

Worklist: 5000

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
M2021-1921	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-1923	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-1975	3	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-2086	3	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-2089	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1229	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1411	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1426	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1442	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1443	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1446	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1458	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1508	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1510	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1533	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1547	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1566	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1581	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1587	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-1588	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: 05/25/21

Analyst: Sarah Collins

Plate lot#: IDP-108-2-210412

Plate Expiration: 10/12/2021

Mobile phase A: 0.1% Formic Acid in LCMS Water

Mobile phase B: 0.1% Formic acid in Acetonitrile

Blank Blood Lot: Lampire 20L20724

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. 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.
- 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 limited: THC 3-100, THC-OH 3-100

80

	1	2	3	4	5	6
A	IS + Cal. 1	negative blood	p2021-1426-2	p2021-1547-1		
B	IS + Cal. 2	m2021-1921-2	p2021-1442-1	p2021-1566-1		
C	IS + Cal. 3	m2021-1923-2*	p2021-1443-1	p2021-1581-1		
D	IS + Cal. 4	m2021-1975-3	p2021-1446-1	p2021-1587-1		
E	IS + Cal. 5	m2021-2086-3	p2021-1458-1	p2021-1588-1		
F	IS + Cal. 6	m2021-2089-2	p2021-1508-1	m2021-1923-2		
G	IS + Cal. 7	p2021-1229-2	p2021-1510-1			
H	IS + QC_1	p2021-1411-1	p2021-1533-1			

All wells to contain 100 µl of residual DMSO

SC

**Idaho State Police
Forensic Services
Toxicology Discipline**

Request for Departure from an Analytical Method

Date of Request

02/24/2021

Forensic Scientist

Anne Nord

Analytical Method

Toxicology AM #27: Quantitative Analysis of THC and Metabolites in Blood and Urine by LCMS-QQQ

Request

The method currently reads:

4.3.2.5 If any points are dropped from the approved quantitative range of the curve, the compound will be reported qualitatively. For calibrators and controls 10 ng and below, the accuracy must be within 30%, for calibrators and controls greater than 10 ng/mL the accuracy must be within 20%. If a control falls outside the accuracy range, at the analyst's discretion, the compound may be reported qualitatively.

I would like to add in the following exception:

If the 1ng/ml point is dropped for THC. If the 1 ng/ml point is dropped the quantitative range will be 3-50 ng/ml.

Discipline Leader Review

Departure approved

Comments: This deviation is approved and will remain in effect until it is changed in the actual method.

Departure Not Approved

Comments:



Celena Shrum

Toxicology Discipline Lead

Date: 02/24/2021

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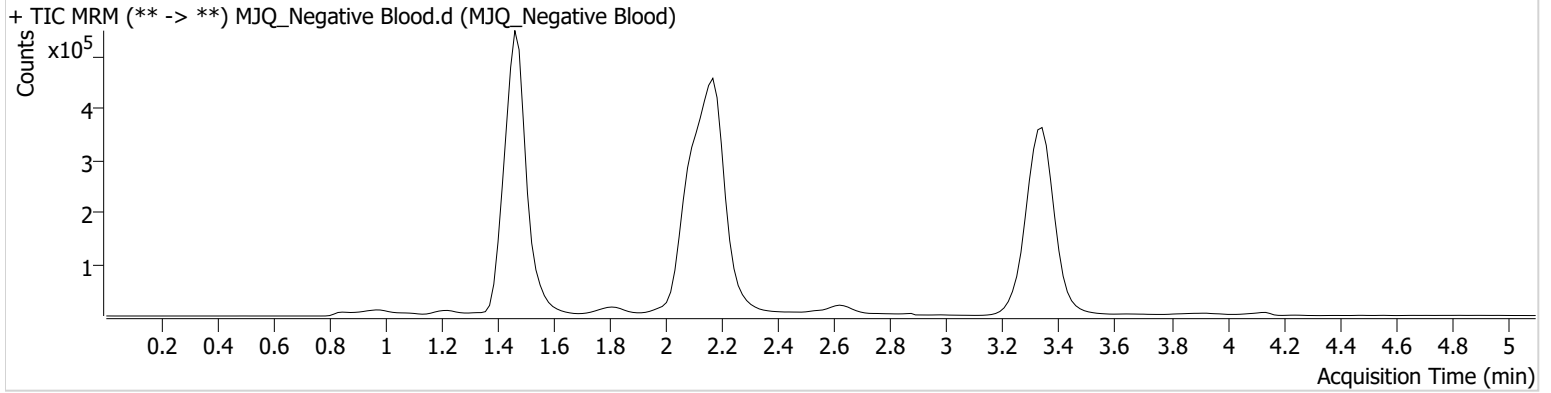


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_Negative Blood.d
Type	Sample	Sample	MJQ_Negative Blood
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-A2	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 11:40:24 AM		
Sample Info.			

Sample Chromatogram



SC

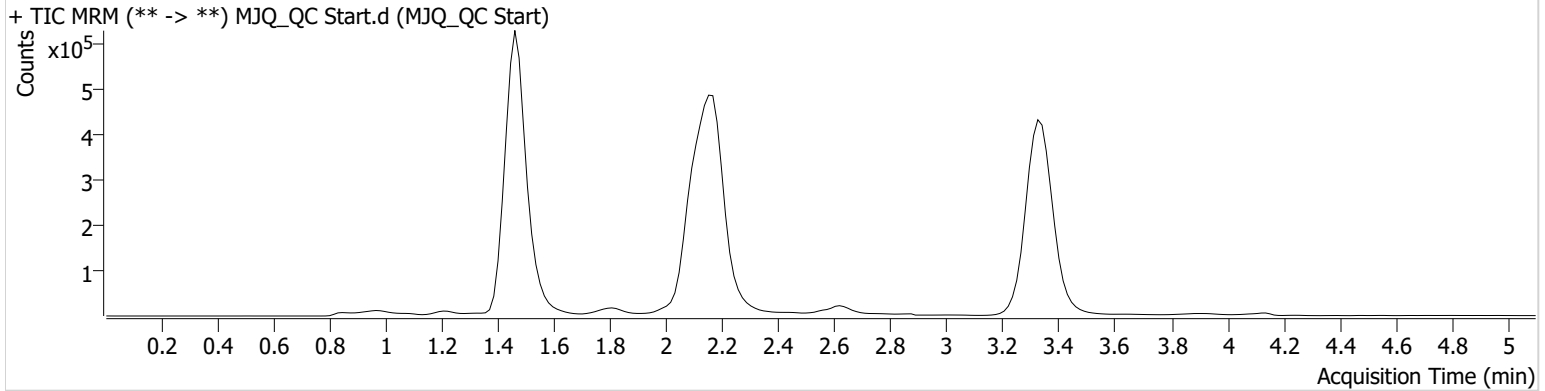


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_QC Start.d
Type	Sample	Sample	MJQ_QC Start
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-H1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 11:55:37 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.483	282536	∞	7.5	∞	2220375	4.8329 ng/ml
THC-COOH	1.504	175489	394.33	52.7	∞	473907	14.6971 ng/ml
THC	3.345	123789	1025.86	32.5	42.82	2787666	4.6966 ng/ml

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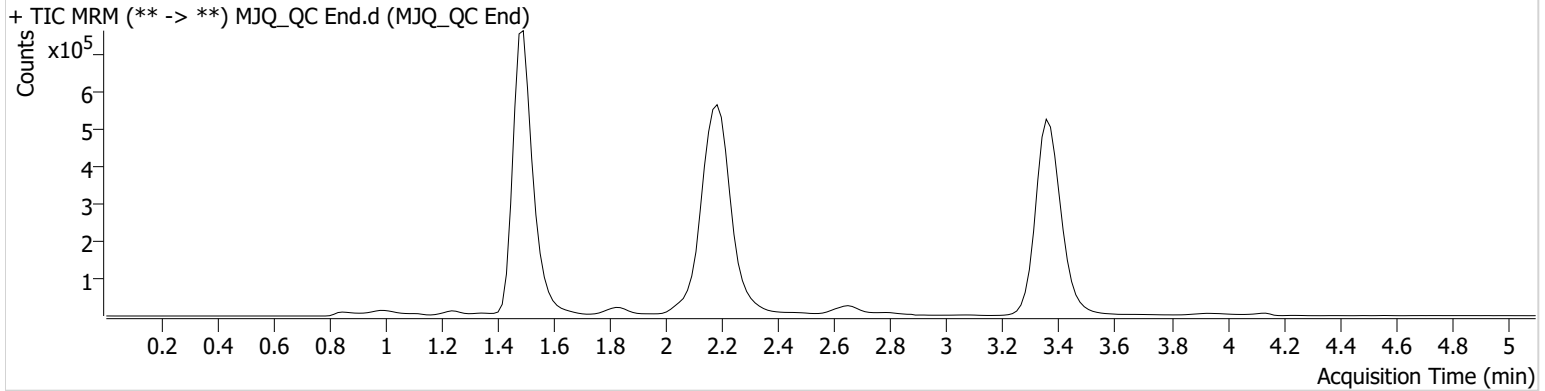


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 12:39:26 PM

Instrument	Falco (069901)	Data File	MJQ_QC End.d
Type	Sample	Sample	MJQ_QC End
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-H1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 5:15:17 PM		

Sample Chromatogram



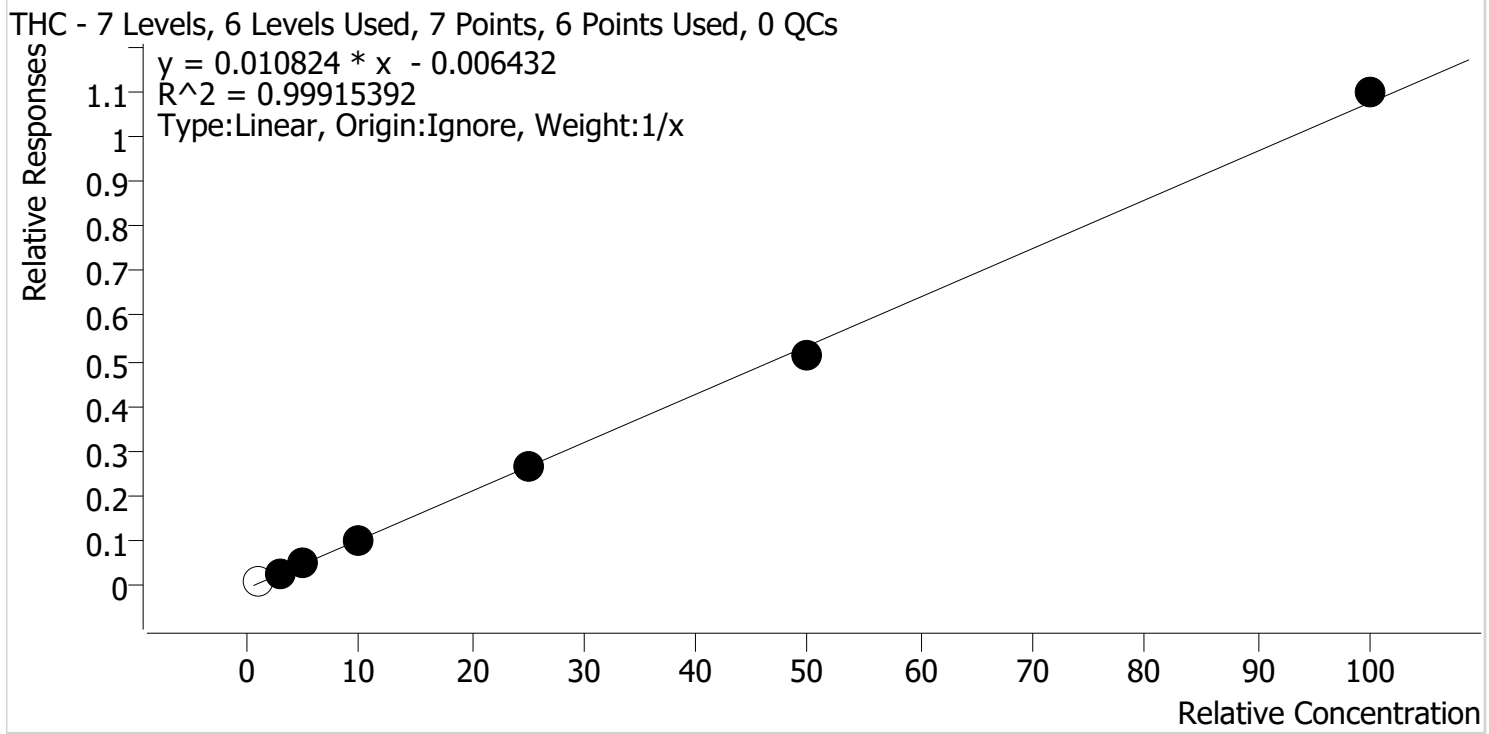
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.498	320479	∞	7.5	167.00	2499635	4.8946 ng/ml
THC-COOH	1.519	207045	∞	51.9	626.28	546292	15.0354 ng/ml
THC	3.375	140866	2109.41	32.5	136.61	3175630	4.6923 ng/ml

SC



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update 5/26/2021 10:01 AM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	x	1.0	1.6	159.4
MJQ_Cal 2	2	✓	3.0	3.1	103.3
MJQ_Cal 3	3	✓	5.0	5.1	102.3
MJQ_Cal 4	4	✓	10.0	9.7	96.6
MJQ_Cal 5	5	✓	25.0	24.8	99.2
MJQ_Cal 6	6	✓	50.0	48.3	96.6
MJQ_Cal 7	7	✓	100.0	102.1	102.1

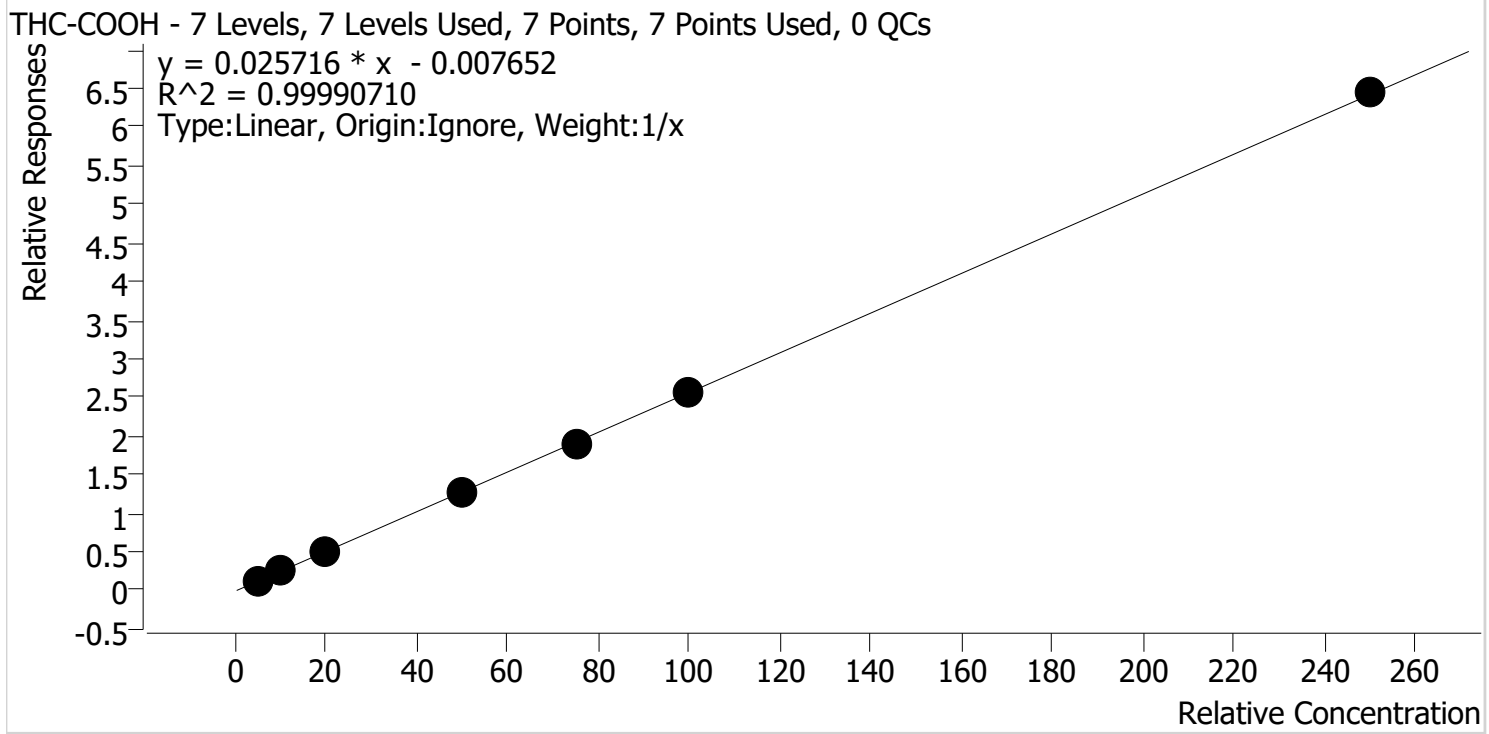
Calibrator 1 dropped due to not meeting ratio requirement

SC



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update 5/26/2021 10:01 AM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✓	5.0	5.2	104.6
MJQ_Cal 2	2	✓	10.0	9.8	97.7
MJQ_Cal 3	3	✓	20.0	19.6	97.9
MJQ_Cal 4	4	✓	50.0	50.3	100.6
MJQ_Cal 5	5	✓	75.0	74.4	99.2
MJQ_Cal 6	6	✓	100.0	99.7	99.7
MJQ_Cal 7	7	✓	250.0	251.0	100.4

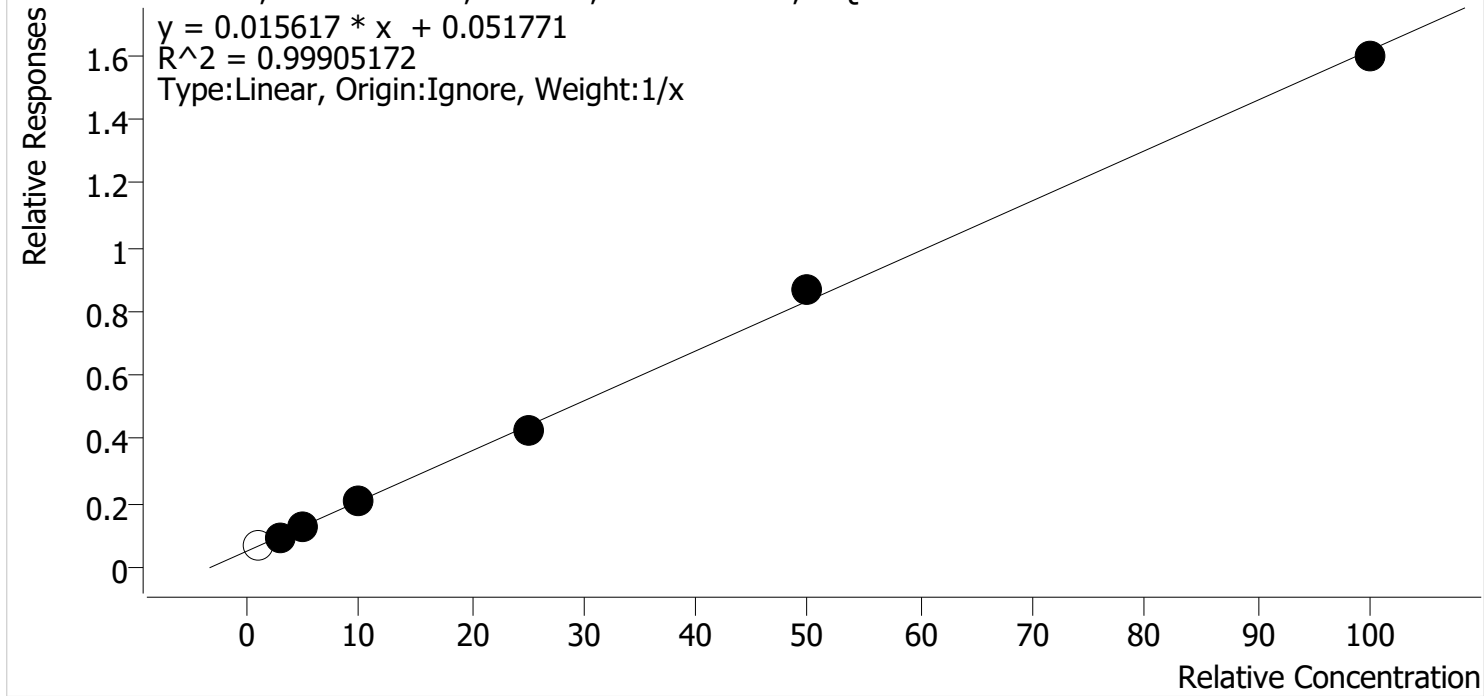
SC



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update 5/26/2021 10:01 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, 0 QCs



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	x	1.0	1.3	129.3
MJQ_Cal 2	2	✓	3.0	3.0	98.7
MJQ_Cal 3	3	✓	5.0	5.1	101.9
MJQ_Cal 4	4	✓	10.0	10.0	99.9
MJQ_Cal 5	5	✓	25.0	24.2	96.6
MJQ_Cal 6	6	✓	50.0	52.1	104.2
MJQ_Cal 7	7	✓	100.0	98.7	98.7

Calibrator 1 dropped due to not meeting ratio requirement

SC



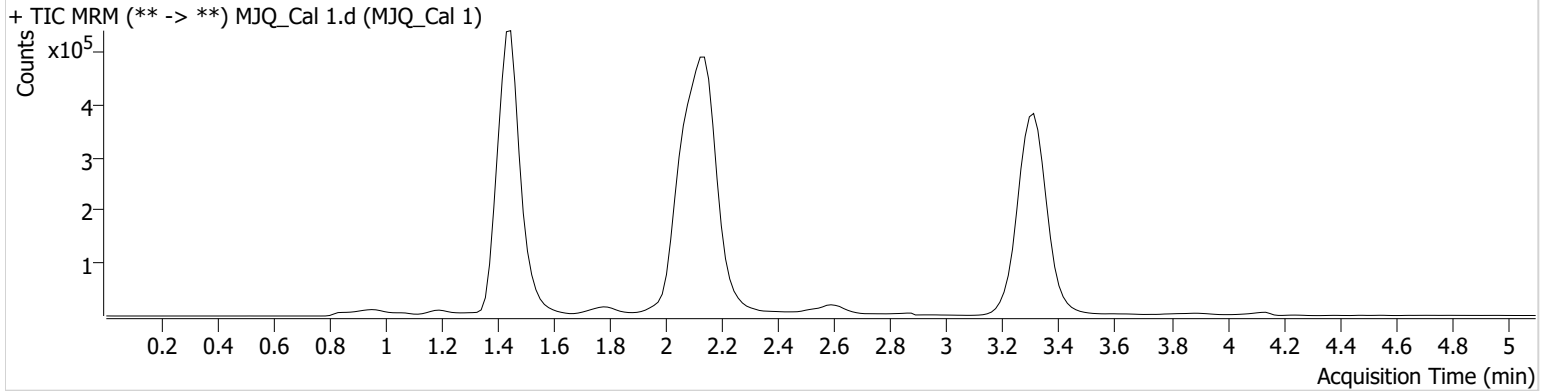
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_Cal 1.d
Type	Cal	Sample	MJQ_Cal 1
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-A1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 10:39:26 AM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.513 High	164995	∞	4.7 Low	∞	2292727	1.2930 ng/ml Low
THC-COOH	1.474	60309	∞	49.1	∞	475588	5.2287 ng/ml
THC	3.315	30182	49.91	33.8	∞	2787860	1.5944 ng/ml

SC

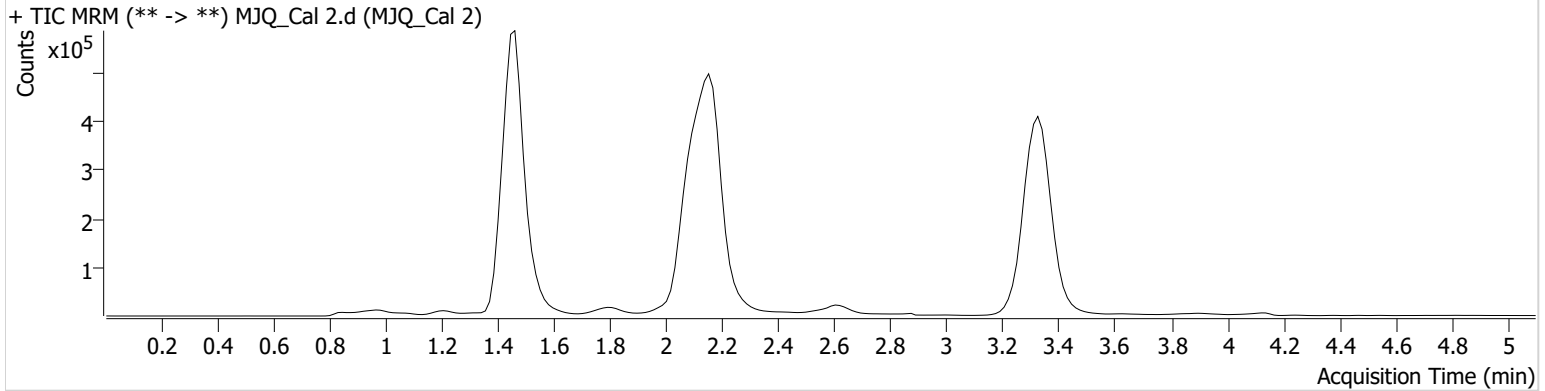


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_Cal 2.d
Type	Cal	Sample	MJQ_Cal 2
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-B1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 10:47:13 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.483	223959	∞	7.7	27.47	2285319	2.9601 ng/ml Low
THC-COOH	1.489	116751	∞	50.9	728.27	479503	9.7657 ng/ml
THC	3.345	75689	372.65	34.2	37.50	2792457	3.0983 ng/ml

SC

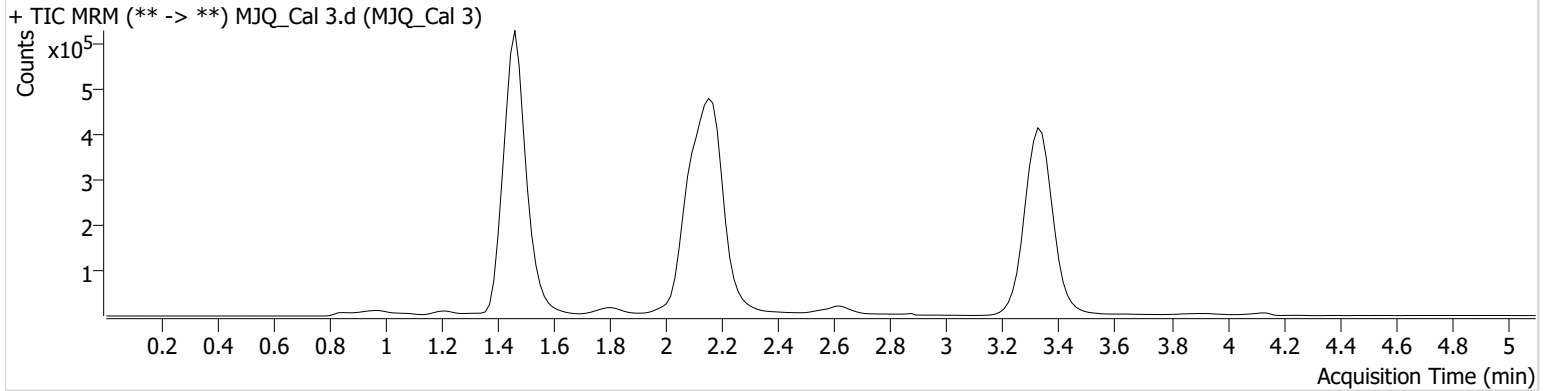


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_Cal 3.d
Type	Cal	Sample	MJQ_Cal 3
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-C1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 10:54:49 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	296744	∞	7.6	390.28	2258881	5.0968 ng/ml
THC-COOH	1.489	234147	190.90	53.1	∞	472396	19.5717 ng/ml
THC	3.345	135603	754.22	28.2	186.24	2770284	5.1164 ng/ml

SC

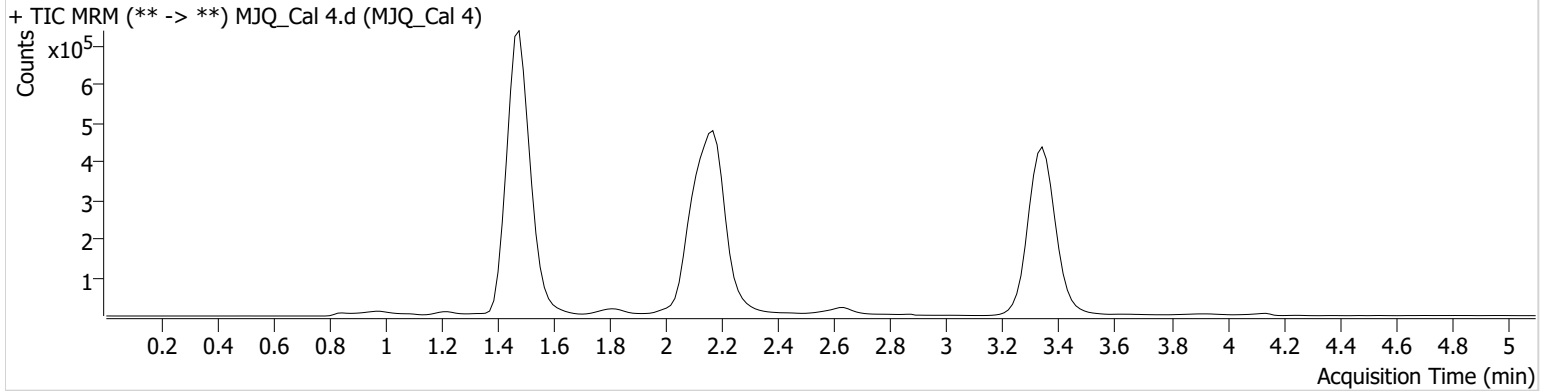


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_Cal 4.d
Type	Cal	Sample	MJQ_Cal 4
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-D1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 11:02:25 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.483	464533	∞	8.9	∞	2236125	9.9872 ng/ml
THC-COOH	1.504	597076	1095.43	52.7	8108.78	464558	50.2762 ng/ml
THC	3.360	264716	2022.78	27.3	∞	2696776	9.6628 ng/ml

SC

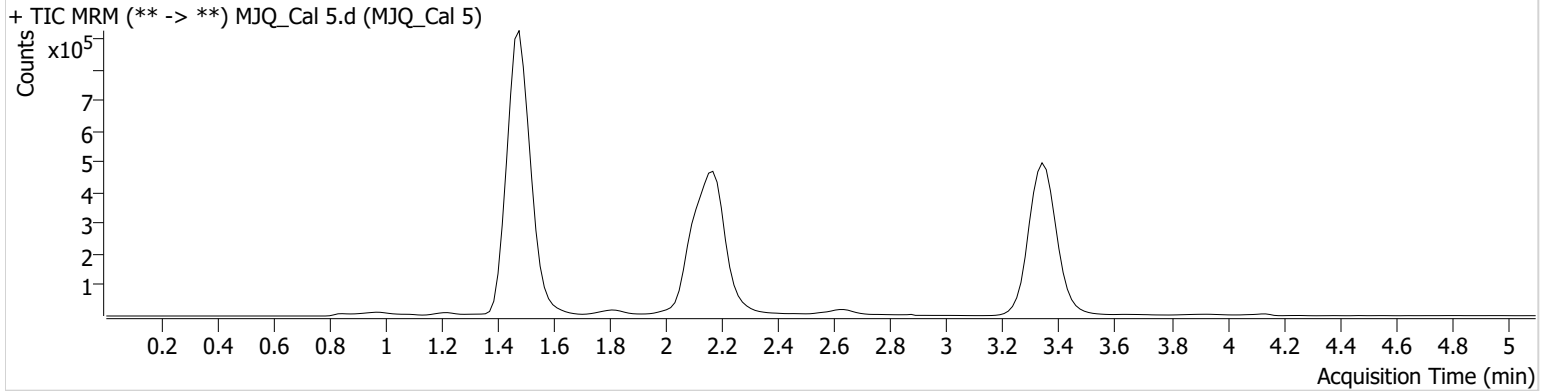


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_Cal 5.d
Type	Cal	Sample	MJQ_Cal 5
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-E1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 11:10:00 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	960485	∞	10.4	646.43	2238566	24.1592 ng/ml
THC-COOH	1.504	872266	972.15	53.6	3556.14	457580	74.4245 ng/ml
THC	3.360	687517	3256.63	24.6	342.37	2625360	24.7877 ng/ml

SC

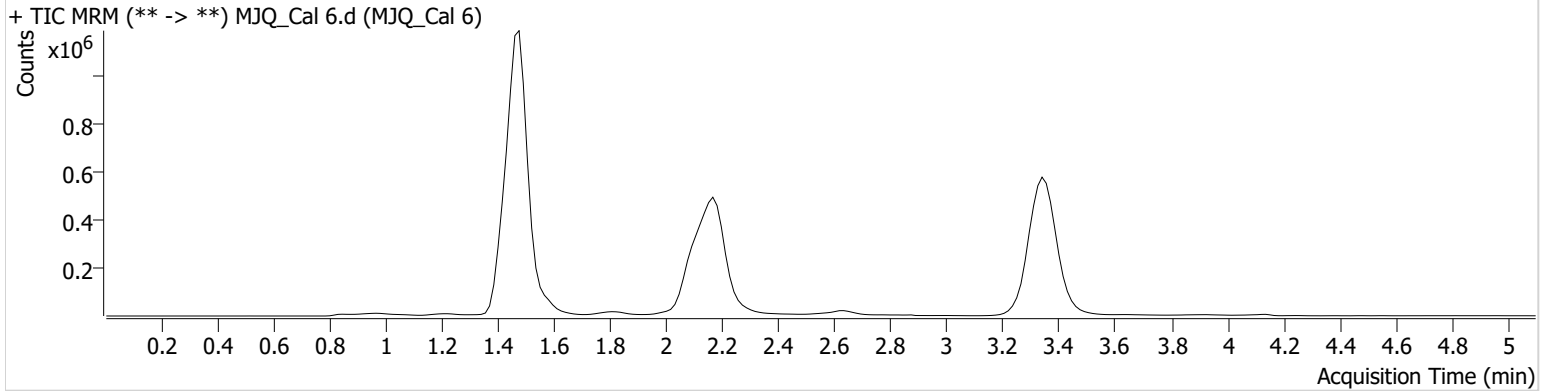


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_Cal 6.d
Type	Cal	Sample	MJQ_Cal 6
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-F1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 11:17:36 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	1907060	∞	10.4	∞	2204109	52.0884 ng/ml
THC-COOH	1.489	1077707	∞	53.1	6261.72	421505	99.7218 ng/ml
THC	3.360	1263539	2408.65	26.1	∞	2447762	48.2837 ng/ml

SC

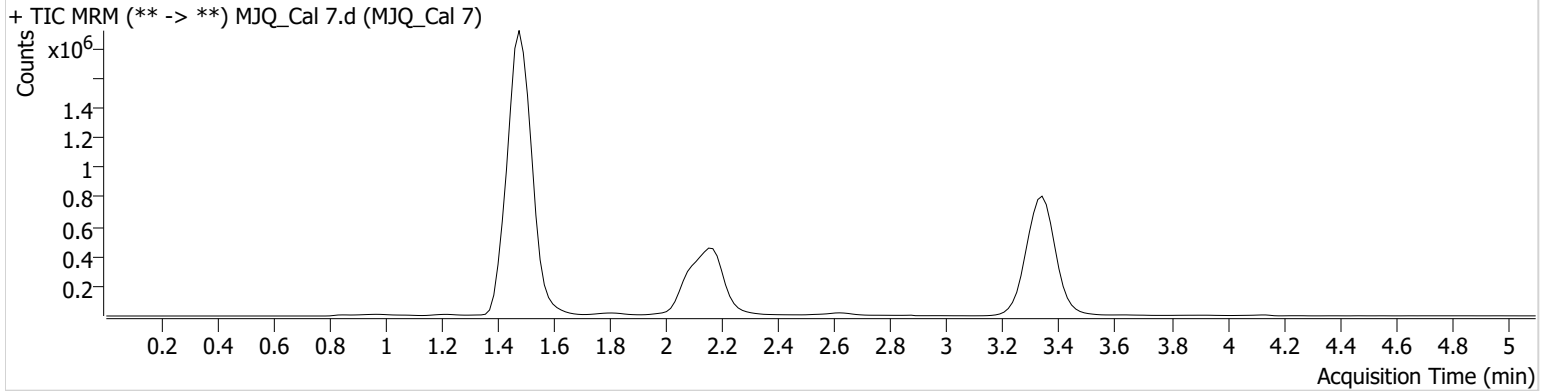


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\052521 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 5/26/2021 10:01:56 AM

Instrument	Falco (069901)	Data File	MJQ_Cal 7.d
Type	Cal	Sample	MJQ_Cal 7
Acq. Method	AM 27 THCQ.m	Operator	Sarah Collins
Sample Position	P1-G1	Comment	
Injection Volume	10		
Acq. Date-Time	5/25/2021 11:25:11 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	3517175	∞	10.7	∞	2207492	98.7084 ng/ml
THC-COOH	1.504	2737926	∞	54.0	∞	424657	251.0115 ng/ml
THC	3.345	2719988	∞	25.3	1135.71	2476788	102.0512 ng/ml