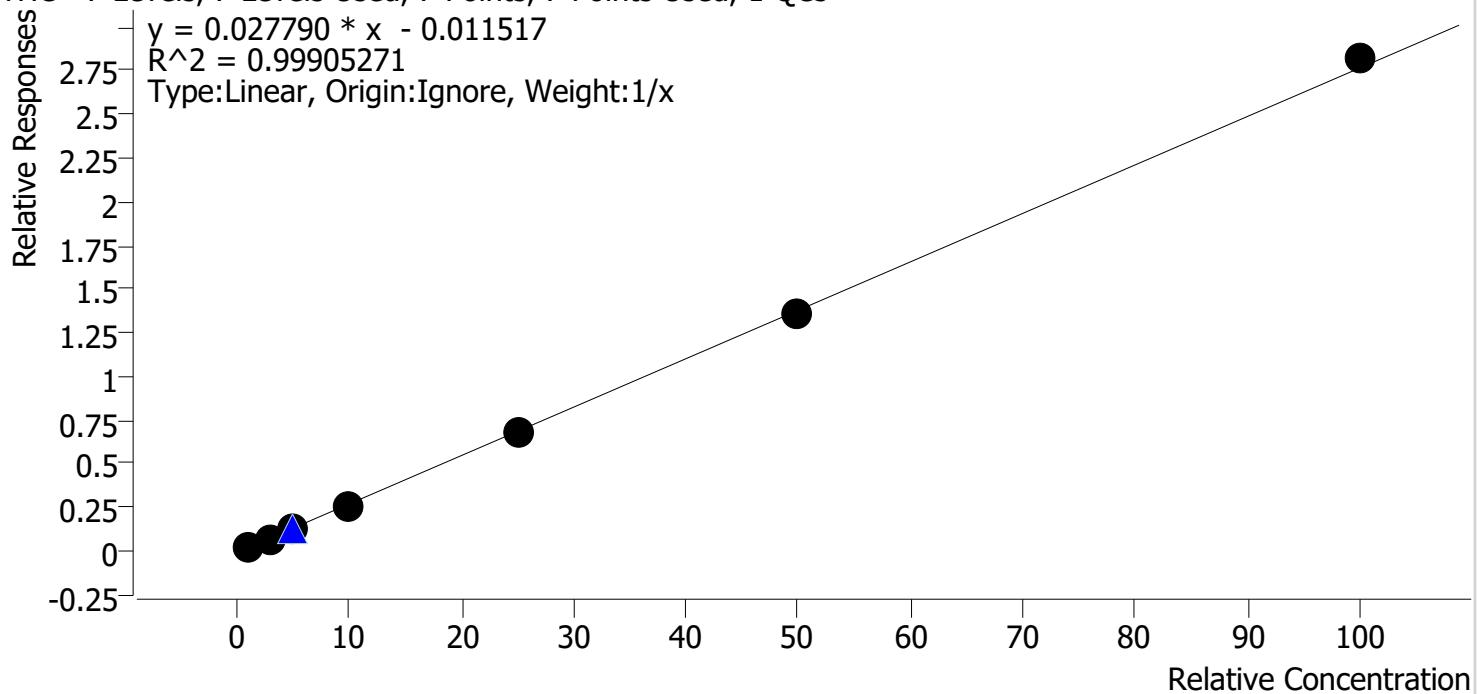
ISP\datastor

Analyte

THC

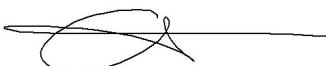
Internal Standard

THC-d3

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

Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✓	1.0	1.2	118.6
mj cal2	2	✓	3.0	2.9	95.5
mj cal 3	3	✓	5.0	4.7	93.5
mj cal 4	4	✓	10.0	9.3	93.2
mj cal 5	5	✓	25.0	24.6	98.5
mj cal 6	6	✓	50.0	49.4	98.8
mj cal 7	7	✓	100.0	101.9	101.9

Compound Calibration Report

**Batch results**

D:\MassHunter\Data\2022\am 27-28\021122\QuantResults\cann.batch.bin

Last Cal. Update

2/15/2022 9:54 AM

Analyst Name

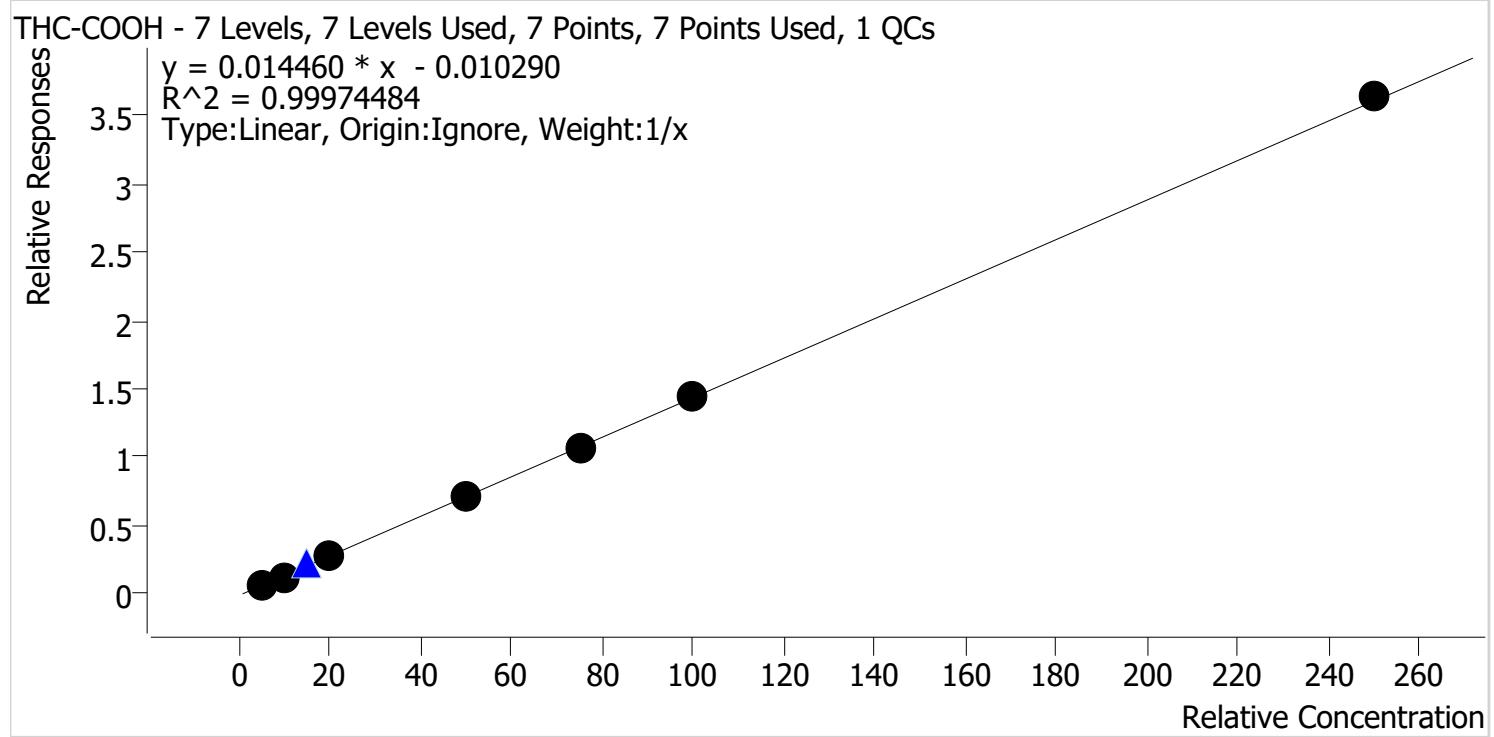
ISP\datastor

Analyte

THC-COOH

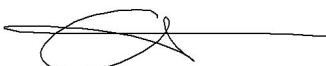
Internal Standard

THC-COOH-d9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✓	5.0	5.4	107.2
mj cal2	2	✓	10.0	9.7	97.2
mj cal 3	3	✓	20.0	19.5	97.7
mj cal 4	4	✓	50.0	49.4	98.7
mj cal 5	5	✓	75.0	73.6	98.2
mj cal 6	6	✓	100.0	100.1	100.1
mj cal 7	7	✓	250.0	252.3	100.9

Compound Calibration Report

**Batch results**

D:\MassHunter\Data\2022\am 27-28\021122\QuantResults\cann.batch.bin

Last Cal. Update

2/15/2022 9:54 AM

Analyst Name

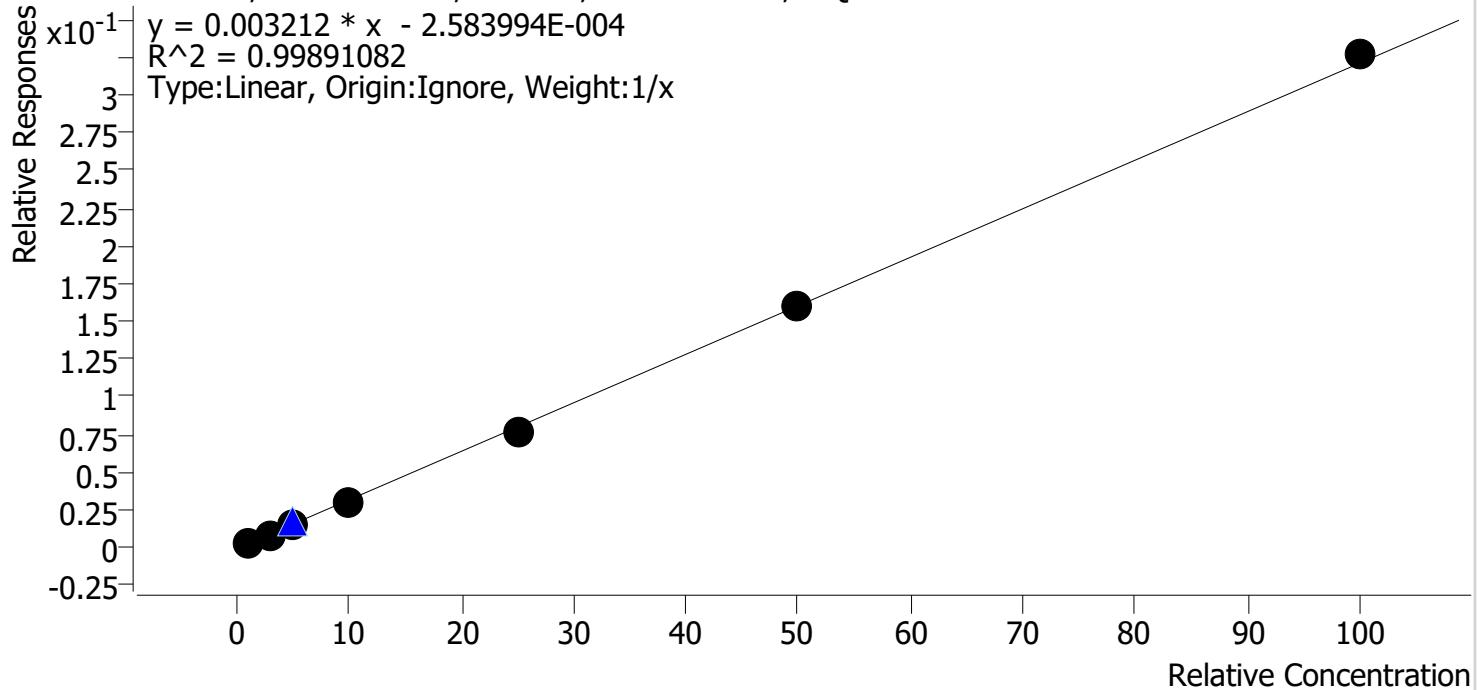
ISP\datastor

Analyte

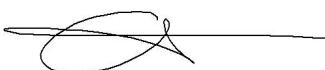
THC-OH

Internal Standard

THC-OH-d3

THC-OH - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 1 QC

Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✓	1.0	1.2	116.2
mj cal2	2	✓	3.0	2.9	97.5
mj cal 3	3	✓	5.0	4.8	96.9
mj cal 4	4	✓	10.0	9.1	91.4
mj cal 5	5	✓	25.0	24.0	95.9
mj cal 6	6	✓	50.0	50.2	100.5
mj cal 7	7	✓	100.0	101.7	101.7

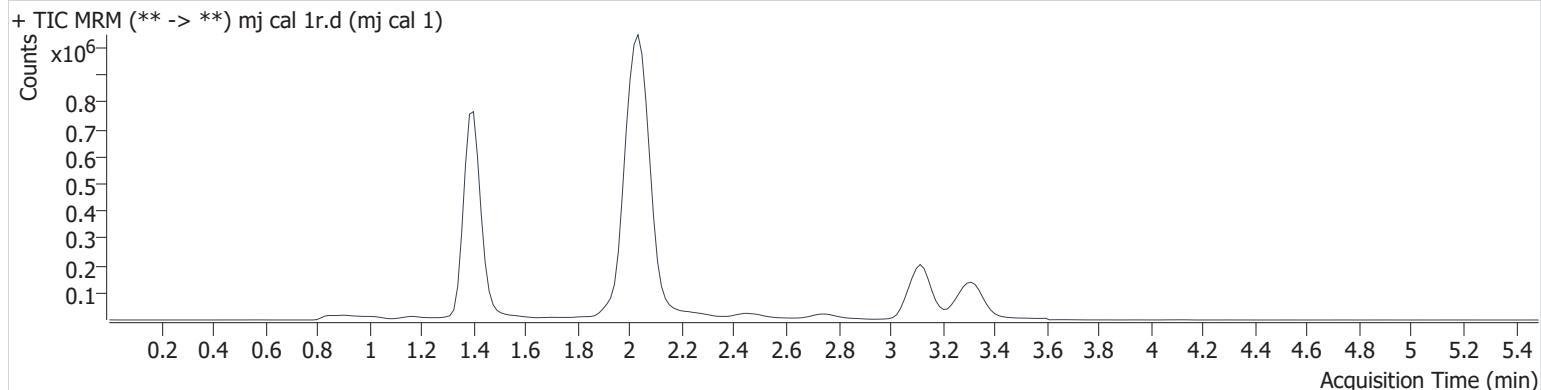


AM #27 Cannabinoids

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Calibration Last Update 2/15/2022 9:54:05 AM

Instrument	69679	Data File	mj cal 1r.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	2/14/2022 3:56:48 PM		
Sample Info.			

Sample Chromatogram



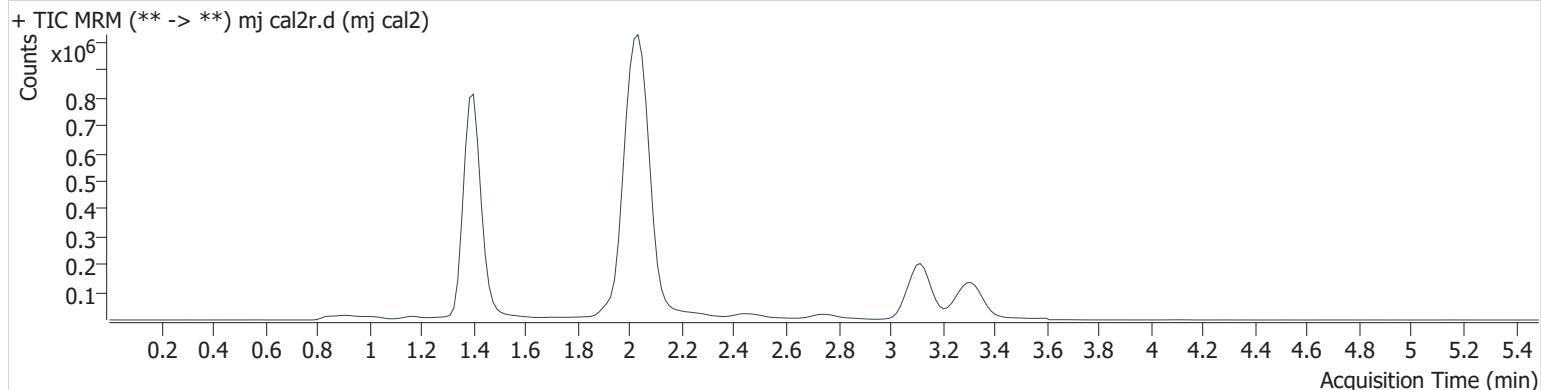
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.394	9099	273.7	681.8	∞	2619582	1.162 ng/ml	Low
THC-COOH	1.431	48183	242.2	36.4	839.7	716829	5.360 ng/ml	
THC	3.137	17506	899.1	27.6	37.2	816762	1.186 ng/ml	

AM #27 Cannabinoids

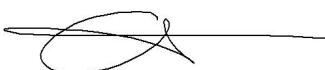
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Calibration Last Update 2/15/2022 9:54:05 AM

Instrument	69679	Data File	mj cal2r.d
Type	Cal	Sample	mj cal2
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-B1	Comment	
Injection Volume	10		
Acq. Date-Time	2/14/2022 4:03:32 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.394	23881	4565.3	777.1	∞	2614703	2.924 ng/ml	Low
THC-COOH	1.431	93157	64.3	36.5	39272. 9	715131	9.720 ng/ml	
THC	3.137	55766	4214.9	28.7	142.7	818748	2.865 ng/ml	

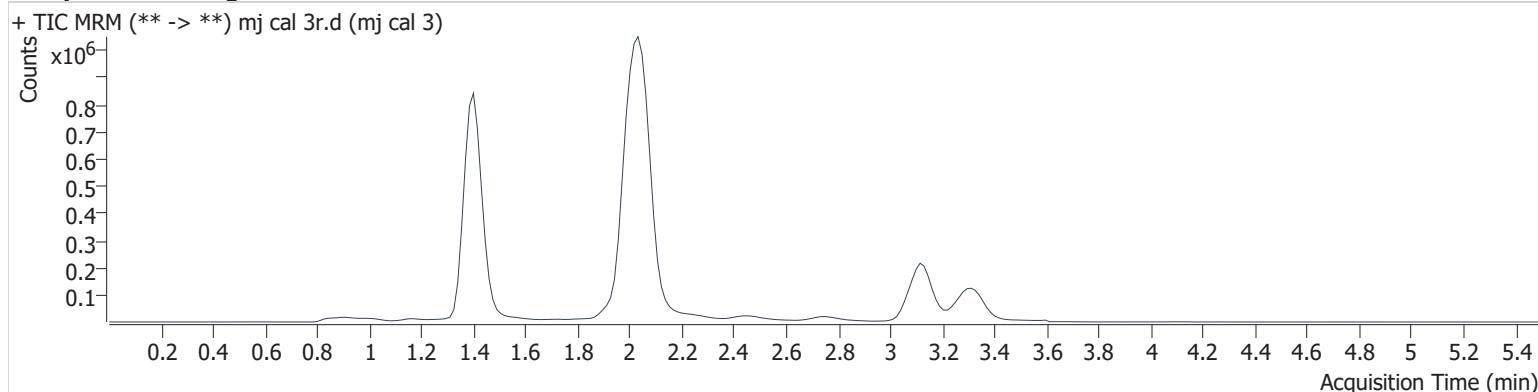


AM #27 Cannabinoids

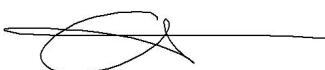
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Calibration Last Update 2/15/2022 9:54:05 AM

Instrument	69679	Data File	mj cal 3r.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	2/14/2022 4:10:14 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.394	37373	∞	782.0	5747.6	2441595	4.845 ng/ml
THC-COOH	1.431	202252	391.8	35.0	90.7	743156	19.533 ng/ml
THC	3.137	100001 4108120417 1726.2		25.0	399.2	845026	4.673 ng/ml

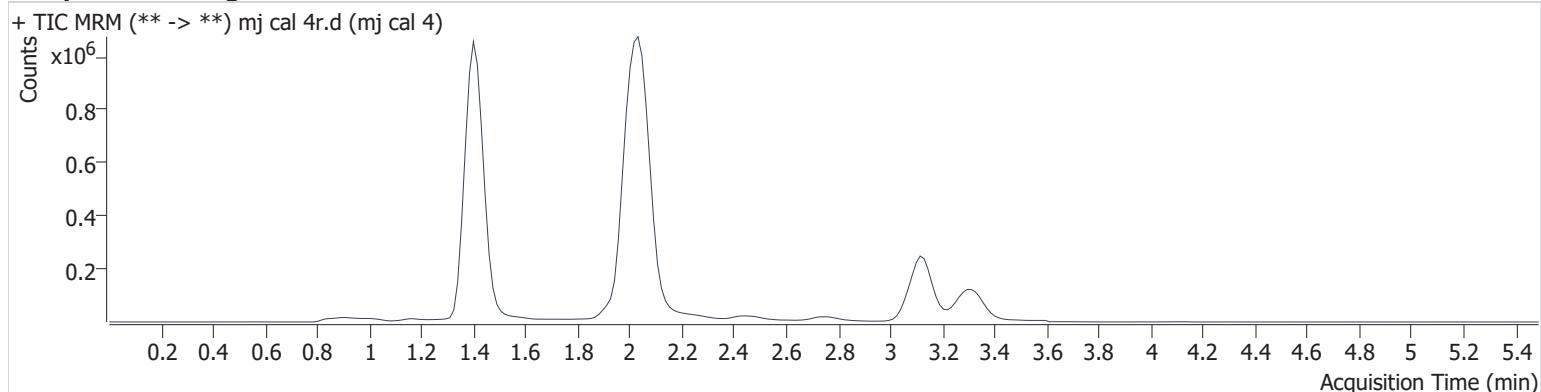


AM #27 Cannabinoids

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Calibration Last Update 2/15/2022 9:54:05 AM

Instrument	69679	Data File	mj cal 4r.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	2/14/2022 4:16:56 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.394	67942	1783.0	873.5	∞	2334394	9.140 ng/ml
THC-COOH	1.431	517955	2010.2	36.7	1579.5	736155	49.370 ng/ml
THC	3.137	222374	∞	22.9	495.4	898279	9.322 ng/ml

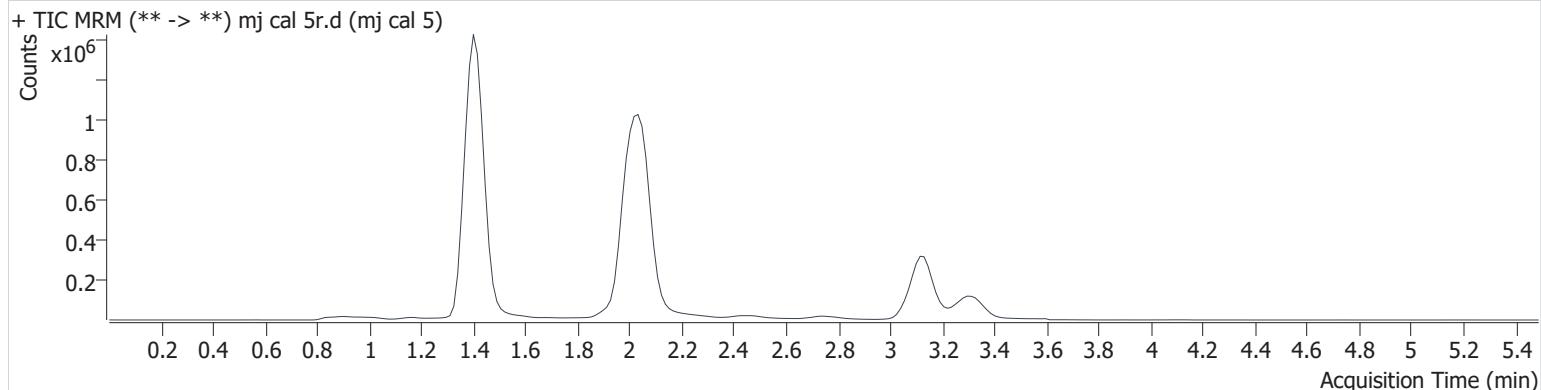


AM #27 Cannabinoids

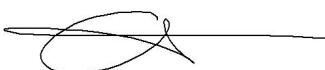
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Type	Cal	Sample	mj cal 5
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-E1	Comment	
Injection Volume	10		
Acq. Date-Time	2/14/2022 4:23:38 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.394	181571	∞	874.2	∞	2366205	23.967 ng/ml
THC-COOH	1.431	793250	796.5	37.4	1424.8	752361	73.627 ng/ml
THC	3.137	608382	1379766077	23.6	∞	904287	24.623 ng/ml



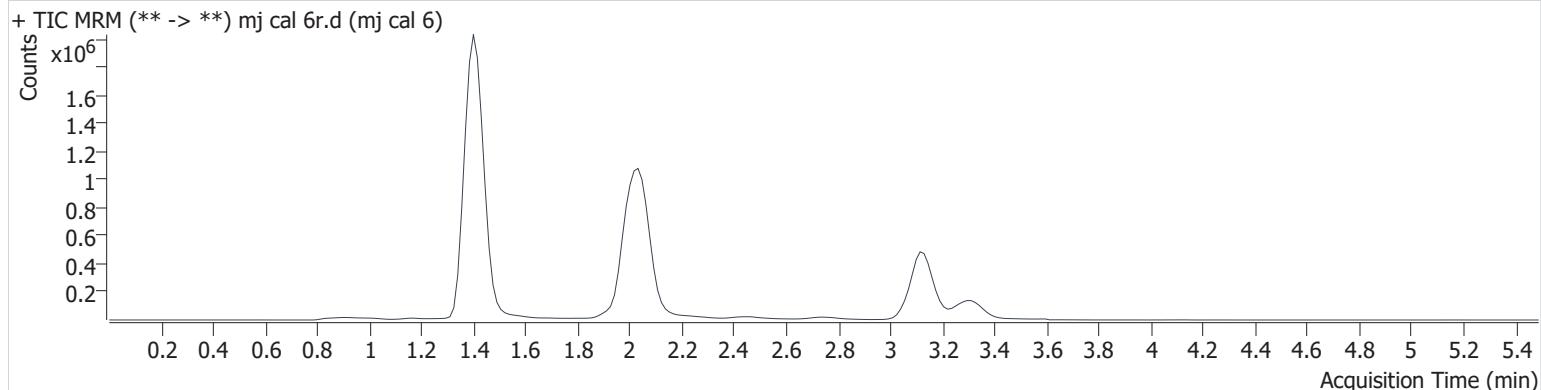
AM #27 Cannabinoids

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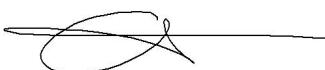
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Type	Cal	Sample	mj cal 6
Acq. Method	AM 27 THC quant.m	Operator	Anne Nord
Sample Position	P3-F1	Comment	
Injection Volume	10		
Acq. Date-Time	2/14/2022 4:30:21 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.394	414676	∞	843.4	∞	2573368	50.242 ng/ml
THC-COOH	1.431	1050452	2025.2	38.3	6307.6	730860	100.109 ng/ml
THC	3.137	1344700	∞	23.4	∞	987350	49.422 ng/ml



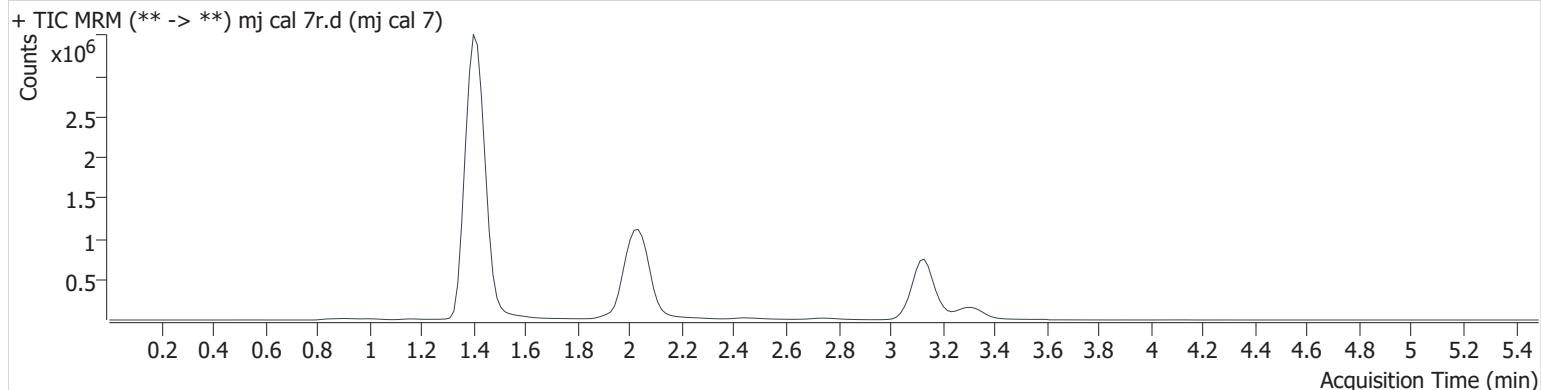
AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\021122\QuantResults\cann.batch.bin
Calibration Last Update 2/15/2022 9:54:05 AM

Instrument	69679	Data File	mj cal 7r.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	2/14/2022 4:37:03 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.394	846223	∞	839.1	∞	2591708	101.720 ng/ml
THC-COOH	1.431	2435334	5427.5	37.8	5001.5	669477	252.281 ng/ml
THC	3.137	2665939	∞	23.7	∞	945176	101.909 ng/ml