








**REVIEWED**

By Tamara Salazar at 8:45 am, Aug 13, 2020

8/12/2020

CS

**Worklist: 4427**

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
P2020-1955	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1960	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-2022	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-2115	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-2141	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-2152	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-2154	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	

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

Extraction Date: 08/07/2020

Analyst: Celena Shrum

Plate lot#: IDP-108-2-200303

Plate Expiration: 09/30/2020

Mobile phase A: 0.1% Formic Acid in LCMS Water

Mobile phase B: 0.1% Formic acid in Acetonitrile

Blank Blood Lot: 445283-4

Column: UCT Selectra DA 100 x 2.1mm 3um

LCMS-QQQ ID: 069901

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.
- 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<sup>2</sup> 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: <sup>5cs</sup>~~10~~ng/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: Only carboxy-THC evaluated.



# Idaho State Police Forensic Services

## AM #26 Screening of THC and Metabolites and AM #27 Confirmation of THC and Metabolites Urine External Control Prep Sheet

### 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 1ug/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		

### Urine External Control Solution (Lot: 042220)

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

*Approximately 20ng/mL each*

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Negative Urine	Pocatello Lab	POC031319
Methanol External Control Solution	-	WS011620
Prepared:	07/07/2020	
Prepared by:	Sophie Jackson	
Expires:	09/30/2020	

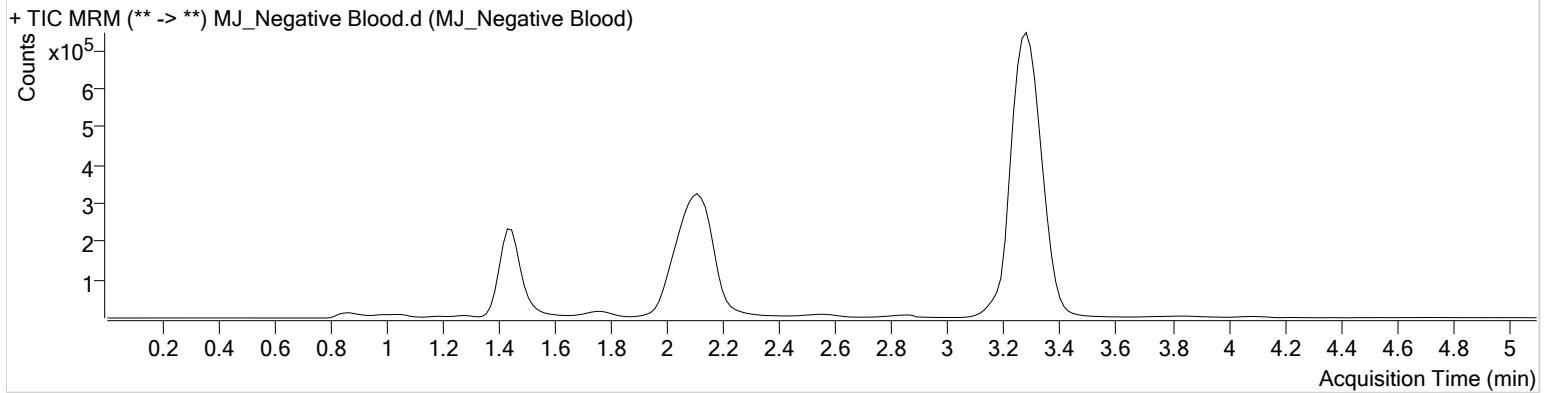
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Negative Blood.d
<b>Type</b>	Sample	<b>Sample</b>	MJ_Negative Blood
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-H5	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 4:27:04 PM		
<b>Sample Info.</b>			

## Sample Chromatogram





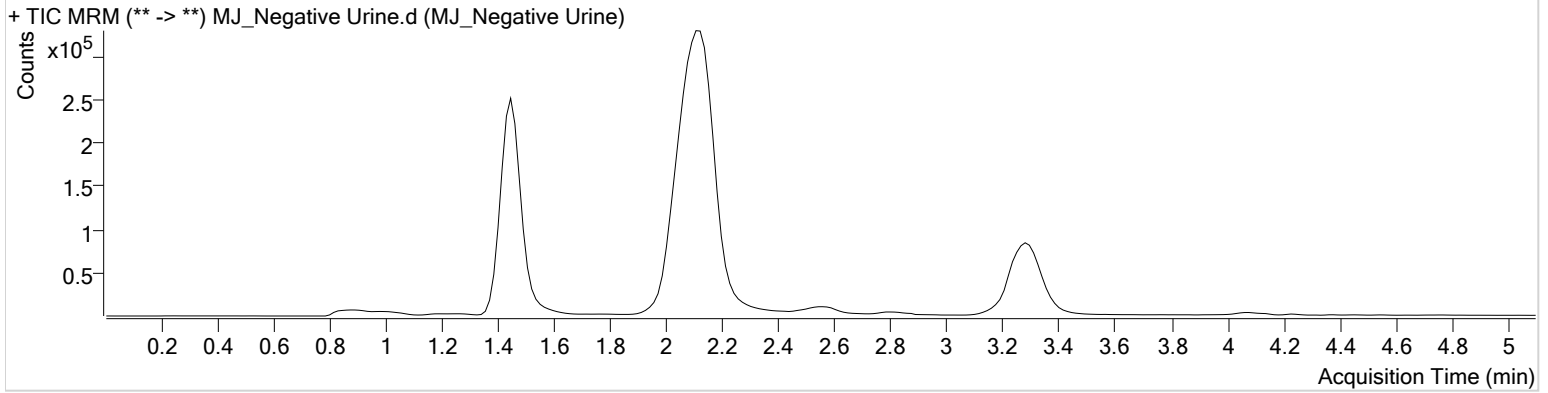
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Negative Urine.d
<b>Type</b>	Sample	<b>Sample</b>	MJ_Negative Urine
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-G5	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 4:34:39 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



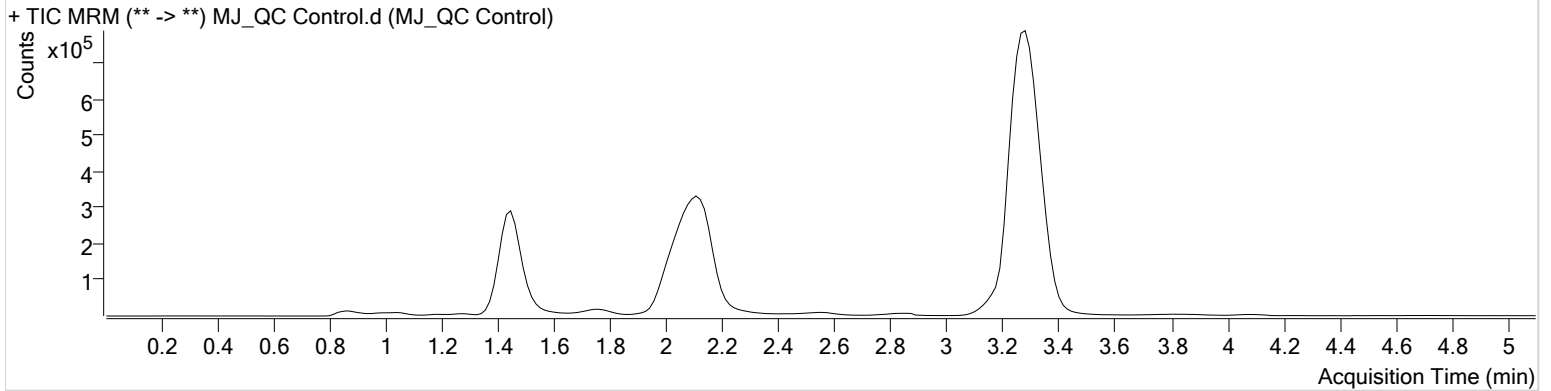
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_QC Control.d
<b>Type</b>	Sample	<b>Sample</b>	MJ_QC Control
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-A6	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 4:11:51 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	124392	∞	57.0	∞	355393	14.2465 ng/ml

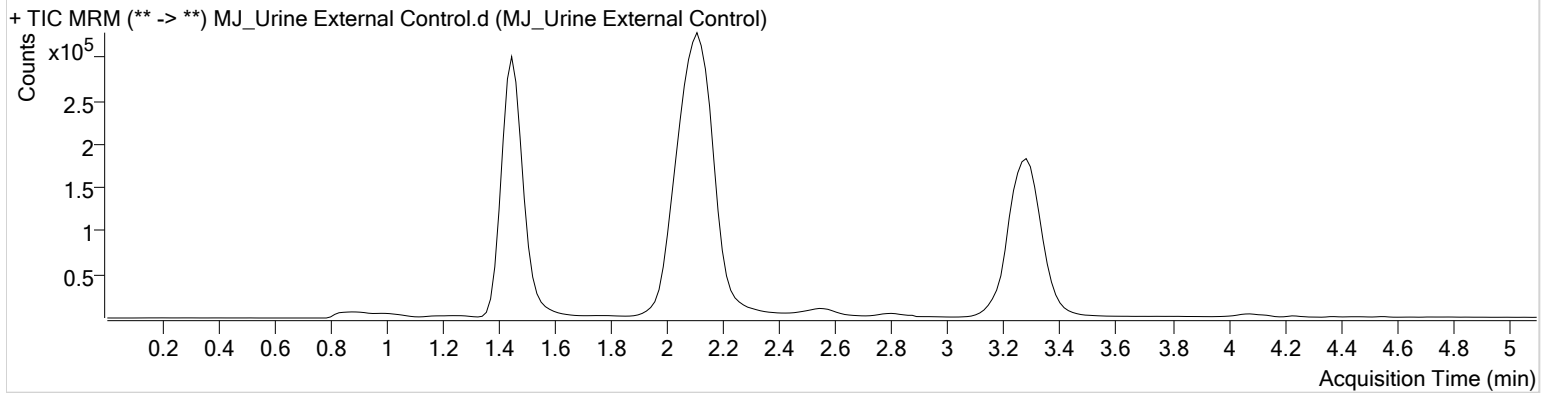
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Urine External Control.d
<b>Type</b>	Sample	<b>Sample</b>	MJ_Urine External Control
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-F5	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 4:42:13 PM		
<b>Sample Info.</b>			

## Sample Chromatogram

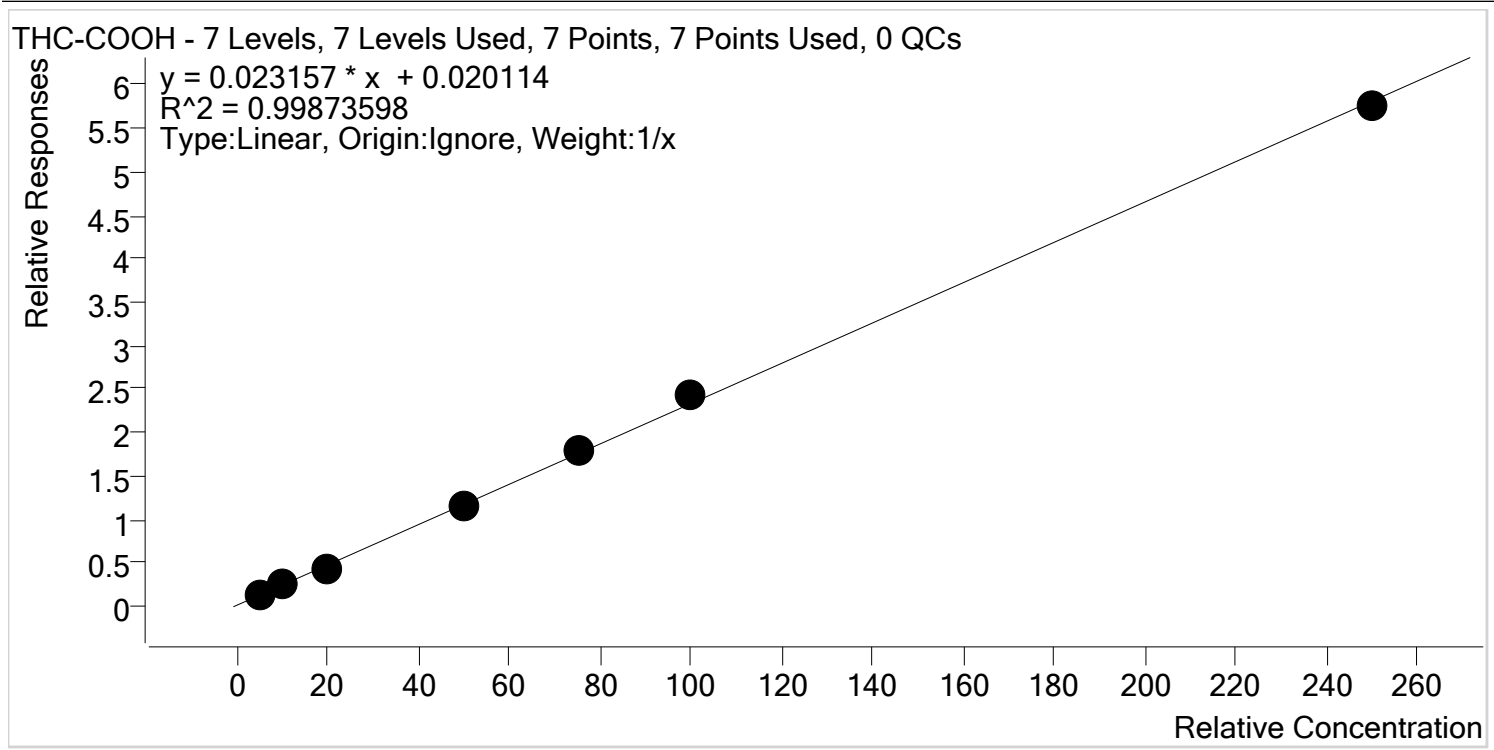


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	94010	∞	65.0	∞	323568	11.6783 ng/ml



# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds  
 Removed.batch.bin  
**Last Cal. Update** 8/12/2020 2:49 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	105.9
MJ_Cal 2	2	✓	10.0	10.2	101.8
MJ_Cal 3	3	✓	20.0	18.1	90.5
MJ_Cal 4	4	✓	50.0	48.5	97.1
MJ_Cal 5	5	✓	75.0	75.9	101.2
MJ_Cal 6	6	✓	100.0	104.5	104.5
MJ_Cal 7	7	✓	250.0	247.4	99.0

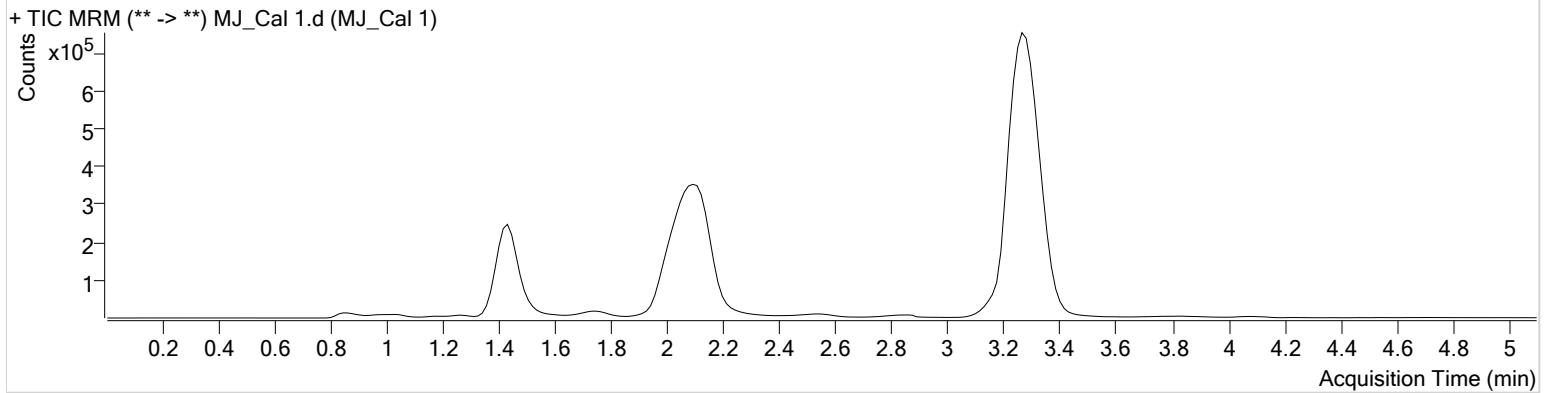
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Cal 1.d
<b>Type</b>	Cal	<b>Sample</b>	MJ_Cal 1
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-H6	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 3:11:01 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.459	49082	∞	49.8	152.85	343917	5.2944 ng/ml

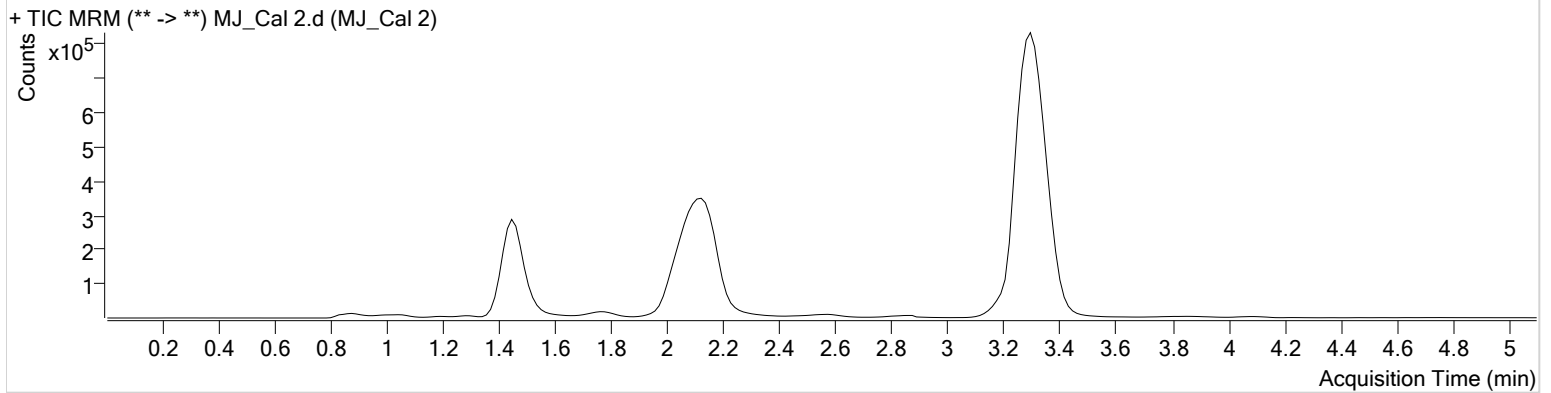
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Cal 2.d
<b>Type</b>	Cal	<b>Sample</b>	MJ_Cal 2
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-G6	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 3:18:46 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	92894	∞	47.9	626.77	362980	10.1832 ng/ml

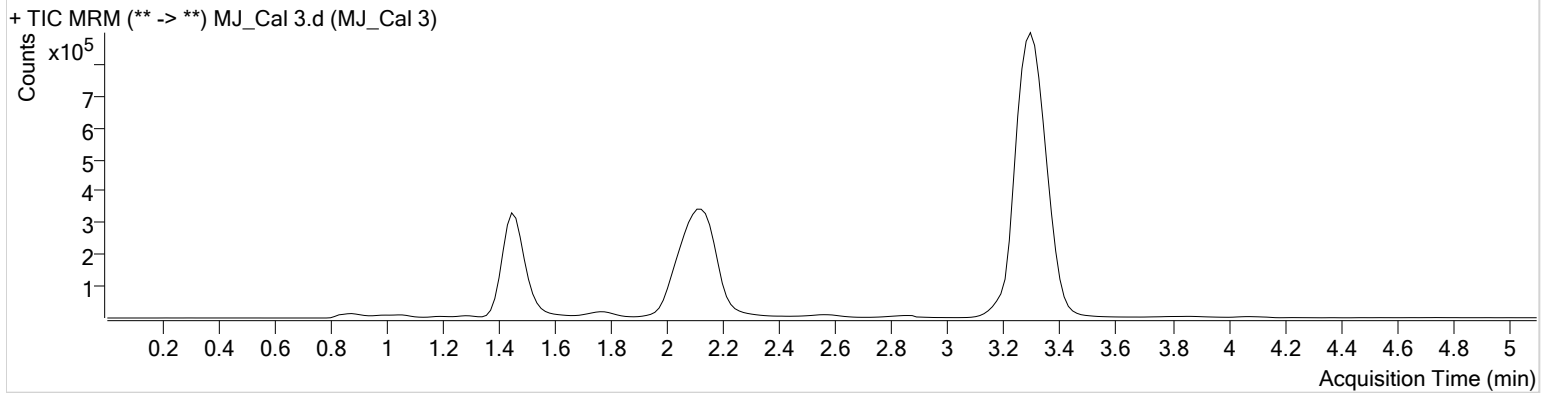
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Cal 3.d
<b>Type</b>	Cal	<b>Sample</b>	MJ_Cal 3
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-F6	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 3:26:20 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	166220	114.01	58.1	∞	378565	18.0928 ng/ml

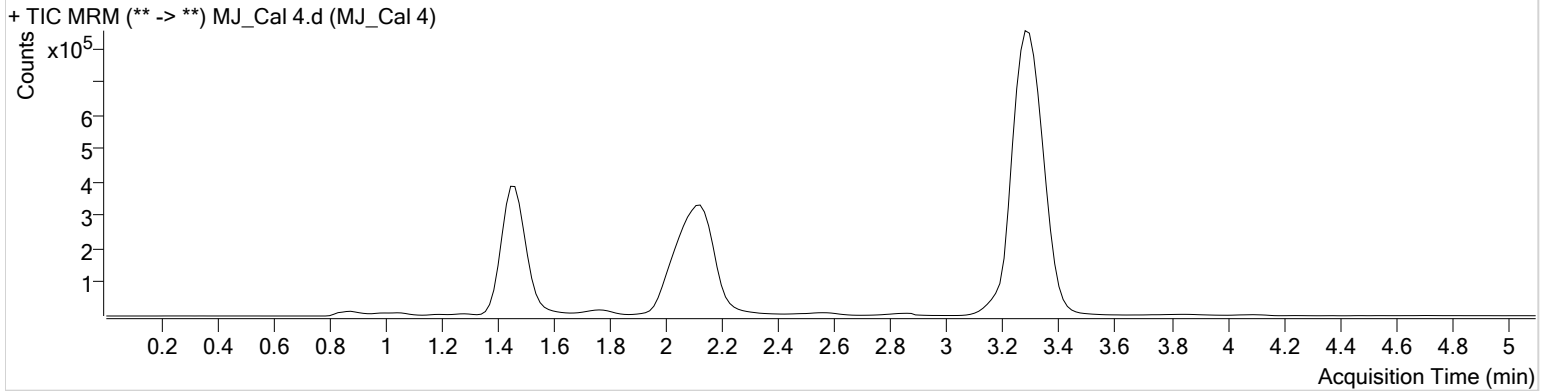
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Cal 4.d
<b>Type</b>	Cal	<b>Sample</b>	MJ_Cal 4
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-E6	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 3:33:55 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	409542	∞	59.8	∞	357989	48.5345 ng/ml



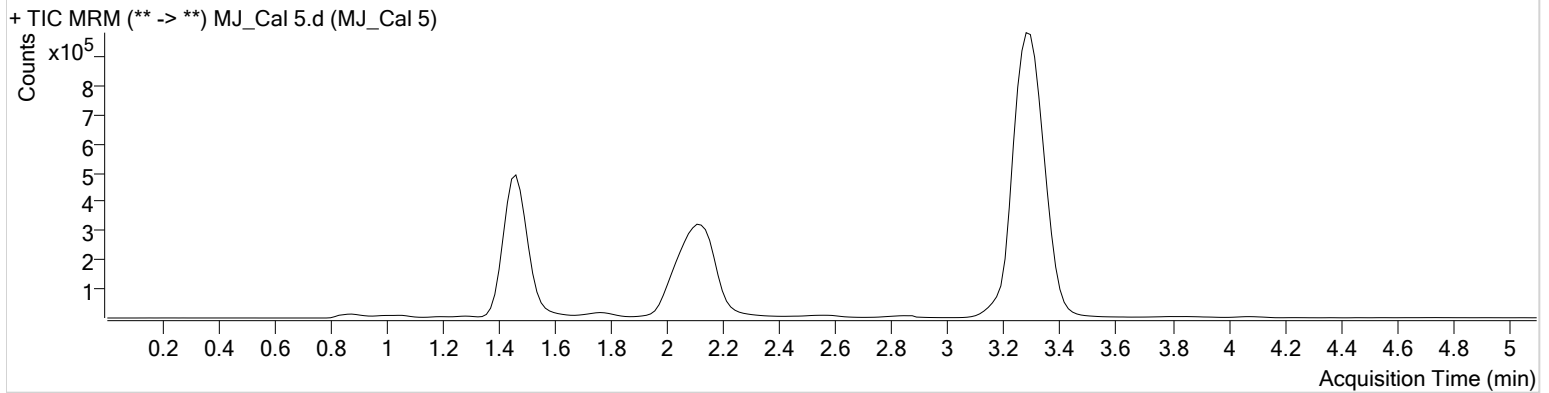
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Cal 5.d
<b>Type</b>	Cal	<b>Sample</b>	MJ_Cal 5
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-D6	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 3:41:29 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	621349	∞	56.9	∞	349394	75.9286 ng/ml

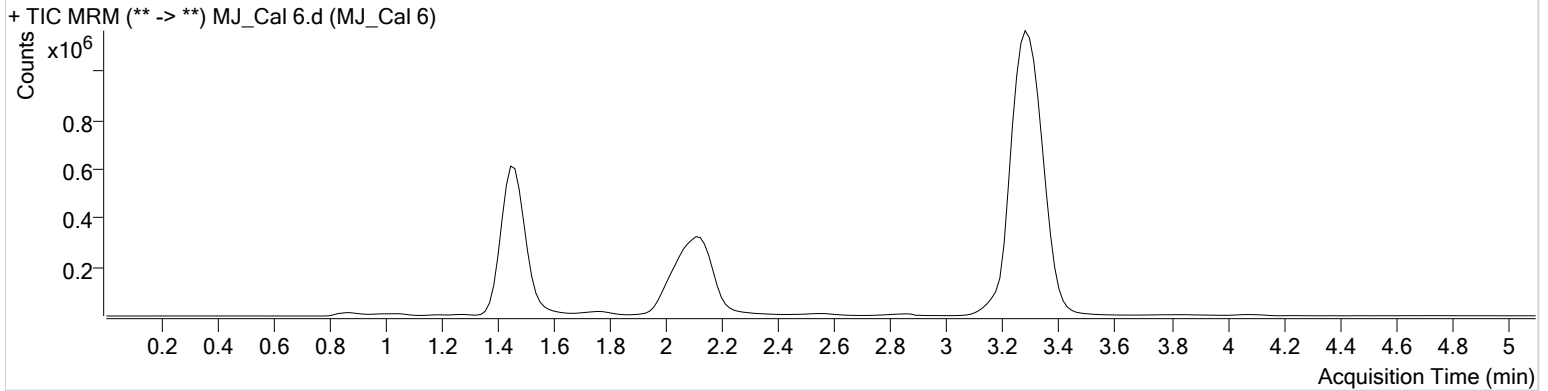
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Cal 6.d
<b>Type</b>	Cal	<b>Sample</b>	MJ_Cal 6
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-C6	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 3:49:04 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	813680	∞	56.4	∞	333359	104.5378 ng/ml

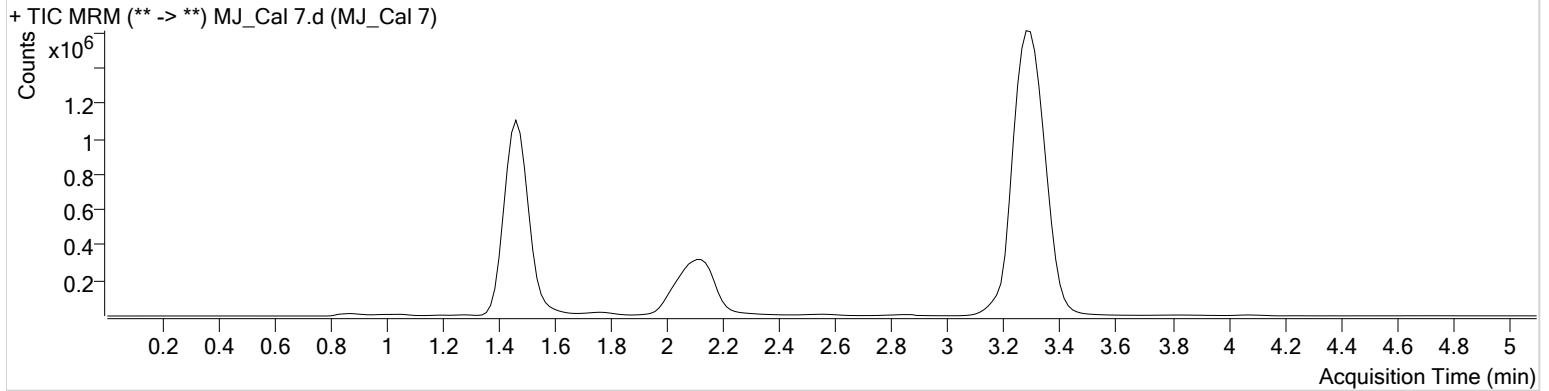
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27-28 080720 CS\QuantResults\THCQ Compounds Removed.batch.bin  
**Calibration Last Update** 8/12/2020 2:49:55 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Cal 7.d
<b>Type</b>	Cal	<b>Sample</b>	MJ_Cal 7
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Celena Shrum
<b>Sample Position</b>	P3-B6	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	8/7/2020 3:56:39 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	1823890	∞	61.1	∞	317214	247.4288 ng/ml