

**REVIEWED**

By Tamara Salazar at 9:46 am, Jun 02, 2020

5/28/2020

CS

**Worklist: 4263**

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
C2020-0784	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
C2020-0789	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2020-1679	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2020-1735	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2020-1738	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2020-1777	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2020-1781	5	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1177	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1186	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1253	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1354	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1368	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1471	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: 05/27/2020

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  $r^2$  values  $\geq 0.98$  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 limited to 3-100.



# Idaho State Police Forensic Services

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

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### Methanol External Control Solution (Lot: WS011620)

10  $\mu$ L of 1mg/mL THC, 100  $\mu$ L of 100  $\mu$ g/mL THC-OH, C-THC in 9790  $\mu$ L 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:	04/22/2020	
Prepared by:	Celena Shrum	
Expires:	09/30/2020	

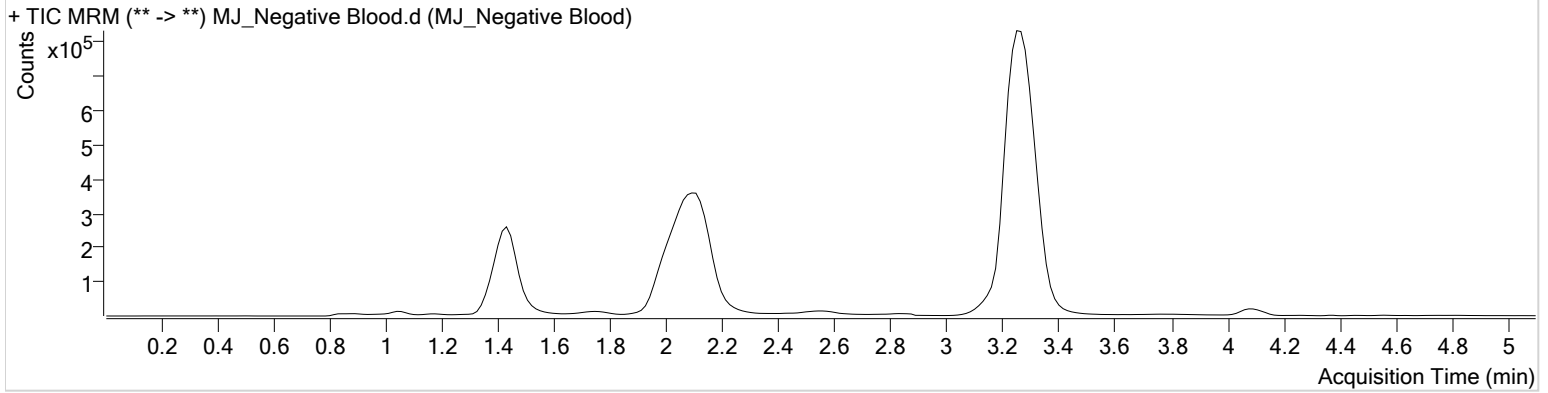
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-A2	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 4:59:36 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



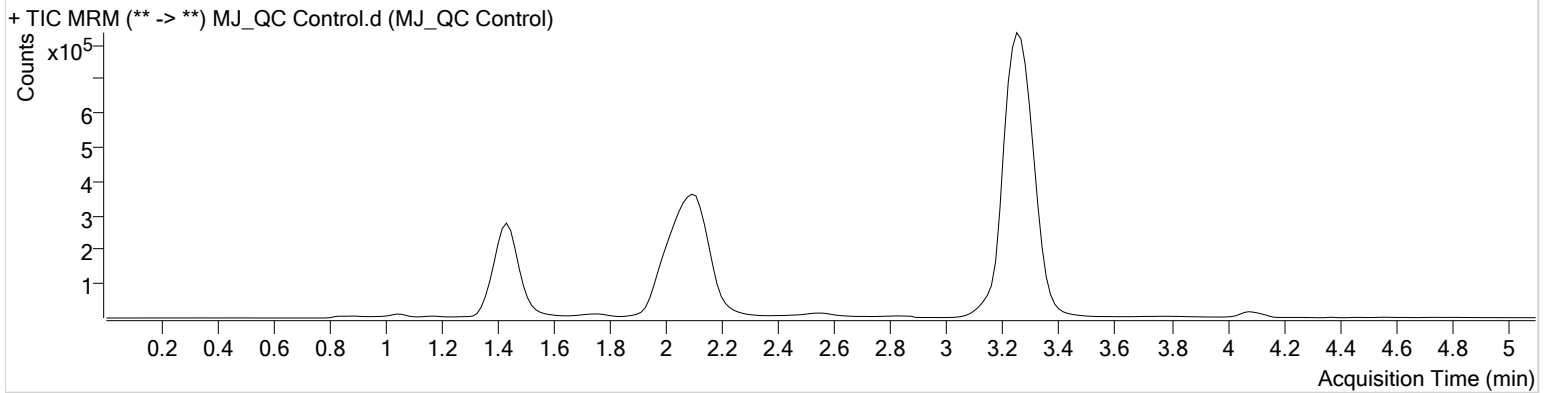
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-H1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 4:44:25 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.438	103826	∞	11.7	107.53	999375	4.2274 ng/ml
THC-COOH	1.459	133946	∞	51.9	535.30	378985	15.7455 ng/ml
THC	3.270	236215	∞	26.8	135.86	6061845	4.2622 ng/ml

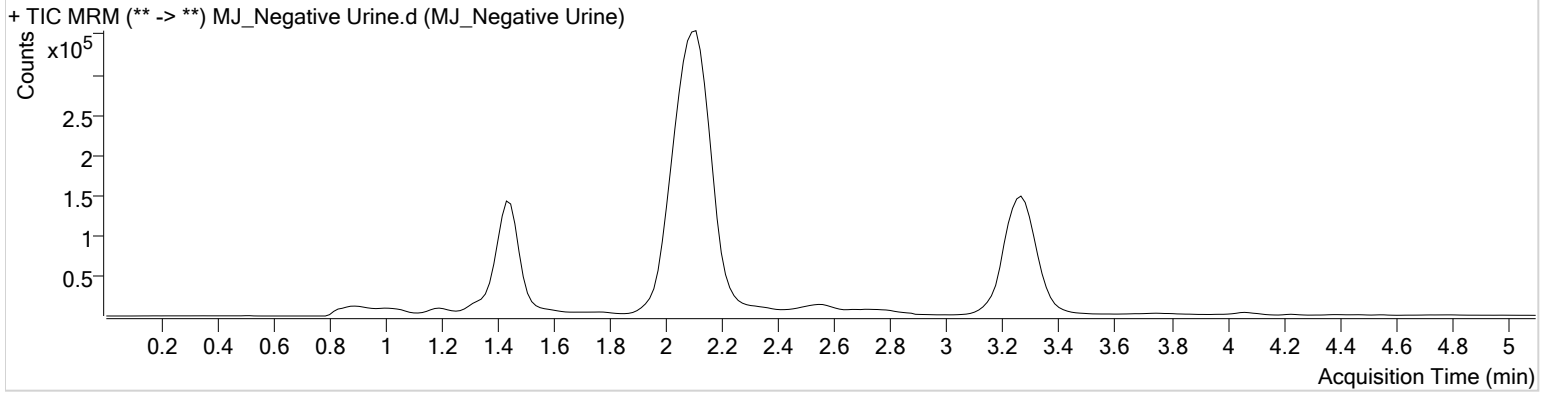


# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-B2	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 5:07:10 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



cg

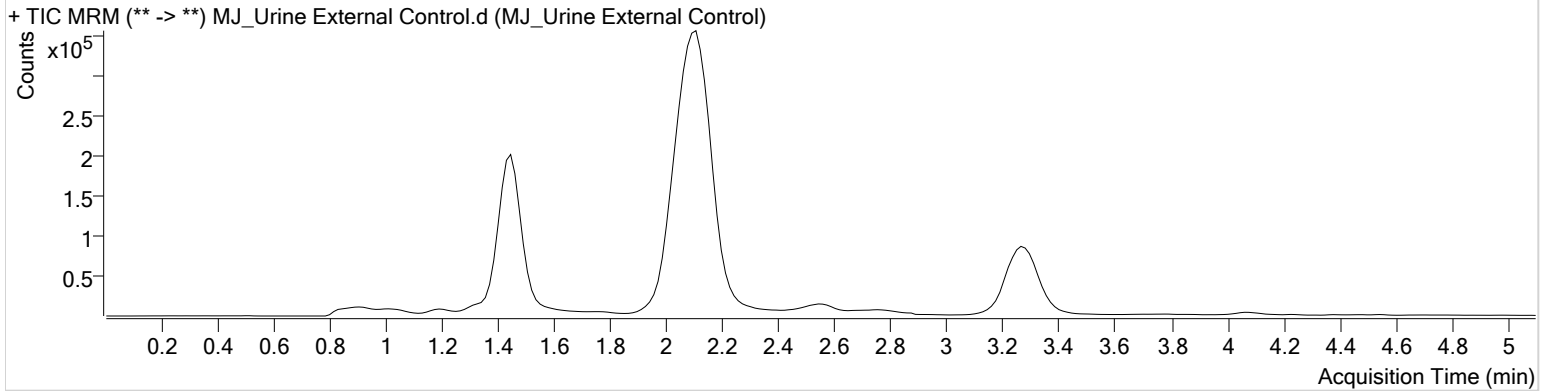


# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-C2	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 5:14:44 PM		

**Sample Chromatogram**

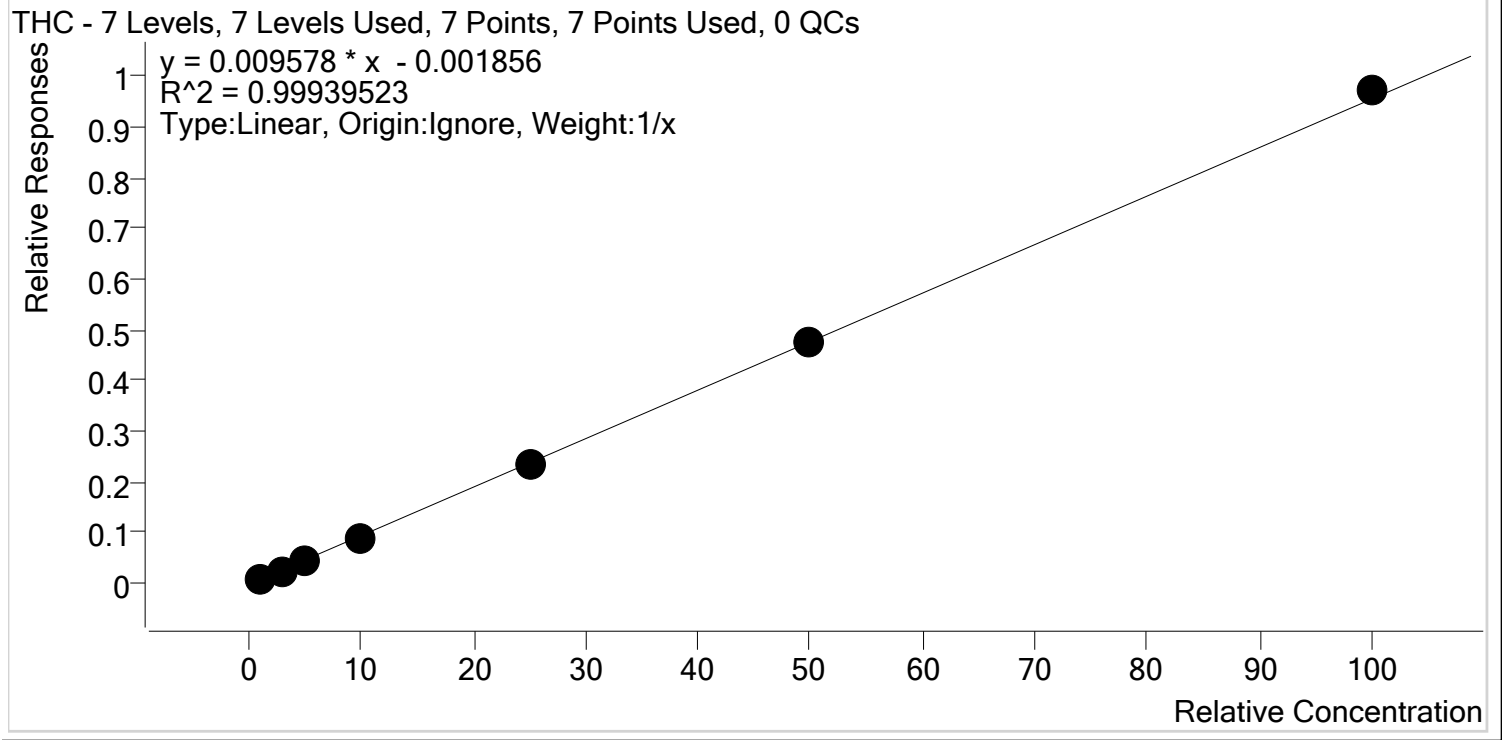


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.438	132950	418.47	13.1	∞	579982	10.8788 ng/ml
THC-COOH	1.474	93525	∞	51.4	∞	256706	16.2387 ng/ml
THC	3.285	70888	414.67	26.1	35.87	629994	11.9417 ng/ml



# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Last Cal. Update** 5/29/2020 10:52 AM  
**Analyst Name** ISP\Datastor  
**Analyte** THC **Internal Standard** THC-D3



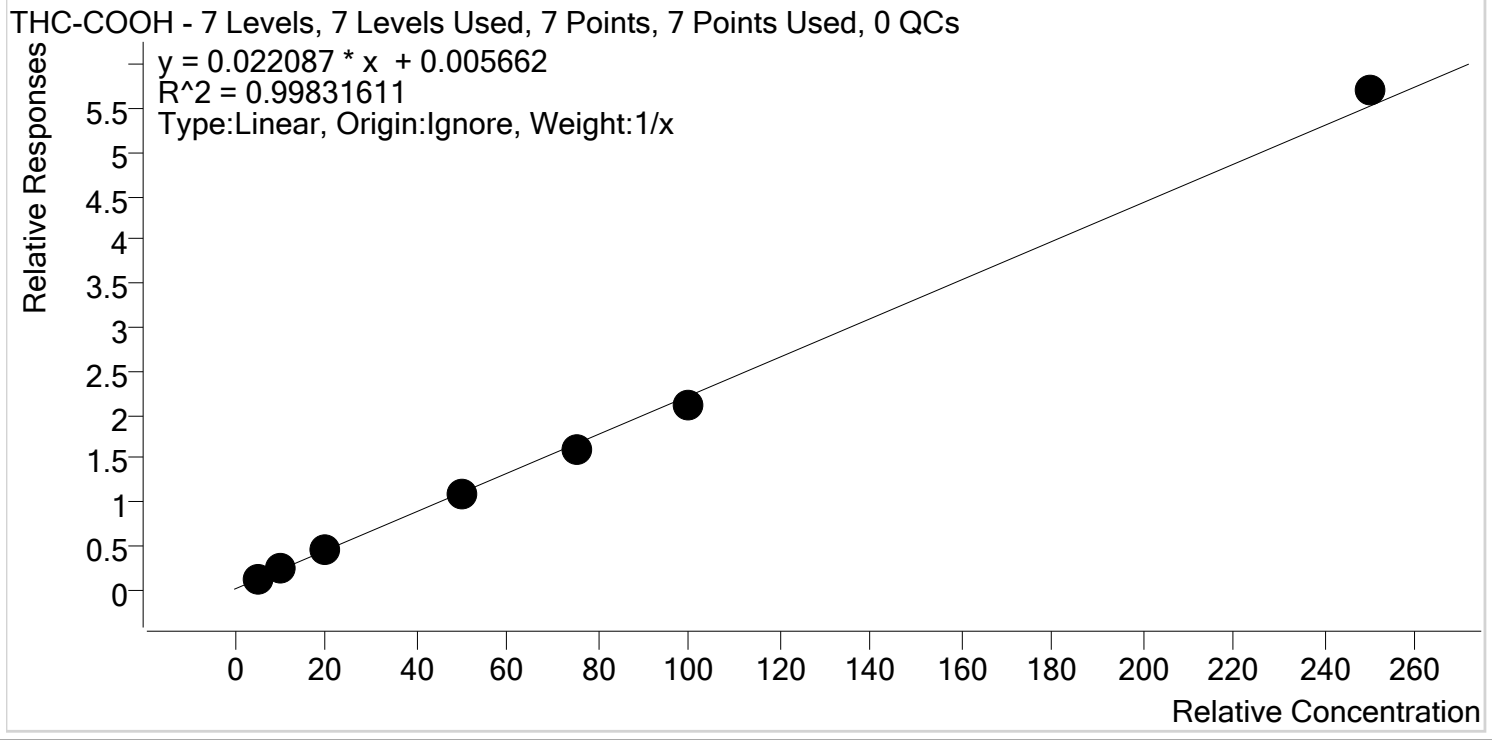
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	1.0	1.1	112.6
MJ_Cal 2	2	✓	3.0	2.9	96.3
MJ_Cal 3	3	✓	5.0	4.9	98.2
MJ_Cal 4	4	✓	10.0	9.5	94.6
MJ_Cal 5	5	✓	25.0	24.4	97.5
MJ_Cal 6	6	✓	50.0	49.5	99.1
MJ_Cal 7	7	✓	100.0	101.7	101.7





# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Last Cal. Update** 5/29/2020 10:52 AM  
**Analyst Name** ISP\Datastor  
**Analyte** THC-COOH **Internal Standard** THC-COOH-D9

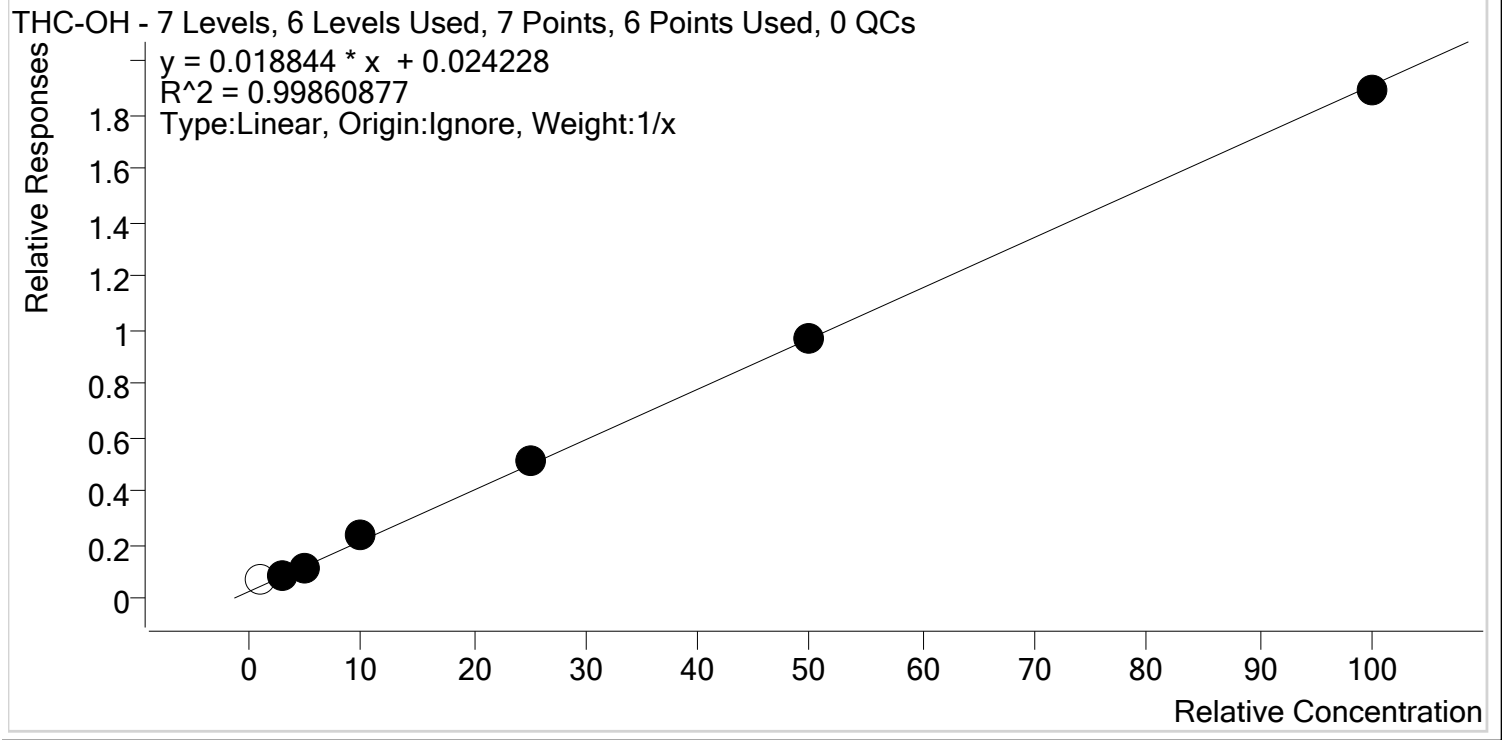


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	5.0	5.2	104.7
MJ_Cal 2	2	✓	10.0	10.5	104.8
MJ_Cal 3	3	✓	20.0	19.4	96.8
MJ_Cal 4	4	✓	50.0	49.6	99.2
MJ_Cal 5	5	✓	75.0	71.9	95.9
MJ_Cal 6	6	✓	100.0	95.4	95.4
MJ_Cal 7	7	✓	250.0	258.0	103.2



# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Last Cal. Update** 5/29/2020 10:52 AM  
**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	2.5	254.7
MJ_Cal 2	2	✓	3.0	2.8	94.7
MJ_Cal 3	3	✓	5.0	4.6	92.6
MJ_Cal 4	4	✓	10.0	11.1	111.3
MJ_Cal 5	5	✓	25.0	25.7	102.9
MJ_Cal 6	6	✓	50.0	49.7	99.5
MJ_Cal 7	7	✓	100.0	98.9	98.9

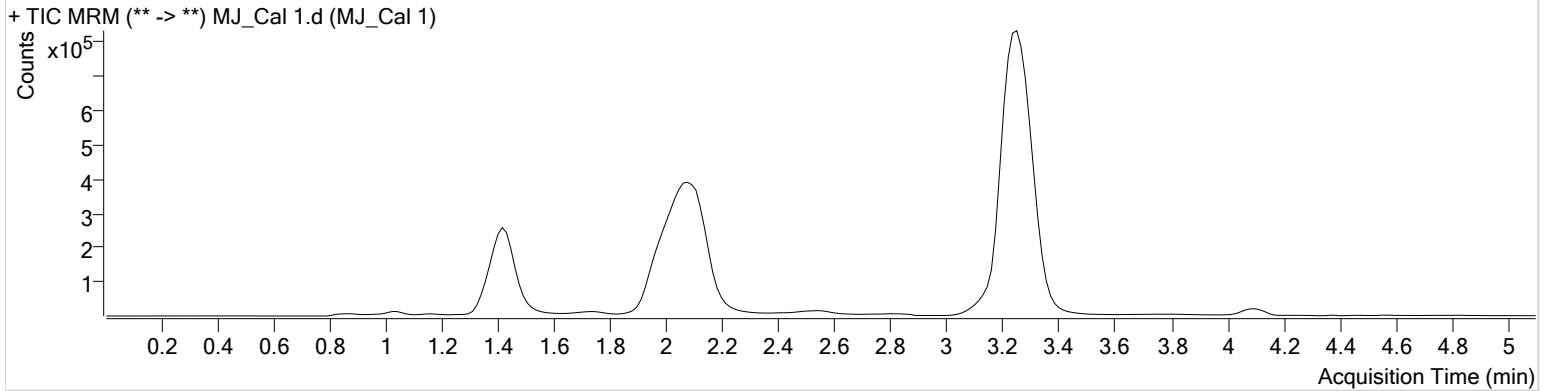


# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

**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** 5/27/2020 3:43:36 PM  
**Sample Info.**

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.483	78661	∞	4.2 <b>Low</b>	14.04	1089104	2.5471 ng/ml <b>Low</b>
THC-COOH	1.459	49523	∞	51.7	∞	408341	5.2345 ng/ml <b>Low</b>
THC	3.254	58088	177.88	29.1	92.48	6503024	1.1264 ng/ml <b>Low</b>

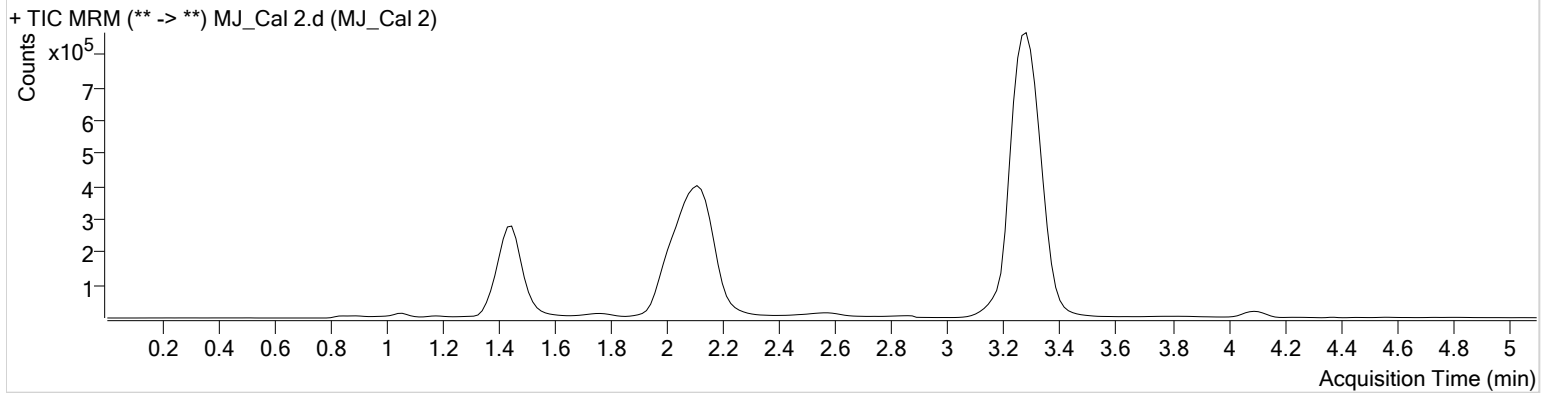
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-B1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 3:51:20 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	85083	∞	11.8	∞	1093839	2.8420 ng/ml <b>Low</b>
THC-COOH	1.474	95748	∞	49.5	∞	403587	10.4849 ng/ml
THC	3.300	166871	373.90	26.3	251.10	6460641	2.8905 ng/ml <b>Low</b>

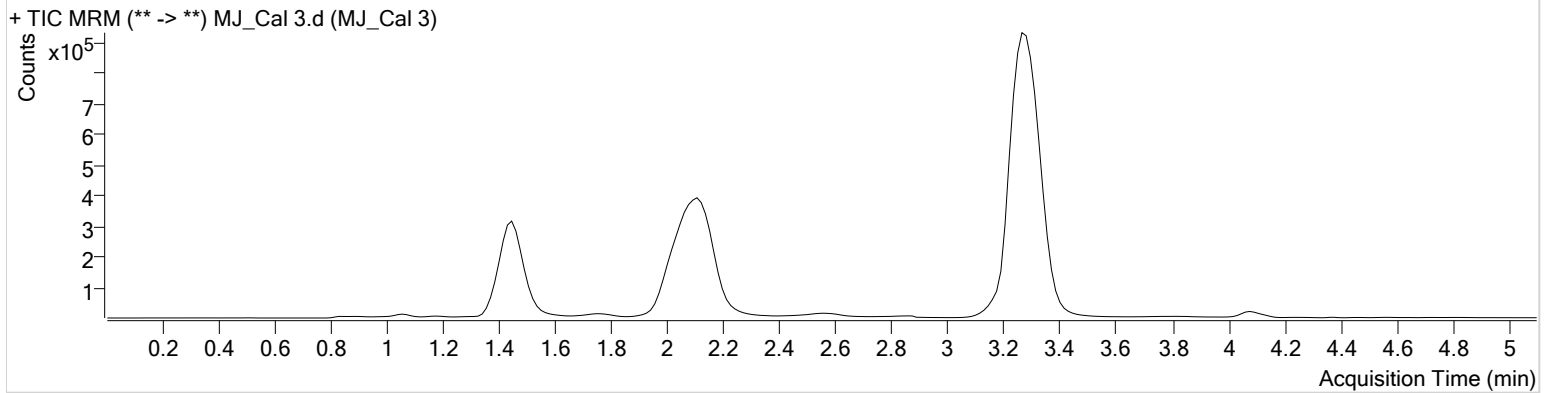
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-C1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 3:58:55 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	118784	∞	12.1	∞	1065212	4.6318 ng/ml
THC-COOH	1.474	172657	∞	60.0	∞	398667	19.3518 ng/ml
THC	3.285	301039	1423.26	25.5	226.73	6667566	4.9077 ng/ml

# AM #27 Cannabinoid Quant. Results

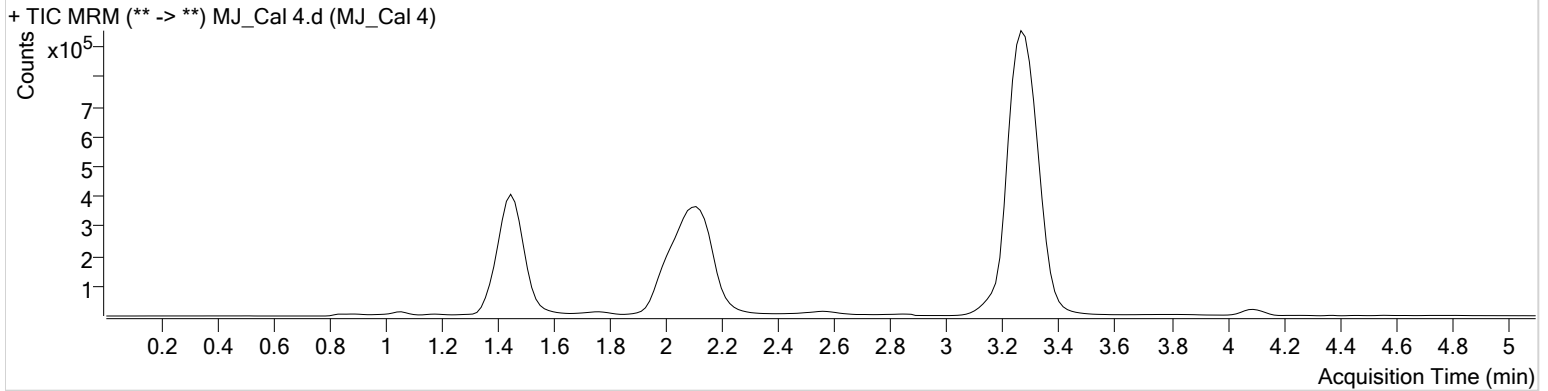


**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-D1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 4:06:29 PM		

**Sample Info.**

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.438	268159	∞	10.9	∞	1145755	11.1342 ng/ml
THC-COOH	1.474	459533	∞	59.8	∞	417368	49.5931 ng/ml
THC	3.285	587679	3588.43	24.9	487.25	6622384	9.4589 ng/ml

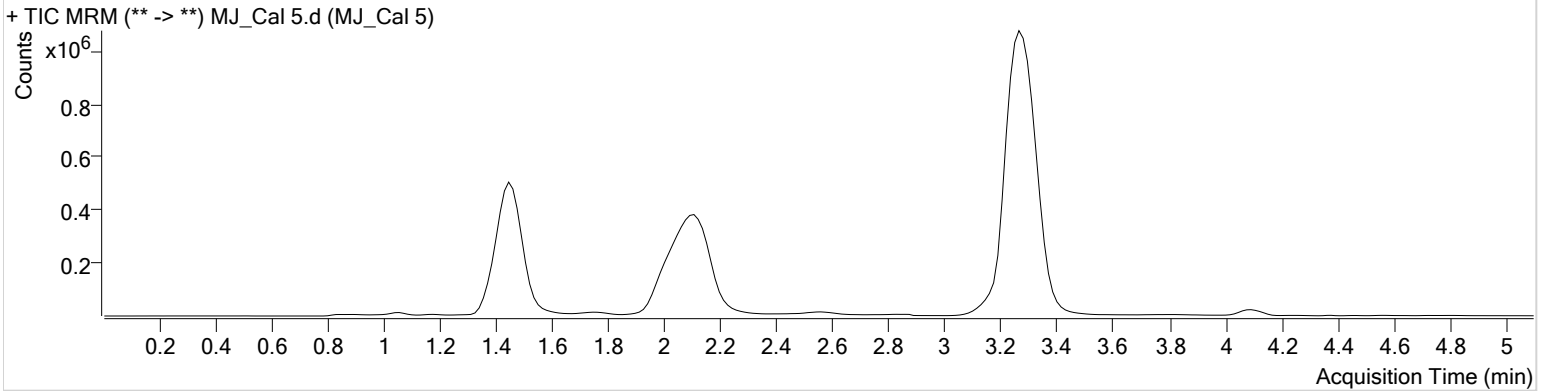
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-E1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 4:14:04 PM		

**Sample Chromatogram**



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.438	557156	∞	12.4	488.63	1094784	25.7206 ng/ml
THC-COOH	1.474	634639	∞	63.0	∞	397972	71.9437 ng/ml
THC	3.285	1494512	1577.40	25.6	3122.85	6454803	24.3674 ng/ml

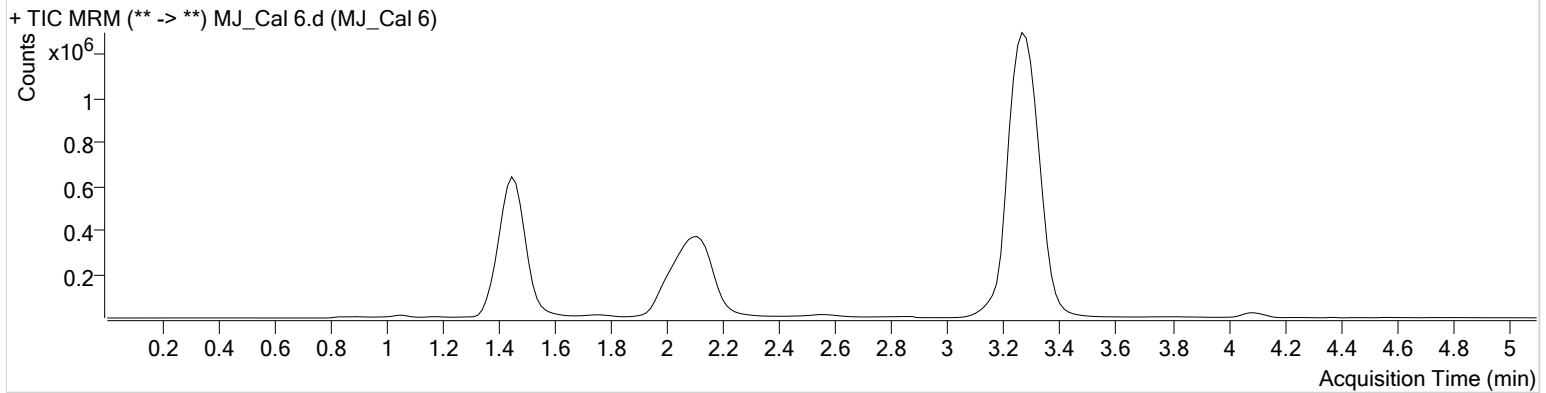
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-F1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 4:21:38 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.438	1041906	∞	13.1	6079.99	1083678	49.7348 ng/ml
THC-COOH	1.474	828215	∞	63.3	∞	392052	95.3888 ng/ml
THC	3.285	3006745	18440.23	25.2	2549.30	6360582	49.5480 ng/ml



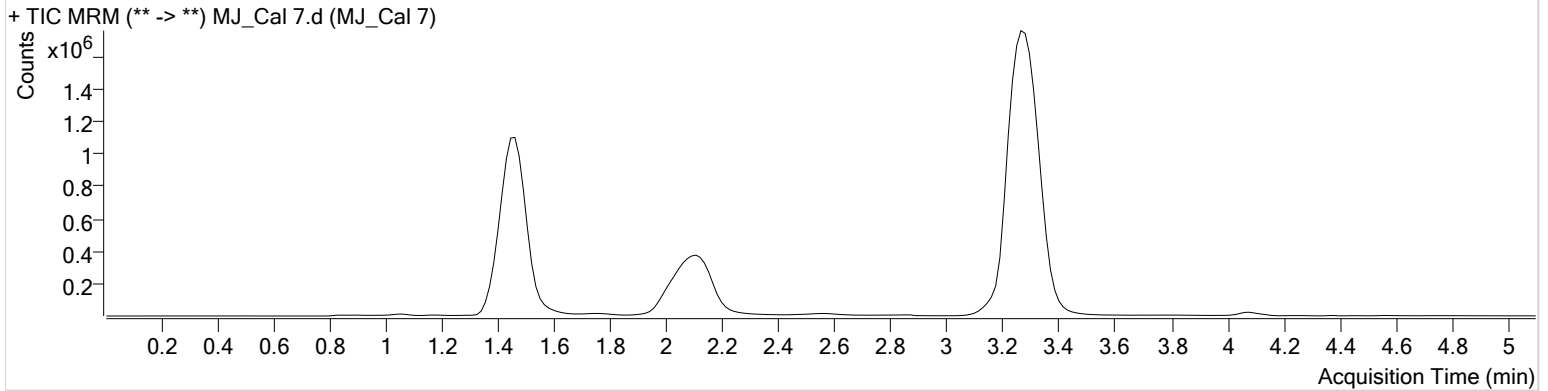
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\AM 27 28 052720 CS\QuantResults\AM 27.batch.bin  
**Calibration Last Update** 5/29/2020 10:52:29 AM

<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-G1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	5/27/2020 4:29:12 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.438	1916055	∞	12.3	∞	1014520	98.9365 ng/ml
THC-COOH	1.474	2021358	∞	56.8	∞	354364	258.0032 ng/ml
THC	3.285	6038499	3398.05	25.8	2638.13	6210927	101.7011 ng/ml