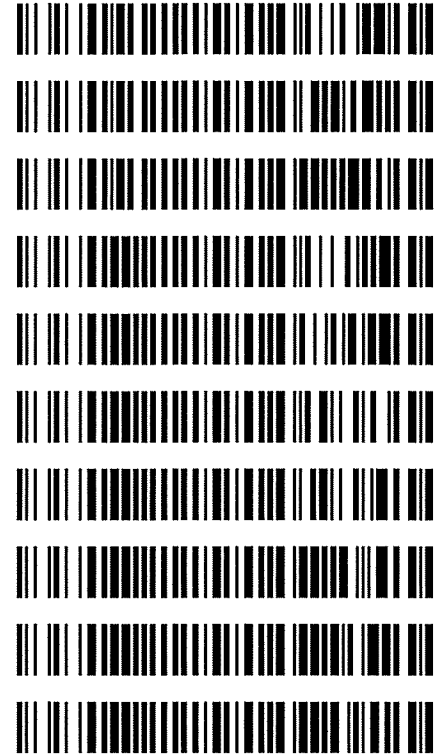


**Worklist: 3968**

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>
M2019-5719	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2020-0264	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2020-0290	2	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0104	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0159	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0172	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0191	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0248	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0249	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0250	1	BCK	AM 27 Blood THC Quant by LC-QQQ



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**Idaho State Police  
Forensic Services  
Toxicology Discipline**

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**Request for Departure from an Analytical Method**

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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 ToxBBox 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

TS

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

Extraction Date: 1/29/2020  
Plate lot#: IDP-108-190716

Analyst: Tamara Salazar  
Plate Expiration: 01/16/2020

**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: 42** 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\012920 AM 27 28 wklsts 3968 3969 TS  
Batch Name: THCQ wklst 3968 TS
- 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? 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-OH 3-100*



# Idaho State Police Forensic Services

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

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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 1  $\mu$ 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: 011620)

100  $\mu$ L of methanol external control solution was added to 9900  $\mu$ L of blood.  
Approximately 10 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:	01/16/2020	
Prepared by:	Tamara Salazar	
Expires:	09/30/2020	

TS



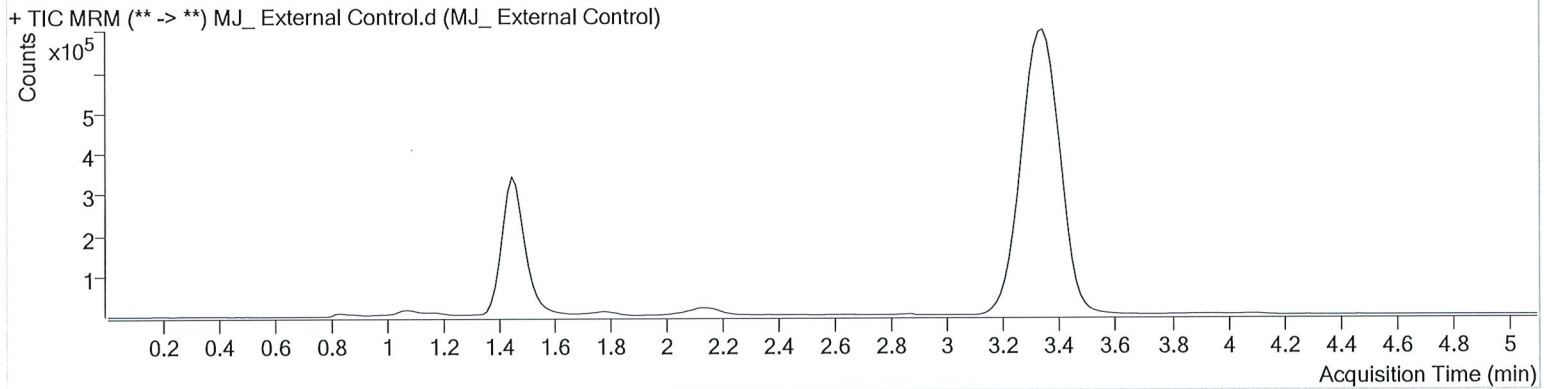
# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ wklst 3968 TS.batch.bin

**Calibration Last Update** 1/29/2020 4:38:32 PM

<b>Instrument</b>	Falco	<b>Data File</b>	MJ_ External Control.d
<b>Type</b>	Sample	<b>Sample</b>	MJ_ External Control
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Tamara Salazar
<b>Sample Position</b>	P3-B2	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 1:43:42 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	223872	∞	9.8	221.10	1237427	10.3345 ng/ml
THC-COOH	1.489	101358	∞	60.8	789.70	316184	14.2918 ng/ml
THC	3.360	431007	3046.91	27.0	∞	6195916	9.2108 ng/ml

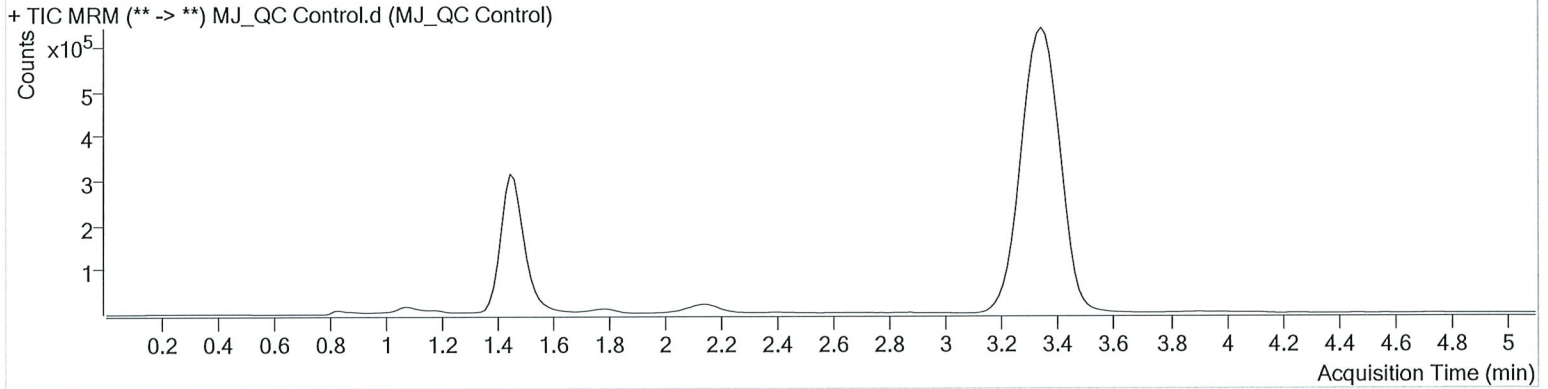
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ wklst 3968  
 TS.batch.bin  
**Calibration Last Update** 1/29/2020 4:38:32 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>	Tamara Salazar
<b>Sample Position</b>	P3-H1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 1:13:19 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	137248	∞	9.2	220.57	1215975	4.4941 ng/ml
THC-COOH	1.489	97011	217.51	60.9	2174.01	307625	14.0541 ng/ml
THC	3.360	197496	∞	27.4	616.91	5891622	4.4250 ng/ml

TS



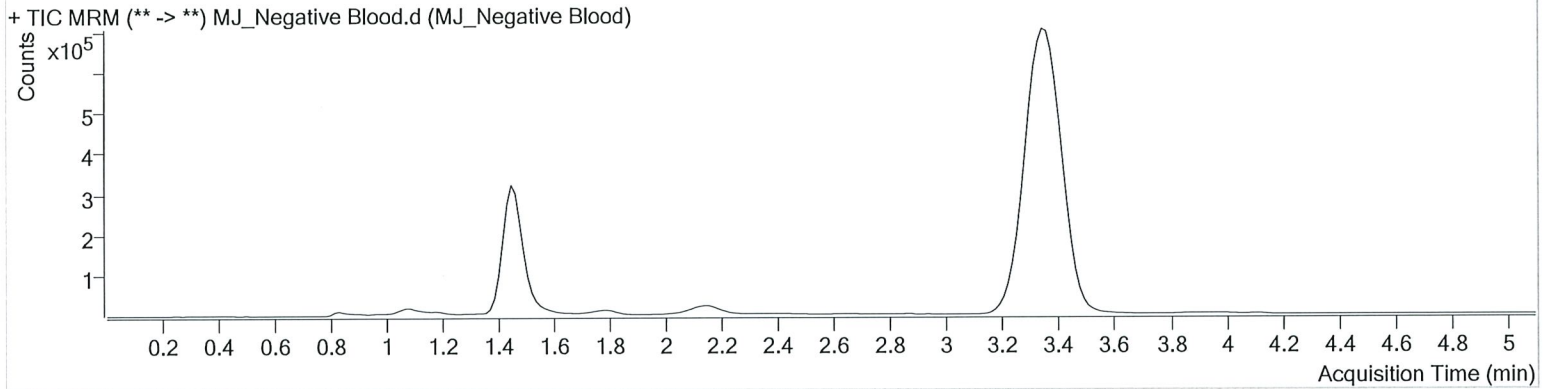
# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ wklst 3968  
TS.batch.bin

**Calibration Last Update** 1/29/2020 4:38:32 PM

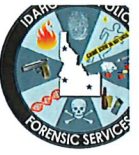
<b>Instrument</b>	Falco	<b>Data File</b>	MJ_Negative Blood.d
<b>Type</b>	Sample	<b>Sample</b>	MJ_Negative Blood
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Tamara Salazar
<b>Sample Position</b>	P3-A2	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 1:28:32 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



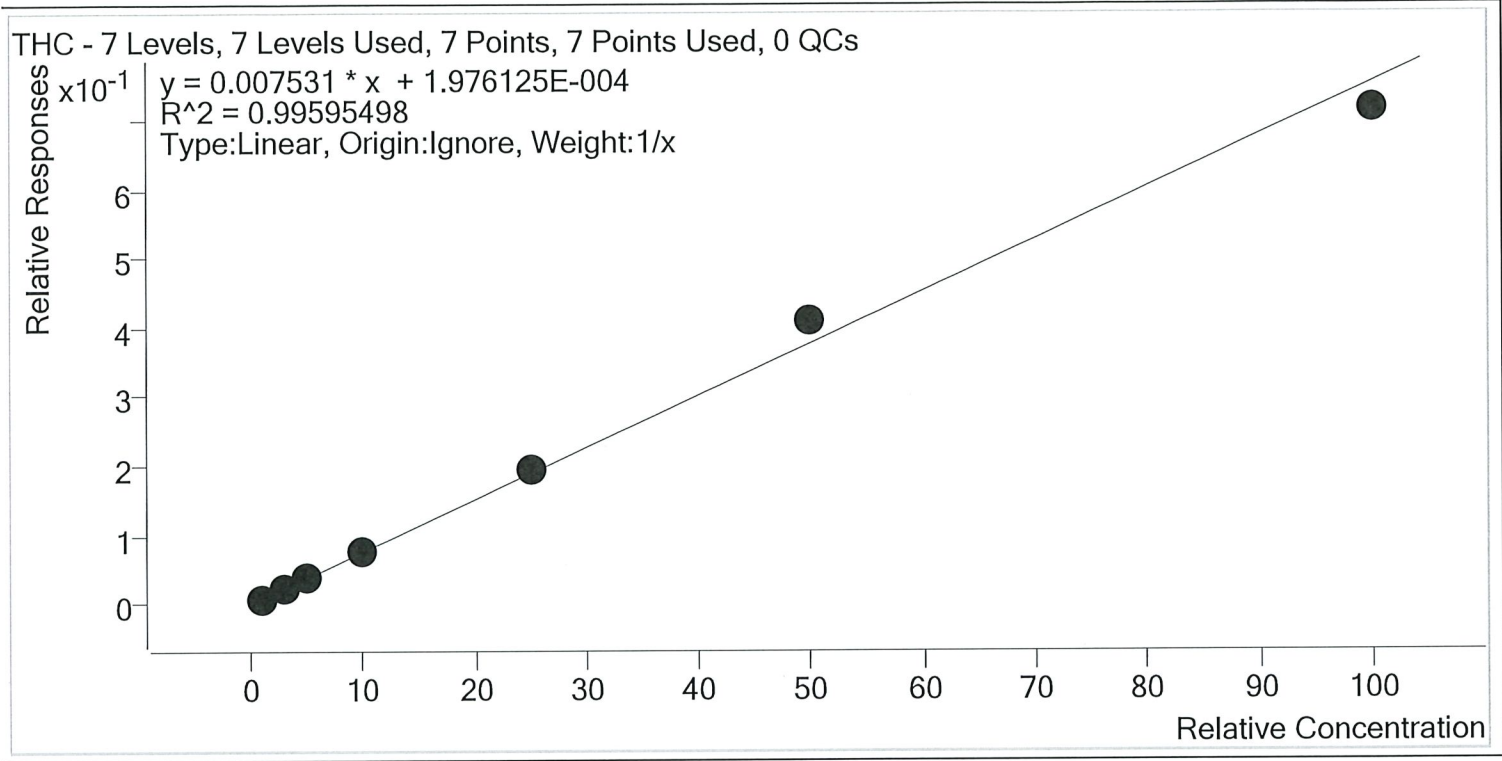


TS



# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ  
 wklst 3968 TS.batch.bin  
**Last Cal. Update** 1/29/2020 4:38 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	1.0	95.9
MJ Cal 2	2	✓	3.0	2.9	97.0
MJ Cal 3	3	✓	5.0	5.0	99.1
MJ Cal 4	4	✓	10.0	10.1	101.0
MJ Cal 5	5	✓	25.0	25.8	103.4
MJ Cal 6	6	✓	50.0	54.3	108.7
MJ Cal 7	7	✓	100.0	94.9	94.9



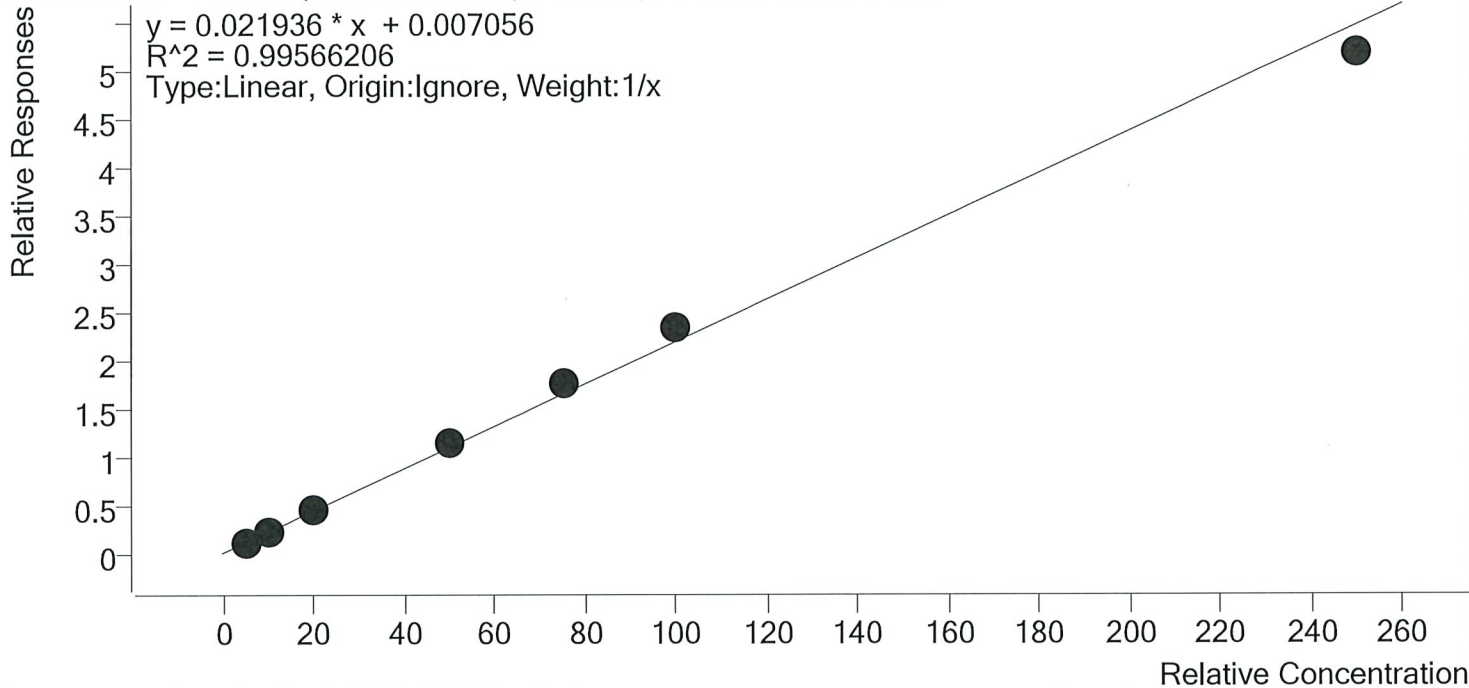
TS



# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ  
 wklt 3968 TS.batch.bin  
**Last Cal. Update** 1/29/2020 4:38 PM  
**Analyst Name** ISP\Datator  
**Analyte** THC-COOH **Internal Standard** THC-COOH-D9

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



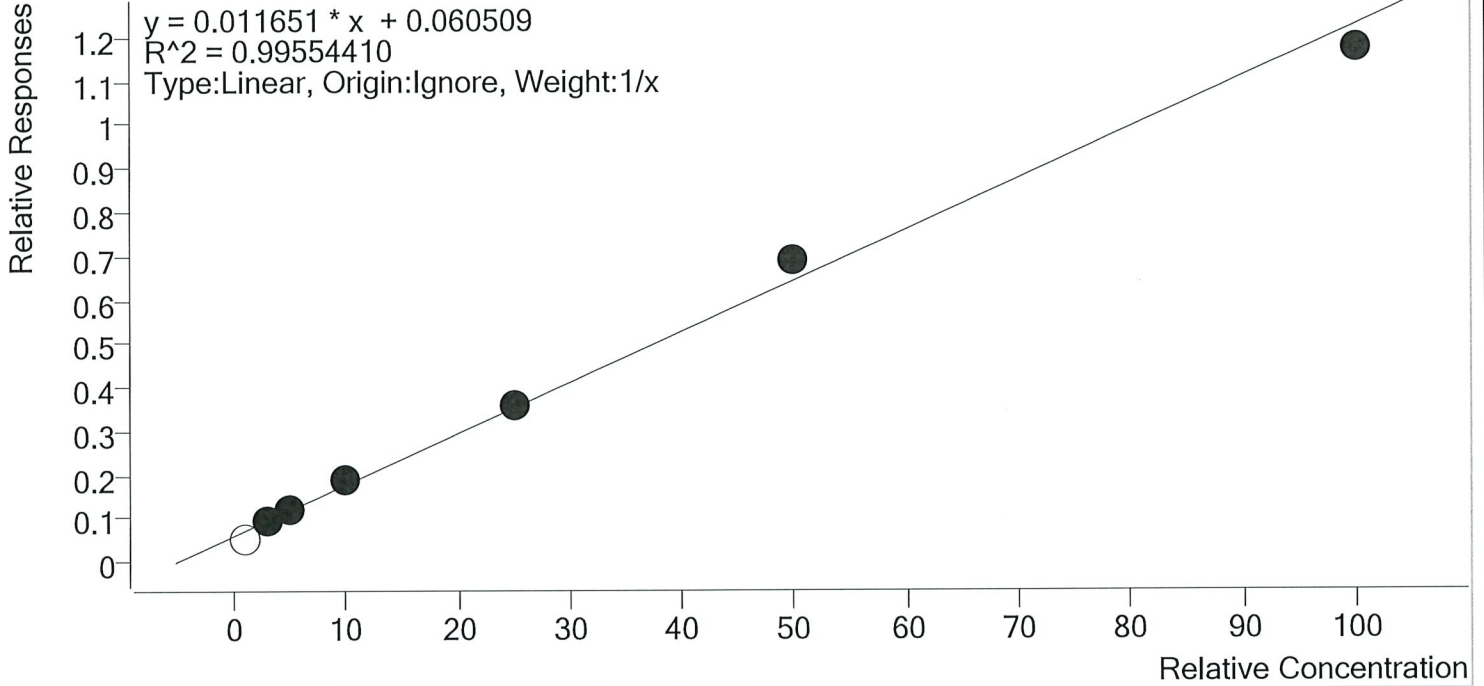
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ Cal 1	1	✓	5.0	4.7	93.8
MJ Cal 2	2	✓	10.0	9.4	93.8
MJ Cal 3	3	✓	20.0	20.1	100.3
MJ Cal 4	4	✓	50.0	51.7	103.4
MJ Cal 5	5	✓	75.0	79.9	106.5
MJ Cal 6	6	✓	100.0	107.5	107.5
MJ Cal 7	7	✓	250.0	236.8	94.7



# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ  
 wklst 3968 TS.batch.bin  
**Last Cal. Update** 1/29/2020 4:38 PM  
**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
MJ Cal 1	1	×	1.0	0.0	0.0
MJ Cal 2	2	✓	3.0	2.7	88.9
MJ Cal 3	3	✓	5.0	4.9	98.8
MJ Cal 4	4	✓	10.0	10.8	108.0
MJ Cal 5	5	✓	25.0	25.2	100.9
MJ Cal 6	6	✓	50.0	54.0	108.0
MJ Cal 7	7	✓	100.0	95.4	95.4

# AM #27 Cannabinoid Quant. Results

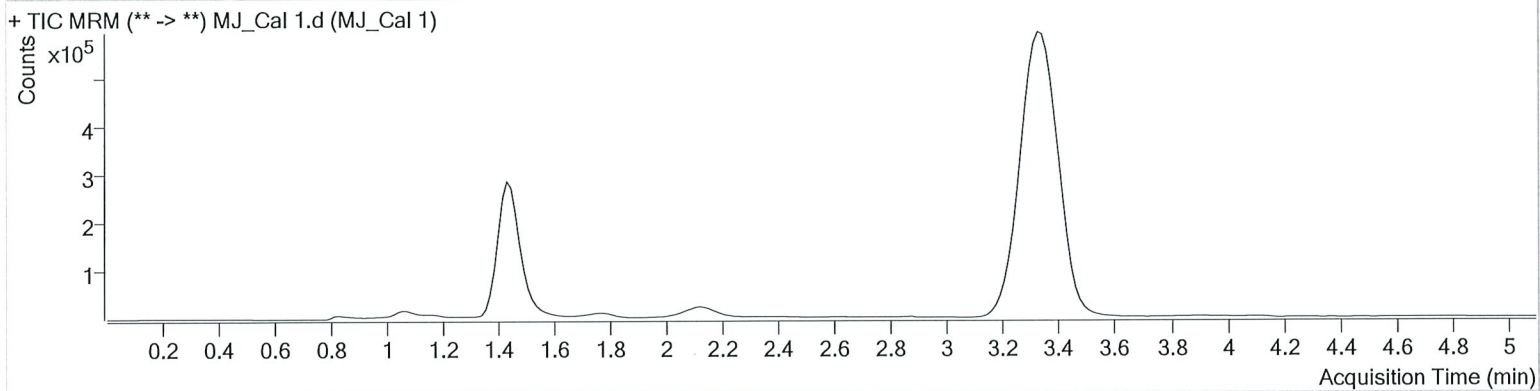
TS



**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ wklst 3968  
 TS.batch.bin  
**Calibration Last Update** 1/29/2020 4:38:32 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>	Tamara Salazar
<b>Sample Position</b>	P3-A1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 12:12:29 PM		
<b>Sample Info.</b>			

**Sample Chromatogram**



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	32944	∞	58.7	108.87	299690	4.6895 ng/ml <b>Low</b>
THC	3.330	41723	128.14	32.0	∞	5620838	0.9594 ng/ml <b>Low</b>

5



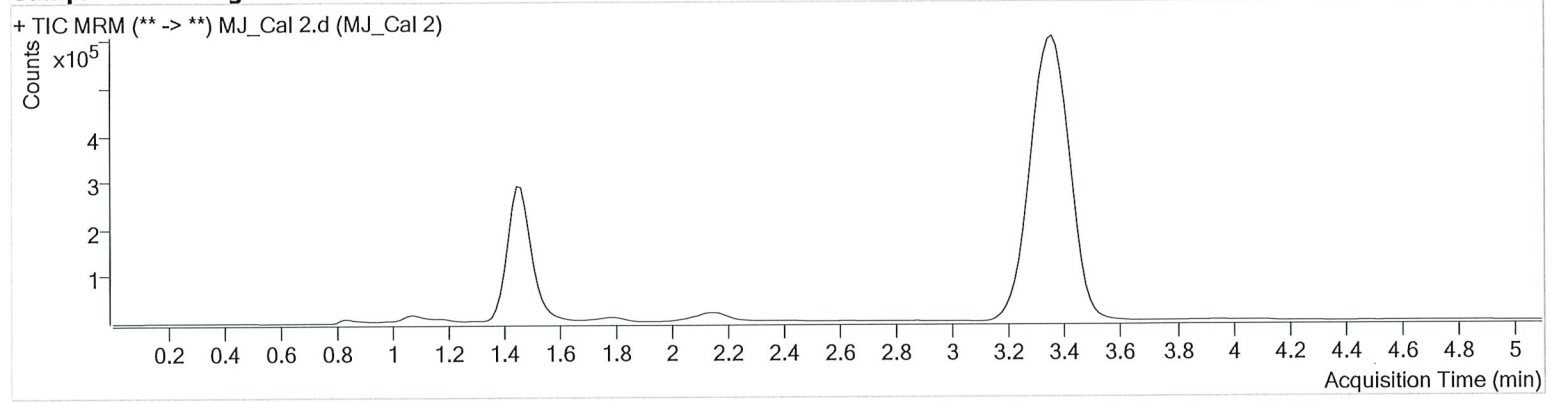
# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklists 3968 3969 TS\QuantResults\THCQ wklst 3968 TS.batch.bin

**Calibration Last Update** 1/29/2020 4:38:32 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>	Tamara Salazar
<b>Sample Position</b>	P3-B1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 12:20:14 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.483	111416	∞	8.8	77.81	1216422	2.6679 ng/ml <b>Low</b>
THC-COOH	1.489	65934	∞	60.5	289.04	309686	9.3839 ng/ml <b>Low</b>
THC	3.375	128235	659.24	27.8	174.10	5800983	2.9091 ng/ml <b>Low</b>

B



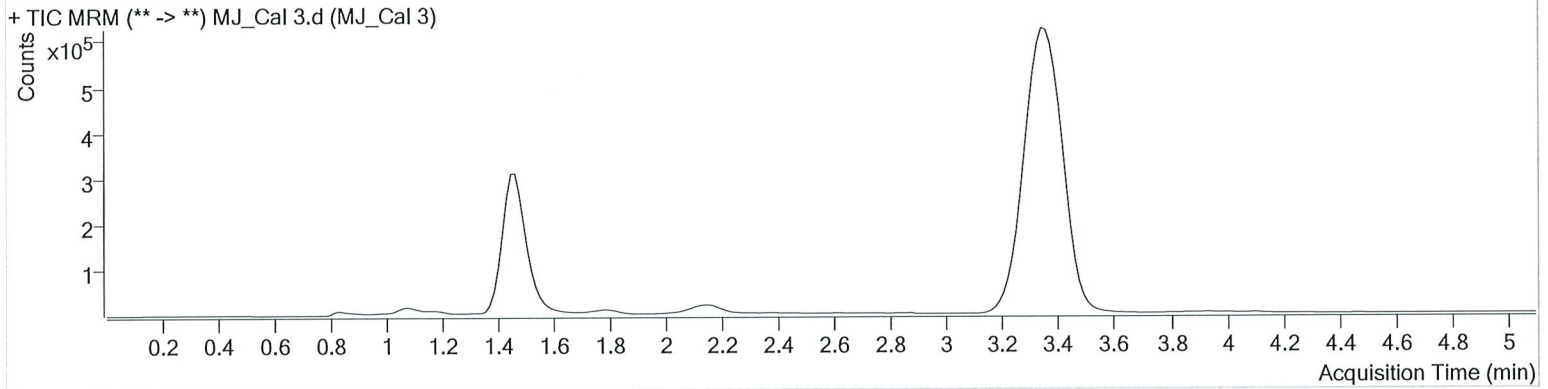
# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ wklst 3968 TS.batch.bin

**Calibration Last Update** 1/29/2020 4:38:32 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>	Tamara Salazar
<b>Sample Position</b>	P3-C1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 12:27:49 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	140372	∞	9.0	1088.80	1189075	4.9388 ng/ml
THC-COOH	1.489	132969	419.78	60.3	1998.49	297469	20.0555 ng/ml
THC	3.375	213612	1751.72	27.9	370.27	5691634	4.9573 ng/ml



B

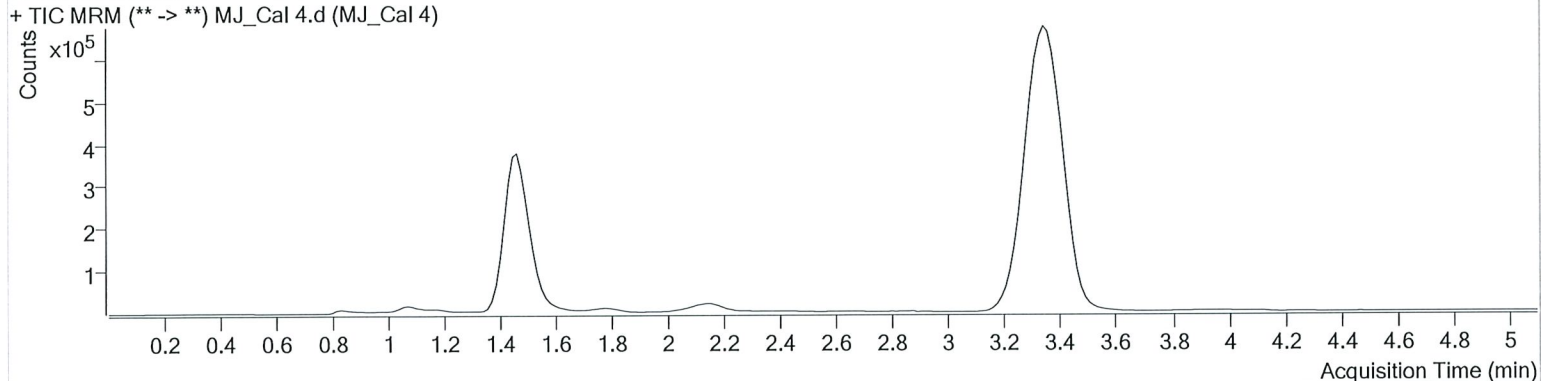


# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklists 3968 3969 TS\QuantResults\THCQ wklst 3968  
 TS.batch.bin  
**Calibration Last Update** 1/29/2020 4:38:32 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>	Tamara Salazar
<b>Sample Position</b>	P3-D1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 12:35:25 PM		
<b>Sample Info.</b>			

**Sample Chromatogram**



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	225882	∞	9.8	491.11	1211850	10.8045 ng/ml
THC-COOH	1.489	345153	∞	61.3	∞	302542	51.6849 ng/ml
THC	3.360	445089	1090.22	27.3	718.33	5836821	10.0994 ng/ml



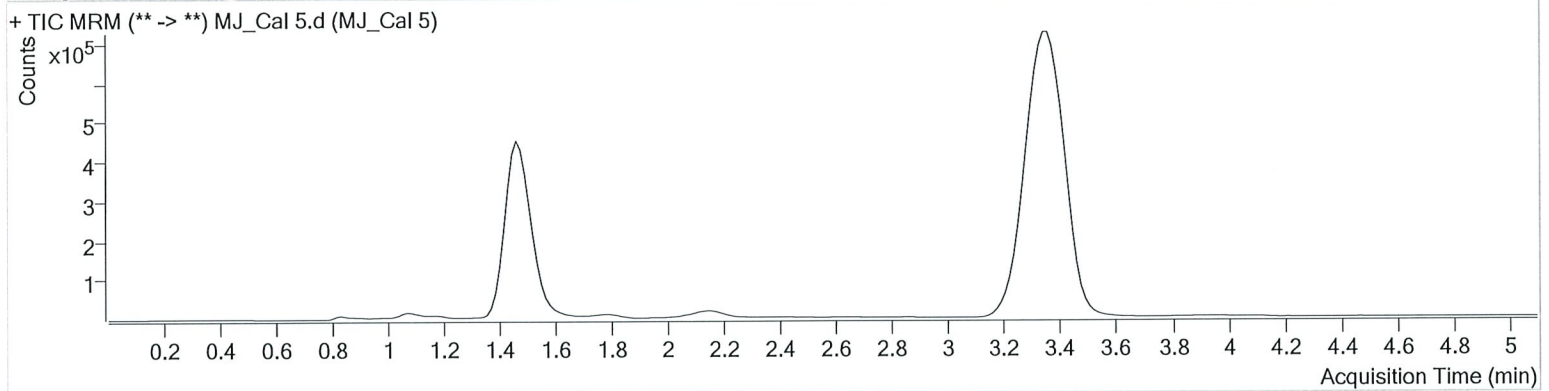
# AM #27 Cannabinoid Quant. Results



**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ wklst 3968  
 TS.batch.bin  
**Calibration Last Update** 1/29/2020 4:38:32 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>	Tamara Salazar
<b>Sample Position</b>	P3-E1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 12:42:59 PM		
<b>Sample Info.</b>			

**Sample Chromatogram**



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	423098	∞	12.6	389.94	1193796	25.2254 ng/ml
THC-COOH	1.489	525502	∞	59.1	3630.31	298607	79.9029 ng/ml
THC	3.375	1114726	3879.26	26.6	699.16	5721467	25.8448 ng/ml



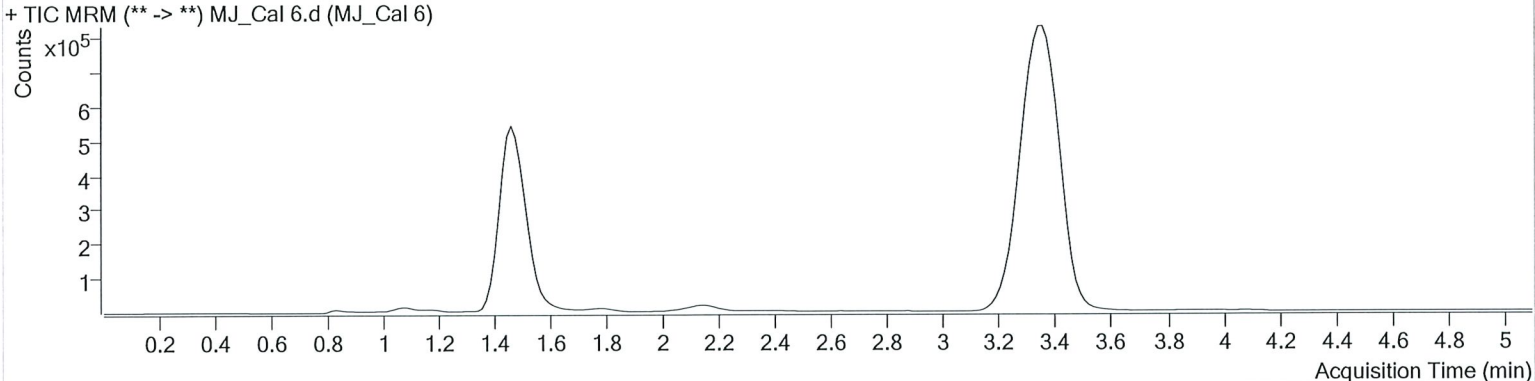
# AM #27 Cannabinoid Quant. Results

**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ wklst 3968  
TS.batch.bin

**Calibration Last Update** 1/29/2020 4:38:32 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>	Tamara Salazar
<b>Sample Position</b>	P3-F1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	1/29/2020 12:50:34 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	781763	∞	12.6	2625.88	1133873	53.9821 ng/ml
THC-COOH	1.489	661657	∞	58.9	2636.59	279858	107.4561 ng/ml
THC	3.360	2177835	7794.50	25.6	2916.98	5319617	54.3361 ng/ml

15



# AM #27 Cannabinoid Quant. Results

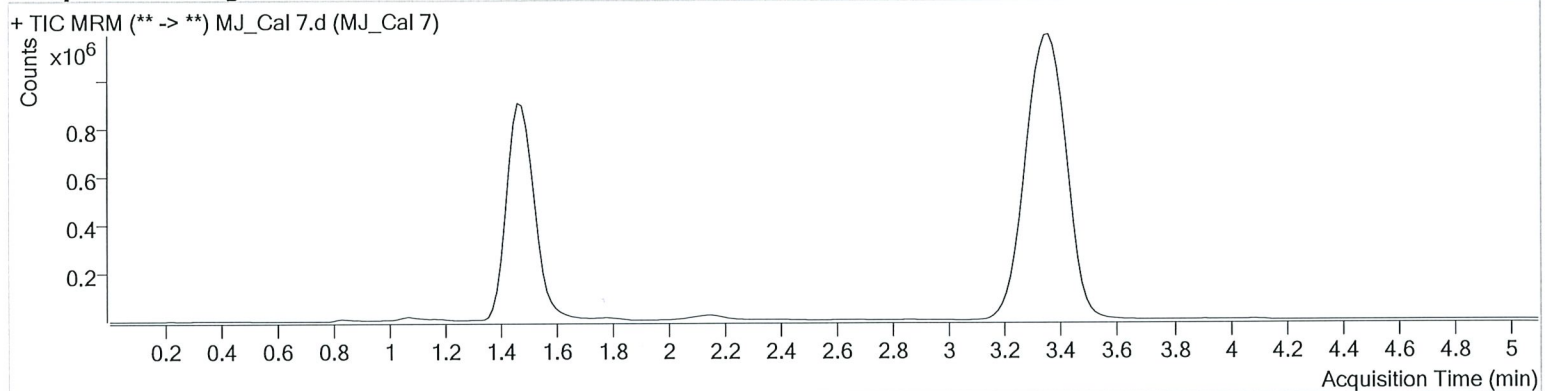
**Batch results** D:\MassHunter\Data\2020\AM 27-28\012920 AM 27 28 wklsts 3968 3969 TS\QuantResults\THCQ wklst 3968  
TS.batch.bin

**Calibration Last Update** 1/29/2020 4:38:32 PM

**Instrument** Falco  
**Type** Cal  
**Acq. Method** AM 27 THC quant.m  
**Sample Position** P3-G1  
**Injection Volume** 10  
**Acq. Date-Time** 1/29/2020 12:58:08 PM  
**Sample Info.**

**Data File** MJ\_Cal 7.d  
**Sample** MJ\_Cal 7  
**Operator** Tamara Salazar  
**Comment**

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	1482969	∞	12.7	4304.95	1265537	95.3812 ng/ml
THC-COOH	1.489	1547162	4097.63	60.3	∞	297404	236.8273 ng/ml
THC	3.360	4506744	∞	26.6	14254.5 1	6304623	94.8937 ng/ml