










REVIEWED

By Amber Gerheart at 10:52 am, Nov 21, 2022

11/17/2022

Worklist: 6166

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
P2022-3188	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-3248	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-3314	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-3364	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-3422	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-3442	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-3452	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-3455	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2022-3477	1	BCK	AM 27 Blood THC Quant by LC-QQQ	

SC

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

Extraction Date: 11/17/2022
Plate lot#: IDP-108-3-220802
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: Lampire 22B52015-2
Column: UCT Selectra DA 100 x 2.1mm 3um
LCMS-QQQ ID: 069901

Analyst: Sarah Collins
Retest Date: 02/02/2023
Mobile phase B: 0.1% Formic acid in Acetonitrile
Blank Urine Lot: POC021022

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 1.5mL urine to blank plate, add 250µl 1N KOH. Shake and incubate at 40 degrees for 15 minutes. Using a calibrated pipette, add **1000µl blood and urine (if applicable) (calibrated pipette)** into the appropriate wells of analytical (standards) plate. **Pipette ID:** 3382167
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 4. Pipette **500µL 0.1% formic acid in water blood sample, 500 µL saturated phosphate buffer in urine** in wells of analytical plate.
- 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 6. Transfer **700-800µL of blood+acid or urine+acid** mixture to corresponding wells of SLE+ plate. Amount transferred: 800 uL
- 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.~~ 2/10/23 SC
- 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: Curve Range: THC-COOH 10-250; calibrator 1 dropped due to ratio
Did not evaluate THC-OH due to interfering peak

SC

	1	2	3	4	5	6
A	IS + Cal. 1	IS + QC_1 urine end	p2022-3455-1			IS + QC_1 blood start
B	IS + Cal. 2	negative blood	p2022-3477-1			IS + Cal. 7
C	IS + Cal. 3	p2022-3248-1	negative urine			IS + Cal. 6
D	IS + Cal. 4	p2022-3314-2	p2022-3188-1			IS + Cal. 5
E	IS + Cal. 5	p2022-3364-1	p2022-3442-1			IS + Cal. 4
F	IS + Cal. 6	p2022-3422-1				IS + Cal. 3
G	IS + Cal. 7	p2022-3442-1*				IS + Cal. 2
H	IS + QC_1 blood start	p2022-3452-1			IS + QC_1 blood end	IS + Cal. 1

All wells to contain 100 µl of residual DMSO

*Sample moved during analytical step 6 due to blood clot

SC

**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 ToxBox 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.

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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



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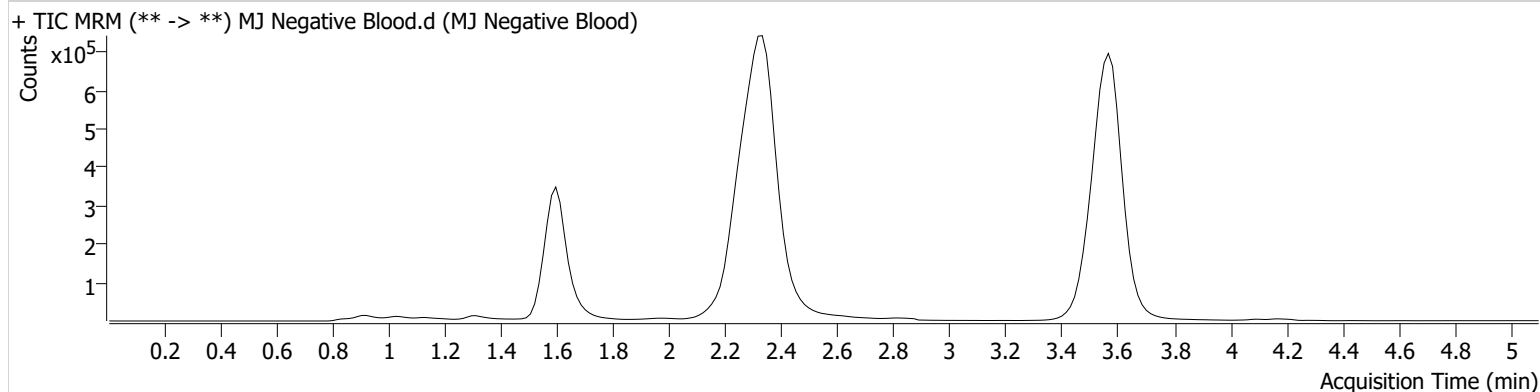


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	MJ Negative Blood.d
Type	Sample	Sample	MJ Negative Blood
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-B2	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 2:46:26 PM		
Sample Info.			

Sample Chromatogram



SC

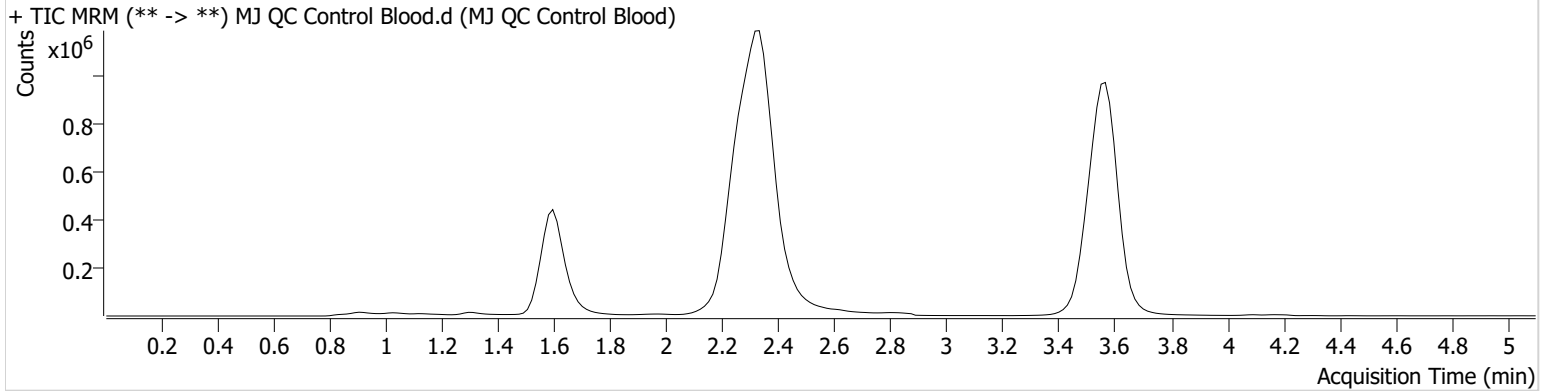


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	MJ QC Control Blood.d
Type	QC	Sample	MJ QC Control Blood
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-H1	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 2:31:12 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.603	162137	∞	9.5	363.66	1627498	5.0128 ng/ml
THC-COOH	1.625	212355	∞	43.2	406.50	470173	16.7245 ng/ml
THC	3.570	312319	32973.85	25.2	∞	6860505	4.9466 ng/ml

SC

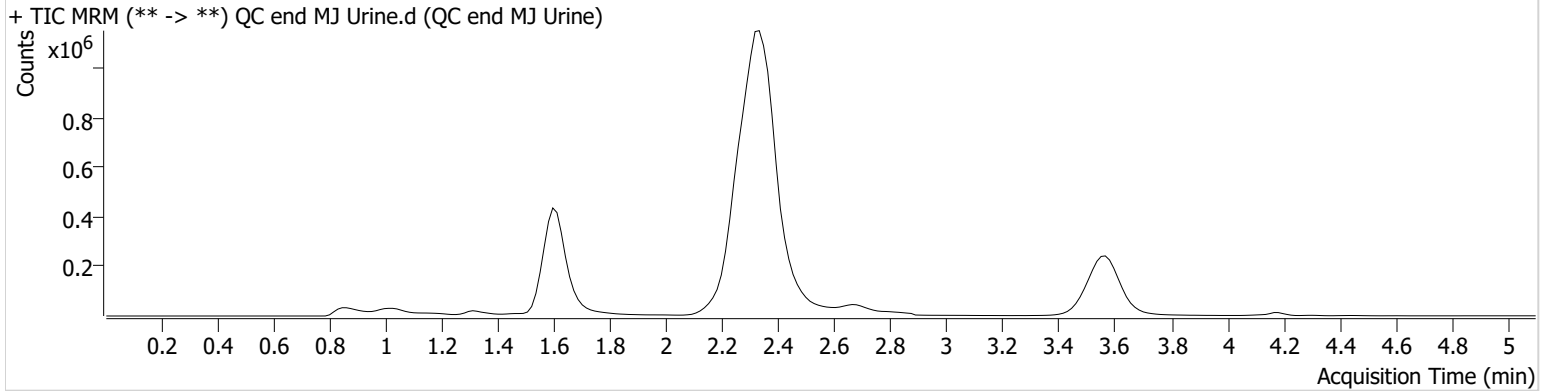


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	QC end MJ Urine.d
Type	QC	Sample	QC end MJ Urine
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-A2	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 5:33:56 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.603	110232	∞	13.4 High	∞	1744064	2.7734 ng/ml Low
THC-COOH	1.640	167868	∞	42.8	226.20	383856	16.1284 ng/ml
THC	3.586	85758	727.40	26.8	78.48	1902112	4.9010 ng/ml

SC

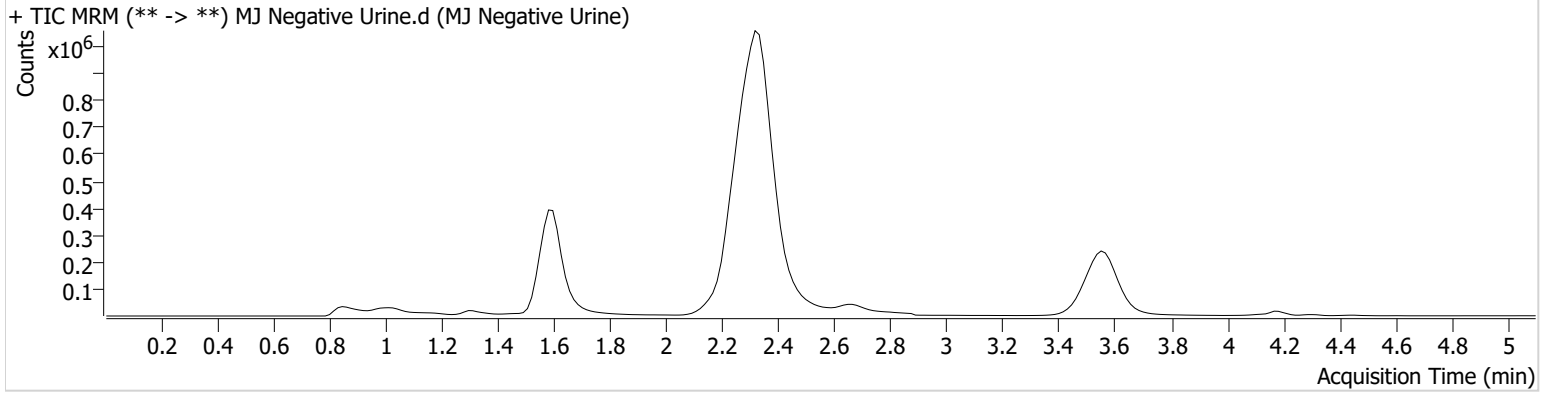


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	MJ Negative Urine.d
Type	Sample	Sample	MJ Negative Urine
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-C3	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 5:03:30 PM		
Sample Info.			

Sample Chromatogram

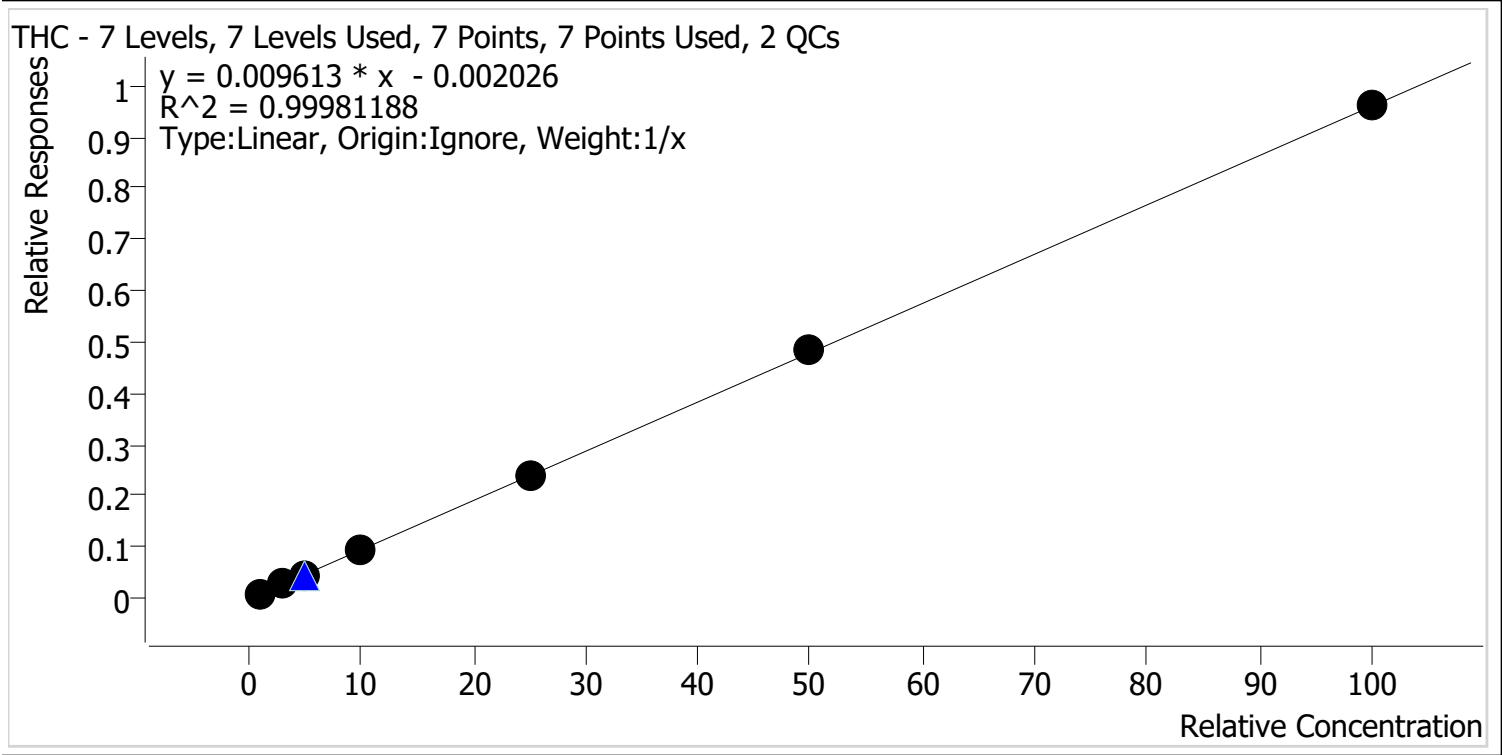


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AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update 11/18/2022 7:34 AM
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	109.6
Cal 2 MJ	2	✓	3.0	2.9	96.1
Cal 3 MJ	3	✓	5.0	4.8	96.1
Cal 4 MJ	4	✓	10.0	9.8	97.6
Cal 5 MJ	5	✓	25.0	24.9	99.7
Cal 6 MJ	6	✓	50.0	50.4	100.9
Cal 7 MJ	7	✓	100.0	100.1	100.1

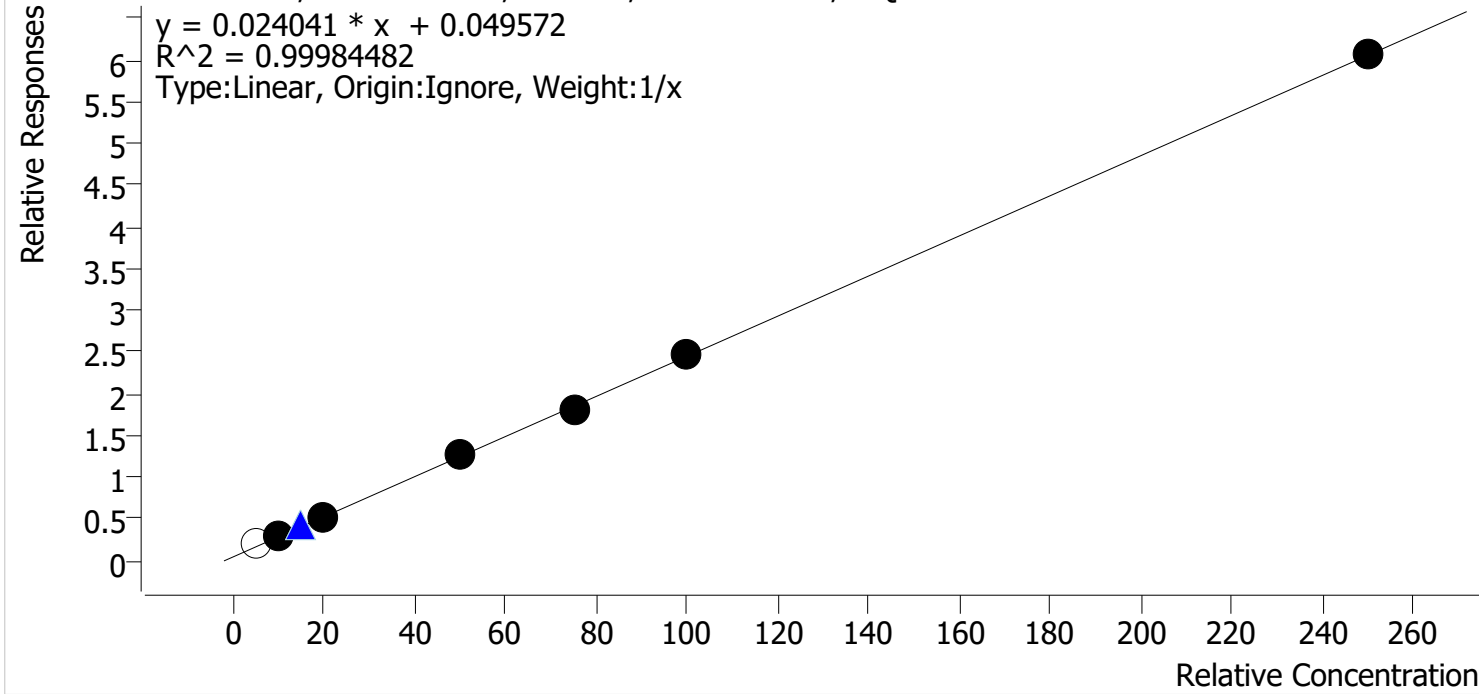
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AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update 11/18/2022 7:34 AM
Analyst Name ISP\Datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9

THC-COOH - 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	5.0	7.0	140.6
Cal 2 MJ	2	✓	10.0	10.0	99.6
Cal 3 MJ	3	✓	20.0	20.0	100.2
Cal 4 MJ	4	✓	50.0	50.9	101.7
Cal 5 MJ	5	✓	75.0	73.4	97.9
Cal 6 MJ	6	✓	100.0	100.4	100.4
Cal 7 MJ	7	✓	250.0	250.3	100.1

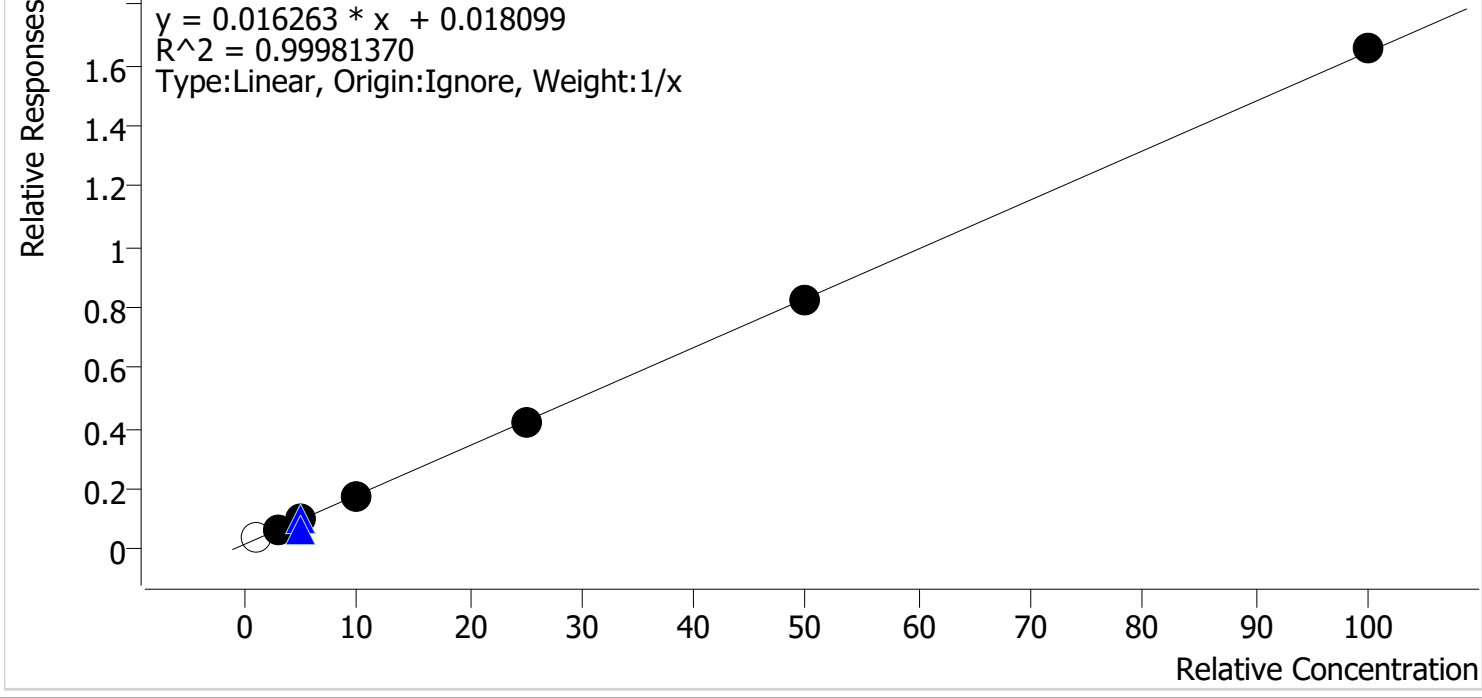
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AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update 11/18/2022 7:34 AM
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	1.5	152.3
Cal 2 MJ	2	✓	3.0	3.1	103.2
Cal 3 MJ	3	✓	5.0	5.0	100.7
Cal 4 MJ	4	✓	10.0	9.8	97.8
Cal 5 MJ	5	✓	25.0	24.4	97.7
Cal 6 MJ	6	✓	50.0	50.0	99.9
Cal 7 MJ	7	✓	100.0	100.7	100.7

SC

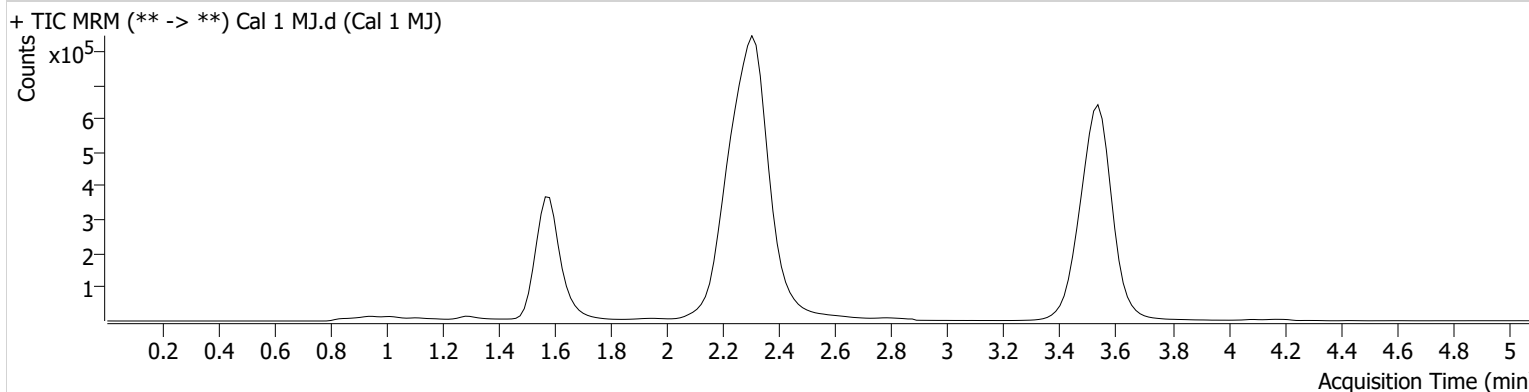


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	Cal 1 MJ.d
Type	Cal	Sample	Cal 1 MJ
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-A1	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 1:30:10 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.648	68668	∞	7.0 Low	45.70	1601922	1.5229 ng/ml Low
THC-COOH	1.610	99588	∞	28.6 Low	320.63	455561	7.0309 ng/ml
THC	3.540	43119	129.23	27.9	20.79	5069910	1.0955 ng/ml

SC

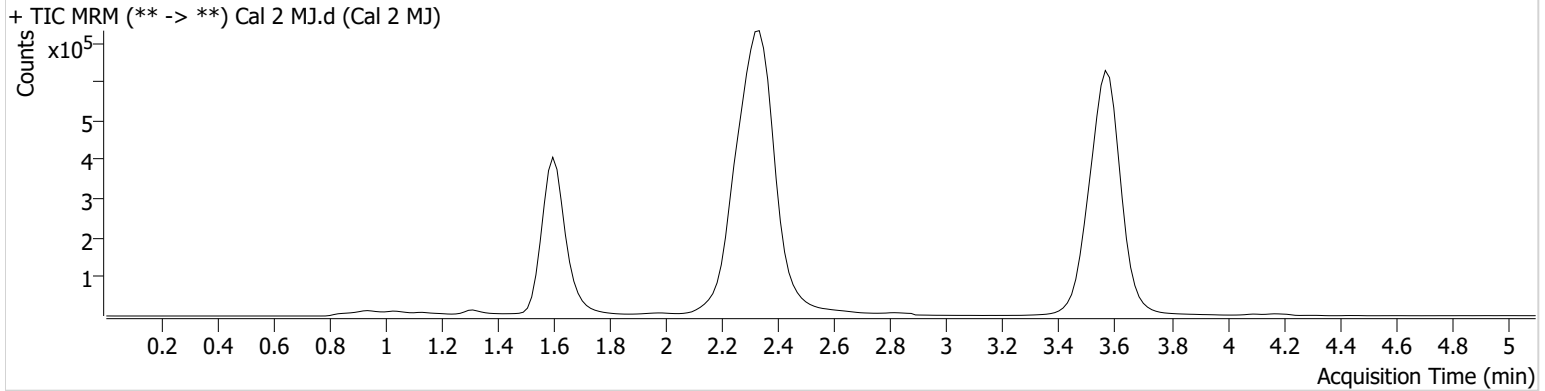


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	Cal 2 MJ.d
Type	Cal	Sample	Cal 2 MJ
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-B1	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 1:37:56 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.618	108783	∞	8.8	∞	1589461	3.0953 ng/ml
THC-COOH	1.640	128456	∞	44.1	627.48	444319	9.9634 ng/ml
THC	3.586	122068	752.76	26.8	44.71	4752907	2.8825 ng/ml

SC

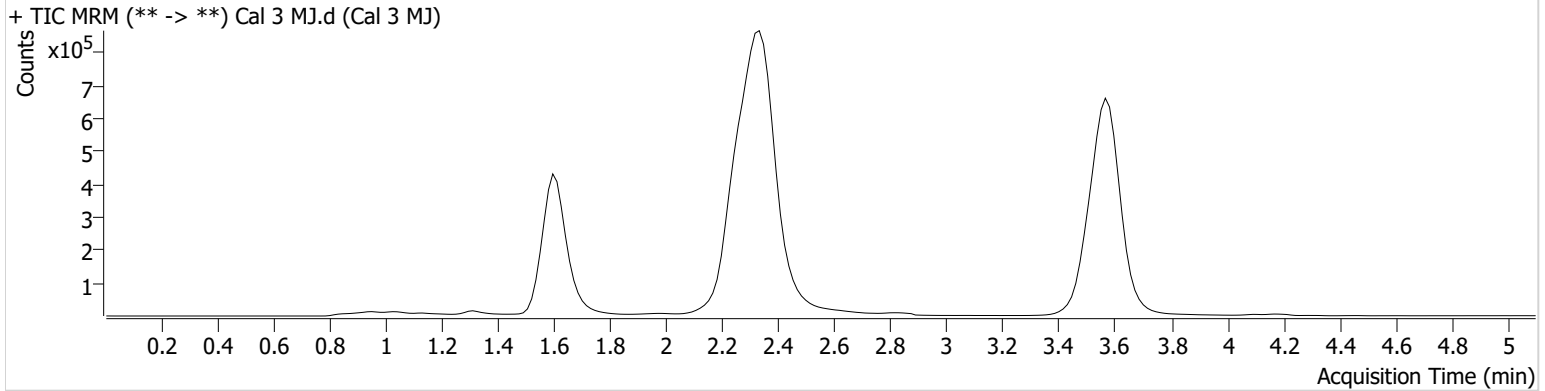


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	Cal 3 MJ.d
Type	Cal	Sample	Cal 3 MJ
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-C1	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 1:45:32 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.603	156088	∞	9.8	252.13	1561168	5.0348 ng/ml
THC-COOH	1.640	235174	∞	48.6	995.51	442546	20.0421 ng/ml
THC	3.586	214479	3072.48	25.9	76.06	4856142	4.8054 ng/ml

SC

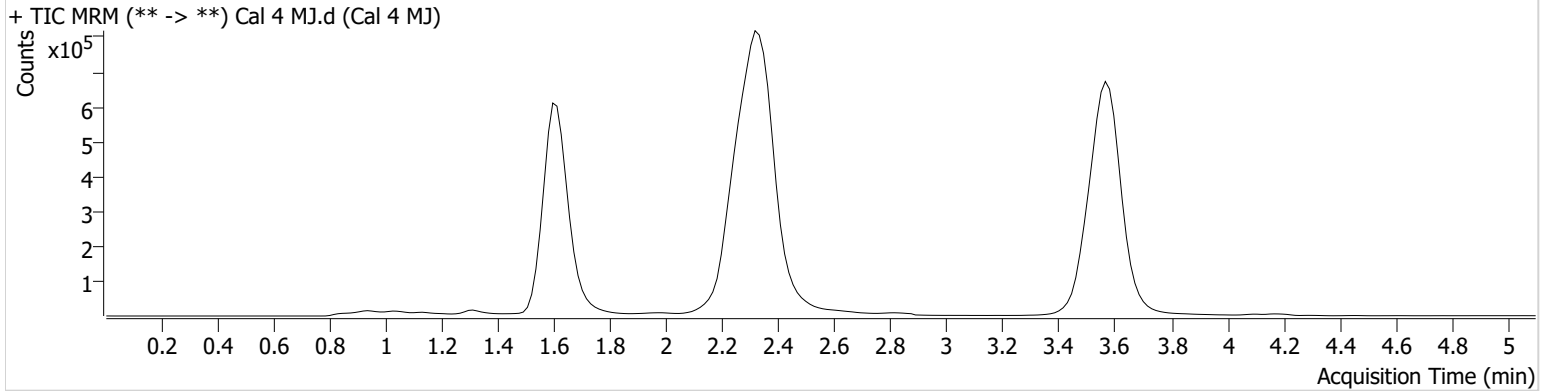


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	Cal 4 MJ.d
Type	Cal	Sample	Cal 4 MJ
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-D1	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 1:53:09 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.603	324428	∞	10.8	∞	1831219	9.7806 ng/ml
THC-COOH	1.625	629658	∞	53.0	∞	494896	50.8594 ng/ml
THC	3.586	451477	2689.97	25.2	237.82	4918709	9.7593 ng/ml

SC

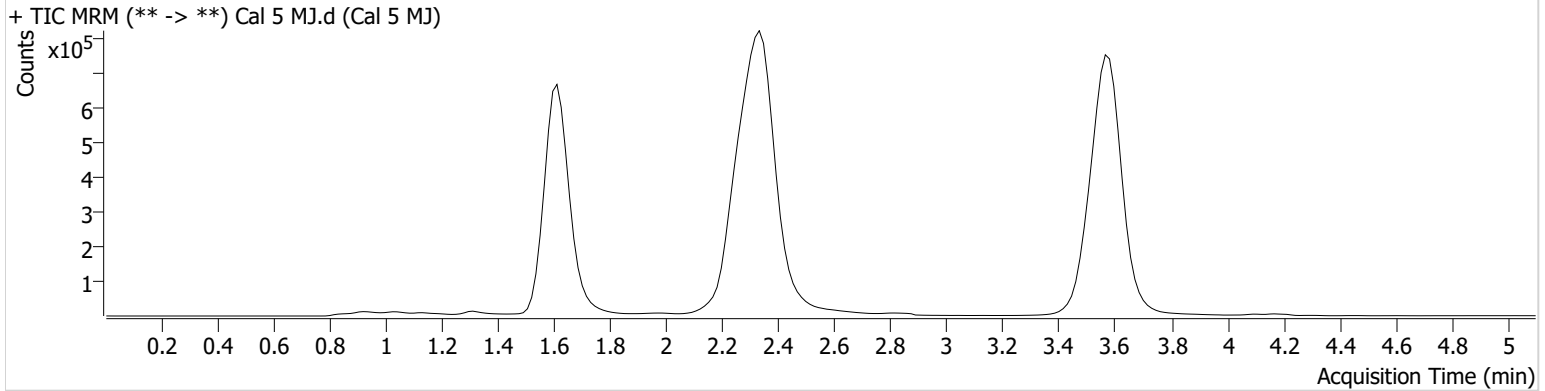


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	Cal 5 MJ.d
Type	Cal	Sample	Cal 5 MJ
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-E1	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 2:00:45 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.603	640044	∞	11.6	∞	1541299	24.4207 ng/ml
THC-COOH	1.625	795915	∞	54.9	3120.15	438553	73.4270 ng/ml
THC	3.586	1094852	∞	24.9	431.84	4608361	24.9258 ng/ml

SC



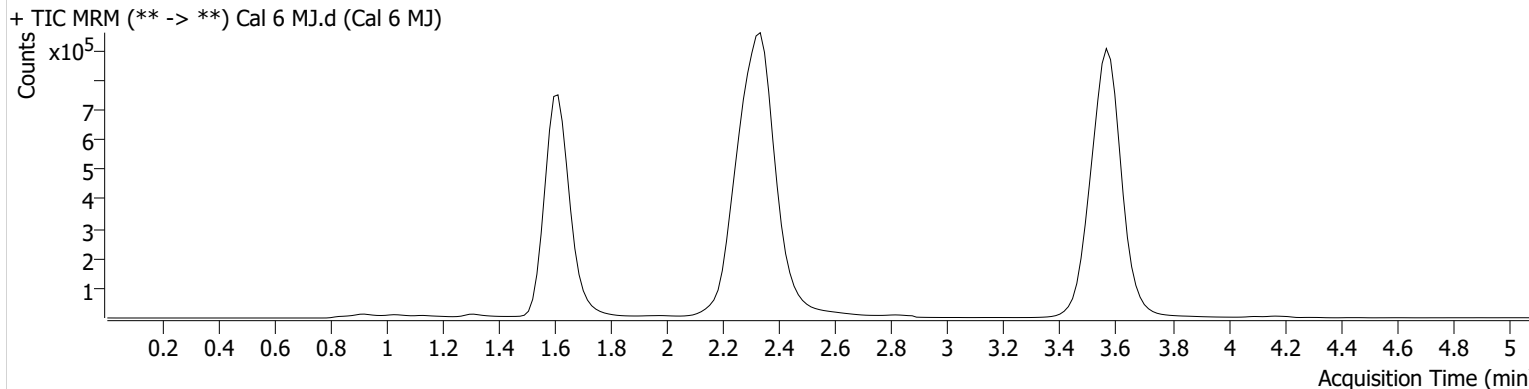
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	Cal 6 MJ.d
Type	Cal	Sample	Cal 6 MJ
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-F1	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 2:08:21 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.603	1117897	∞	11.8	2512.20	1345633	49.9687 ng/ml
THC-COOH	1.625	950487	2252.06	54.2	6225.35	385788	100.4176 ng/ml
THC	3.586	2073817	38391.06	25.1	2166.49	4296043	50.4282 ng/ml

SC

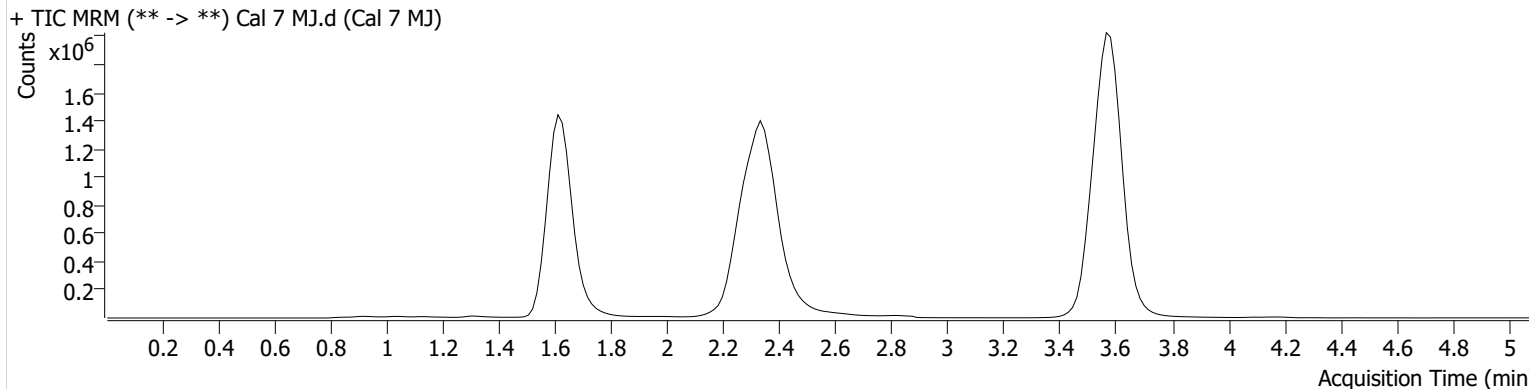


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\111722 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 11/18/2022 7:34:21 AM

Instrument	Falco (069901)	Data File	Cal 7 MJ.d
Type	Cal	Sample	Cal 7 MJ
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-G1	Comment	
Injection Volume	10		
Acq. Date-Time	11/17/2022 2:15:57 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.603	2497384	∞	11.9	5346.63	1508247	100.6998 ng/ml
THC-COOH	1.640	2435939	∞	55.7	∞	401512	250.2905 ng/ml
THC	3.586	6472737	∞	25.3	11655.2 6	6740741	100.1033 ng/ml