









**REVIEWED**  
By Sarah Pickle at 3:03 pm, Mar 11, 2020

3/10/2020 BW

**Worklist: 4076**

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
C2020-0367	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0371	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0381	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0382	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0388	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0418	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0419	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0421	1	BCK	AM 27 Blood THC Quant by LC-QQQ	

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

Extraction Date: 3/10/20  
Plate lot#: 190716

Analyst: Britany Wylie  
Plate Expiration: 1-16-2020

**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:** 20A52255 **Urine Blank:** 11420

**Column:** UCT Selectra DA 100 x 2.1mm 3um

**LCMS-QQQ ID:** 69679

### 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 1.5 ml urine to blank plate, add 250 ul 1N KOH mix and incubate at 40 degrees for 15 minutes.  
Pipette **1000µL blood (calibrated pipette) Pipette ID: k52558g** in wells of analytical (standards) plate.
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 66759*
- 4. Pipette **500µL 0.1% formic acid in water blood sample** 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: 66792*
- 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: 66819*
- 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 blood), 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 is it 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: *Bloods only in this batch:  
curves limited: 3-100- THC*

**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 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

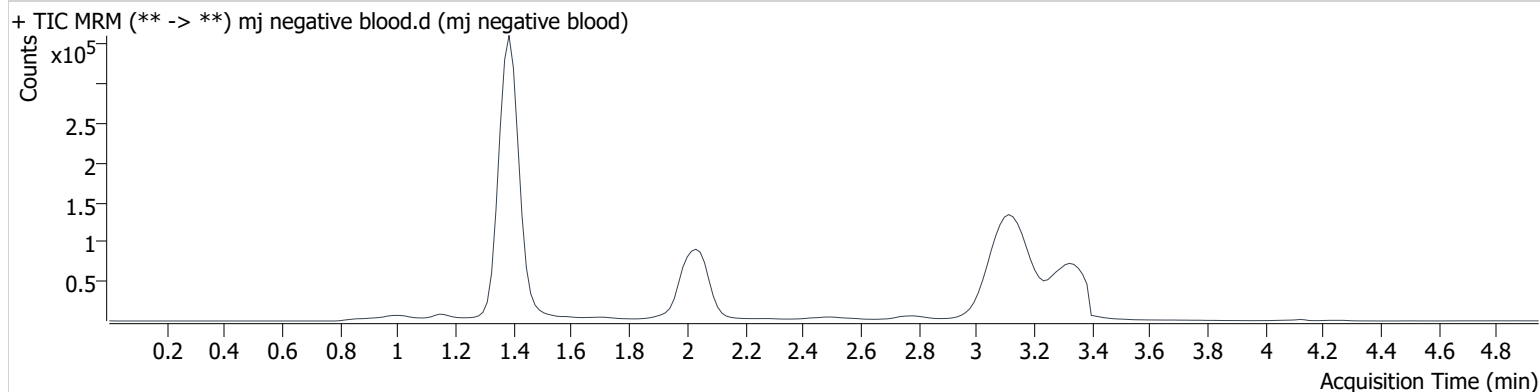
Toxicology Discipline Lead

# AM #27 Cannabinoids

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<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>	Britany Wylie
<b>Sample Position</b>	P3-A2	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 2:07:05 PM		
<b>Sample Info.</b>			

**Sample Chromatogram**



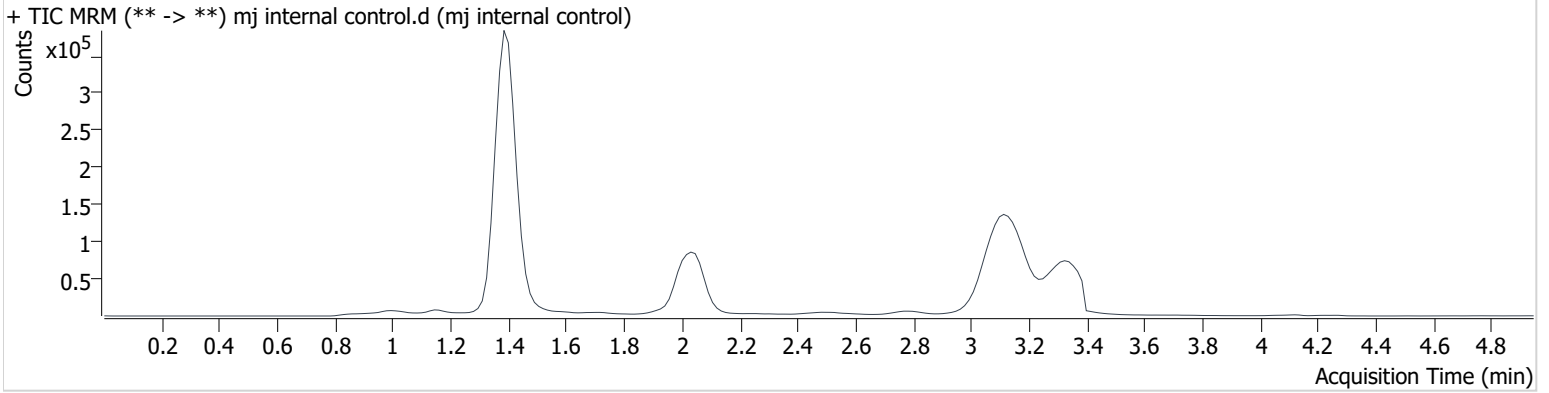
# AM #27 Cannabinoids

BW

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<b>Data File</b>	mj internal control.d
<b>Type</b>	QC	<b>Sample</b>	mj internal control
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Britany Wylie
<b>Sample Position</b>	P3-H1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 1:59:23 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	69128	∞	10.4	∞	1094756	4.492 ng/ml
THC-COOH	1.415	90974	972.9	200.5	119.6	535020	14.135 ng/ml
THC	3.153	20691	∞	28.7	∞	667331	4.543 ng/ml

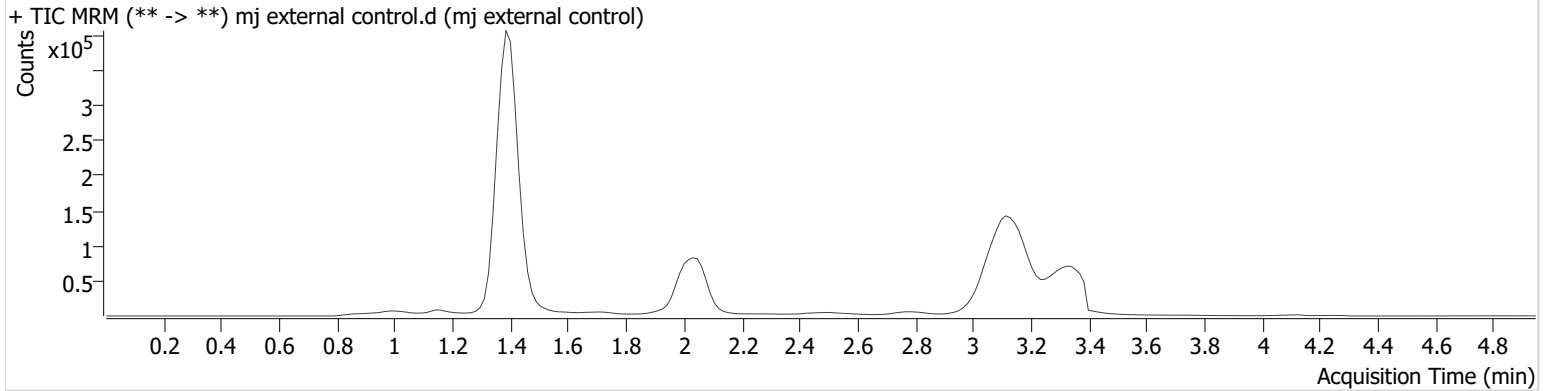
# AM #27 Cannabinoids

BW

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<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>	Britany Wylie
<b>Sample Position</b>	P3-B2	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 2:22:25 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	127437	∞	10.1	∞	1154233	7.782 ng/ml
THC-COOH	1.415	96481	1133.4	206.2	1477.5	566756	14.151 ng/ml
THC	3.153	39240	∞	26.4	∞	701773	7.625 ng/ml

**Toxicology AM method 27/26 external prep information**

working solution 1 ug/ml in meoh C-THC, THC-OH, THC

Stock solution 1mg/ml 7.5 ul each THC, 100 ug/ml 150 ul C-THC, 75 ul THC-OH in 9767.5 ul meOH

Ppd 2/13/20 Exp: 8/13/20 lot 21320 by AMN

Drug	lot	expiration
C-THC	FE07171501	9/1/2020
THC-OH	FE07721601	7/1/2021
THC	FE001041701	3/1/2022

AM 27/26 blood control 100 ul working solution lot (91319) in 9900 ul blood lot (20A52255)

ppd 02/13/20 Exp 08/13/20 lot b81320 Concentration 7.5 ng/ml THC, THC-OH and 15 ng/ml C-THC by AMN

AM 27/26 urine control 400 ul working solution lot (21320) in 9600 ul urine lot (11420)

ppd 02/13/20 Exp 08/13/20 lot u81320 Concentration 30 ng/ml THC, THC-OH and 60 ng/ml C-THC by AMN





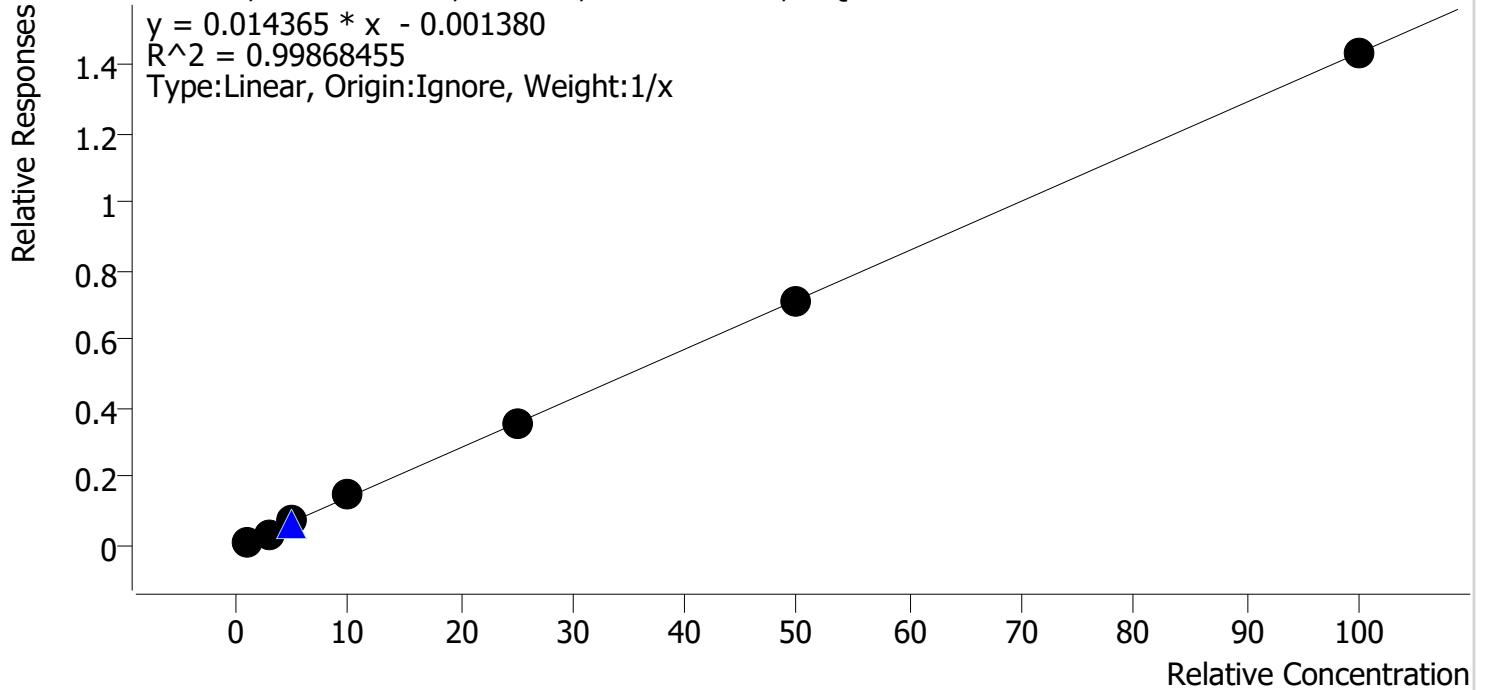


# Compound Calibration Report

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Last Cal. Update** 3/11/2020 8:15 AM  
**Analyst Name** ISP\datastor  
**Analyte** THC-OH *BW*

**Internal Standard** THC-OH-d3

THC-OH - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 1 QCs



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj qc1	1	✓	1.0	1.0	99.2
mj cal2	2	✓	3.0	2.5	83.4
mj cal 3	3	✓	5.0	5.6	111.3
mj cal 4	4	✓	10.0	10.8	108.0
mj cal 5	5	✓	25.0	24.9	99.6
mj cal 6	6	✓	50.0	49.4	98.9
mj cal 7	7	✓	100.0	99.8	99.8

# AM #27 Cannabinoids

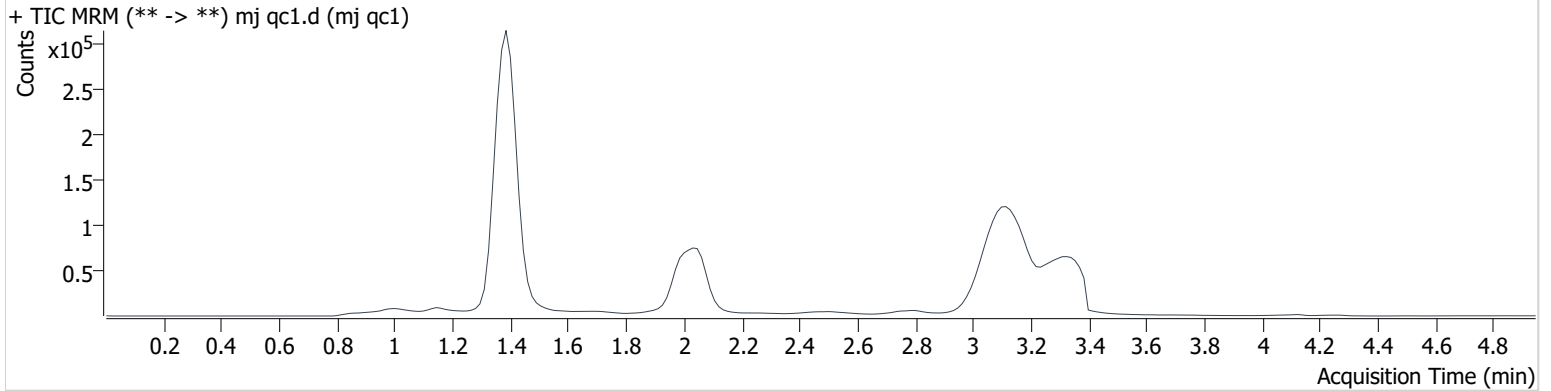
BW

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<b>Data File</b>	mj qc1.d
<b>Type</b>	Cal	<b>Sample</b>	mj qc1
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Britany Wylie
<b>Sample Position</b>	P3-G1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 1:05:20 PM		

**Sample Info.**

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	13394	∞	12.6	∞	1041179	0.992 ng/ml <b>Low</b>
THC-COOH	1.415	33847	57.9	232.8	54616.0	511711	5.751 ng/ml <b>Low</b>
THC	3.168	4385	∞	34.9 <b>High</b>	1065052 481032. 2	623444	1.576 ng/ml <b>Low</b>

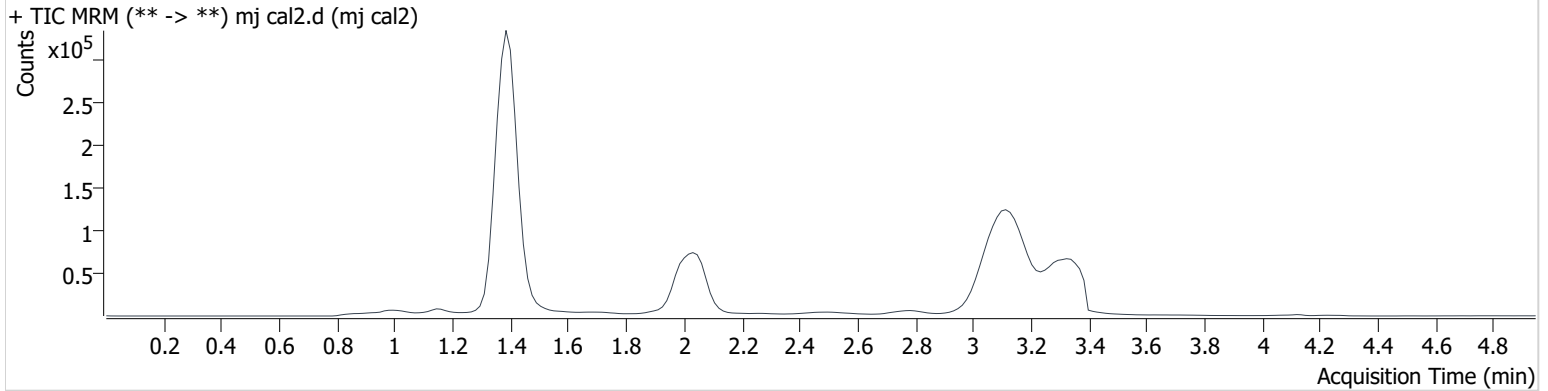
# AM #27 Cannabinoids

BW

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<b>Data File</b>	mj cal2.d
<b>Type</b>	Cal	<b>Sample</b>	mj cal2
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Operator</b>	Britany Wylie
<b>Sample Position</b>	P3-F1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 1:13:05 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	35904	∞	12.7	∞	1039510	2.501 ng/ml <b>Low</b>
THC-COOH	1.415	56454	72.8	226.6	225.6	512938	9.295 ng/ml <b>Low</b>
THC	3.153	12921	∞	27.6	∞	629621	3.245 ng/ml

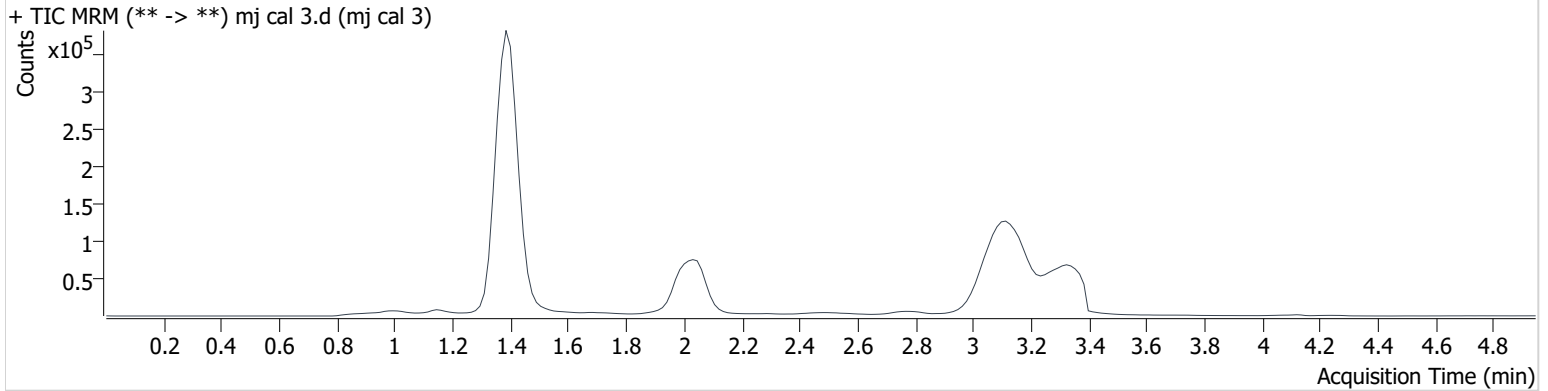
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BW

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<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>	Britany Wylie
<b>Sample Position</b>	P3-E1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 1:20:47 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	85226	∞	9.4	∞	1085029	5.564 ng/ml
THC-COOH	1.415	121139	163.5	195.4	221.4	527416	18.948 ng/ml
THC	3.168	22328	∞	27.2	∞	647444	4.973 ng/ml

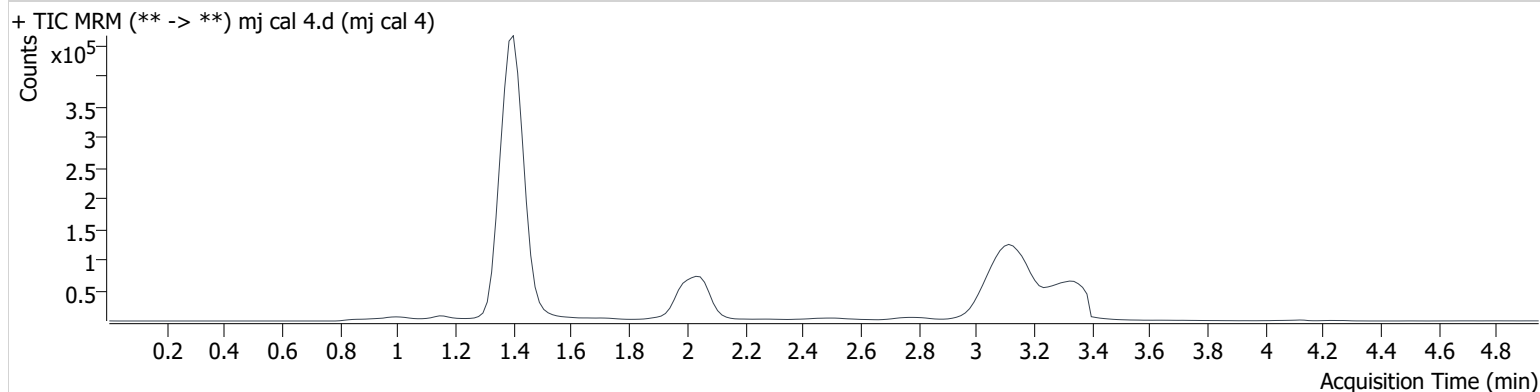
# AM #27 Cannabinoids

BW

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<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>	Britany Wylie
<b>Sample Position</b>	P3-D1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 1:28:29 PM		
<b>Sample Info.</b>			

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	160781	∞	10.3	∞	1045945	10.797 ng/ml
THC-COOH	1.415	307280	361.0	185.6	504.3	506967	49.325 ng/ml
THC	3.153	45507	∞	27.3	∞	618897	9.804 ng/ml

# AM #27 Cannabinoids

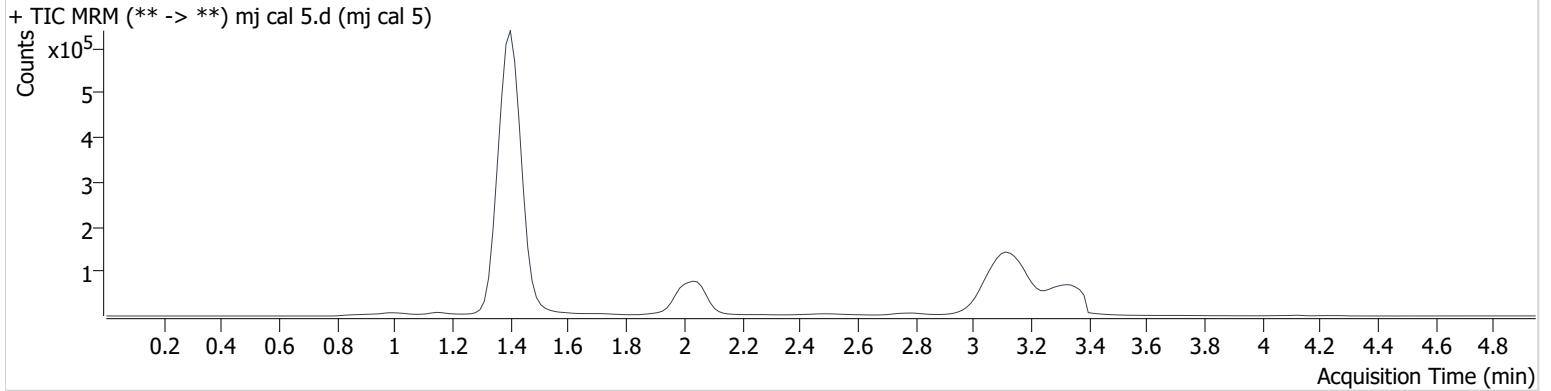
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**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<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>	Britany Wylie
<b>Sample Position</b>	P3-C1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 1:36:13 PM		

**Sample Info.**

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	400017	∞	10.9	∞	1123226	24.888 ng/ml
THC-COOH	1.415	477943	6023.5	184.5	263.2	527457	73.536 ng/ml
THC	3.168	121767	∞	25.3	∞	663055	23.431 ng/ml

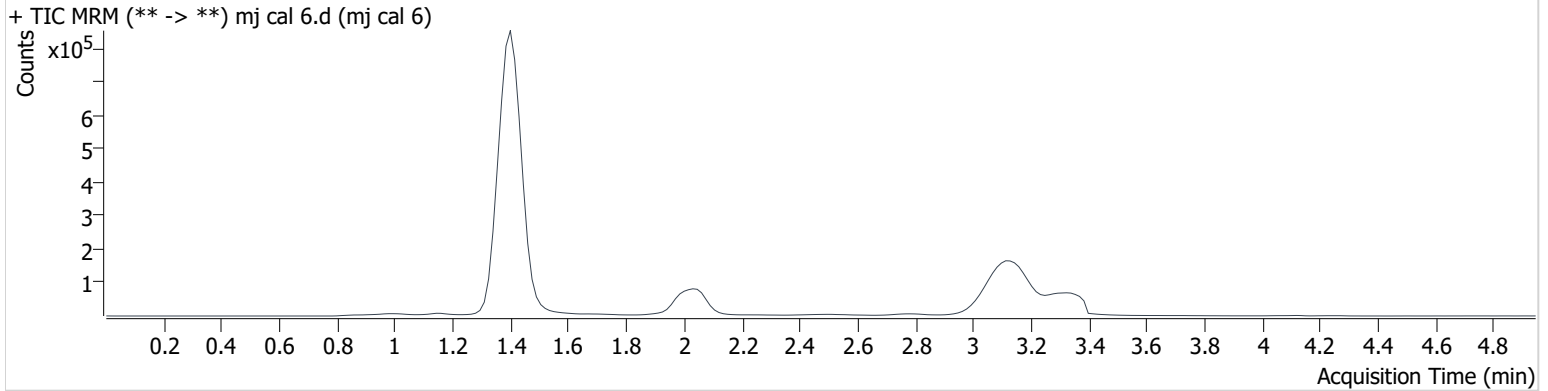
# AM #27 Cannabinoids

BW

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<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>	Britany Wylie
<b>Sample Position</b>	P3-B1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 1:43:55 PM		

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	863043	∞	11.6	∞	1217797	49.430 ng/ml
THC-COOH	1.415	672728	2052.9	179.0	581.0	551325	98.881 ng/ml
THC	3.153	277580	∞	24.5	∞	710562	49.045 ng/ml



# AM #27 Cannabinoids

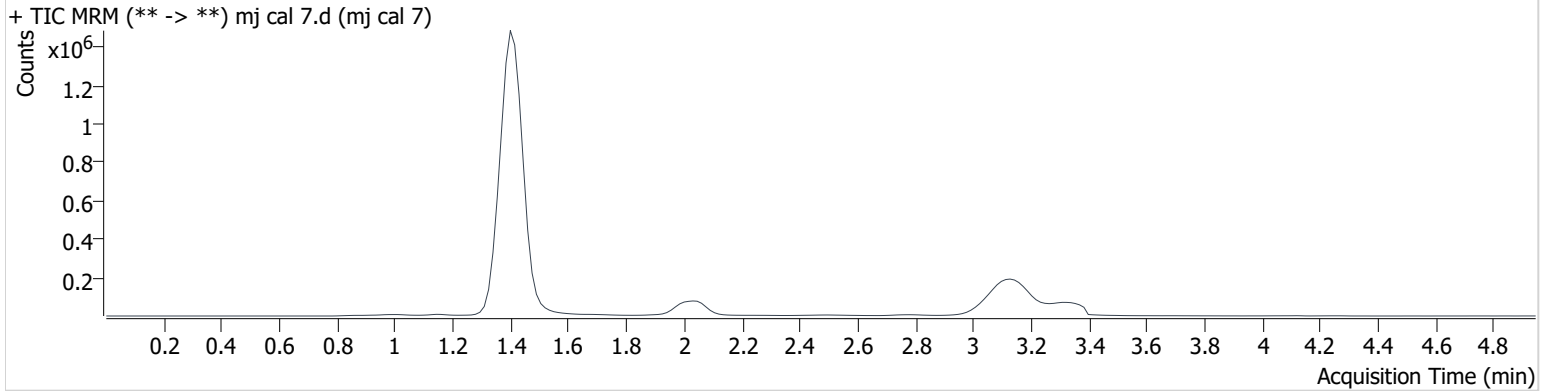
BW

**Batch results** D:\MassHunter\Data\2020 Data\am 27-28 3-10-2020\QuantResults\thcq.batch.bin  
**Calibration Last Update** 3/11/2020 8:15:49 AM

<b>Instrument</b>	69679	<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>	Britany Wylie
<b>Sample Position</b>	P3-A1	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	3/10/2020 1:51:39 PM		

**Sample Info.**

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	1658204	∞	11.9	∞	1157421	99.829 ng/ml
THC-COOH	1.415	1550455	9012.3	175.9	4967.0	492883	254.263 ng/ml
THC	3.153	530070	∞	24.8	∞	644350	102.501 ng/ml