

REVIEWED

By Tamara Salazar at 9:00 am, May 04, 2022

4/27/2022

CS

Worklist: 5821

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
M2022-1156	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2022-1224	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2022-1262	3	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2022-1303	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2022-1304	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2022-1443	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2022-1461	3	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-0832	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-0863	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-0887	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-0958	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-0989	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-1019	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-1021	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-1034	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-1036	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-1038	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-1114	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: 04/26/2022

Analyst: Celena Shrum

Plate lot#: 220309

Plate Retest Date: 09/09/2022

Mobile phase A: 0.1% Formic Acid in LCMS Water

Mobile phase B: 0.1% Formic acid in Acetonitrile

Blank Blood Lot: Lampire 22B52016-2

Column: UCT Selectra DA 100 x 2.1mm 3um

LCMS-QQQ ID: 069901

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. Using a calibrated pipette, add **1000µl blood and urine (if applicable) (calibrated pipette)** into the appropriate wells of analytical (standards) plate. **Pipette ID: #42**
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 4. Pipette **500µL 0.1% formic acid in water blood sample** 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)**
- 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.
- 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 and OH-THC 3ng/mL (quantitative), Carboxy-THC: 5ng/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, 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-OH curve range: 3-100 5/4/22 04

	1	2	3	4	5	6
a	cal 1ng	QC 2	M2022-1461-3	P2022-1034-1		
b	cal 3 ng	Blood NEG	P2022-0832-1	P2022-1036-1		
c	cal 5 ng	M2022-1156-2	P2022-0863-1	P2022-1038-1		
d	cal 10ng	M2022-1224-1	P2022-0887-1	P2022-1114-1		
e	cal 25 ng	M2022-1262-3	P2022-0958-1			
f	cal 50 ng	M2022-1303-1	P2022-0989-1			
g	cal 100 ng	M2022-1304-1	P2022-1019-1			
h	QC 1	M2022-1443-2	P2022-1021-1			

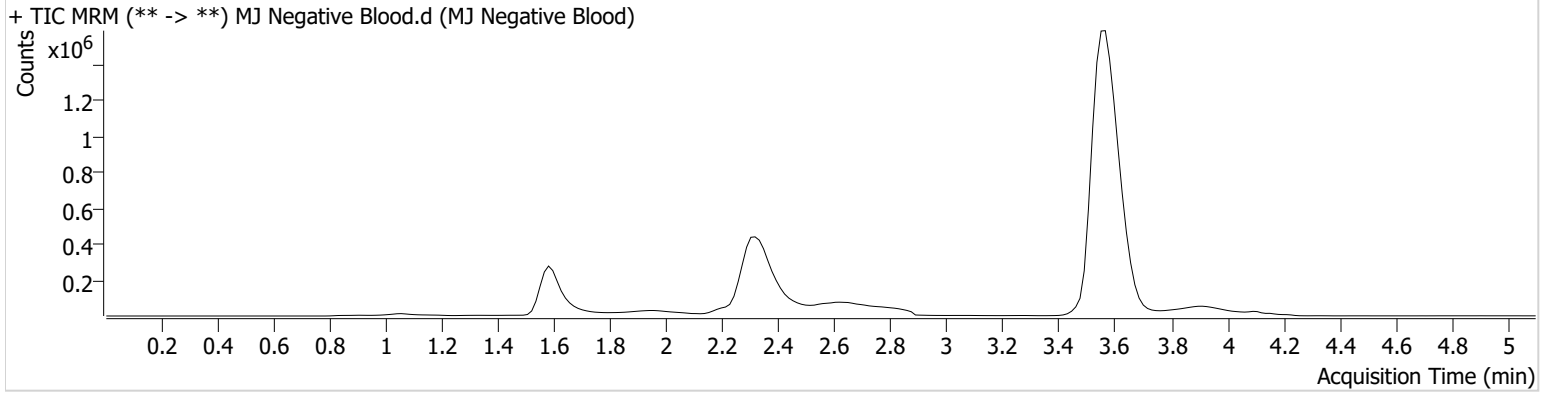
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	MJ Negative Blood.d
Type	Sample	Sample	MJ Negative Blood
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-B2	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 2:11:22 PM		
Sample Info.			

Sample Chromatogram



AM #27 Cannabinoid Quant. Results

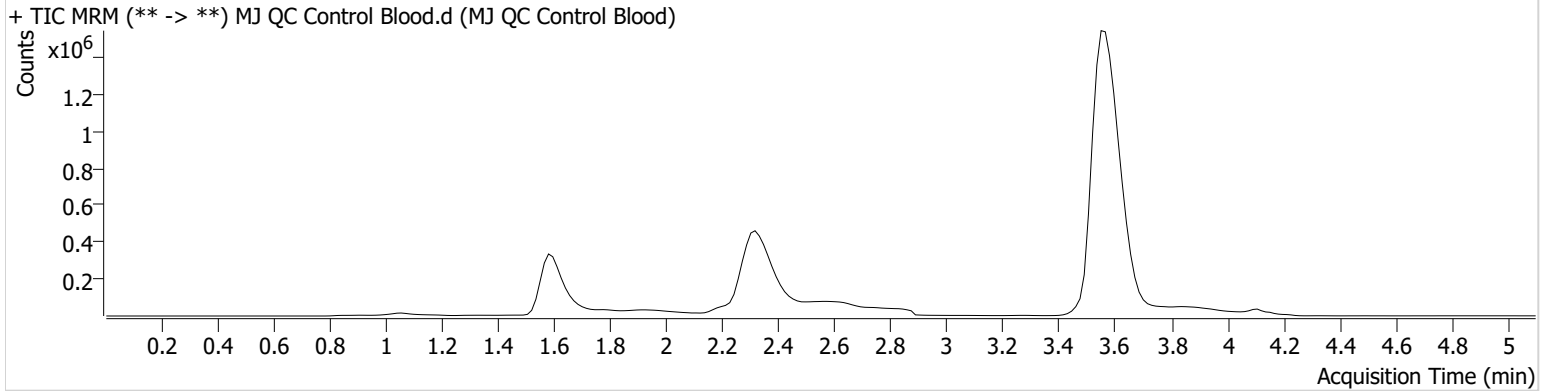


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Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	MJ QC Control Blood.d
Type	QC	Sample	MJ QC Control Blood
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-H1	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 1:56:08 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.625	144084	∞	45.0	511.47	304340	15.2528 ng/ml
THC-OH	1.588	93640	∞	11.5	197.05	1298333	4.2510 ng/ml
THC	3.570	427497	∞	24.8	206.74	10401839	4.8855 ng/ml

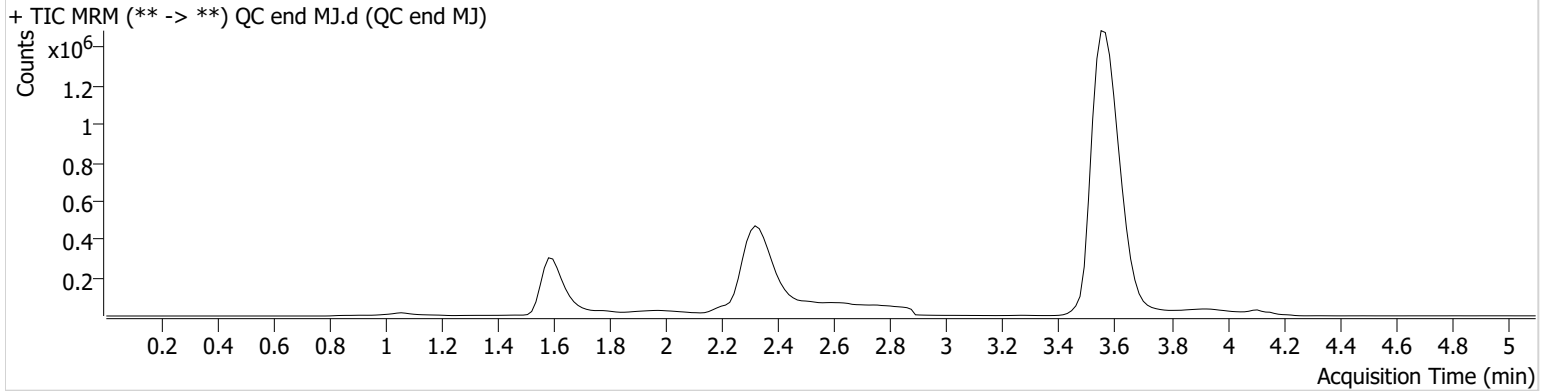
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	QC end MJ.d
Type	QC	Sample	QC end MJ
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-A2	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 7:00:45 PM		

Sample Chromatogram

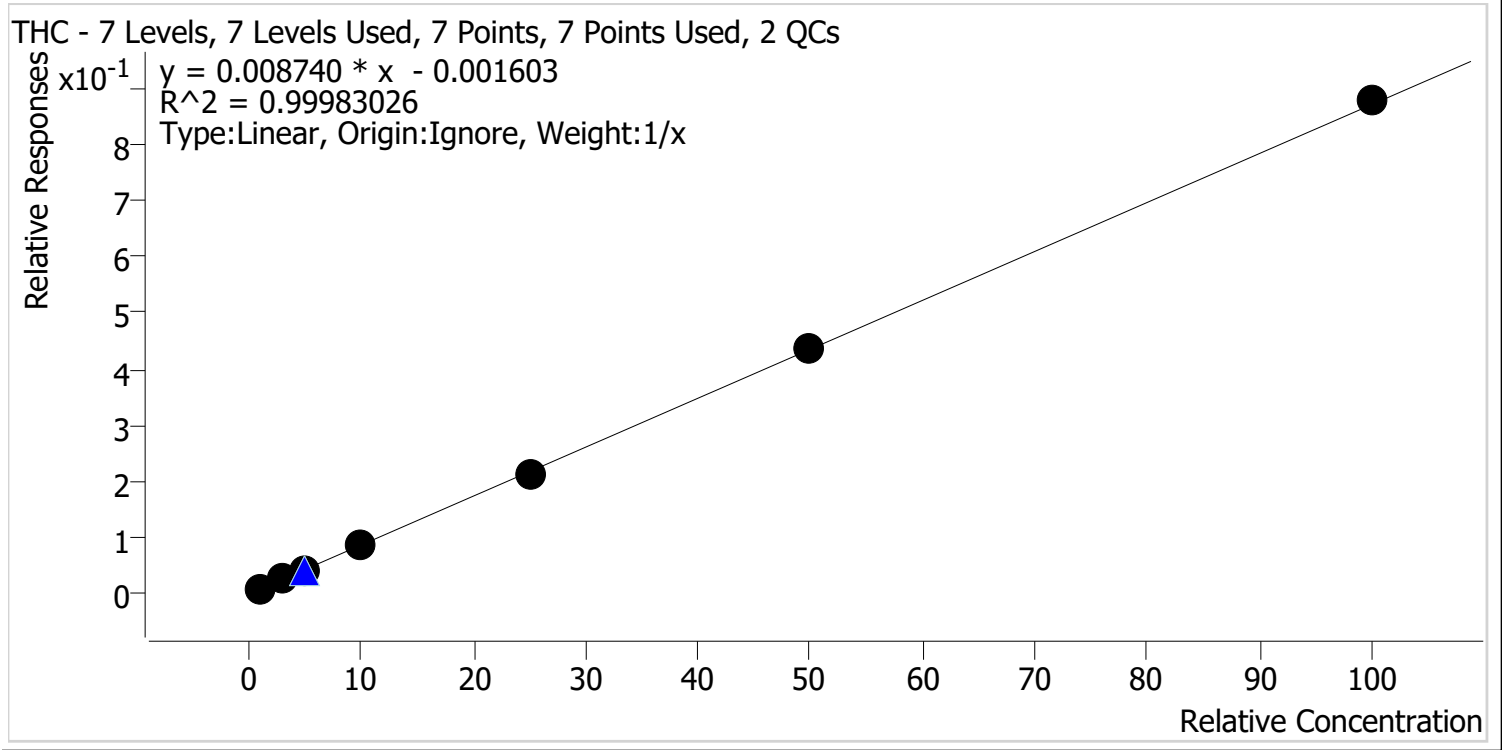


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.625	133004	752.35	46.6	∞	299621	14.0513 ng/ml
THC-OH	1.588	82018	∞	11.6	∞	1137580	4.2493 ng/ml
THC	3.570	413433	∞	25.3	∞	10054830	4.8878 ng/ml



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update 4/28/2022 1:01 PM
Analyst Name ISP\Datastor
Analyte THC **Internal Standard** THC-D3

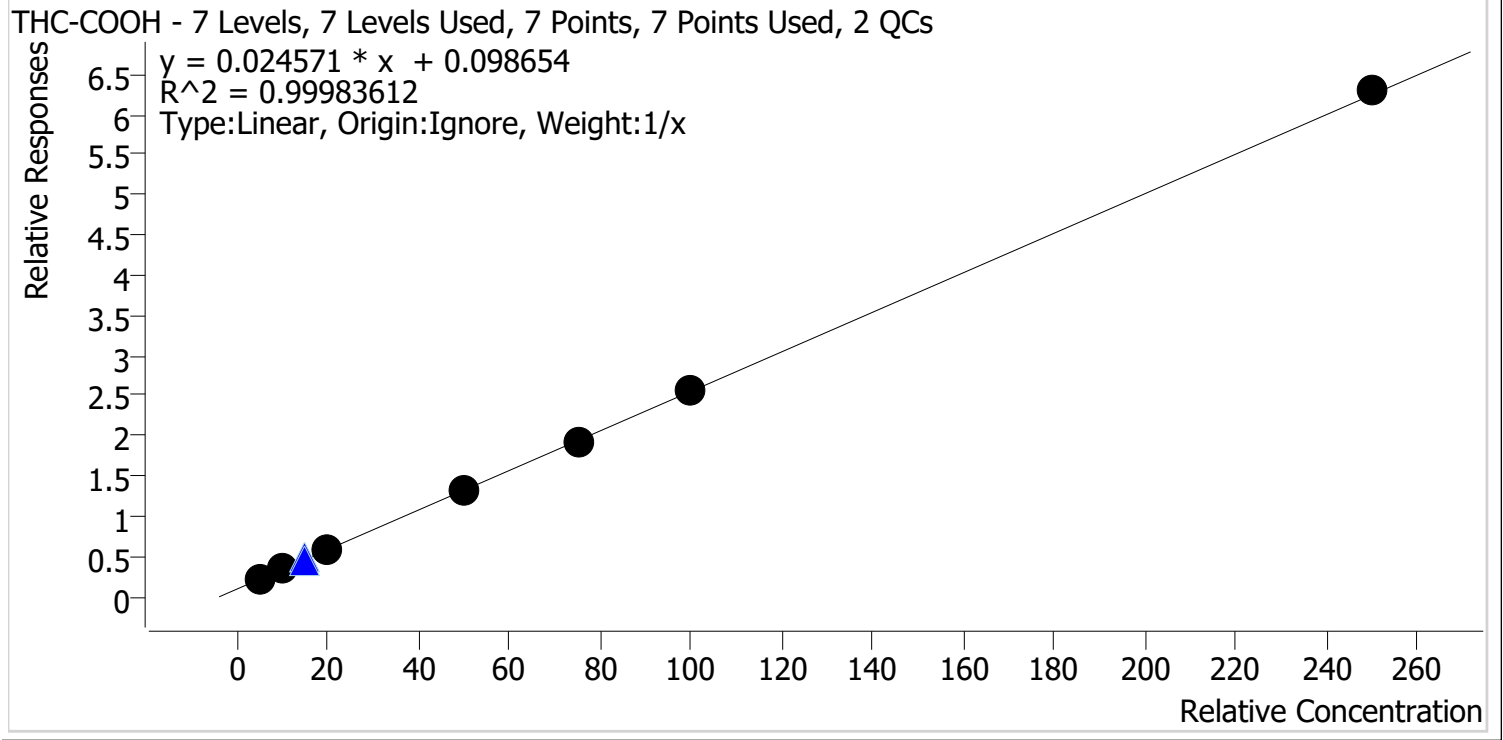


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✓	1.0	1.1	107.9
Cal 2 MJ	2	✓	3.0	2.9	98.3
Cal 3 MJ	3	✓	5.0	4.8	96.9
Cal 4 MJ	4	✓	10.0	9.8	97.7
Cal 5 MJ	5	✓	25.0	24.7	98.6
Cal 6 MJ	6	✓	50.0	49.9	99.8
Cal 7 MJ	7	✓	100.0	100.8	100.8



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update 4/28/2022 1:01 PM
Analyst Name ISP\Datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✓	5.0	5.2	103.3
Cal 2 MJ	2	✓	10.0	10.1	101.1
Cal 3 MJ	3	✓	20.0	19.5	97.5
Cal 4 MJ	4	✓	50.0	49.6	99.1
Cal 5 MJ	5	✓	75.0	73.7	98.2
Cal 6 MJ	6	✓	100.0	99.9	99.9
Cal 7 MJ	7	✓	250.0	252.1	100.8

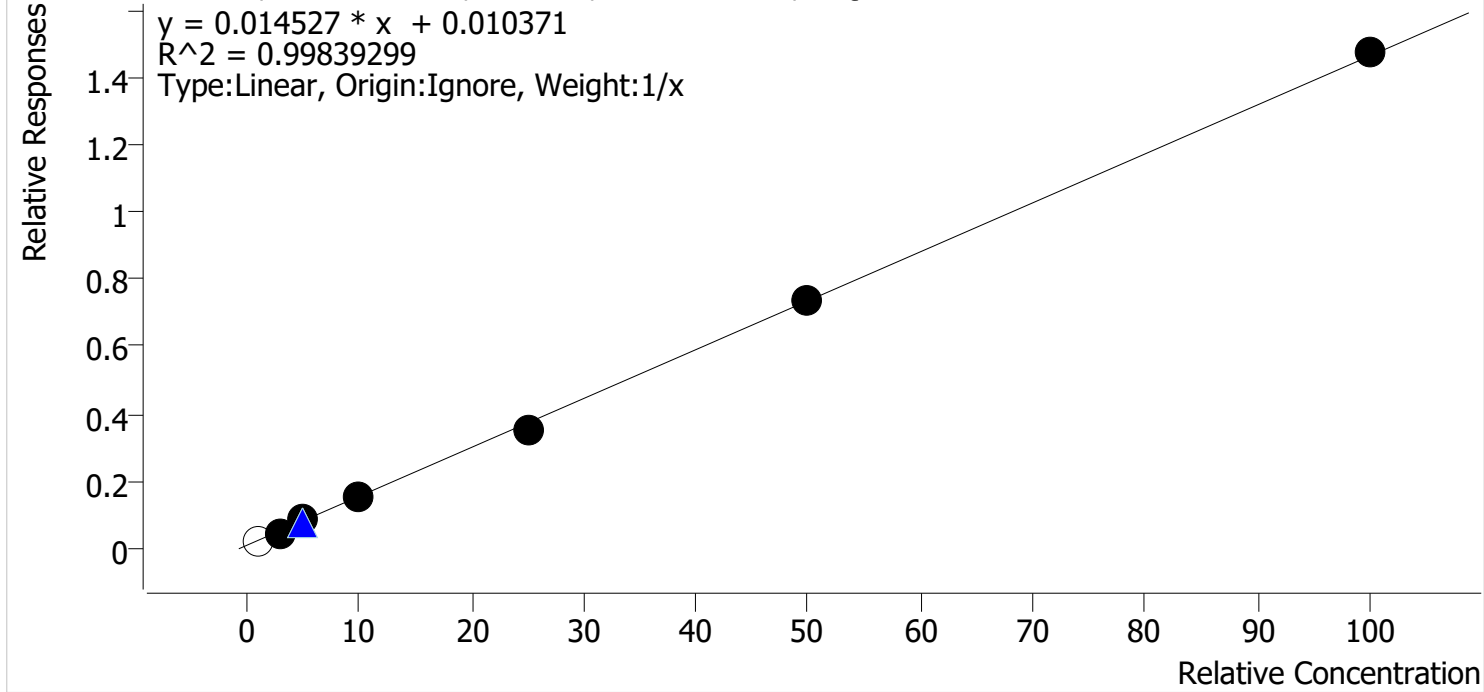
5/4/22



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update 4/28/2022 1:01 PM
Analyst Name ISP\Datastor
Analyte THC-OH **Internal Standard** THC-OH-D3

THC-OH - 7 Levels, 6 Levels Used, 7 Points, 6 Points Used, 2 QCs



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	x	1.0	0.9	93.9
Cal 2 MJ	2	✓	3.0	2.7	89.6
Cal 3 MJ	3	✓	5.0	5.7	113.1
Cal 4 MJ	4	✓	10.0	10.2	102.2
Cal 5 MJ	5	✓	25.0	23.4	93.7
Cal 6 MJ	6	✓	50.0	50.3	100.7
Cal 7 MJ	7	✓	100.0	100.7	100.7

Cal 1 removed due to peak shape (it is also not within the curve range for the method).

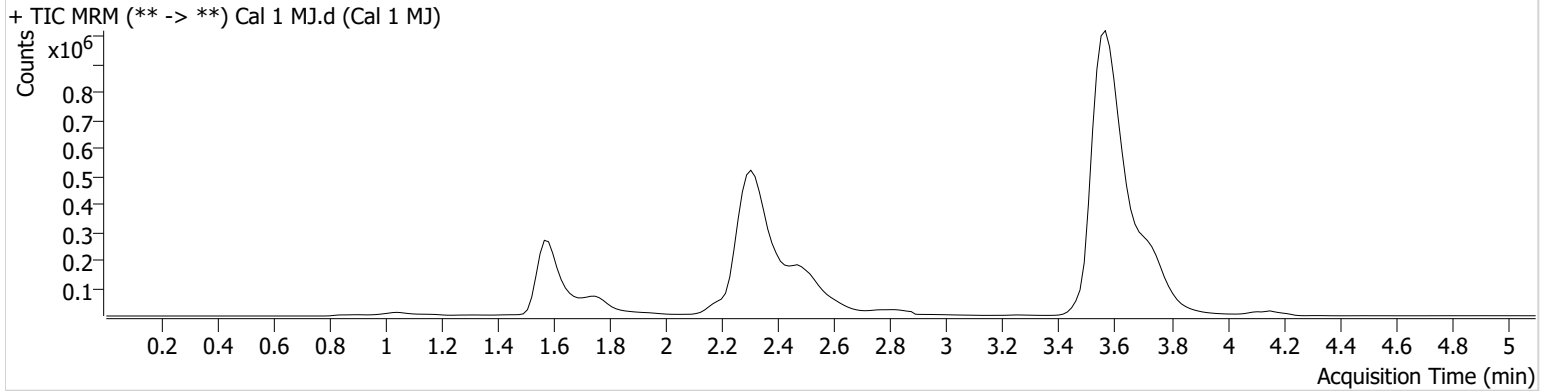
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	Cal 1 MJ.d
Type	Cal	Sample	Cal 1 MJ
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-A1	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 12:55:08 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.610	68957	∞	40.0	∞	305760	5.1635 ng/ml
THC-OH	1.648	27936	28.28	13.8	10.77	1163710	0.9386 ng/ml Low
THC	3.570	69960	∞	28.6	∞	8938016	1.0789 ng/ml

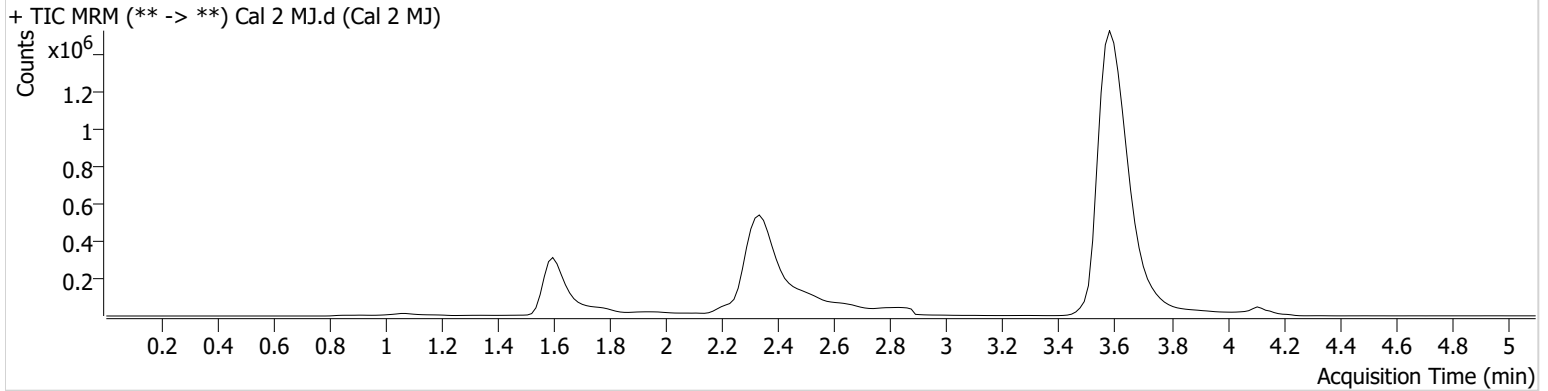
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	Cal 2 MJ.d
Type	Cal	Sample	Cal 2 MJ
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-B1	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 1:02:54 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.640	107351	∞	42.9	224.45	309333	10.1089 ng/ml
THC-OH	1.603	61546	∞	11.3	89.21	1245506	2.6877 ng/ml Low
THC	3.601	269654	∞	25.7	285.66	11158896	2.9481 ng/ml

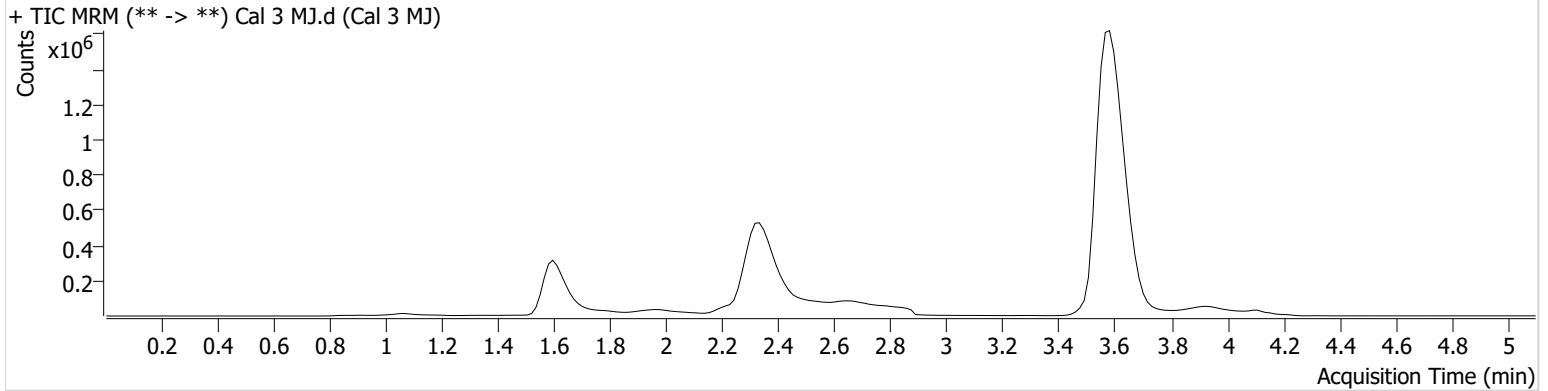
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	Cal 3 MJ.d
Type	Cal	Sample	Cal 3 MJ
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-C1	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 1:10:29 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.640	168619	410.85	49.0	762.26	291715	19.5097 ng/ml
THC-OH	1.603	108123	∞	10.0	84.80	1168235	5.6573 ng/ml
THC	3.586	440161	∞	25.5	∞	10806288	4.8436 ng/ml

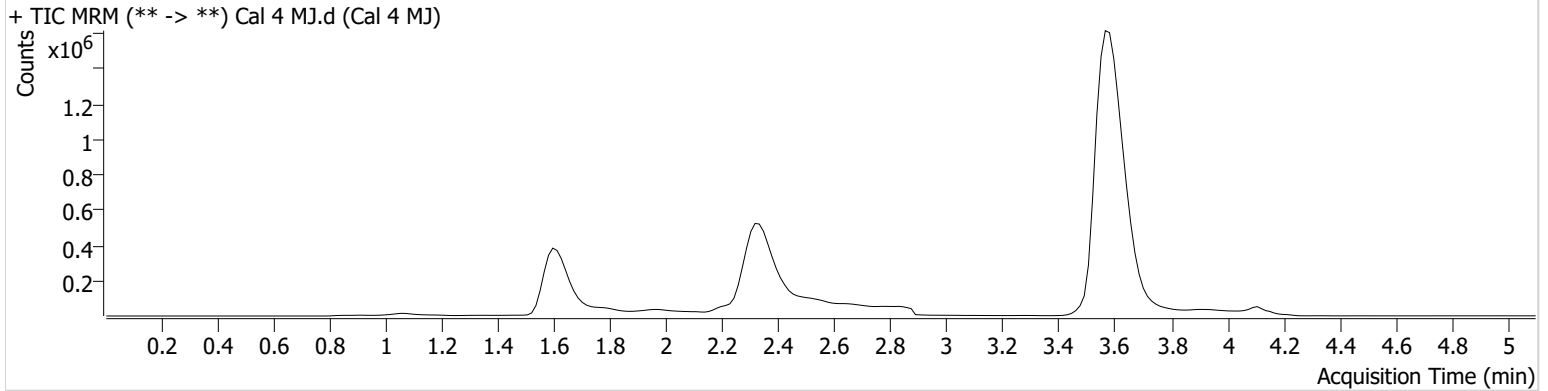
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	Cal 4 MJ.d
Type	Cal	Sample	Cal 4 MJ
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-D1	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 1:18:05 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.625	391869	1374.14	55.6	∞	297610	49.5731 ng/ml
THC-OH	1.603	192630	∞	11.1	340.19	1213048	10.2176 ng/ml
THC	3.586	888918	∞	24.7	∞	10608567	9.7703 ng/ml

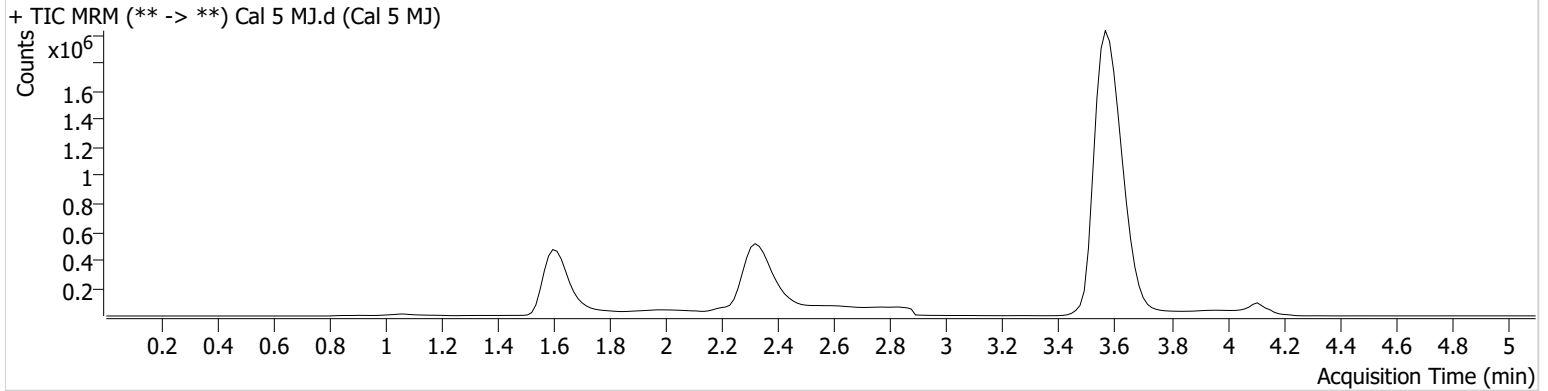
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	Cal 5 MJ.d
Type	Cal	Sample	Cal 5 MJ
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-E1	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 1:25:41 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.625	561982	∞	55.1	∞	294409	73.6718 ng/ml
THC-OH	1.588	423835	∞	13.1	605.03	1208168	23.4354 ng/ml
THC	3.586	2408986	∞	24.8	∞	11259716	24.6616 ng/ml

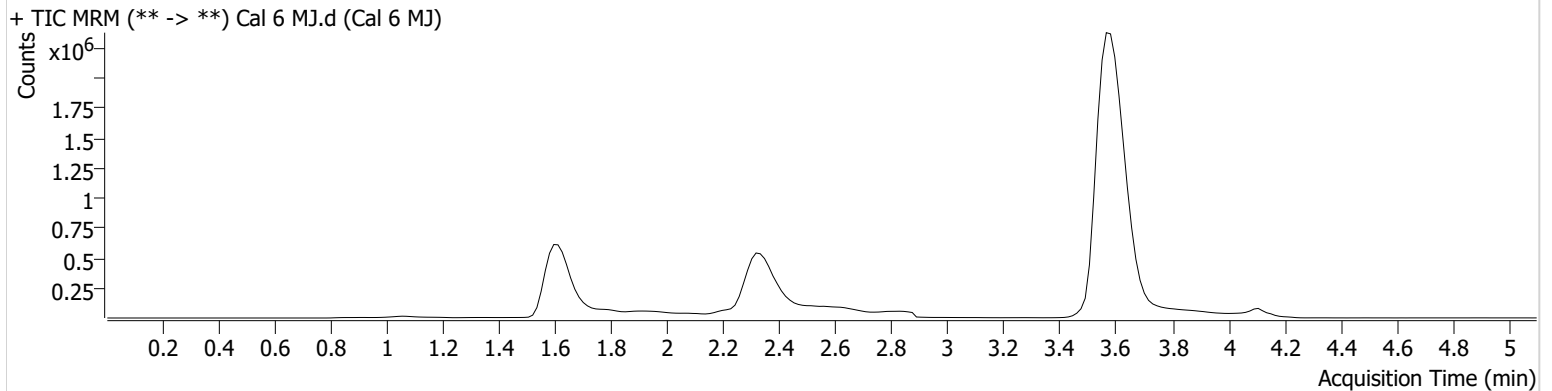
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	Cal 6 MJ.d
Type	Cal	Sample	Cal 6 MJ
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-F1	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 1:33:17 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.625	751667	∞	57.0	∞	294445	99.8805 ng/ml
THC-OH	1.603	939326	∞	12.5	11056.1	1266417	50.3454 ng/ml
THC	3.586	4851882	∞	25.1	∞	11161130	49.9198 ng/ml

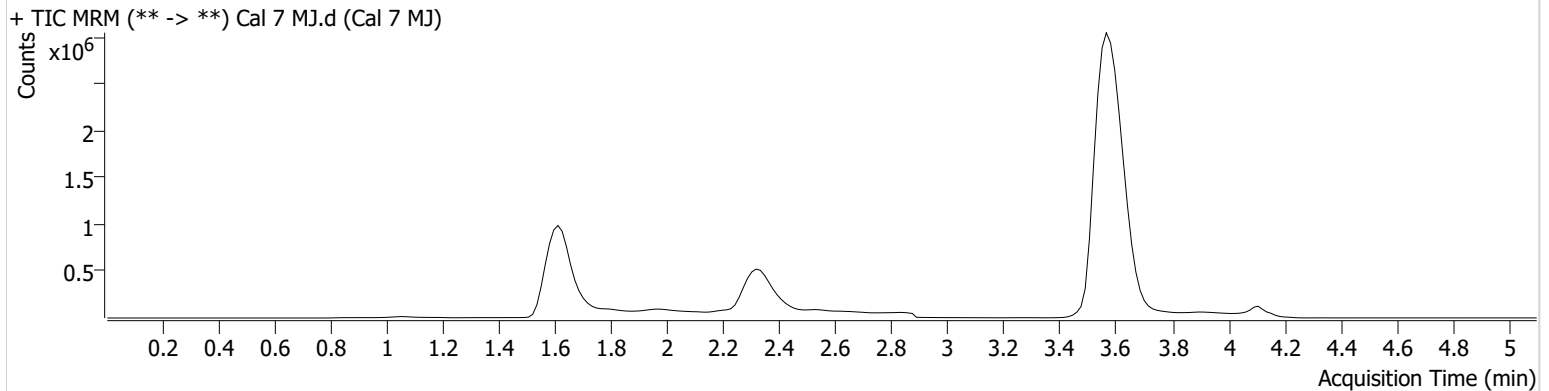
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2022\AM 27-28\042622 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 4/28/2022 1:01:33 PM

Instrument	Falco (069901)	Data File	Cal 7 MJ.d
Type	Cal	Sample	Cal 7 MJ
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P5-G1	Comment	
Injection Volume	10		
Acq. Date-Time	4/26/2022 1:40:52 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.625	1654757	∞	40.3	4804.12	262958	252.0926 ng/ml
THC-OH	1.588	1711512	1726.94	12.6	2307.07	1162261	100.6567 ng/ml
THC	3.586	9132687	∞	24.8	∞	10387174	100.7777 ng/ml