4/24/2020

REVIEWED
By Sarah Pickle at 9:05 am, Apr 27, 2020

Worklist: 4191

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
M2020-0358	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2020-0981	4	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1042	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1085	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-1135	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: 04/22/2020
Plate lot#: IDP-108-2-200303
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: 445283-3
LCMS-QQQ ID: 069901

Analyst: Celena Shrum
Plate Expiration: 09/30/2020
Mobile phase B: 0.1% Formic acid in Acetonitrile
Column: UCT Selectra DA 100 x 2.1mm 3um
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 ≥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: Curve ranges are THC: 3-100, THC-COOH: 5-250. THC-OH not evaluated for this run.

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.

Component	*Source*	*Source Lot Number*	*Expiration Date*
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

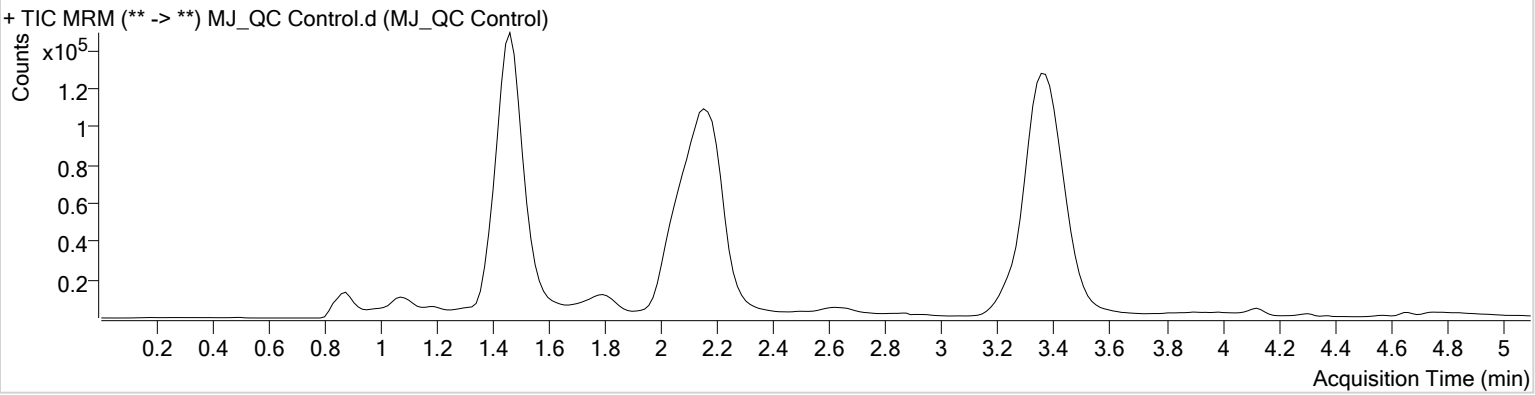
Component	*Source*	*Source Lot Number*
Negative Urine	Pocatello Lab	POC031319
Methanol External Control Solution	-	WS011620
Prepared:	04/22/2020	
Prepared by:	Celena Shrum	
Expires:	09/30/2020	

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_QC Control.d
Type	Sample	**Sample**	MJ_QC Control
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 3:22:45 PM		
Sample Info.			

Sample Chromatogram



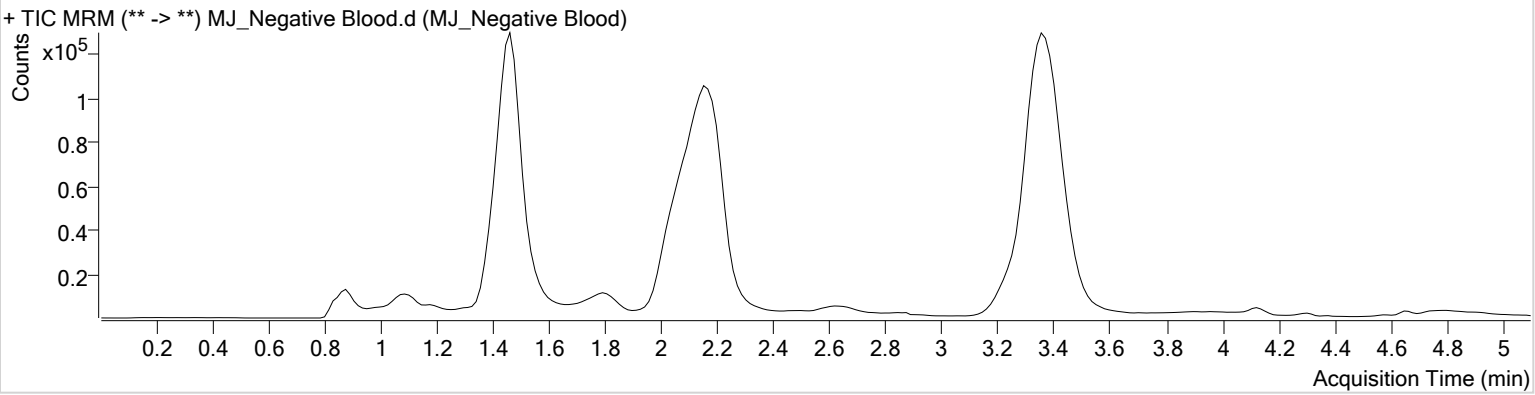
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.489	68093	∞	48.4	∞	176193	15.0093	ng/ml
THC	3.375	47125	425.36	32.5	111.00	1226632	4.4008	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update	4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Negative Blood.d
Type	Sample	**Sample**	MJ_Negative Blood
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 3:37:57 PM		
Sample Info.			

Sample Chromatogram

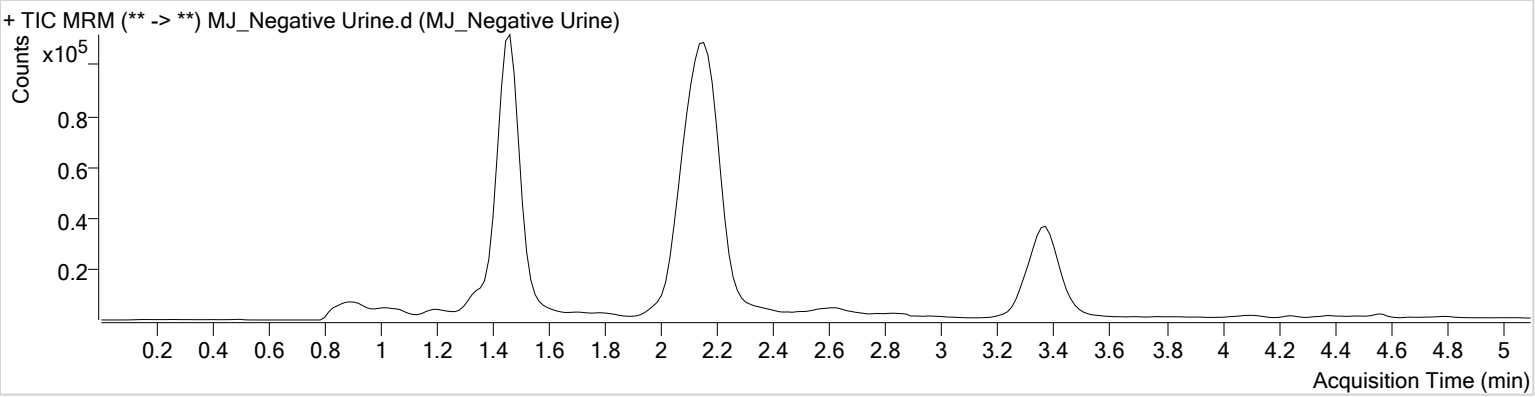


AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update	4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Negative Urine.d
Type	Sample	**Sample**	MJ_Negative Urine
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-G5	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 3:53:07 PM		
Sample Info.			

Sample Chromatogram

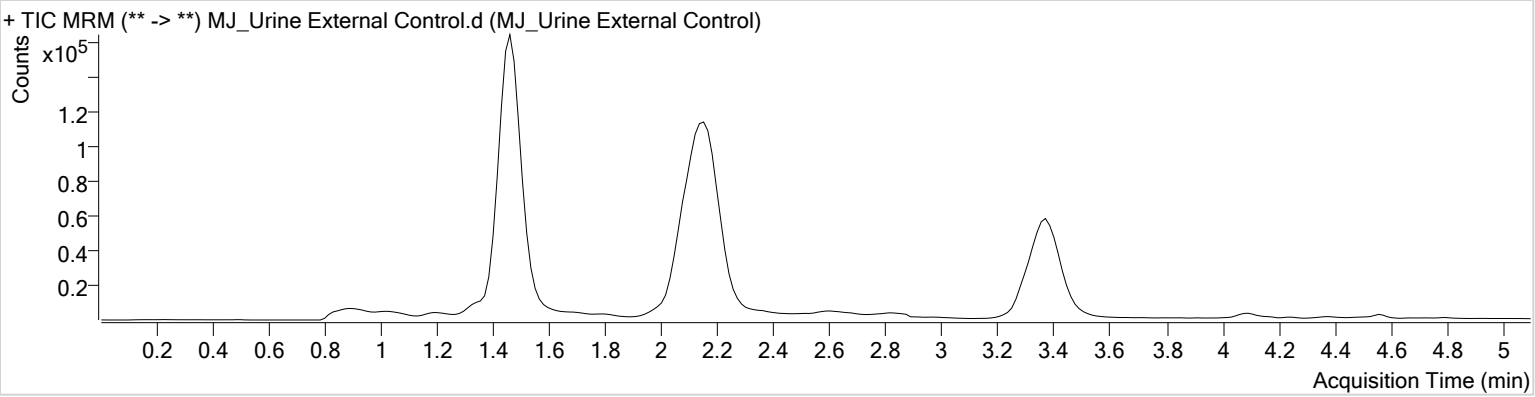


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Urine External Control.d
Type	Sample	**Sample**	MJ_Urine External Control
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-F5	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 4:08:18 PM		
Sample Info.			

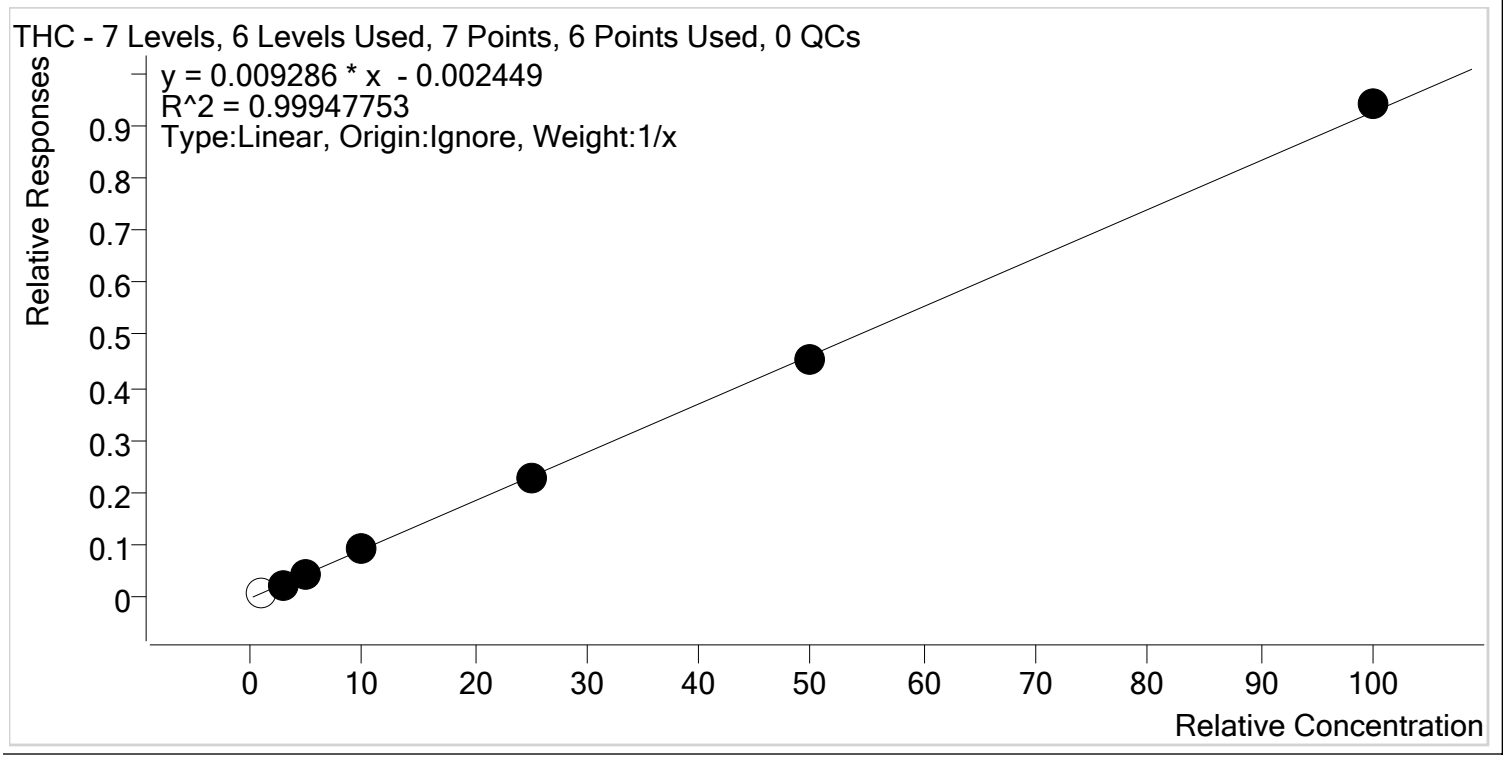
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.504	58473	∞	55.5	∞	149741	15.1933	ng/ml
THC	3.375	52984	139.78	28.8	35.23	403642	14.3991	ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

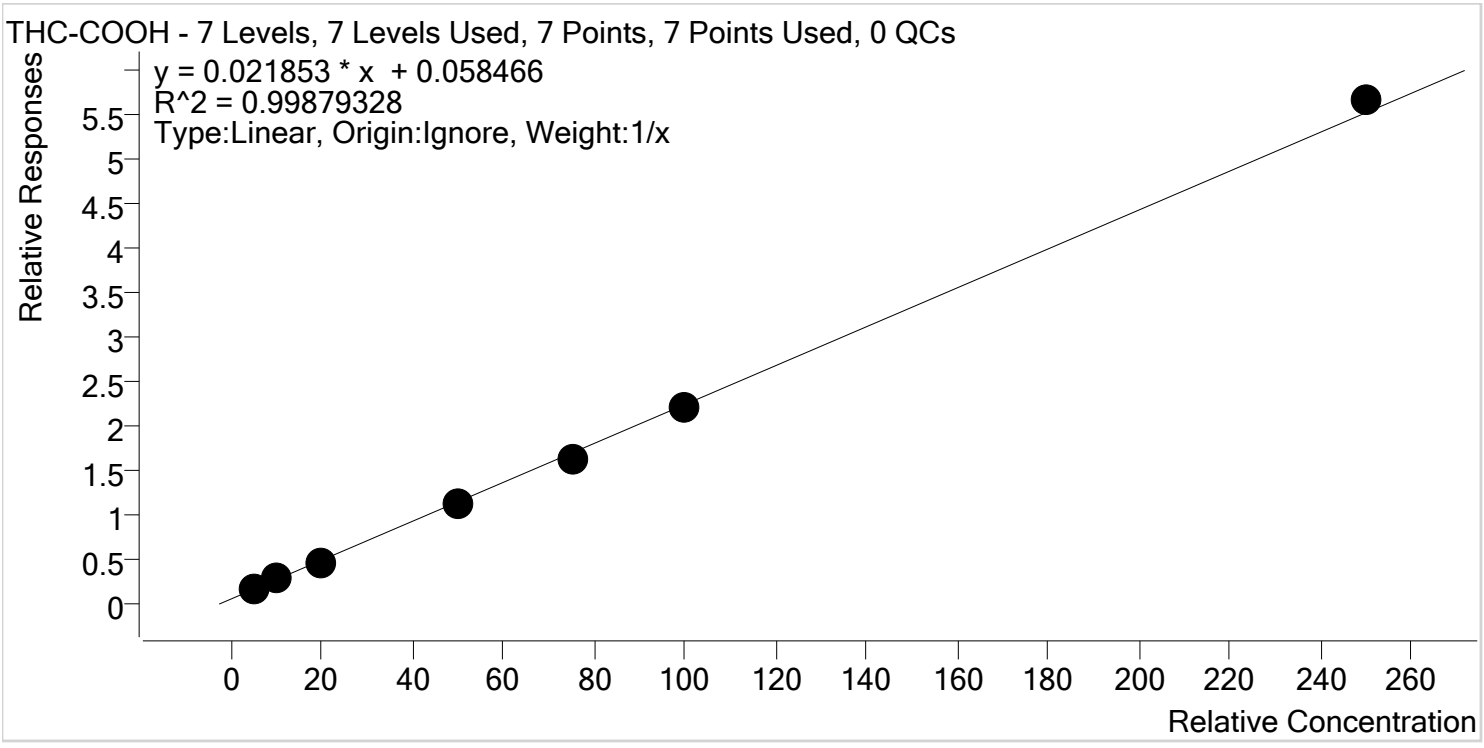
Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Last Cal. Update	4/27/2020 8:43 AM
Analyst Name	ISP\Datastor
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✗	1.0	1.2	122.6
MJ_Cal 2	2	✔	3.0	3.0	99.5
MJ_Cal 3	3	✔	5.0	5.2	104.5
MJ_Cal 4	4	✔	10.0	9.9	98.8
MJ_Cal 5	5	✔	25.0	24.5	98.0
MJ_Cal 6	6	✔	50.0	48.8	97.6
MJ_Cal 7	7	✔	100.0	101.6	101.6

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Last Cal. Update	4/27/2020 8:43 AM
Analyst Name	ISP\Datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



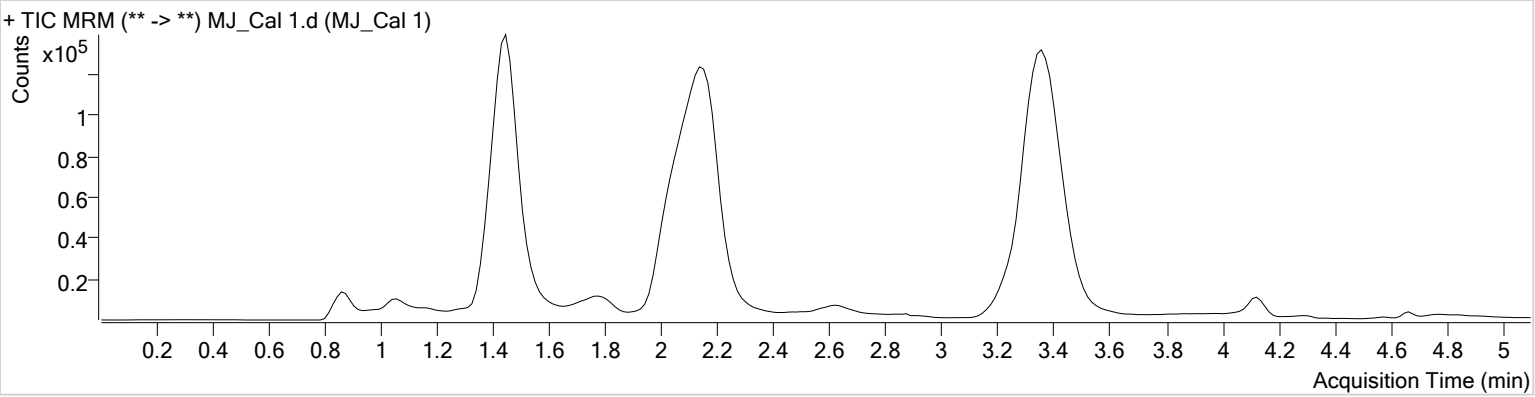
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✔	5.0	5.4	108.1
MJ_Cal 2	2	✔	10.0	10.4	104.0
MJ_Cal 3	3	✔	20.0	18.8	93.8
MJ_Cal 4	4	✔	50.0	48.4	96.8
MJ_Cal 5	5	✔	75.0	72.2	96.2
MJ_Cal 6	6	✔	100.0	98.5	98.5
MJ_Cal 7	7	✔	250.0	256.4	102.5

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 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-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 2:21:54 PM		
Sample Info.			

Sample Chromatogram



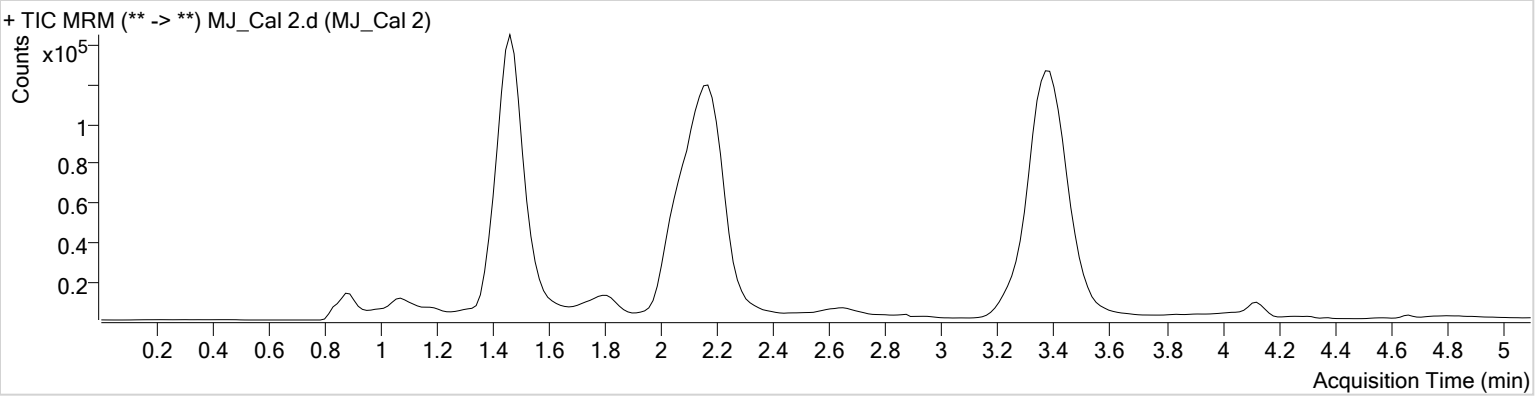
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.474	32526	∞	45.5	∞	184211	5.4045	ng/ml **Low**
THC	3.375	11825	∞	30.4	8.13 **Low**	1323031	1.2262	ng/ml **Low**

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Cal 2.d
Type	Cal	**Sample**	MJ_Cal 2
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 2:29:39 PM		
Sample Info.			

Sample Chromatogram



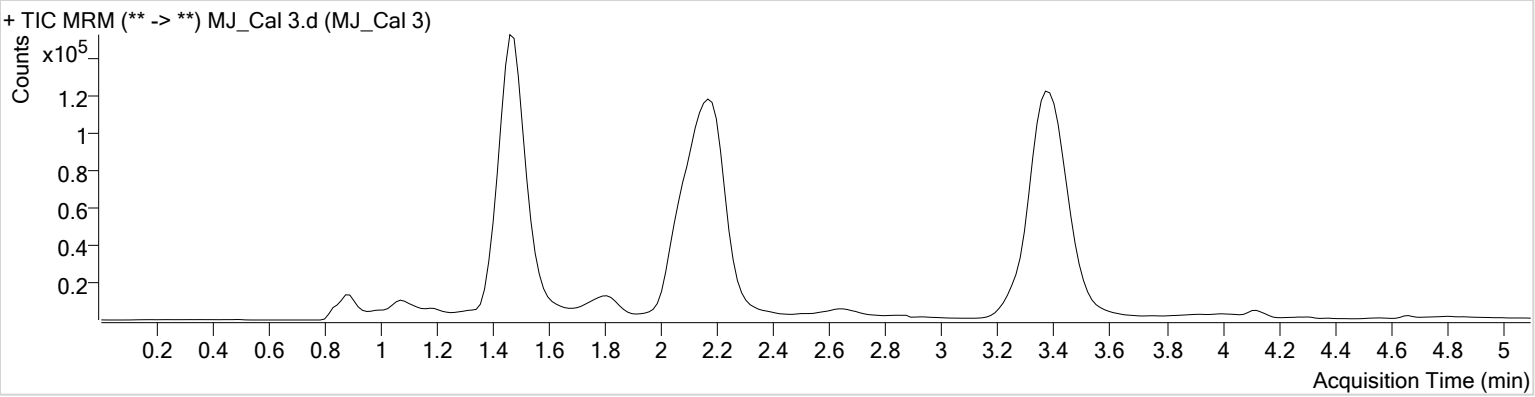
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.504	50663	∞	46.2	88.26	177314	10.3993	ng/ml
THC	3.390	31059	52.67	35.2	37.46	1229333	2.9844	ng/ml **Low**

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Cal 3.d
Type	Cal	**Sample**	MJ_Cal 3
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 2:37:13 PM		
Sample Info.			

Sample Chromatogram



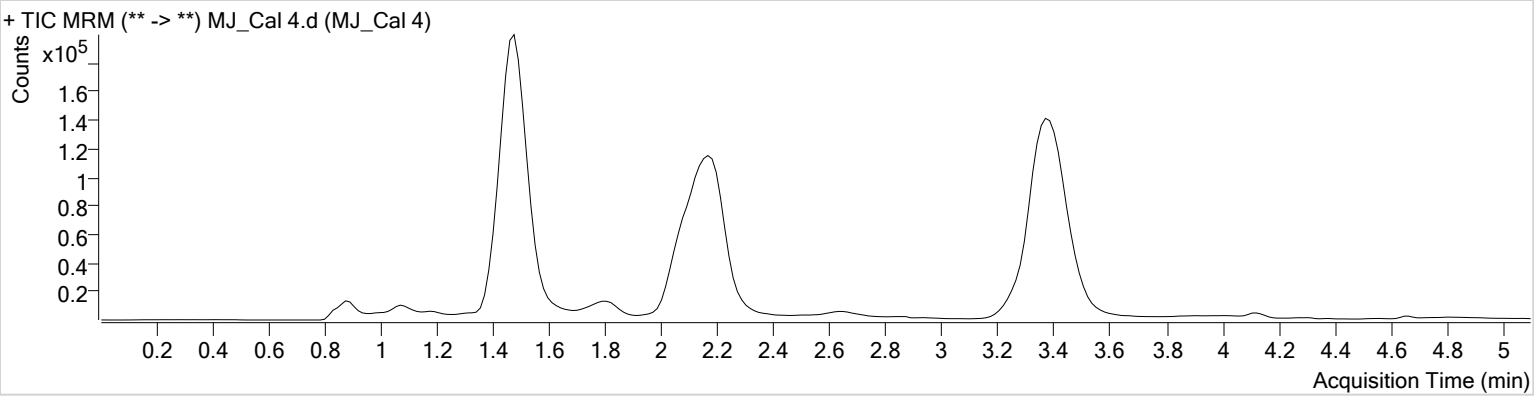
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.504	80074	∞	52.6	∞	170867	18.7691	ng/ml
THC	3.405	52001	181.79	26.5	30.71	1128983	5.2237	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Cal 4.d
Type	Cal	**Sample**	MJ_Cal 4
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-E6	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 2:44:47 PM		
Sample Info.			

Sample Chromatogram



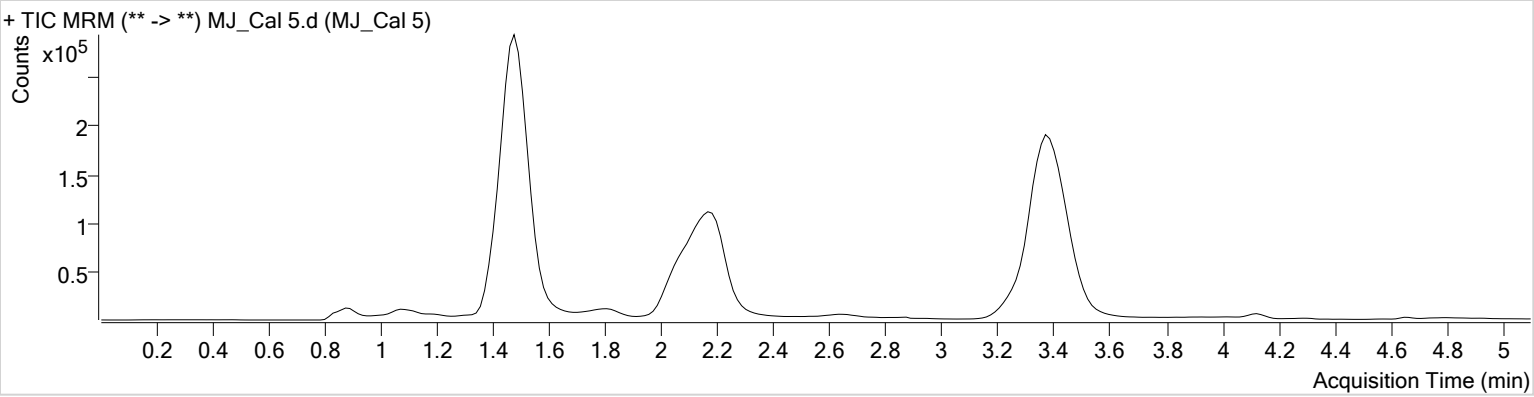
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.504	203273	∞	57.3	1200.51	182083	48.4097	ng/ml
THC	3.390	108100	150.12	29.7	197.10	1210602	9.8794	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Cal 5.d
Type	Cal	**Sample**	MJ_Cal 5
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 2:52:22 PM		
Sample Info.			

Sample Chromatogram



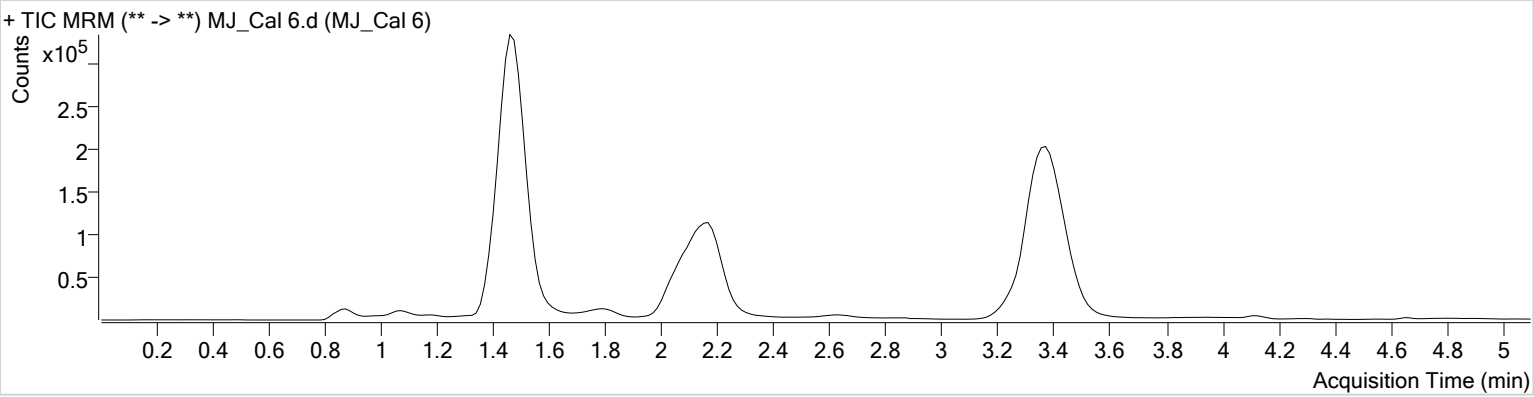
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.504	354723	∞	59.2	∞	216888	72.1648	ng/ml
THC	3.390	330163	702.84	26.1	∞	1466622	24.5056	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Cal 6.d
Type	Cal	**Sample**	MJ_Cal 6
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 2:59:58 PM		
Sample Info.			

Sample Chromatogram



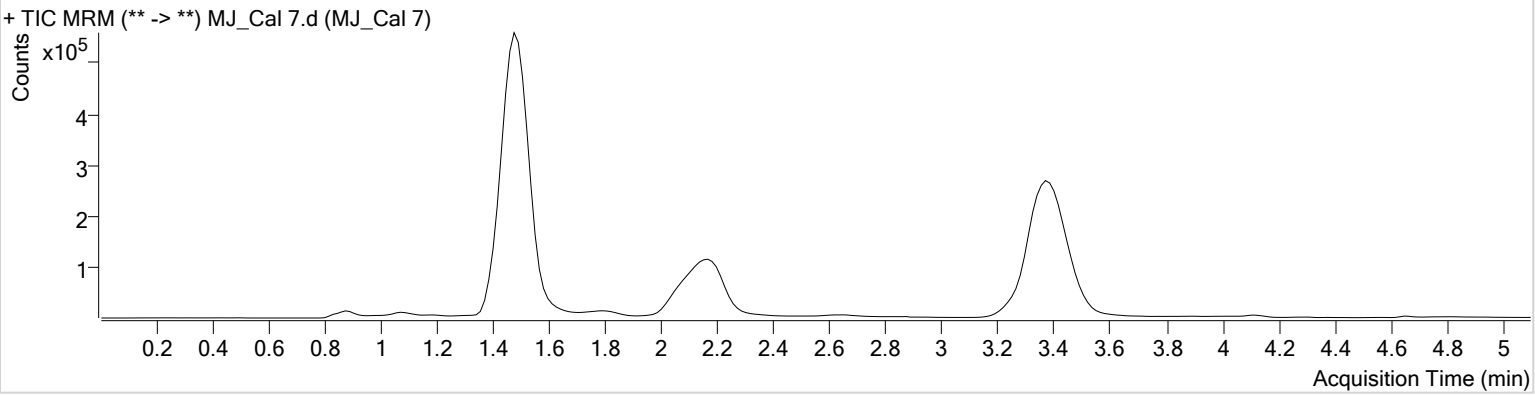
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.489	405968	∞	58.6	6460.55	183635	98.4868	ng/ml
THC	3.375	583365	3276.78	26.1	164.22	1293675	48.8229	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 042220 CS\QuantResults\AM 27 THC-OH removed.batch.bin
Calibration Last Update 4/27/2020 8:43:20 AM

Instrument	Falco	**Data File**	MJ_Cal 7.d
Type	Cal	**Sample**	MJ_Cal 7
Acq. Method	AM 27 THC quant.m	**Operator**	Celena Shrum
Sample Position	P3-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/22/2020 3:07:32 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.504	950033	∞	58.7	∞	167823	256.3659	ng/ml
THC	3.390	1168830	4301.33	25.0	124.65	1242254	101.5841	ng/ml