









REVIEWED

By Tamara Salazar at 12:19 pm, Feb 25, 2020

2/21/2020

CS

Worklist: 4022

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
M2020-0306	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2020-0448	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2020-0591	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-0028	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-0055	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-0262	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-0488	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-0489	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	

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 ToxBox 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

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

Extraction Date: 02/19/2020

Plate lot#: 190716

Mobile phase A: 0.1% Formic Acid in LCMS Water

Blank Blood Lot: 445283-3

LCMS-QQQ ID: 069901

Analyst: Celena Shrum

Plate Expiration: 01/16/2020- OK, deviation in place

Mobile phase B: 0.1% Formic acid in Acetonitrile

Column: UCT Selectra DA 100 x 2.1mm 3um

Blank Urine Lot: POC031319

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: #42**
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 067105*
- 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 **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)** 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.
- 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 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? (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 is 5-100.



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 µL of 1mg/mL THC, 100 µL of 100 µg/mL THC-OH, C-THC in 9790 µL MeOH
Approximate concentration 1µg/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 µL of methanol external control solution was added to 9800 µL 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	



Idaho State Police Forensic Services

AM #27 Confirmation of THC and Metabolites in Blood and Urine by LCMS-QQQ

Methanol External Control Solution (Lot: WS021320)

100 ul of 100 ug/mL C-THC in 9900 ul MeOH

Approximate concentration 1ug/mL.

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>	<i>Expiration Date</i>
Methanol (LCMS)	Fisher	193941	
C-THC	Cerilliant	FE07171501	09/30/2020
Prepared:	02/13/2020		
Prepared By:	Celena Shrum		
Expires:*	02/13/2020 <small>02/25/2020</small>		

*Per AM 21- no set expiration for references for qualitative compounds

Urine External Control Solution (Lot: 021320)

200 ul of methanol external control solution was added to 9800 ul of urine.

Approximately 20ng/mL

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Negative Urine	Pocatello Lab	POC031319
Methanol External Control Solution	-	WS021320
Prepared:	02/13/2020	
Prepared by:	Celena Shrum	
Expires:	09/30/2020	

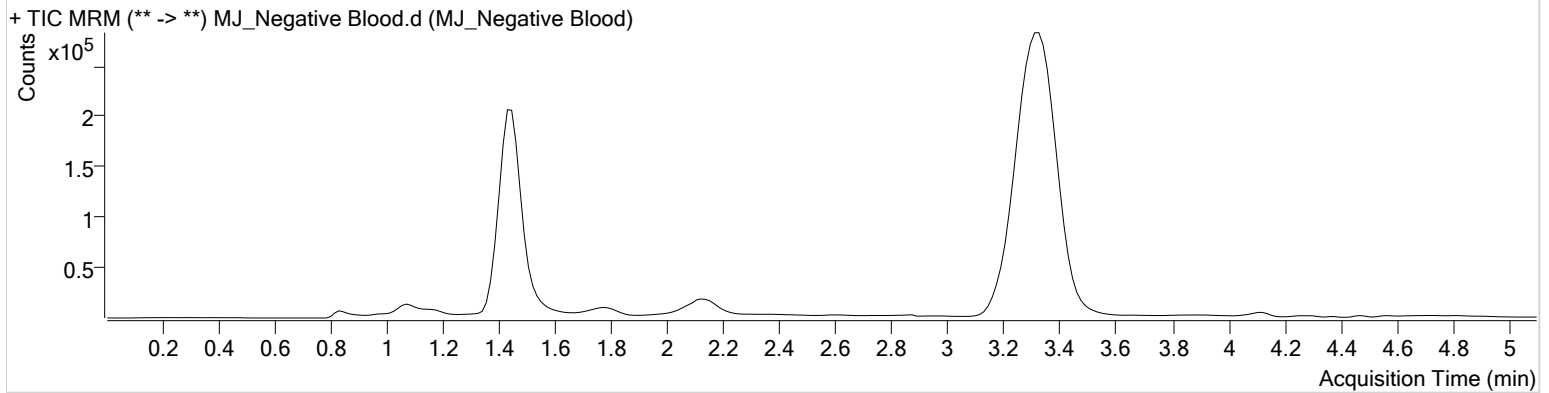
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Negative Blood.d
Type	Sample	Sample	MJ_Negative Blood
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-A2	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 3:34:51 PM		
Sample Info.			

Sample Chromatogram



CS

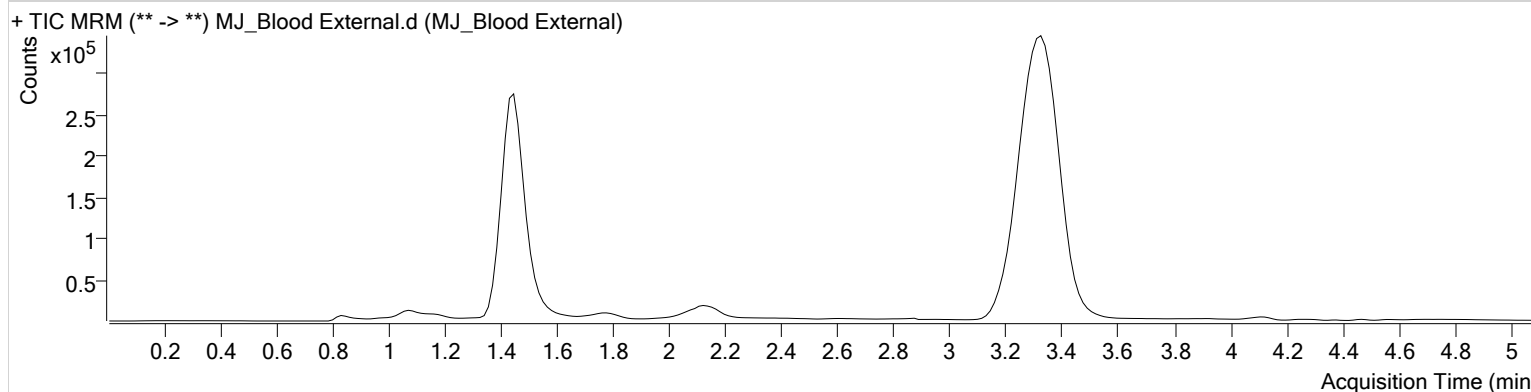


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Blood External.d
Type	Sample	Sample	MJ_Blood External
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-B2	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 3:50:02 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	308385	∞	11.1	460.65	937352	19.7254 ng/ml
THC-COOH	1.474	83614	∞	60.3	849.68	197841	16.6203 ng/ml
THC	3.345	428834	1009.65	26.9	180.24	2957800	18.1473 ng/ml

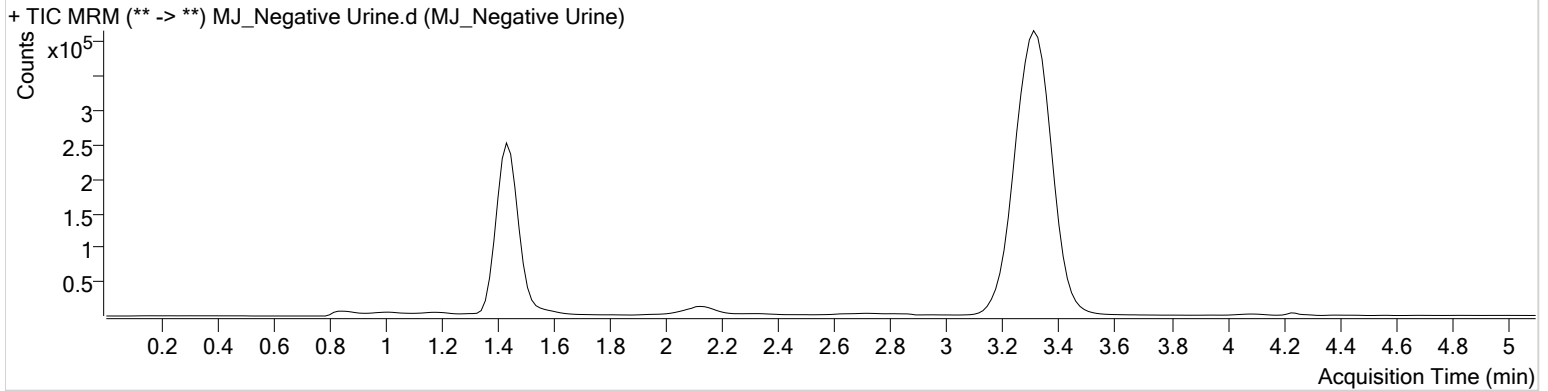
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Negative Urine.d
Type	Sample	Sample	MJ_Negative Urine
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-C2	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 4:05:14 PM		
Sample Info.			

Sample Chromatogram



CS

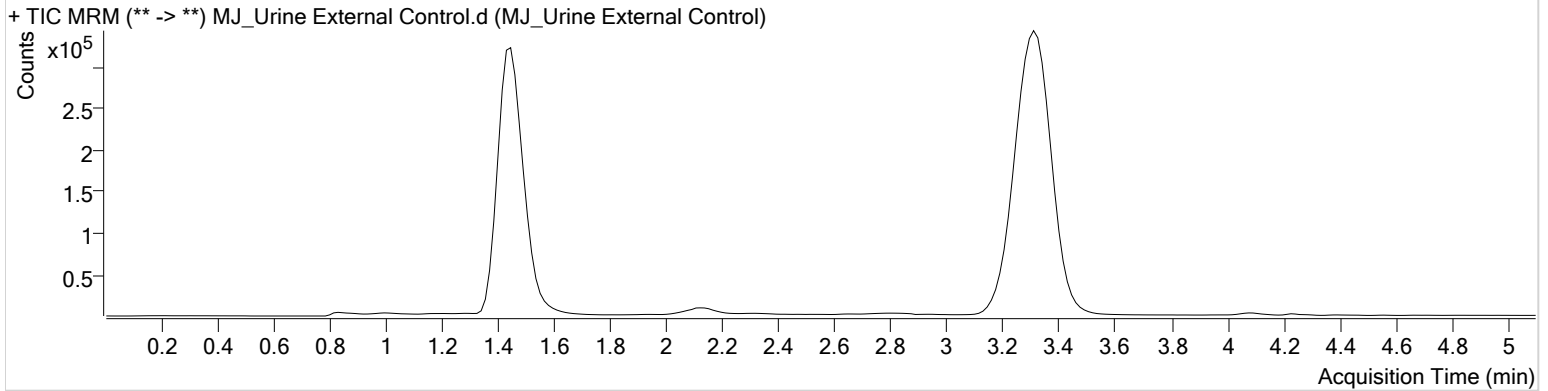


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Urine External Control.d
Type	Sample	Sample	MJ_Urine External Control
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-D2	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 4:20:26 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	300237	∞	59.4	∞	228474	56.1057 ng/ml

cg

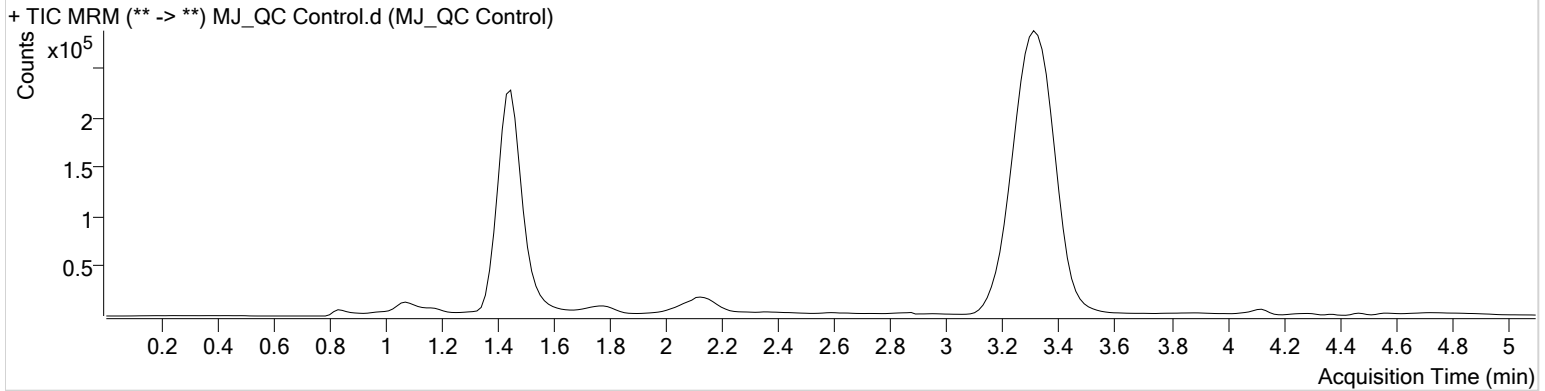


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_QC Control.d
Type	Sample	Sample	MJ_QC Control
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-H1	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 3:19:40 PM		

Sample Chromatogram



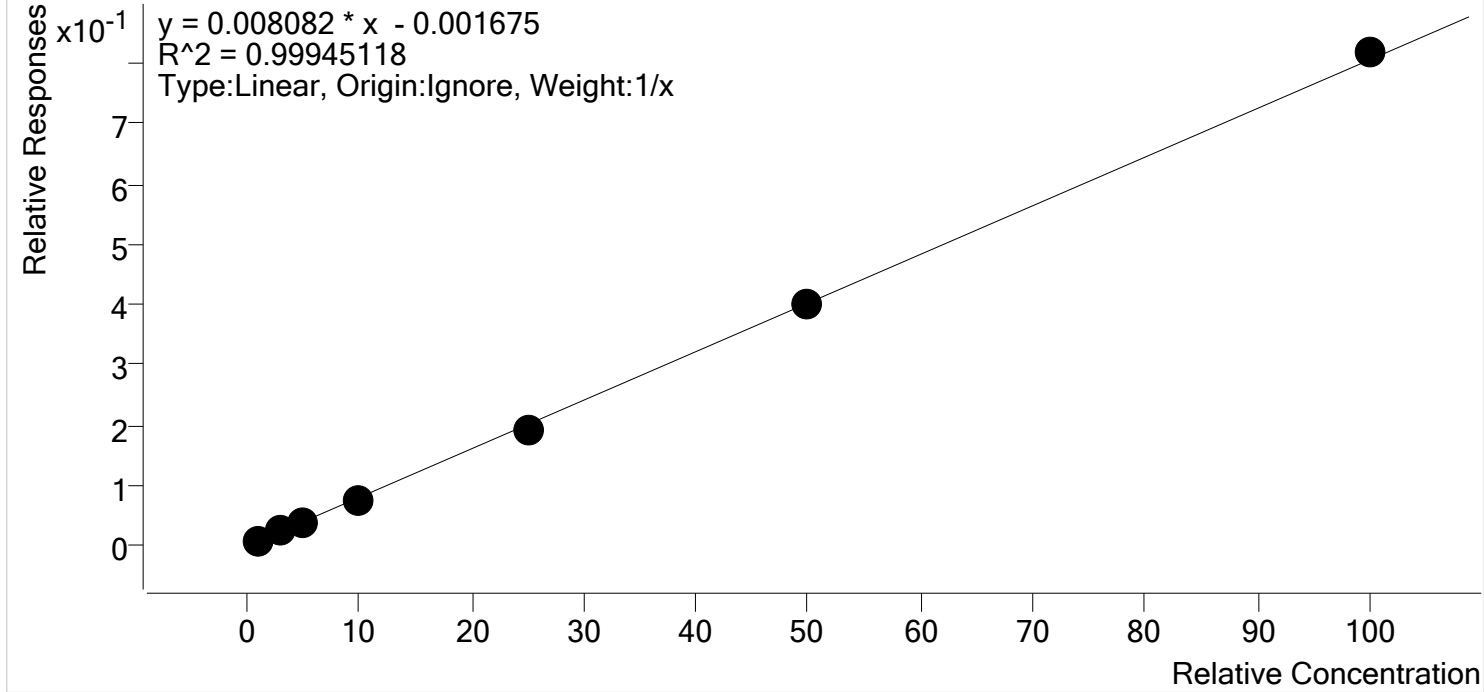
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	113002	∞	8.6	126.05	953621	4.5681 ng/ml
THC-COOH	1.474	71962	∞	51.3	438.45	201296	13.7351 ng/ml
THC	3.345	96922	131.87	27.6	∞	2893507	4.3520 ng/ml



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Last Cal. Update 2/21/2020 12:52 PM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-D3

THC - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 0 QCs

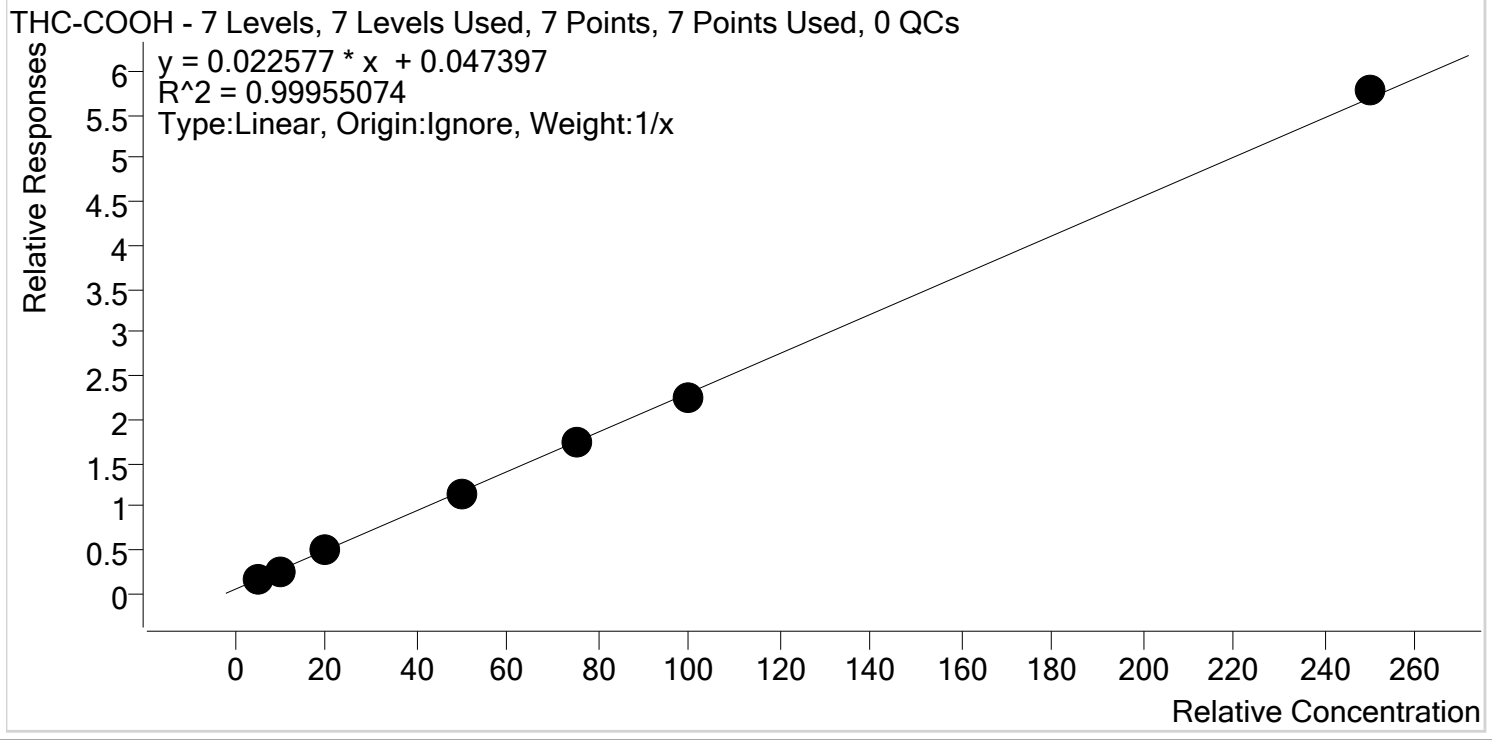


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	1.0	1.1	111.2
MJ_Cal 2	2	✓	3.0	3.0	98.9
MJ_Cal 3	3	✓	5.0	4.8	96.4
MJ_Cal 4	4	✓	10.0	9.6	95.7
MJ_Cal 5	5	✓	25.0	24.1	96.4
MJ_Cal 6	6	✓	50.0	50.0	100.0
MJ_Cal 7	7	✓	100.0	101.4	101.4



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Last Cal. Update 2/21/2020 12:52 PM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9

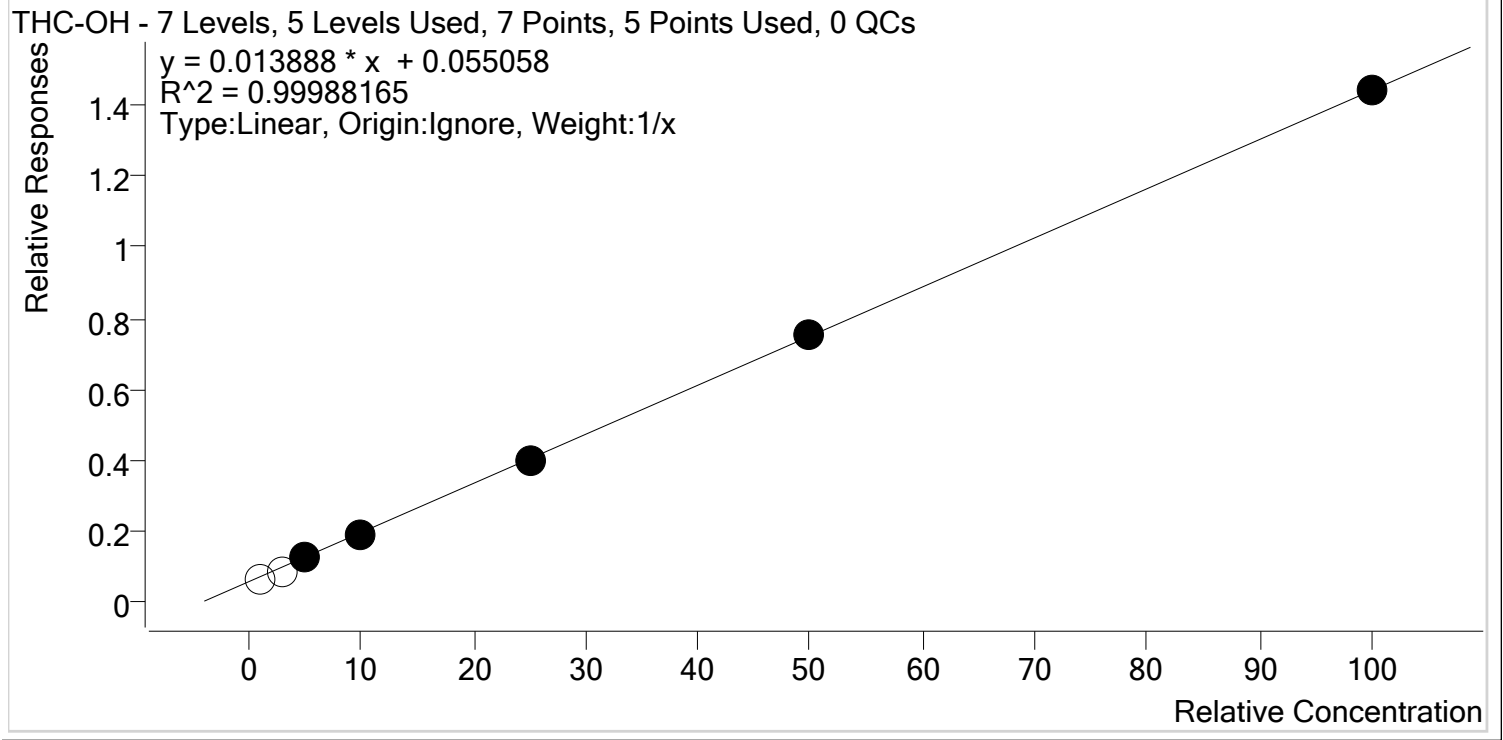


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	5.0	5.4	107.0
MJ_Cal 2	2	✓	10.0	9.6	96.2
MJ_Cal 3	3	✓	20.0	20.2	100.9
MJ_Cal 4	4	✓	50.0	48.7	97.3
MJ_Cal 5	5	✓	75.0	73.9	98.6
MJ_Cal 6	6	✓	100.0	98.4	98.4
MJ_Cal 7	7	✓	250.0	253.8	101.5



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Last Cal. Update 2/21/2020 12:52 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	x	1.0	0.6	60.1
MJ_Cal 2	2	x	3.0	2.1	68.9
MJ_Cal 3	3	✓	5.0	5.1	102.7
MJ_Cal 4	4	✓	10.0	9.7	97.3
MJ_Cal 5	5	✓	25.0	24.9	99.4
MJ_Cal 6	6	✓	50.0	50.4	100.7
MJ_Cal 7	7	✓	100.0	99.9	99.9

CS

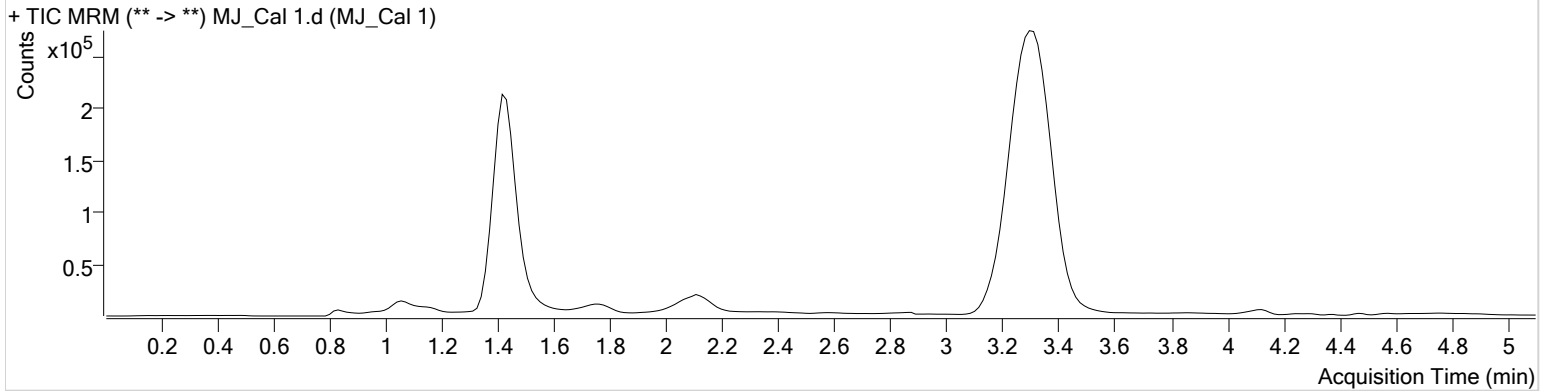


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Cal 1.d
Type	Cal	Sample	MJ_Cal 1
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-A1	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 2:18:52 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.498	61203	∞	5.6 Low	10.70	965321	0.6008 ng/ml Low
THC-COOH	1.459	33407	∞	44.3	85.16	198629	5.3501 ng/ml Low
THC	3.330	21110	68.27	33.1	13.23	2887400	1.1119 ng/ml Low

cg

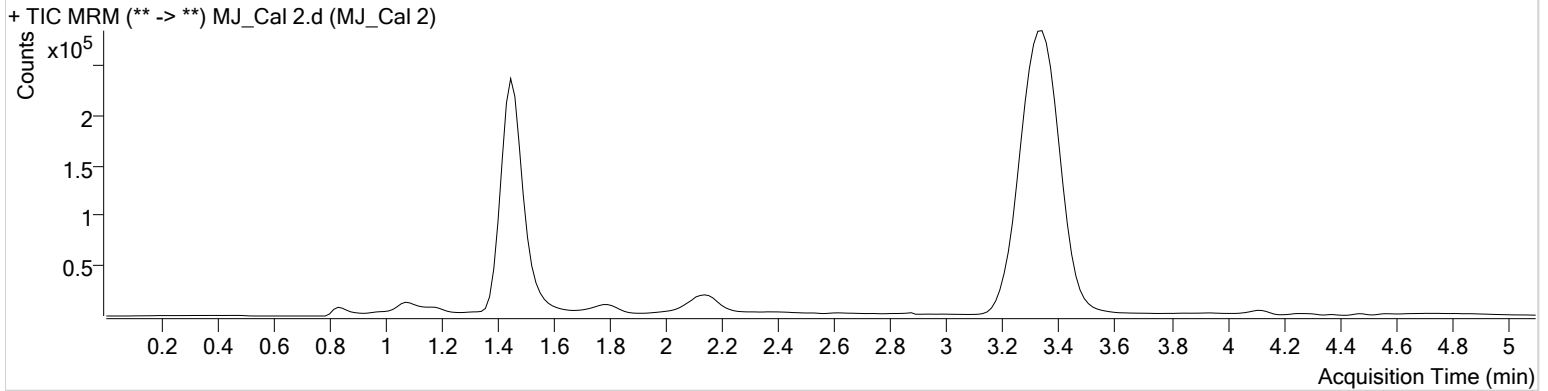


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Cal 2.d
Type	Cal	Sample	MJ_Cal 2
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-B1	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 2:26:36 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.513 High	80217	∞	7.3 Low	129.28	957819	2.0660 ng/ml Low
THC-COOH	1.489	51612	∞	48.3	∞	194988	9.6246 ng/ml Low
THC	3.360	60762	∞	28.0	62.16	2725789	2.9655 ng/ml Low

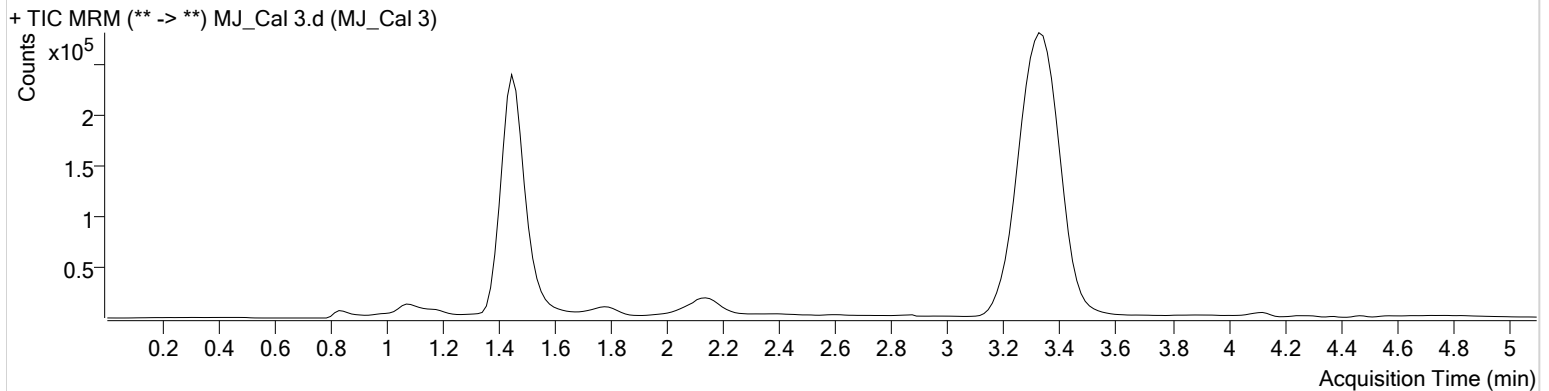
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Cal 3.d
Type	Cal	Sample	MJ_Cal 3
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-C1	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 2:34:11 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	120894	∞	8.4	76.43	956834	5.1334 ng/ml
THC-COOH	1.489	97663	∞	52.1	397.75	194195	20.1760 ng/ml
THC	3.345	103003	350.13	26.6	35.24	2762944	4.8202 ng/ml

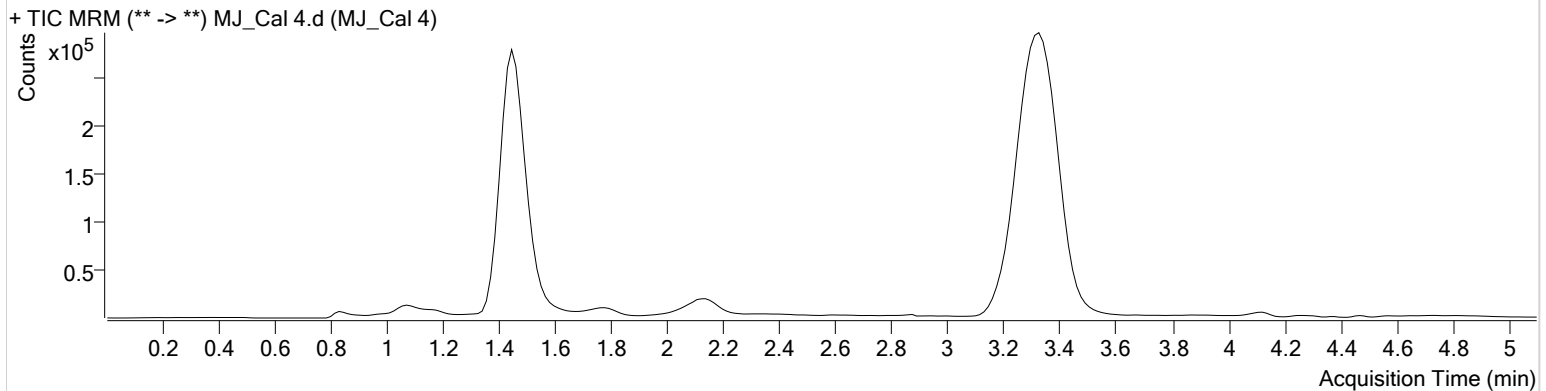
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Cal 4.d
Type	Cal	Sample	MJ_Cal 4
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-D1	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 2:41:46 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	186327	∞	10.8	∞	979859	9.7281 ng/ml
THC-COOH	1.489	226771	680.95	58.3	1794.60	197831	48.6729 ng/ml
THC	3.345	212698	∞	27.2	137.84	2809623	9.5746 ng/ml

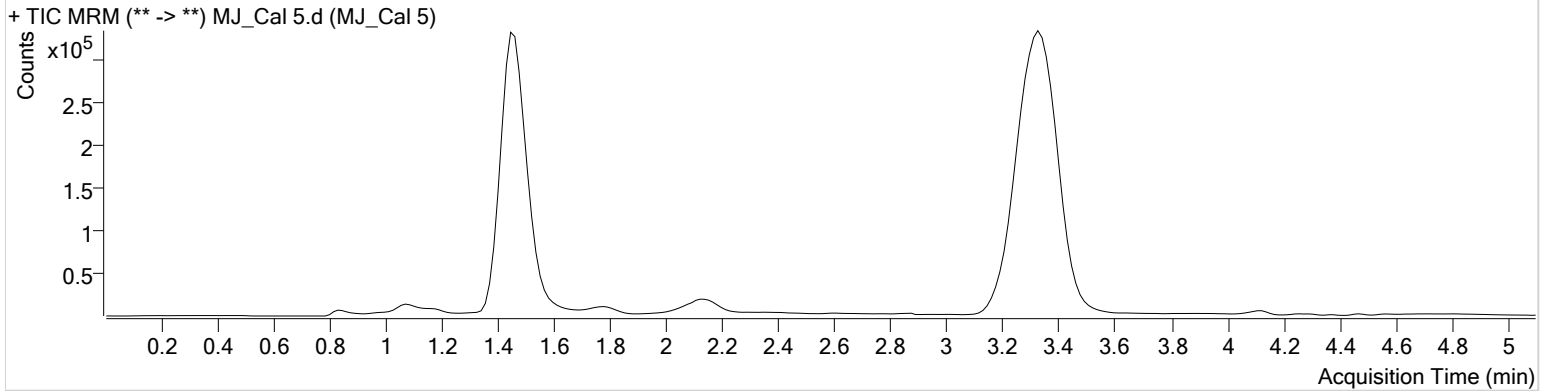
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Cal 5.d
Type	Cal	Sample	MJ_Cal 5
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-E1	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 2:49:21 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	379200	∞	12.1	806.82	947578	24.8510 ng/ml
THC-COOH	1.489	332800	706.28	58.9	915.40	193898	73.9232 ng/ml
THC	3.345	535767	2449.18	26.0	191.91	2774633	24.1004 ng/ml

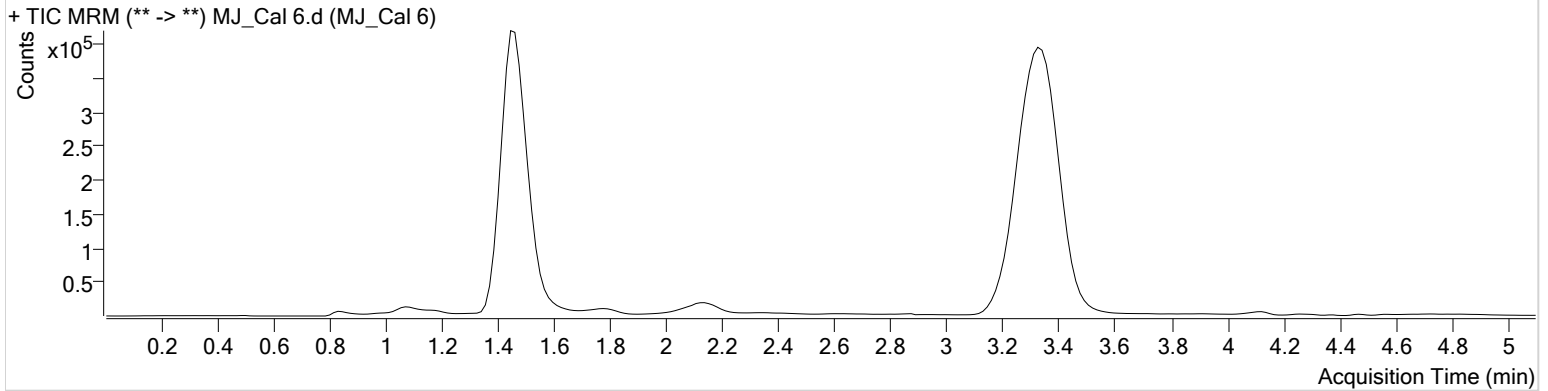


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Cal 6.d
Type	Cal	Sample	MJ_Cal 6
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-F1	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 2:56:55 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	718008	∞	12.5	4106.40	951706	50.3605 ng/ml
THC-COOH	1.489	440547	1102.61	59.8	1358.53	194098	98.4326 ng/ml
THC	3.345	1091588	5373.74	26.0	550.11	2713644	49.9820 ng/ml

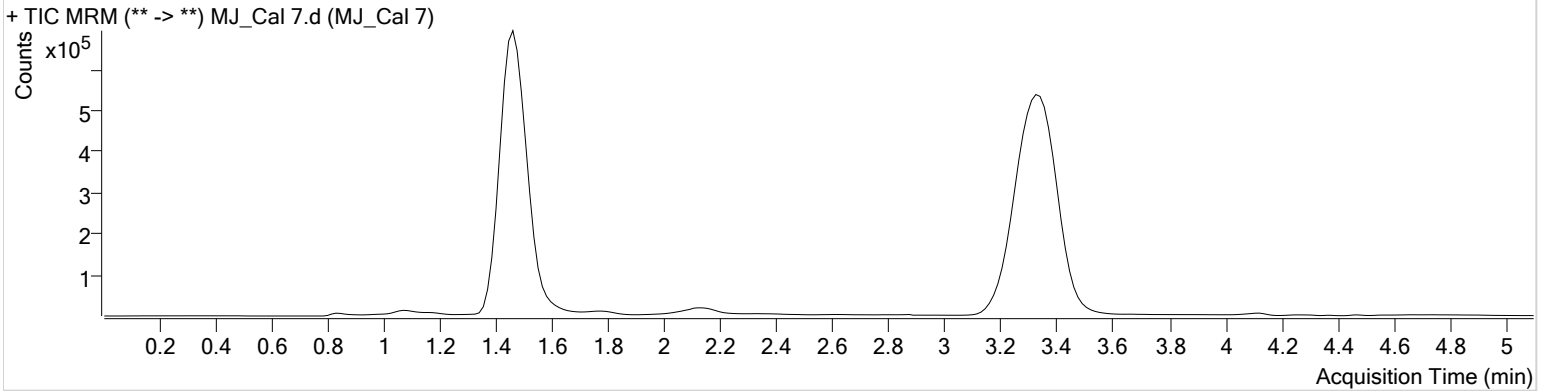
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ CS.batch.bin
Calibration Last Update 2/21/2020 12:52:55 PM

Instrument	Falco	Data File	MJ_Cal 7.d
Type	Cal	Sample	MJ_Cal 7
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-G1	Comment	
Injection Volume	10		
Acq. Date-Time	2/19/2020 3:04:30 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	1370282	∞	12.6	12819.8 5	949736	99.9271 ng/ml
THC-COOH	1.489	1083640	∞	59.2	10764.1 3	187549	253.8205 ng/ml
THC	3.345	2286391	44880.13	26.1	∞	2794540	101.4453 ng/ml