







REVIEWED

By Sarah Pickle at 10:30 am, Feb 25, 2020

Byylee
2/21/2020

Worklist: 4025

<u>LAB_CASE</u>	<u>ITEM</u>	<u>ITEM_TYPE</u>	<u>DESCRIPTION</u>	
C2020-0101	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
C2020-0164	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
C2020-0186	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0210	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
C2020-0267	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2020-0301	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: 2/20/20
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
LCMS-QQQ ID: 69679

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.

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, 500 ul 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: 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 ≥ 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:

BW

Curves limited to : 3-100 for THC and THC-OH

2-25-20

**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

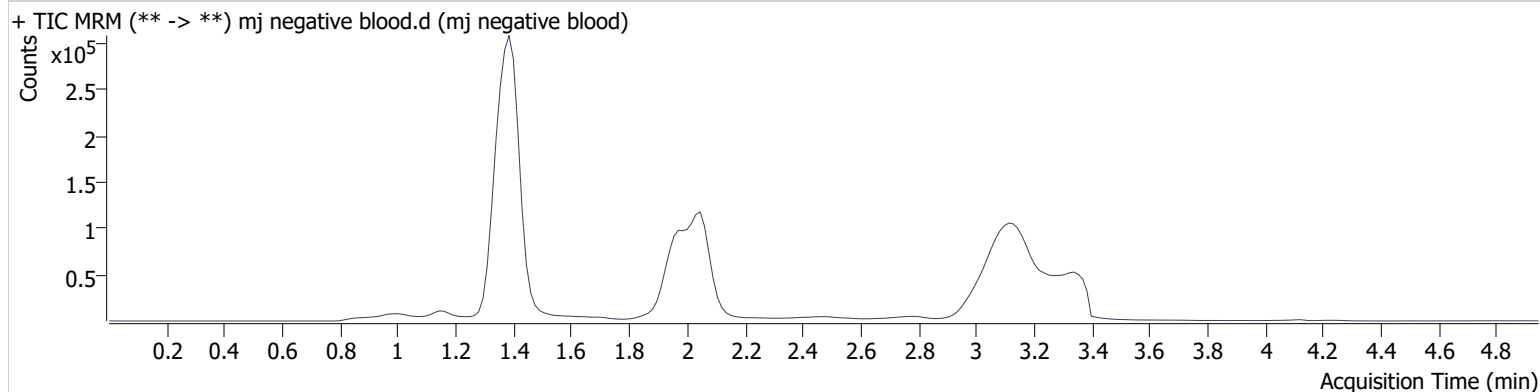
Toxicology Discipline Lead

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj negative blood.d
Type	Sample	Sample	mj negative blood
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-A2	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 1:37:34 PM		
Sample Info.			

Sample Chromatogram



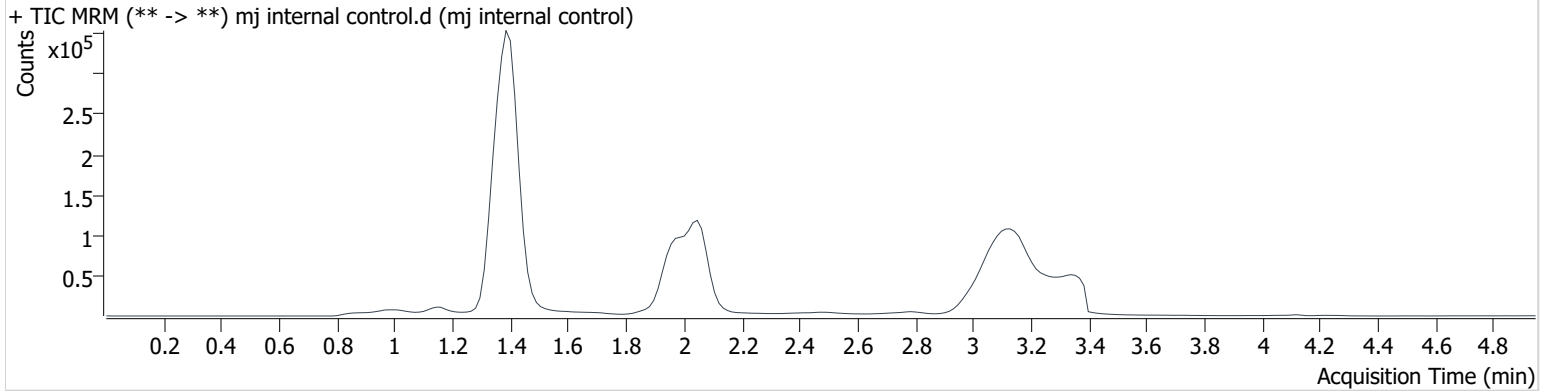
AM #27 Cannabinoids

Byylee

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj internal control.d
Type	QC	Sample	mj internal control
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-H1	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 1:29:52 PM		
Sample Info.			

Sample Chromatogram



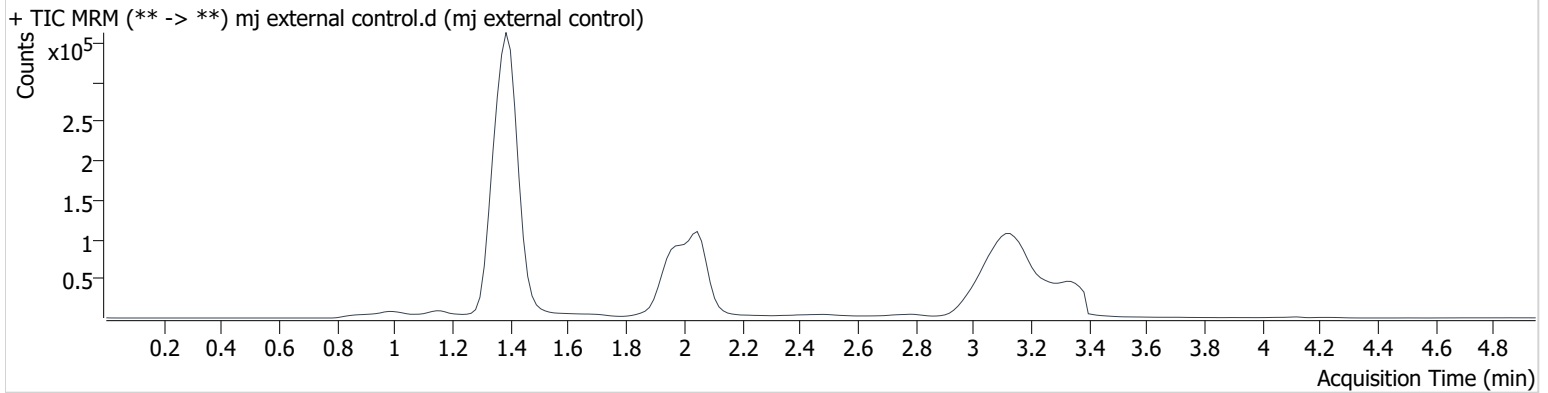
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	84841	∞	9.9	∞	1141459	4.530 ng/ml
THC-COOH	1.415	95938	177.2	205.9	191.1	585141	14.103 ng/ml
THC	3.168	20822	137808795541 45400.0	28.6	76.9	691478	4.444 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj external control.d
Type	Sample	Sample	mj external control
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-B2	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 1:45:18 PM		
Sample Info.			

Sample Chromatogram



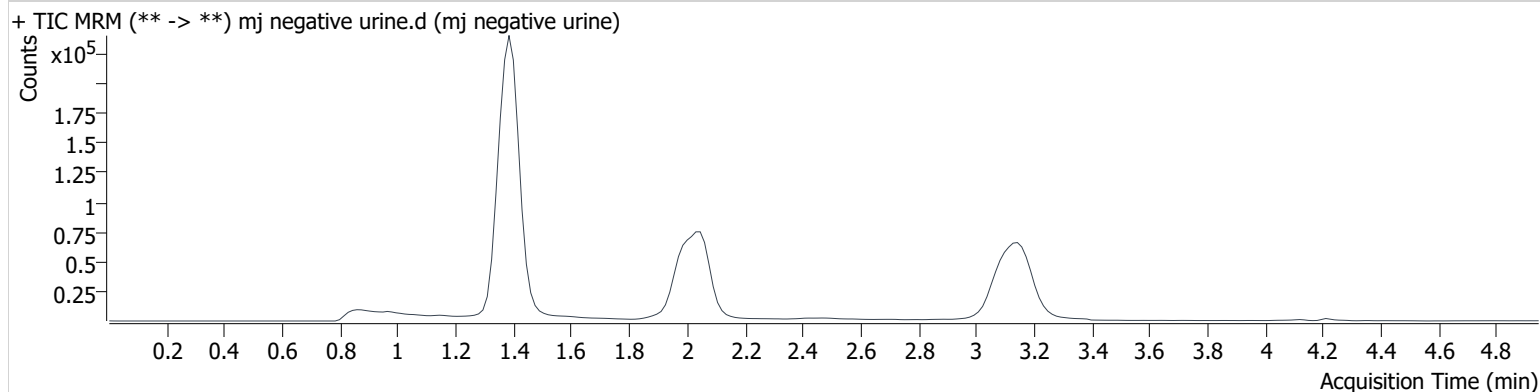
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	104826	∞	11.3	∞	1136902	5.744 ng/ml
THC-COOH	1.415	101404	41588.6	205.3	181.3	595082	14.622 ng/ml
THC	3.168	39201	∞	26.0	1267.0	676130	7.857 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj negative urine.d
Type	Sample	Sample	mj negative urine
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-F2	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 2:46:47 PM		
Sample Info.			

Sample Chromatogram



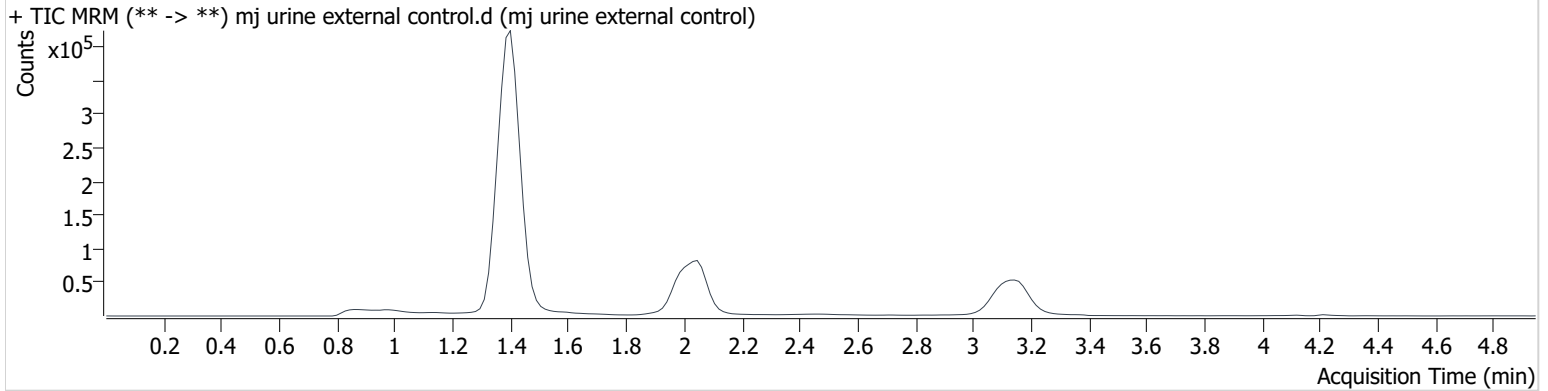
AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj urine external control.d
Type	Sample	Sample	mj urine external control
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-G2	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 3:02:09 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	271444	∞	11.4	∞	939020	19.108 ng/ml
THC-COOH	1.415	213261	412.2	192.4	539.2	410028	42.779 ng/ml
THC	3.168	16785	555.1	30.0	220.1	438494	5.444 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-TH 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

BW

2-25-20

BW

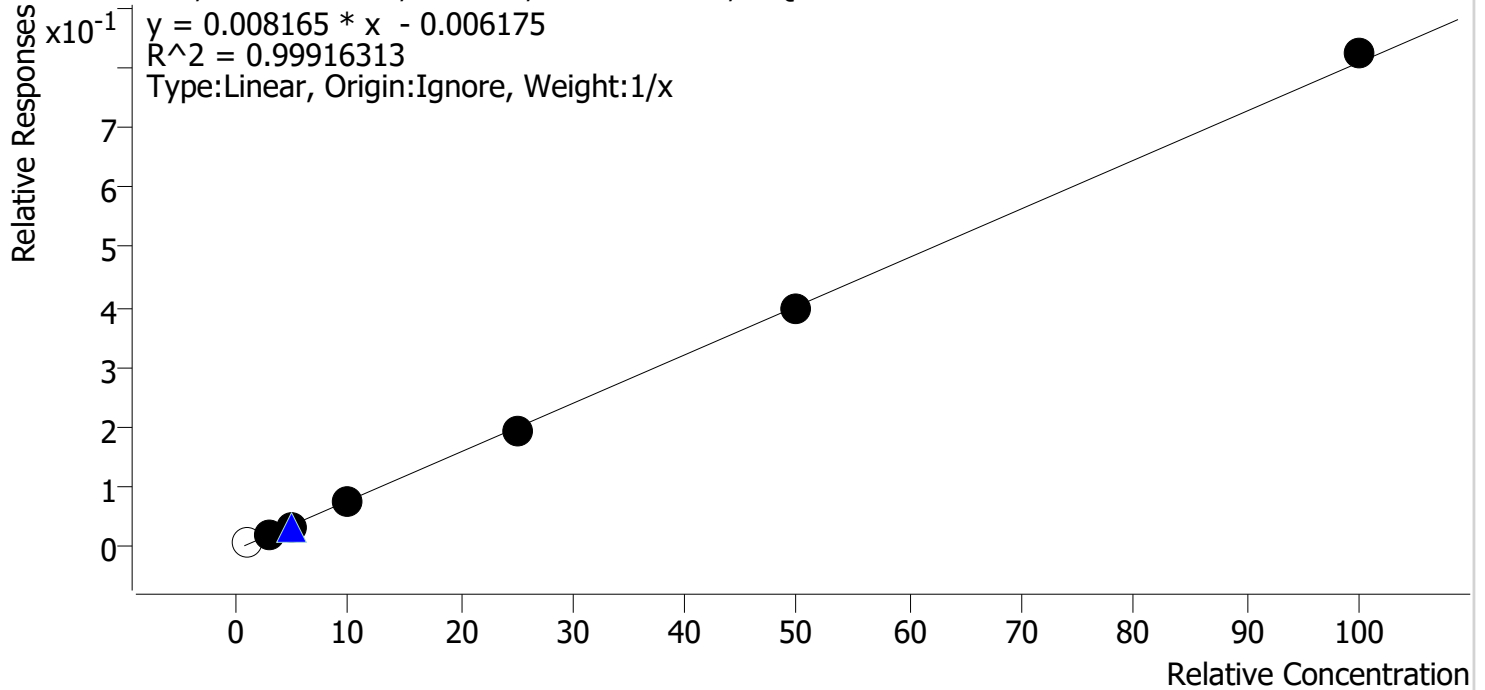
2-25-20

Compound Calibration Report



Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Last Cal. Update 2/24/2020 11:21 AM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-d3

THC - 7 Levels, 6 Levels Used, 7 Points, 6 Points Used, 1 QCs

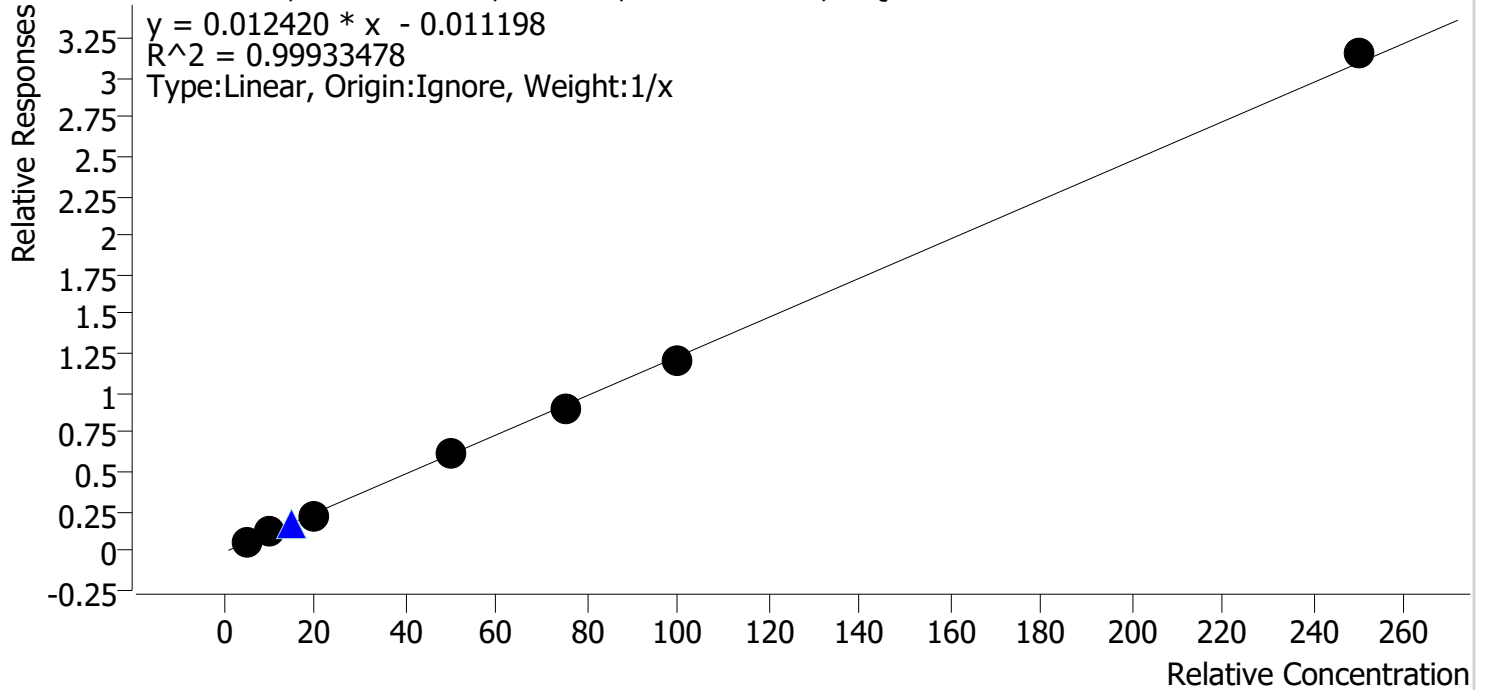


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj qc1	1	x	1.0	1.6	160.0
mj cal2	2	✓	3.0	3.3	109.5
mj cal 3	3	✓	5.0	4.8	95.7
mj cal 4	4	✓	10.0	9.8	98.2
mj cal 5	5	✓	25.0	24.1	96.3
mj cal 6	6	✓	50.0	49.2	98.4
mj cal 7	7	✓	100.0	101.8	101.8

Compound Calibration Report

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Last Cal. Update 2/24/2020 11:21 AM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-d9

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

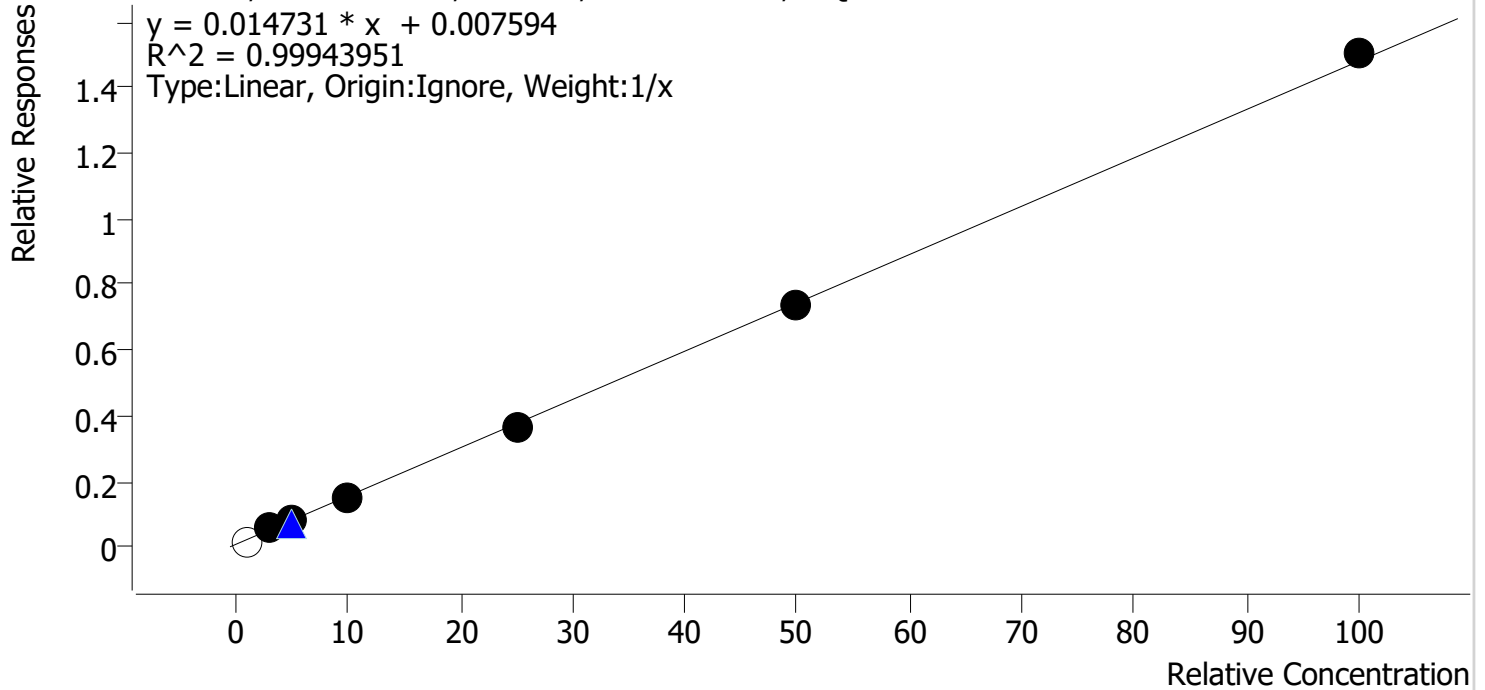


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj qc1	1	✓	5.0	5.3	106.4
mj cal2	2	✓	10.0	10.2	102.0
mj cal 3	3	✓	20.0	19.0	94.9
mj cal 4	4	✓	50.0	49.7	99.4
mj cal 5	5	✓	75.0	72.8	97.1
mj cal 6	6	✓	100.0	98.3	98.3
mj cal 7	7	✓	250.0	254.7	101.9

Compound Calibration Report

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Last Cal. Update 2/24/2020 11:21 AM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-d3

THC-OH - 7 Levels, 6 Levels Used, 7 Points, 6 Points Used, 1 QCs



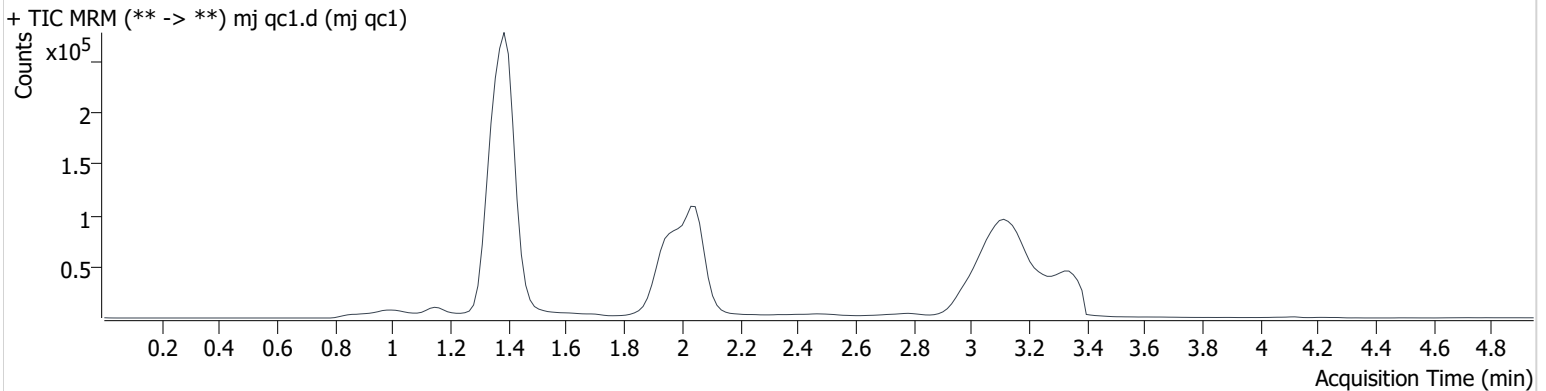
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj qc1	1	x	1.0	0.5	54.7
mj cal2	2	✓	3.0	3.2	105.6
mj cal 3	3	✓	5.0	4.9	98.9
mj cal 4	4	✓	10.0	9.9	99.0
mj cal 5	5	✓	25.0	24.0	96.1
mj cal 6	6	✓	50.0	49.4	98.8
mj cal 7	7	✓	100.0	101.5	101.5

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj qc1.d
Type	Cal	Sample	mj qc1
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-G1	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 12:35:45 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	16519	∞	9.3	∞	1055772	0.547 ng/ml Low
THC-COOH	1.415	29204	16.4	204.2	17.3	532232	5.320 ng/ml Low
THC	3.183	4406	136.6	23.0	6.3 Low	639629	1.600 ng/ml Low

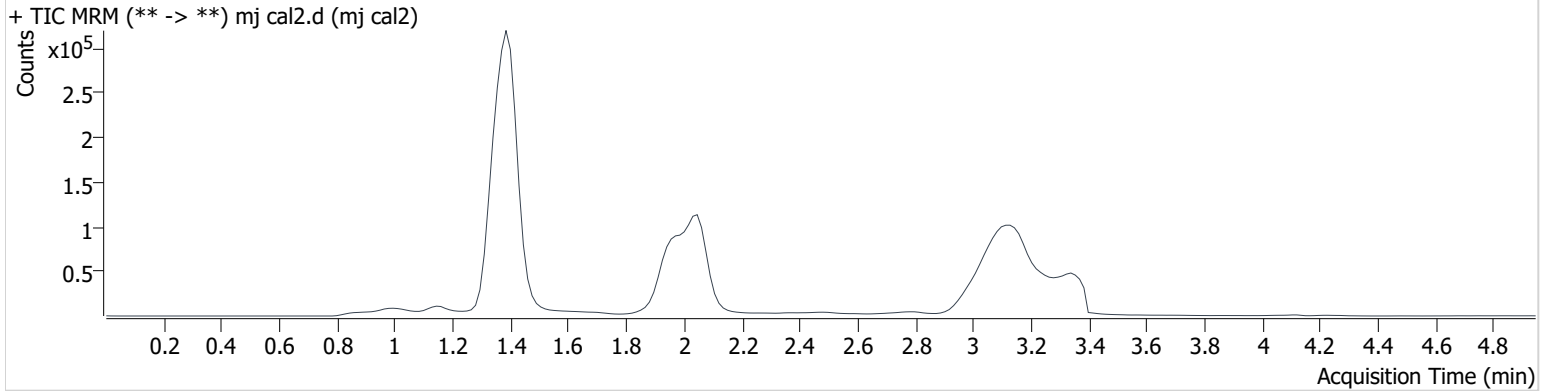
AM #27 Cannabinoids

BWylie

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj cal2.d
Type	Cal	Sample	mj cal2
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-F1	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 12:43:29 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	61075	∞	9.2	∞	1125441	3.168 ng/ml
THC-COOH	1.415	64135	126.1	223.5	1213.6	555095	10.204 ng/ml
THC	3.183	13863	751.7	27.0	61.7	670981	3.286 ng/ml

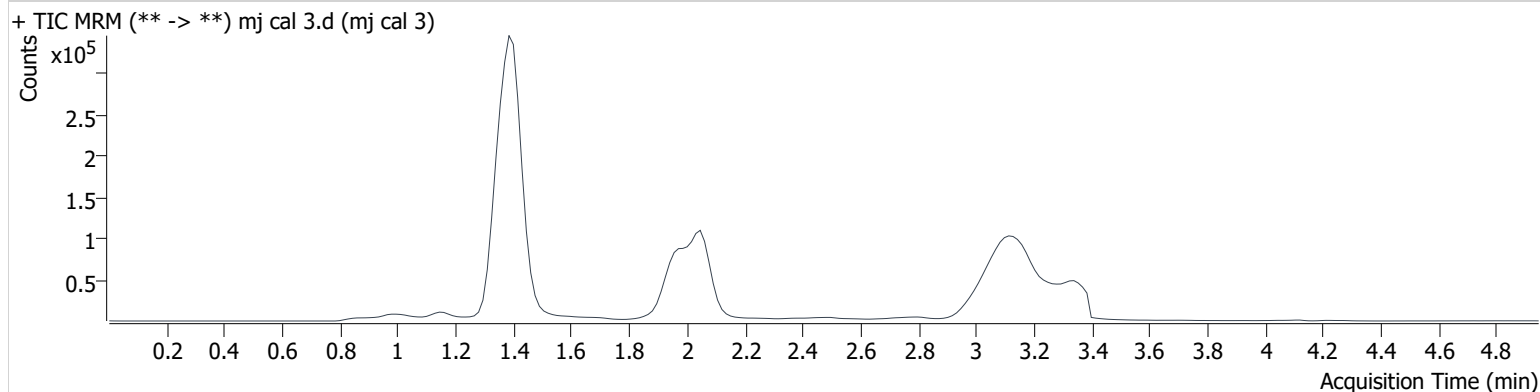
AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj cal 3.d
Type	Cal	Sample	mj cal 3
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-E1	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 12:51:14 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	87256	∞	10.5	∞	1084806	4.945 ng/ml
THC-COOH	1.415	124550	530.9	202.6	355822.2	554509	18.986 ng/ml
THC	3.168	21745	316202940771 90.2	26.6	59.9	660920	4.786 ng/ml

AM #27 Cannabinoids

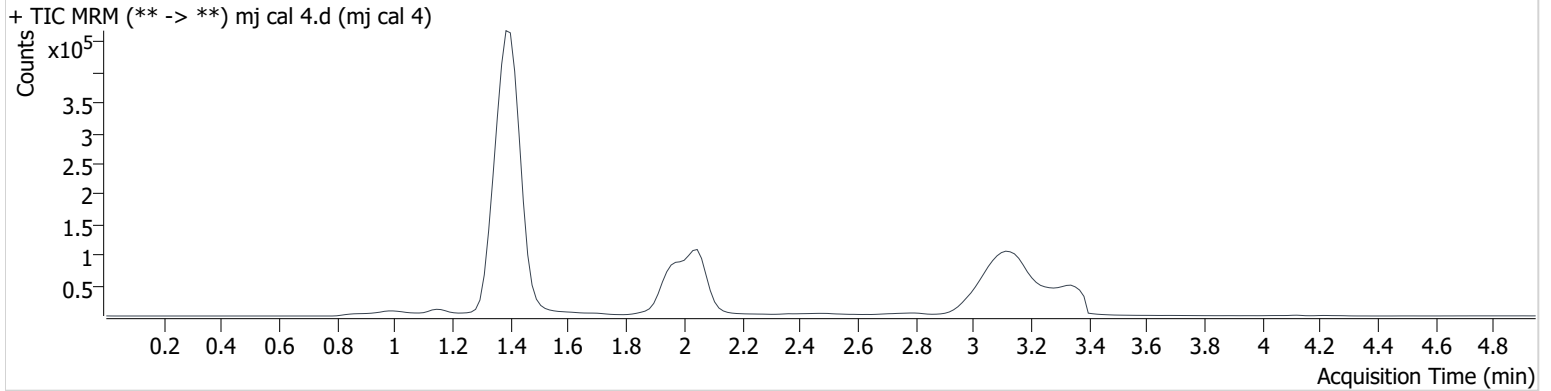
BWylie

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj cal 4.d
Type	Cal	Sample	mj cal 4
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-D1	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 12:58:56 PM		

Sample Info.

Sample Chromatogram



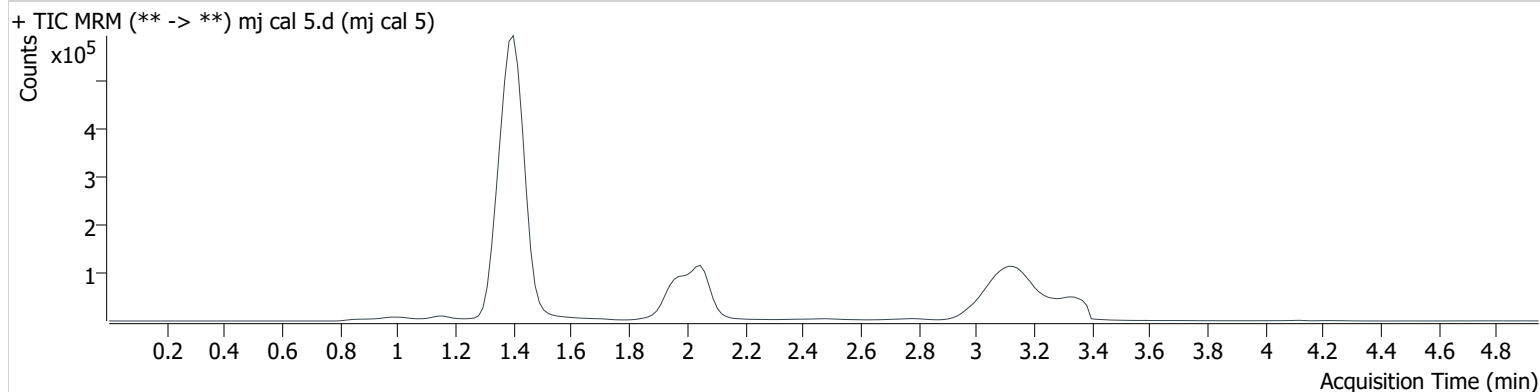
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	170325	∞	10.8	∞	1110066	9.901 ng/ml
THC-COOH	1.415	338202	427.0	185.6	2257.6	558302	49.675 ng/ml
THC	3.168	48357	∞	25.3	387.0	653610	9.817 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj cal 5.d
Type	Cal	Sample	mj cal 5
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-C1	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 1:06:40 PM		

Sample Chromatogram



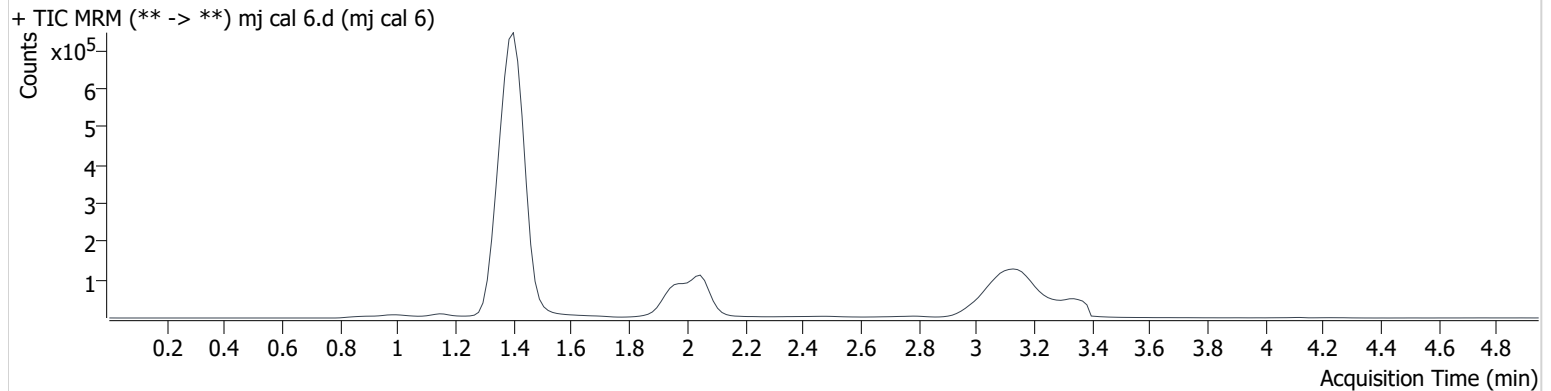
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	407531	∞	11.1	∞	1127365	24.024 ng/ml
THC-COOH	1.415	488764	477.9	186.8	1843.3	547157	72.825 ng/ml
THC	3.153	123120	∞	24.6	∞	646505	24.079 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj cal 6.d
Type	Cal	Sample	mj cal 6
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-B1	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 1:14:24 PM		

Sample Chromatogram



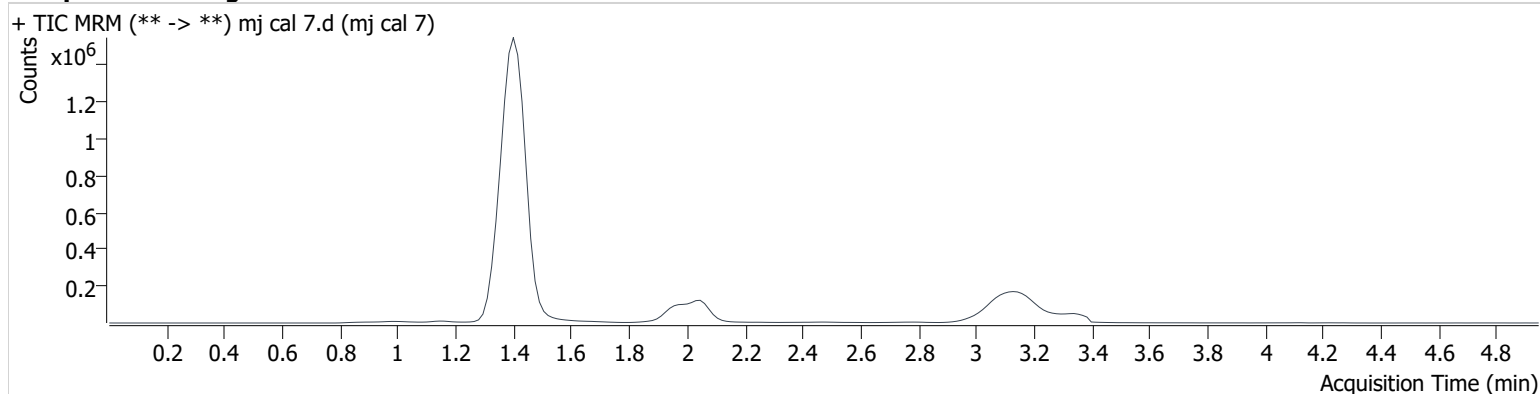
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	836299	∞	11.2	∞	1136869	49.422 ng/ml
THC-COOH	1.415	662870	1458.0	183.5	1320.3	547918	98.309 ng/ml
THC	3.168	261063	∞	24.1	13404.2	659694	49.221 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2020 Data\am 27-28 2-20-2020\QuantResults\thcq.batch.bin
Calibration Last Update 2/24/2020 11:21:40 AM

Instrument	69679	Data File	mj cal 7.d
Type	Cal	Sample	mj cal 7
Acq. Method	AM 27 THC quant.m	Operator	Britany Wylie
Sample Position	P3-A1	Comment	
Injection Volume	10		
Acq. Date-Time	2/20/2020 1:22:08 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.390	1914141	∞	11.8	∞	1273250	101.540 ng/ml
THC-COOH	1.415	1757538	5929.1	178.7	1002.4	557608	254.680 ng/ml
THC	3.168	575387	∞	24.9	∞	697305	101.811 ng/ml