

REVIEWED

By Celena Shrum at 1:40 pm, Mar 04, 2020

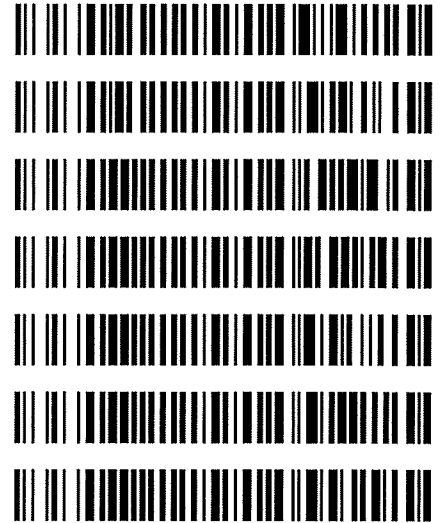
CS

3/3/2020

CS

Worklist: 4044

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>
M2020-0553	3	BCK	AM 27 Blood THC Quant by LC-QQQ
M2020-0641	2	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0062	2	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0203	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0510	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0523	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0524	1	BCK	AM 27 Blood THC Quant by LC-QQQ



15

**Idaho State Police
Forensic Services
Toxicology Discipline**

Request for Departure from an Analytical Method

Date of Request
01/13/2020

Forensic Scientist
Celena Shrum

Analytical Methods
Toxicology AM #25, Toxicology AM #26/27, and AM #28

Deviation

The expiration dates listed for the current batch of PinPoint ToxBx extraction plates are as follows:

- *MDS (batch IDP-107-190725)- Expiration is 1/25/2020
- *THC (batch IDP-108-190716)- Expiration is 1/16/2020
- *MDQ P1 (batch IDP-111-190729)- Expiration is 1/29/2020
- *MDQ P2 (batch IDP-112-190730)- Expiration is 1/30/2020

I am issuing a deviation to allow for the use of the remaining plates of these batches. The controls will be used to evaluate if the plate is working as intended. In addition, at least one external control must be included for each run.

Celena Shrum

Date: 01/13/2020
Celena Shrum
Toxicology Discipline Lead

15

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

Extraction Date: 03/03/2020
Plate lot#: IDP-108-190716

Analyst: Tamara Salazar
Plate Expiration: 01/16/2020—Ok, Deviation in place

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: Hemostat 445283-3
LCMS-QQQ ID: 069901

Column: UCT Selectra DA 100 x 2.1mm 3um

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.
- 3. Create worklist:

Analytic:

- 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
- 2. Pipette **1000µL blood/urine (calibrated pipette) Pipette ID: 16** in wells of analytical (standards) plate.
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 067105*
- 4. Pipette **500µL 0.1% formic acid in water for blood samples, 500µl saturated phosphate buffer for urine samples** in wells of analytical plate.
- 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 6. Transfer **800µL of blood+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: 067104*
- 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: 067103*
- 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.
Worklist path: D:\MassHunter\Data\2020\AM 27-28\AM 27-28 030320 AM 27 28 wklsts 4044 4045 TS
Batch Name: THCQ TS
- 2. Make any necessary integration changes, Curve weighting of Linear 1/x with r² 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 and OH-THC 3ng/mL (quantitative), Carboxy-THC: 10ng/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? Y / N
- 6. Enter QCs into control charting.
- 7. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: *Curves limited: THC: 3-100, THC-COOH: 10-250, THC-OH: 3-100*



Idaho State Police Forensic Services

AM #26 Blood THC and Metabolites Screen by LCMS-QQQ and AM #27 Quantitative Analysis of THC and Metabolites in Blood by LCMS-QQQ

Methanol External Control Solution (Lot: WS011620)

*10 μ L of 1mg/mL THC, 100 μ L of 100 μ g/mL THC-OH, C-THC in 9790 μ L MeOH
Approximate concentration 1 μ g/mL.*

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>	<i>Expiration Date</i>
Methanol (LCMS)	Fisher	193941	
THC	Cerilliant	FE09101501	11/30/2020
C-THC	Cerilliant	FE07171501	09/30/2020
THC-OH	Cerilliant	FE07221601	07/31/2021
Prepared:	01/16/2020		
Prepared By:	Tamara Salazar		
Expires:	09/30/2020		

Blood External Control Solution (Lot: 021320)

*200 μ L of methanol external control solution was added to 9800 μ L of blood.
Approximately 20 ng/mL of each compound.*

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Negative Blood	Hemostat	445283-3
Methanol External Control Solution	-	WS011620
Prepared:	02/13/2020	
Prepared by:	Celena Shrum	
Expires:	09/30/2020	

TS

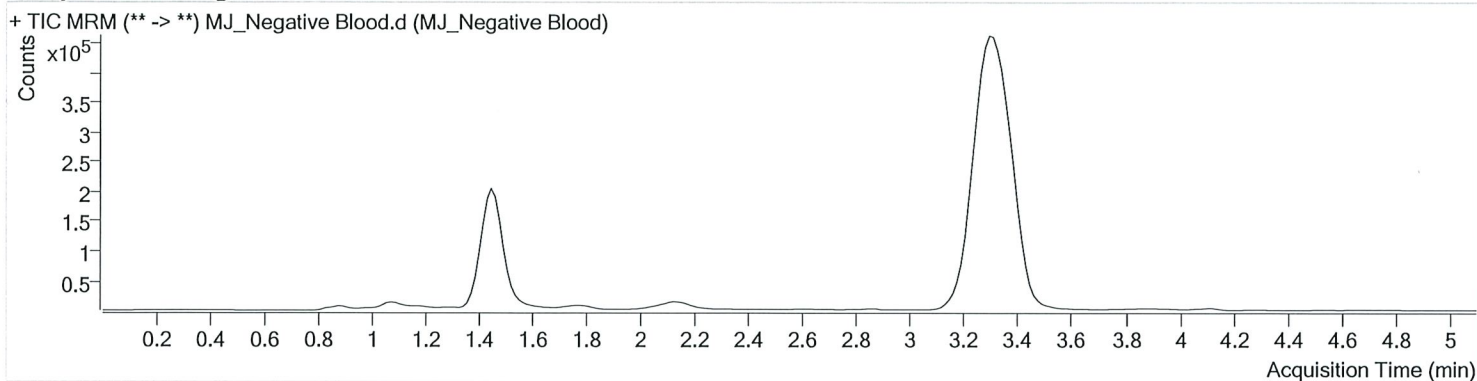


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wklsts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_Negative Blood.d
Type	Sample	Sample	MJ_Negative Blood
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-H5	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 12:33:08 PM		
Sample Info.			

Sample Chromatogram



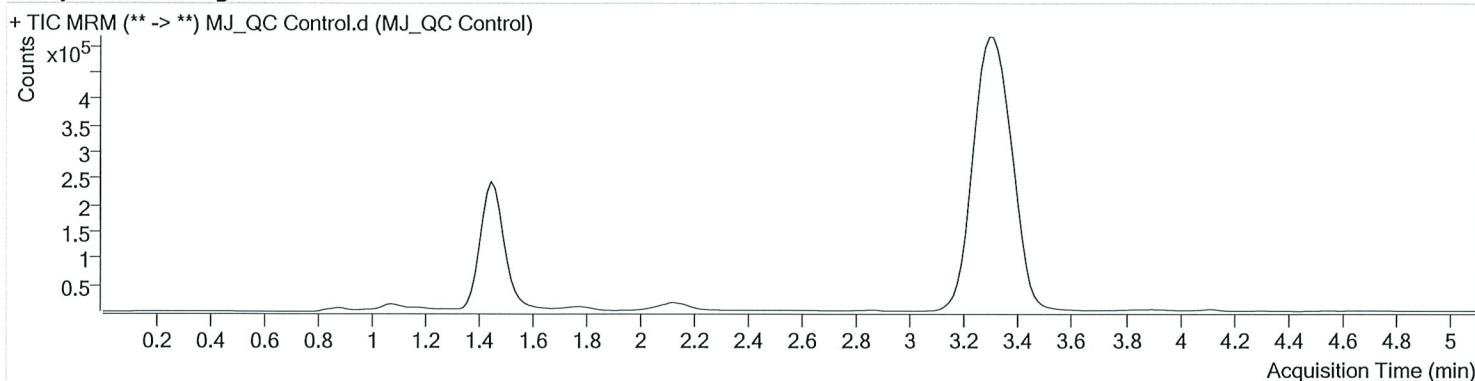


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wk1sts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_QC Control.d
Type	Sample	Sample	MJ_QC Control
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-A6	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 12:17:55 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	104899	∞	9.3	124.29	921044	5.0312 ng/ml
THC-COOH	1.489	130892	∞	44.7	1692.97	277233	16.4319 ng/ml
THC	3.315	158882	890.79	27.4	86.62	4970334	4.3881 ng/ml

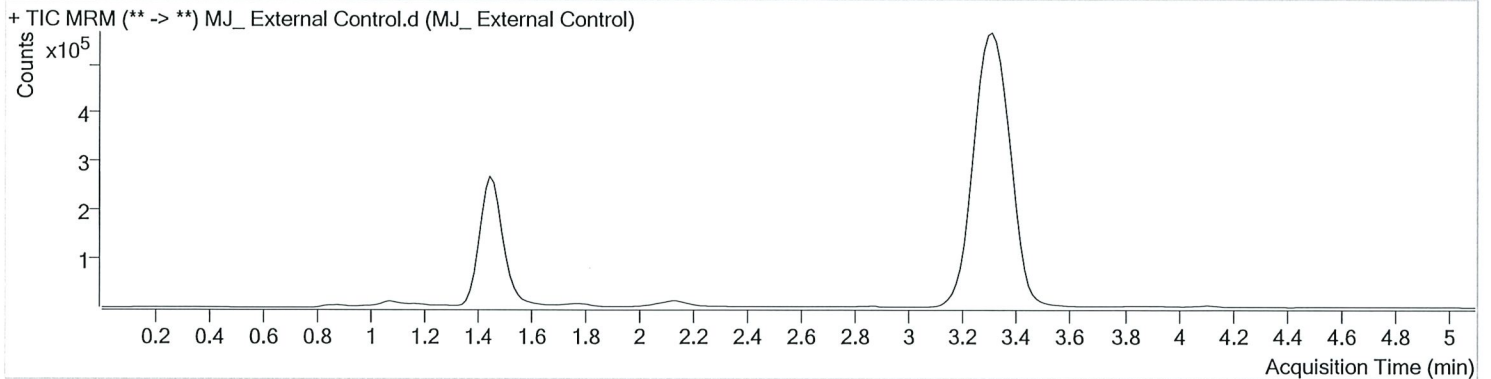
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wklsts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_ External Control.d
Type	Sample	Sample	MJ_ External Control
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-G5	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 12:48:18 PM		
Sample Info.			

Sample Chromatogram

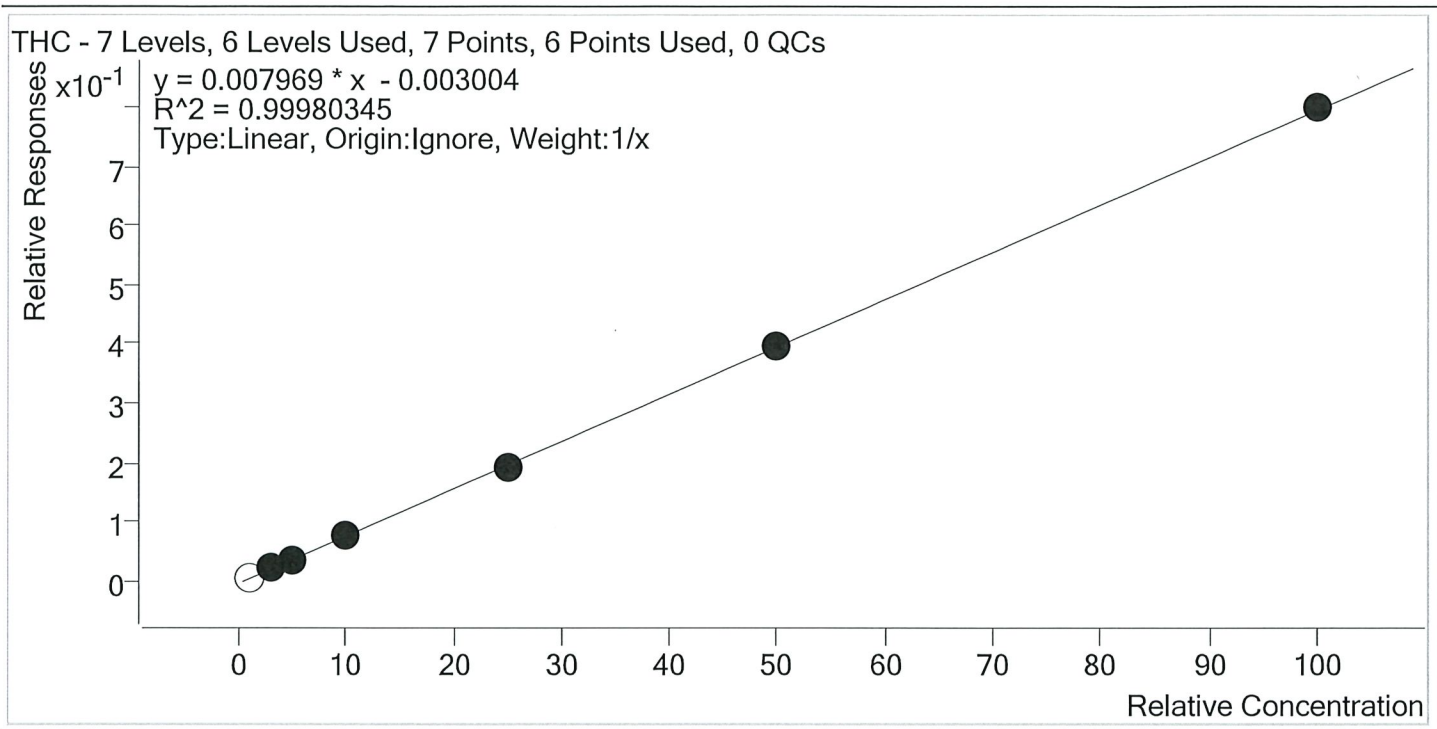


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	242183	∞	12.3	∞	849813	17.6508 ng/ml
THC-COOH	1.489	147810	∞	46.3	195.37	264294	19.8801 ng/ml
THC	3.330	587524	1779.09	26.2	∞	4643158	16.2549 ng/ml



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wklsts 4044 4045 TS\QuantResults\THCQ
 TS.batch.bin
Last Cal. Update 3/3/2020 3:37 PM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-D3

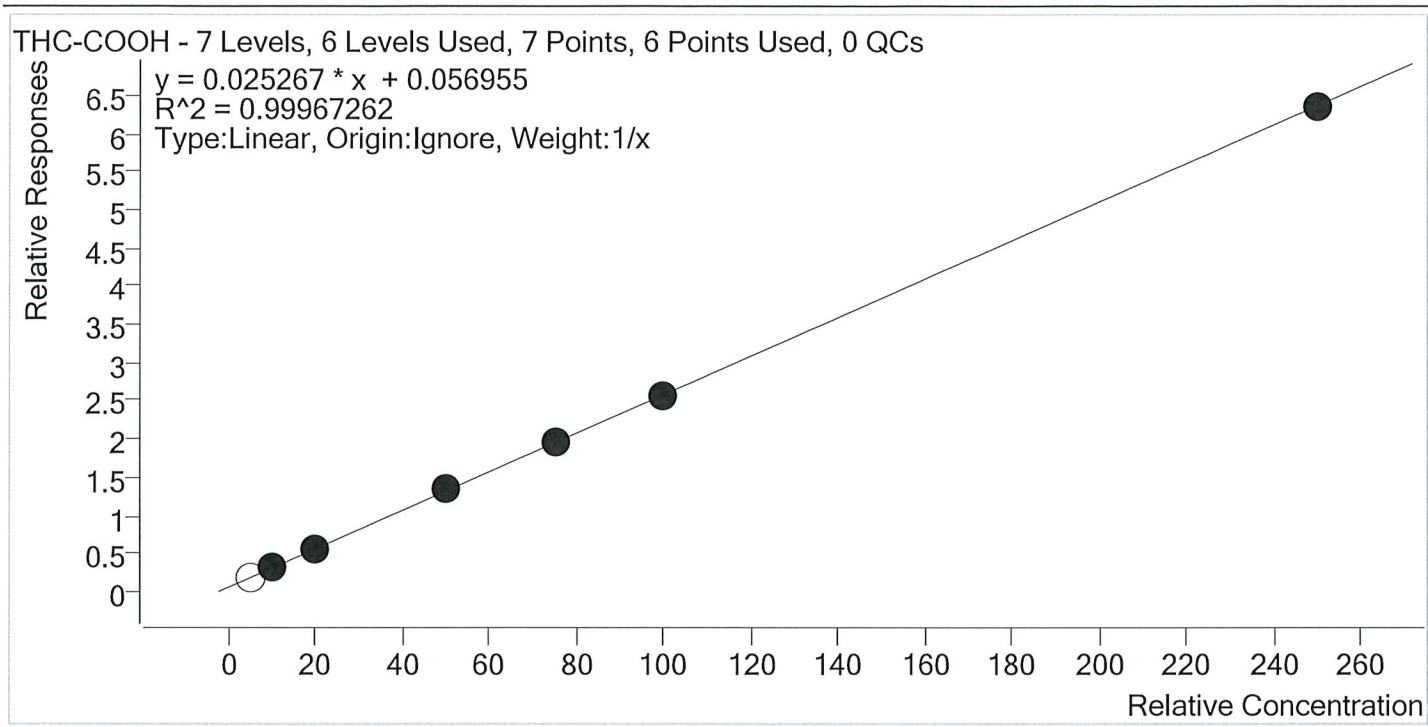


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ Cal 1	1	×	1.0	1.3	128.1
MJ Cal 2	2	✓	3.0	3.1	104.2
MJ Cal 3	3	✓	5.0	4.9	98.2
MJ Cal 4	4	✓	10.0	9.9	99.5
MJ Cal 5	5	✓	25.0	24.3	97.4
MJ Cal 6	6	✓	50.0	50.1	100.2
MJ Cal 7	7	✓	100.0	100.6	100.6



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wk\lsts 4044 4045 TS\QuantResults\THCQ
 TS.batch.bin
Last Cal. Update 3/3/2020 3:37 PM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9



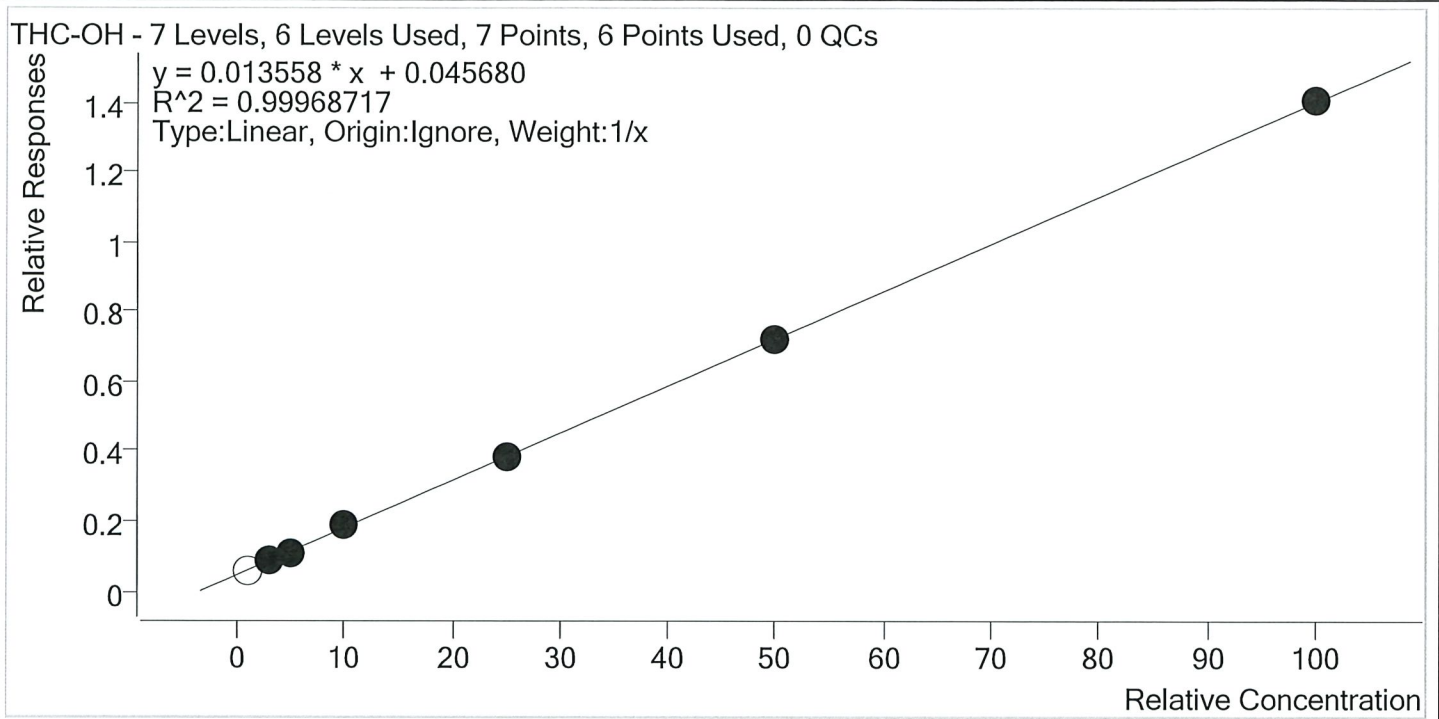
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ Cal 1	1	×	5.0	5.3	105.5
MJ Cal 2	2	✓	10.0	10.1	100.6
MJ Cal 3	3	✓	20.0	19.3	96.3
MJ Cal 4	4	✓	50.0	51.9	103.8
MJ Cal 5	5	✓	75.0	75.0	100.0
MJ Cal 6	6	✓	100.0	99.6	99.6
MJ Cal 7	7	✓	250.0	249.2	99.7

TS



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wk\sts 4044 4045 TS\QuantResults\THCQ
 TS.batch.bin
Last Cal. Update 3/3/2020 3:37 PM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ Cal 1	1	×	1.0	1.0	97.3
MJ Cal 2	2	✓	3.0	3.0	98.9
MJ Cal 3	3	✓	5.0	4.9	97.4
MJ Cal 4	4	✓	10.0	10.6	105.6
MJ Cal 5	5	✓	25.0	24.6	98.5
MJ Cal 6	6	✓	50.0	49.7	99.3
MJ Cal 7	7	✓	100.0	100.3	100.3

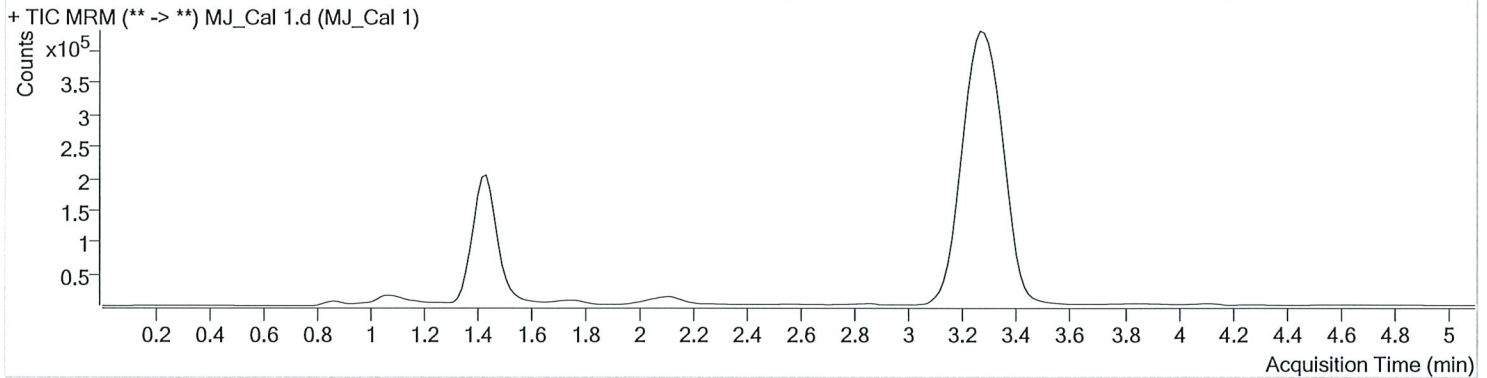
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wklsts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_Cal 1.d
Type	Cal	Sample	MJ_Cal 1
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-B6	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 11:17:06 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.483	53627	1.14 Low	5.2 Low	15.63	910907	0.9731 ng/ml Low
THC-COOH	1.459	50115	∞	36.5 Low	∞	263423	5.2753 ng/ml Low
THC	3.270	32560	87.73	28.1	9.24 Low	4517212	1.2814 ng/ml Low

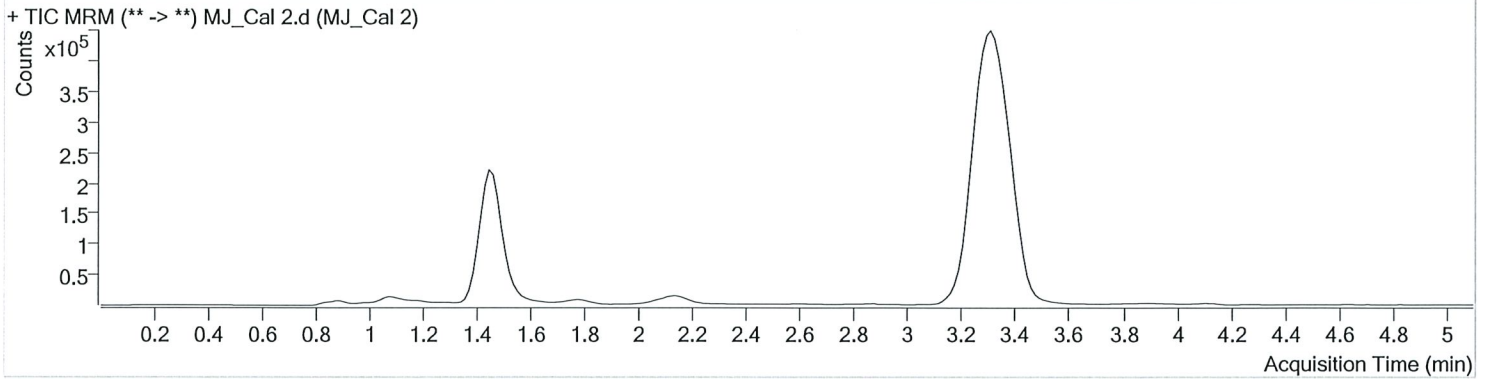
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wklsts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
 Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_Cal 2.d
Type	Cal	Sample	MJ_Cal 2
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-C6	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 11:24:51 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.498	73876	∞	9.4	67.09	860071	2.9662 ng/ml Low
THC-COOH	1.489	77742	788.68	44.8	279.17	249817	10.0622 ng/ml
THC	3.315	92775	285.36	29.6	∞	4235142	3.1258 ng/ml

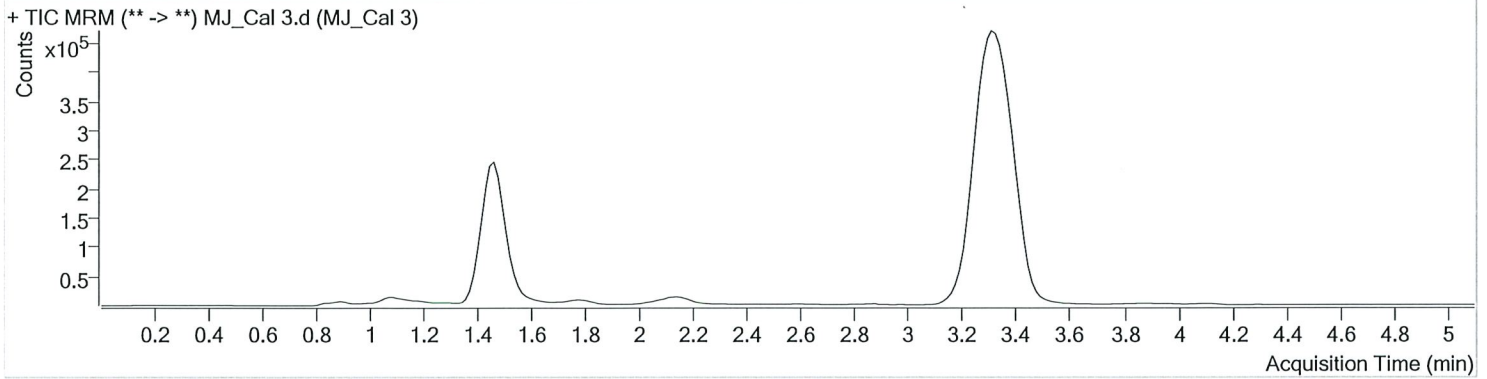
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wklsts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_Cal 3.d
Type	Cal	Sample	MJ_Cal 3
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-D6	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 11:32:25 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.483	102018	∞	9.8	205.44	913429	4.8686 ng/ml
THC-COOH	1.489	144671	∞	51.6	∞	266201	19.2549 ng/ml
THC	3.345	163935	752.59	27.2	83.01	4539619	4.9084 ng/ml

TS

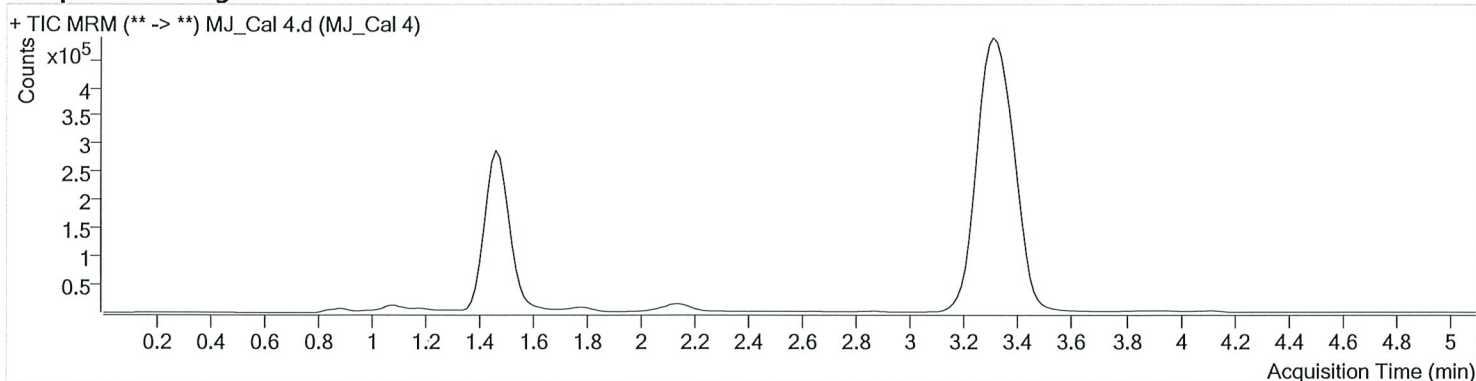


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wk\lsts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_Cal 4.d
Type	Cal	Sample	MJ_Cal 4
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-E6	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 11:40:00 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	157590	∞	10.4	∞	834362	10.5620 ng/ml
THC-COOH	1.489	334223	655.55	53.6	4012.77	244202	51.9128 ng/ml
THC	3.330	326518	∞	27.1	198.22	4281089	9.9474 ng/ml

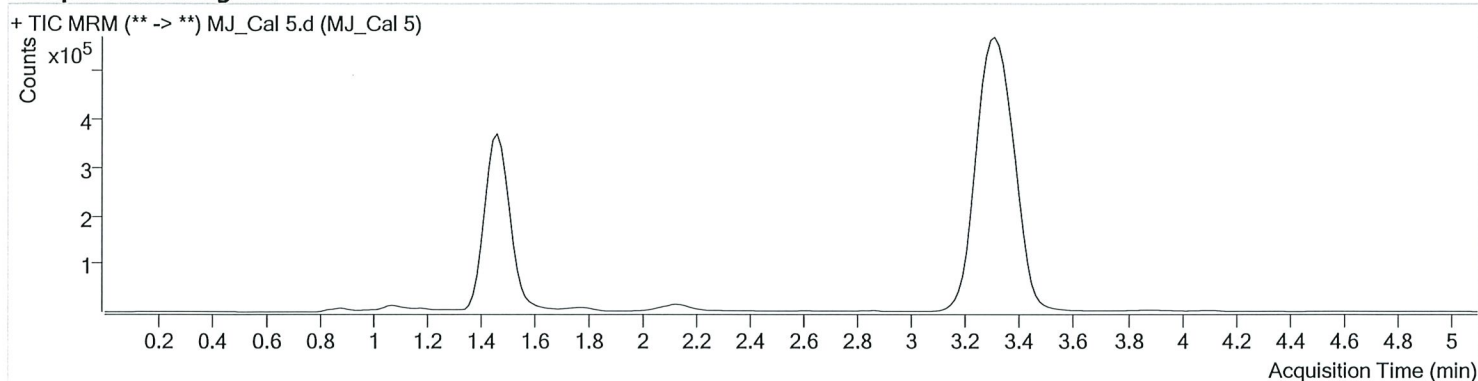


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wk1sts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
 Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_Cal 5.d
Type	Cal	Sample	MJ_Cal 5
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-F6	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 11:47:36 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	331218	∞	12.9	∞	872809	24.6212 ng/ml
THC-COOH	1.489	493217	∞	56.5	3445.36	252686	74.9968 ng/ml
THC	3.330	856798	1013.68	26.3	∞	4486092	24.3428 ng/ml

TS

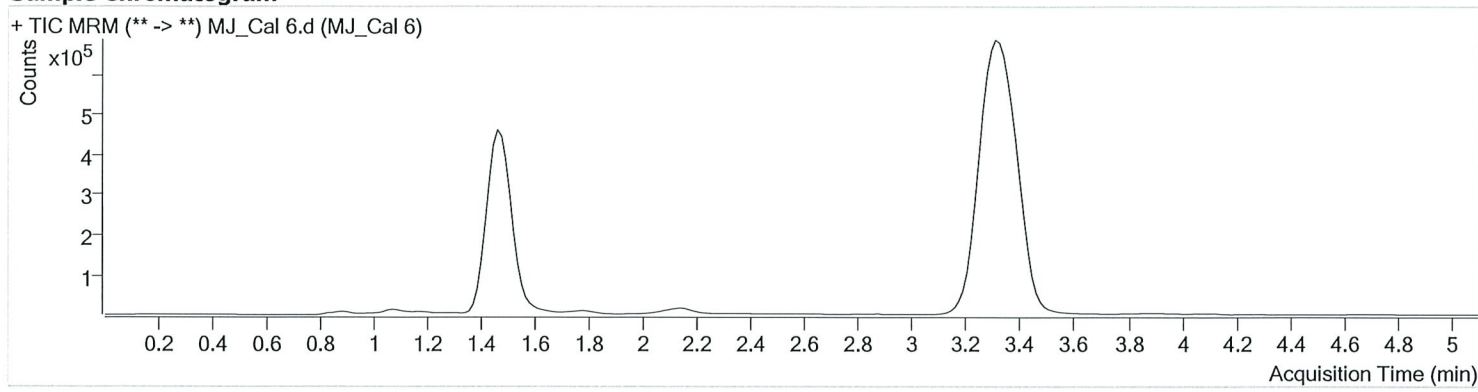


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wk1sts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_Cal 6.d
Type	Cal	Sample	MJ_Cal 6
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-G6	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 11:55:10 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	619201	431.74	13.3	∞	861154	49.6661 ng/ml
THC-COOH	1.489	638374	4597.63	56.1	2652.21	248001	99.6212 ng/ml
THC	3.330	1763073	7028.13	25.9	780.43	4447548	50.1200 ng/ml

TS

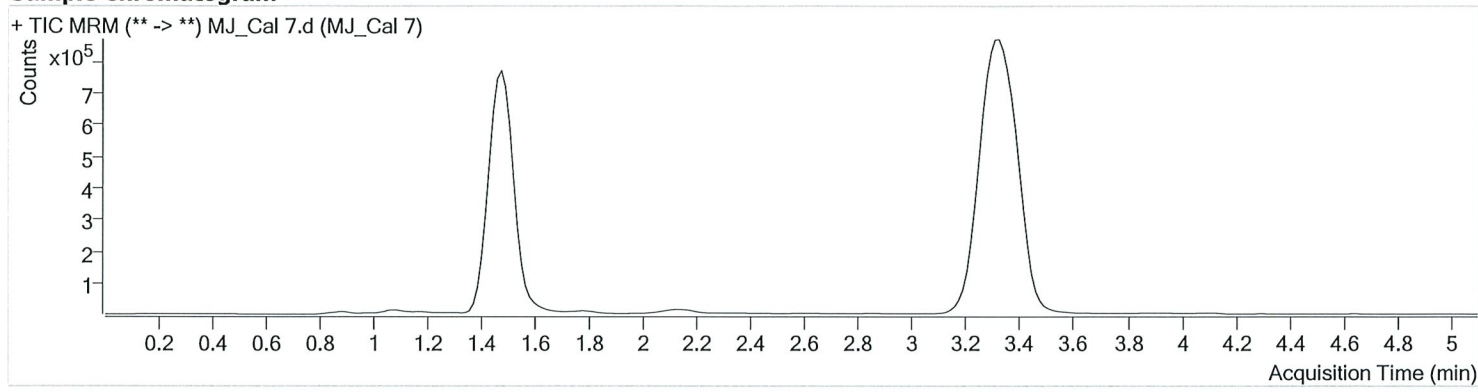


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28 030320 AM 27 28 wk1sts 4044 4045 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 3/3/2020 3:37:13 PM

Instrument	Falco	Data File	MJ_Cal 7.d
Type	Cal	Sample	MJ_Cal 7
Acq. Method	AM 27 THC quant.m	Operator	Tamara Salazar
Sample Position	P3-H6	Comment	
Injection Volume	10		
Acq. Date-Time	3/3/2020 12:02:45 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	1150505	∞	13.4	∞	818441	100.3158 ng/ml
THC-COOH	1.489	1442748	∞	59.0	2643.02	227123	249.1521 ng/ml
THC	3.330	3416969	2482.98	26.3	2157.92	4280047	100.5557 ng/ml