REVIEWED
By Tamara Salazar at 11:06 am, Dec 09, 2020

12/2/2020

Worklist: 4645

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2020-4705	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
P2020-3062	4	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
P2020-3165	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
P2020-3289	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: 12/03/2020
Plate lot#: IDP-108-2-200723
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: ~~20K20702~~ 20L20725
LCMS-QQQ ID: 069901

Analyst: Celena Shrum
Plate Expiration: 01/23/2021
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: 5ng/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:

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		

Urine External Control Solution (Lot: 110220)

200 ul of methanol external control solution was added to 9800 ul of urine.

Approximately 20ng/mL each

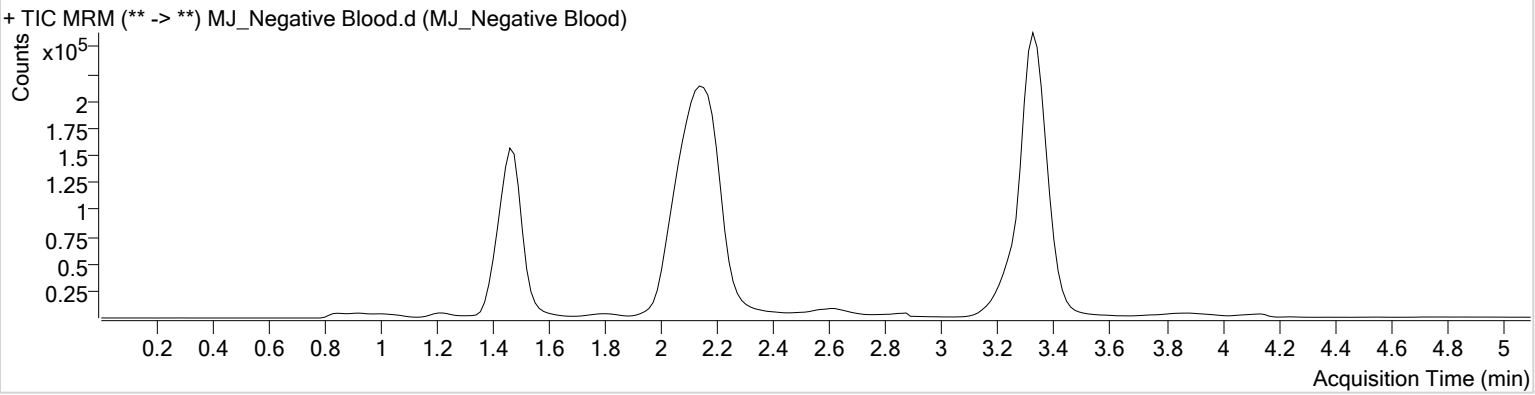
Component	*Source*	*Source Lot Number*
Negative Urine	Pocatello Lab	POC031319
Methanol External Control Solution	-	WS011620
Prepared:	11/02/2020	
Prepared by:	Celena Shrum	

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Negative Blood.d
Type	Sample	**Sample**	MJ_Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 4:56:42 PM		
Sample Info.			

Sample Chromatogram

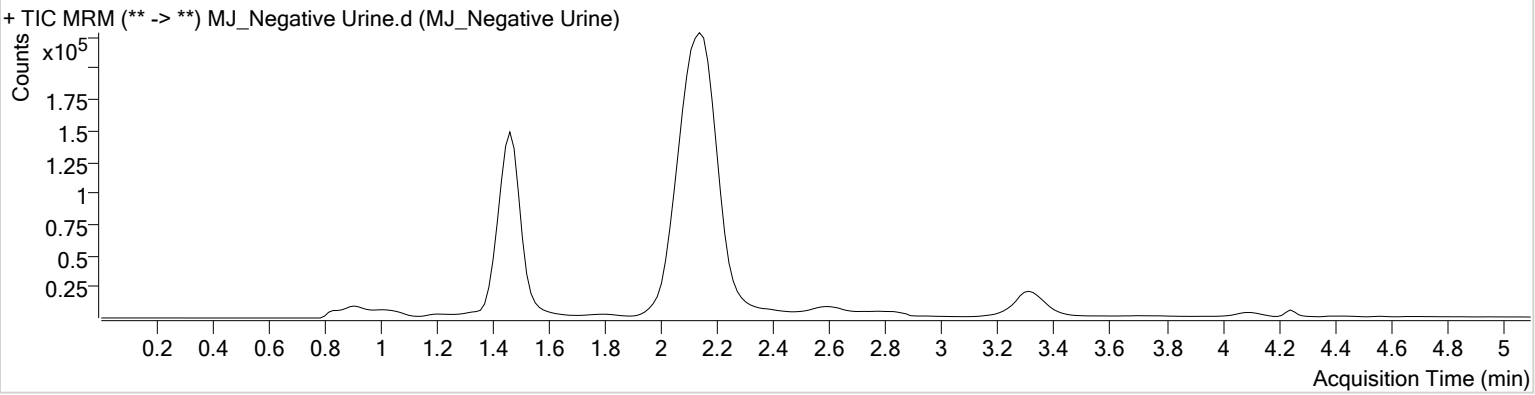


AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Negative Urine.d
Type	Sample	**Sample**	MJ_Negative Urine
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-G5	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 5:11:54 PM		
Sample Info.			

Sample Chromatogram

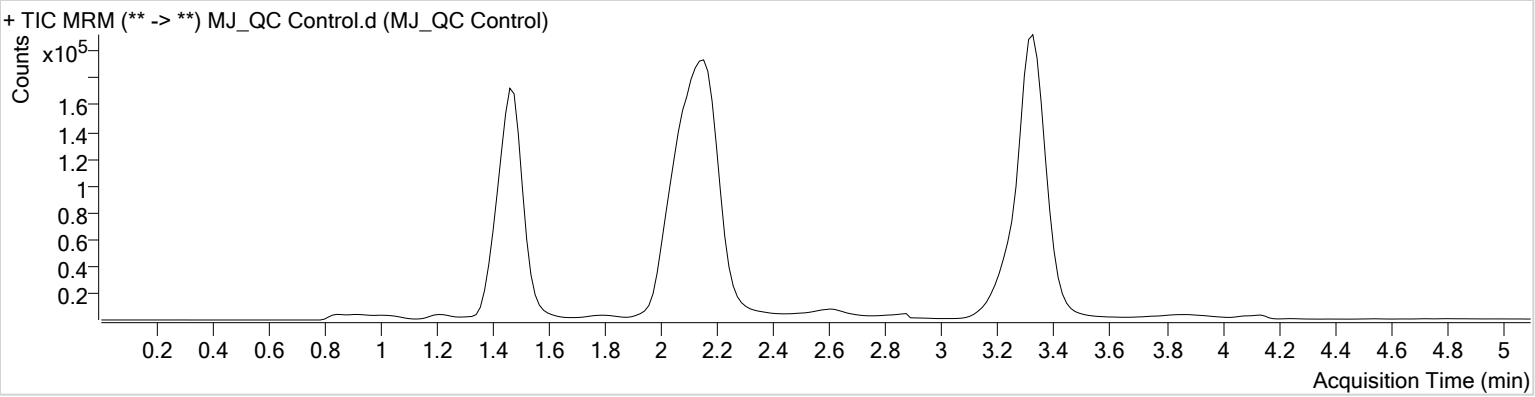


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_QC Control.d
Type	Sample	**Sample**	MJ_QC Control
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 4:41:29 PM		
Sample Info.			

Sample Chromatogram



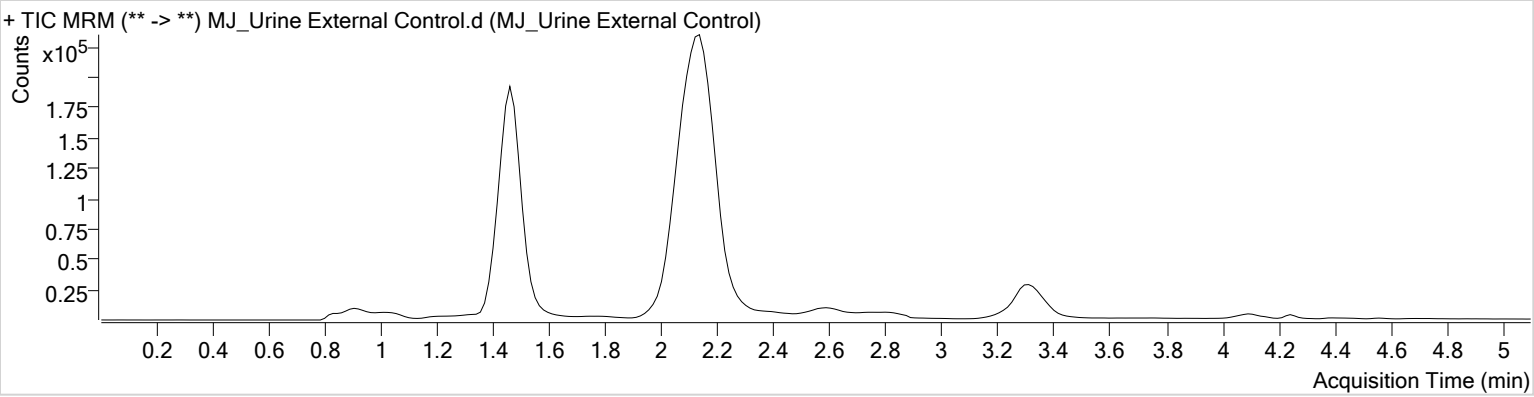
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	49197	∞	13.4	55.66	638393	4.5390	ng/ml
THC-COOH	1.489	87972	∞	59.4	∞	228100	14.8424	ng/ml
THC	3.330	59617	∞	35.9	∞	1528010	4.6390	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Urine External Control.d
Type	Sample	**Sample**	MJ_Urine External Control
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-F5	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 5:19:30 PM		
Sample Info.			

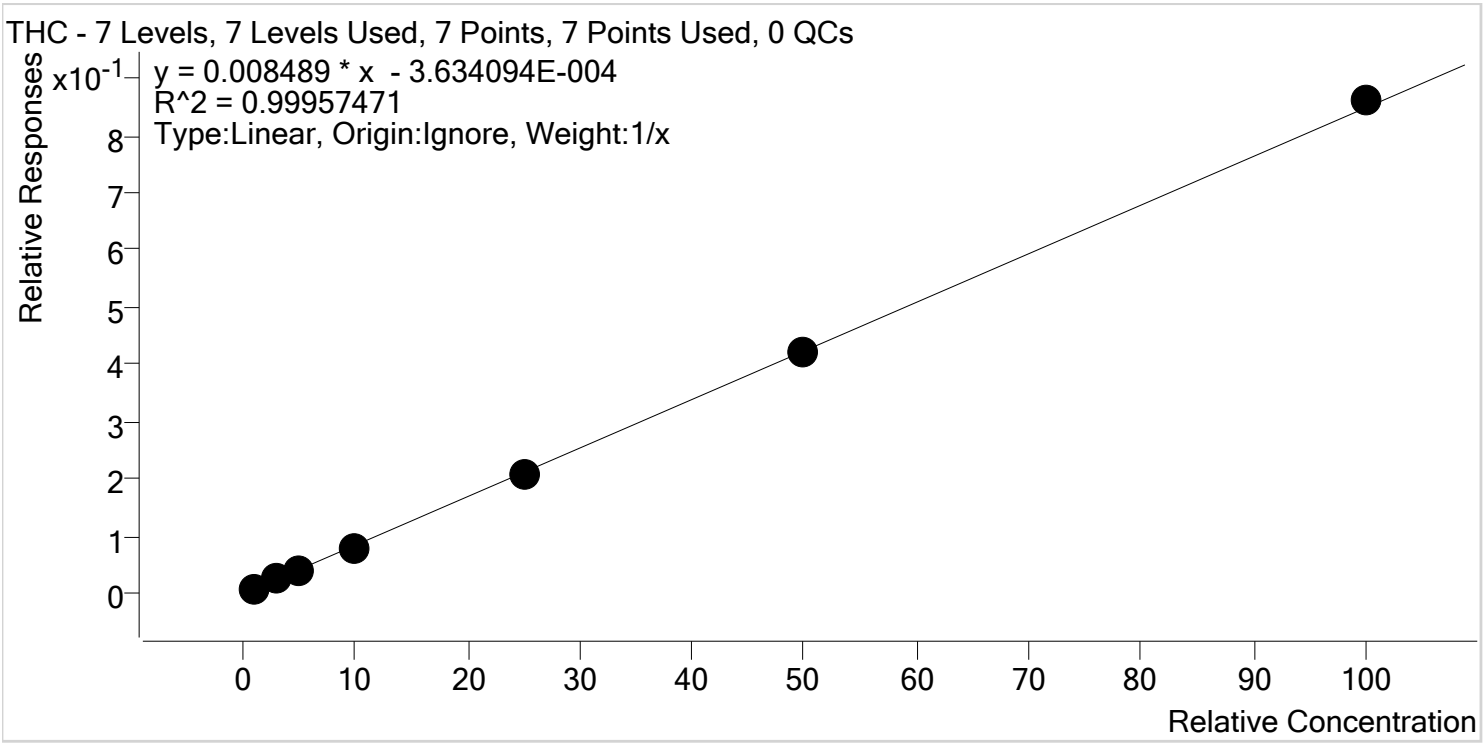
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	137973	∞	13.1	178.60	605343	13.4737	ng/ml
THC-COOH	1.489	87154	∞	60.8	∞	164623	20.8138	ng/ml
THC	3.330	15073	∞	27.9	22.97	210336	8.4849	ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

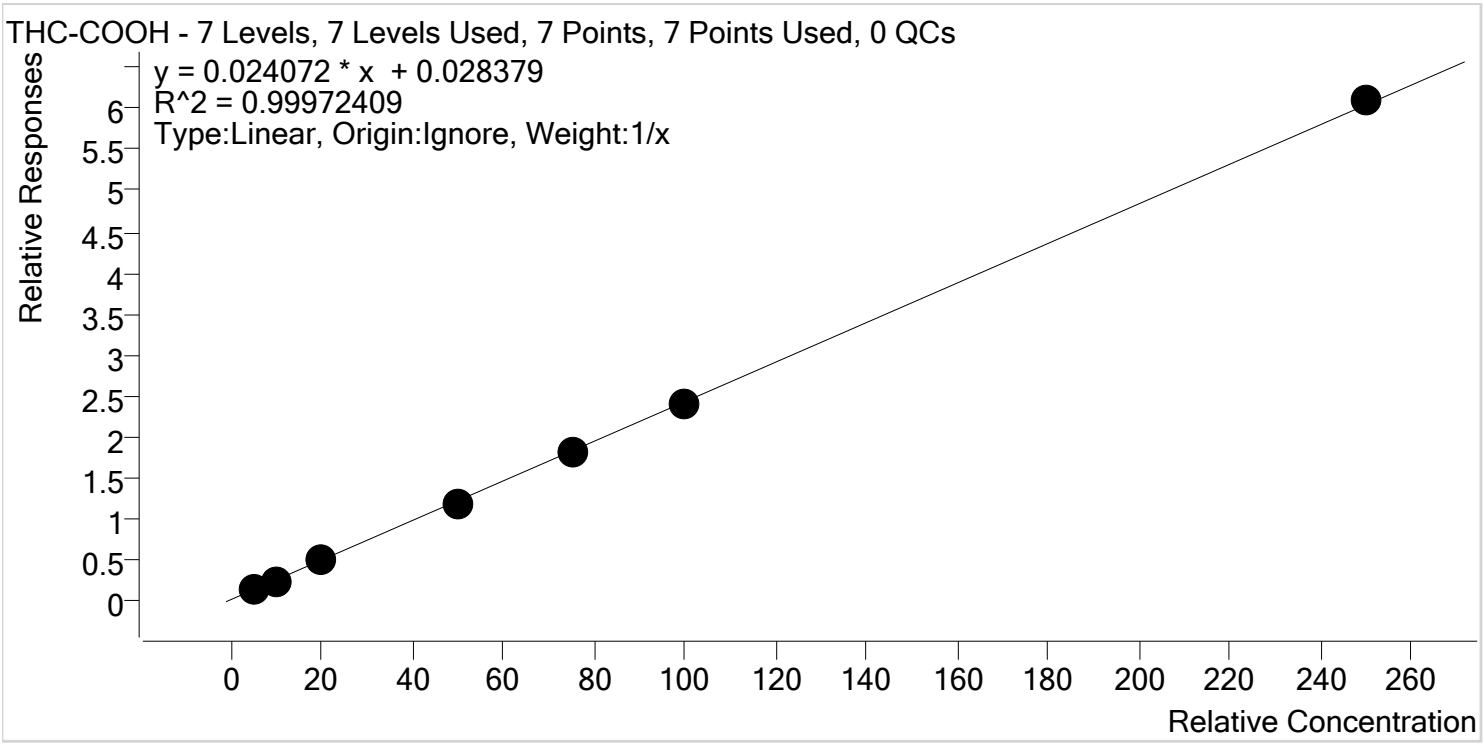
Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Last Cal. Update	12/7/2020 9:19 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✔	1.0	1.1	107.8
MJ_Cal 2	2	✔	3.0	2.9	97.3
MJ_Cal 3	3	✔	5.0	5.0	101.0
MJ_Cal 4	4	✔	10.0	9.6	96.3
MJ_Cal 5	5	✔	25.0	24.2	96.9
MJ_Cal 6	6	✔	50.0	49.7	99.3
MJ_Cal 7	7	✔	100.0	101.4	101.4

AM #27 Cannabinoids Quant. Calibration Curve Report

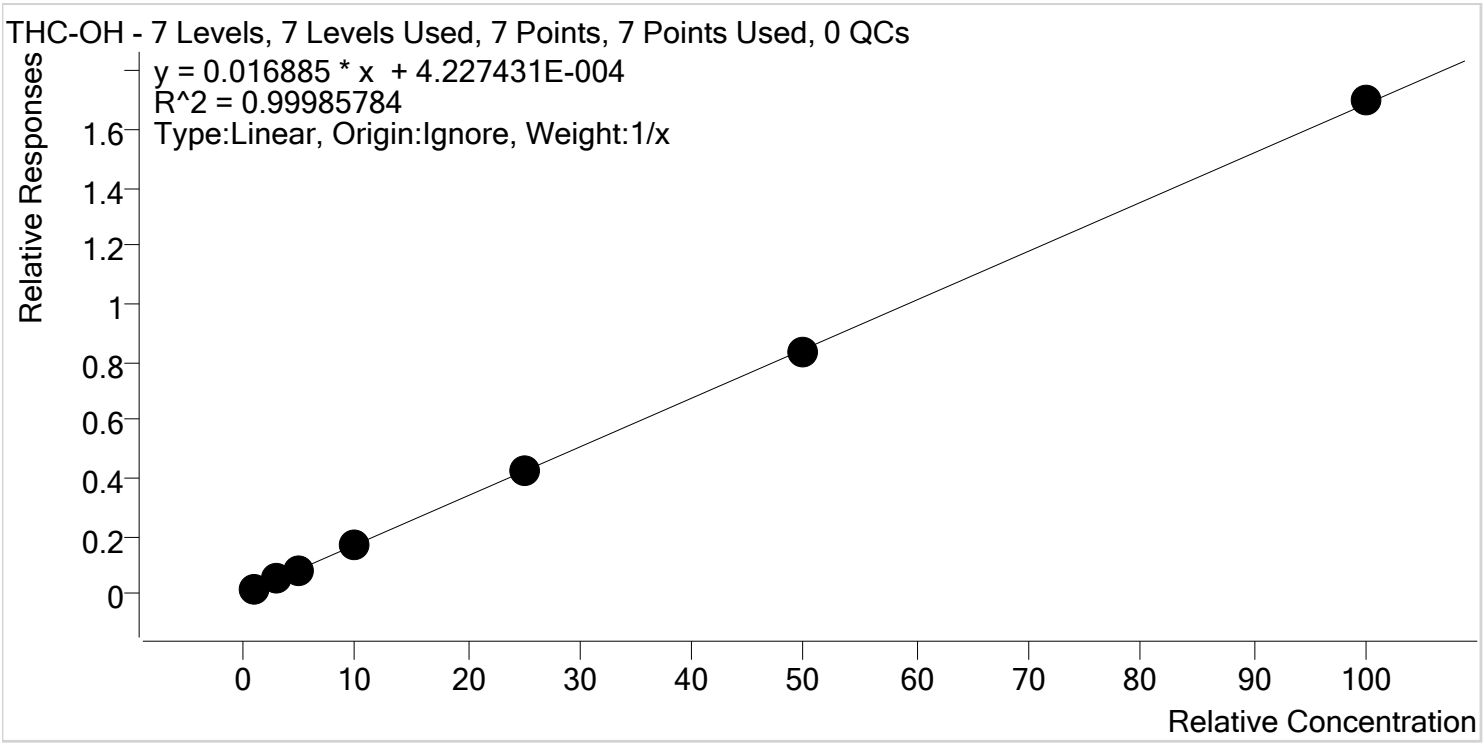
Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Last Cal. Update	12/7/2020 9:19 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✔	5.0	5.4	108.3
MJ_Cal 2	2	✔	10.0	9.6	96.5
MJ_Cal 3	3	✔	20.0	19.4	96.9
MJ_Cal 4	4	✔	50.0	49.0	98.1
MJ_Cal 5	5	✔	75.0	75.0	100.0
MJ_Cal 6	6	✔	100.0	99.4	99.4
MJ_Cal 7	7	✔	250.0	252.1	100.8

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Last Cal. Update	12/7/2020 9:19 AM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



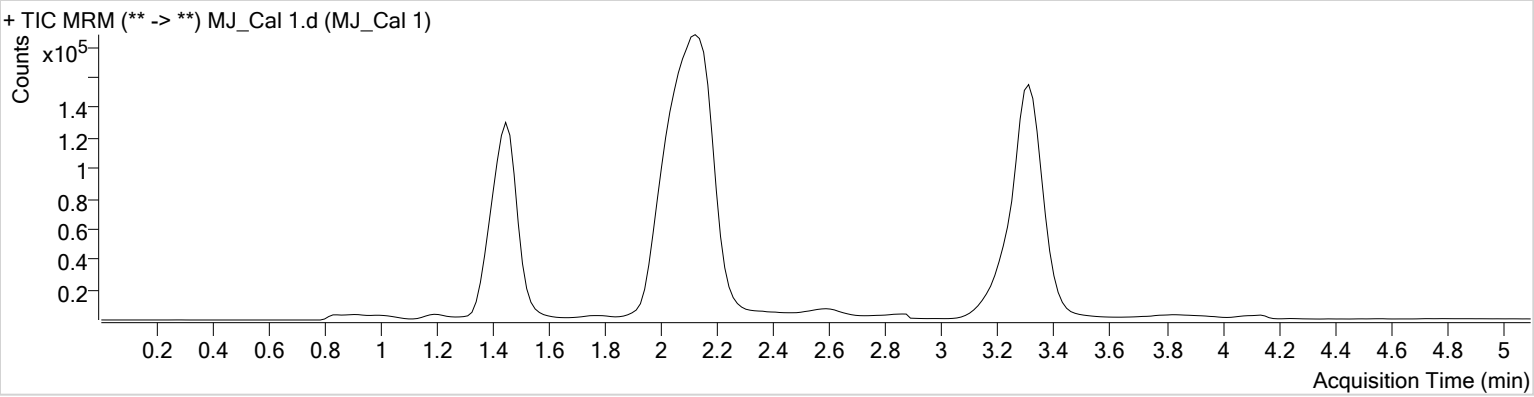
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✔	1.0	1.1	107.3
MJ_Cal 2	2	✔	3.0	2.9	95.9
MJ_Cal 3	3	✔	5.0	5.0	99.6
MJ_Cal 4	4	✔	10.0	9.7	97.4
MJ_Cal 5	5	✔	25.0	24.9	99.6
MJ_Cal 6	6	✔	50.0	49.7	99.5
MJ_Cal 7	7	✔	100.0	100.7	100.7

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Cal 1.d
Type	Cal	**Sample**	MJ_Cal 1
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 3:40:25 PM		
Sample Info.			

Sample Chromatogram



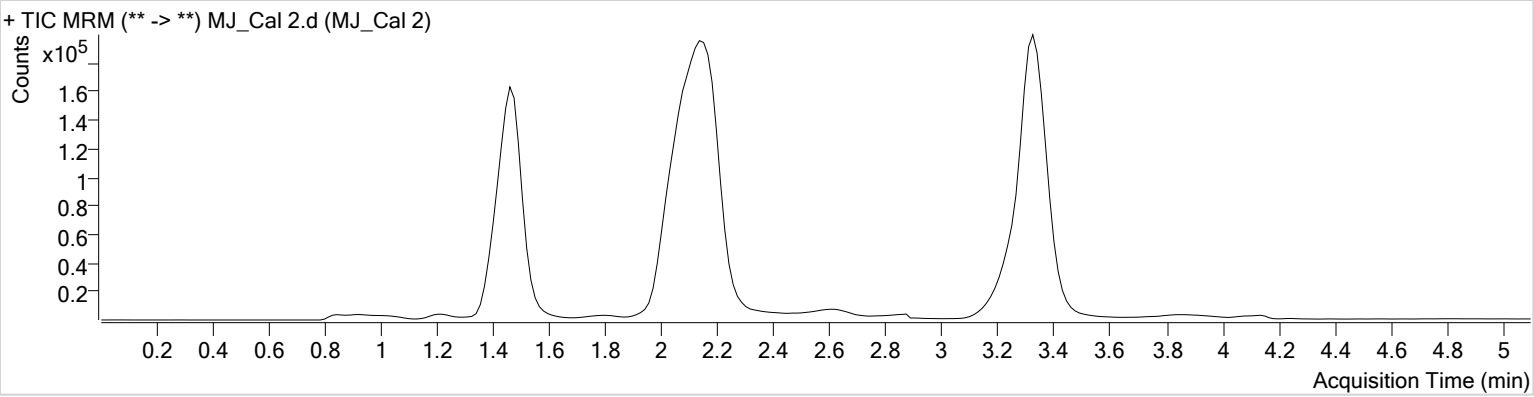
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.		
THC-OH	1.453	10626	∞	13.2	∞	573301	1.0727	ng/ml	Low
THC-COOH	1.474	32540	∞	50.8	∞	204987	5.4154	ng/ml	
THC	3.315	11067	∞	36.2	21.61	1259638	1.0778	ng/ml	Low

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Cal 2.d
Type	Cal	**Sample**	MJ_Cal 2
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 3:48:11 PM		
Sample Info.			

Sample Chromatogram



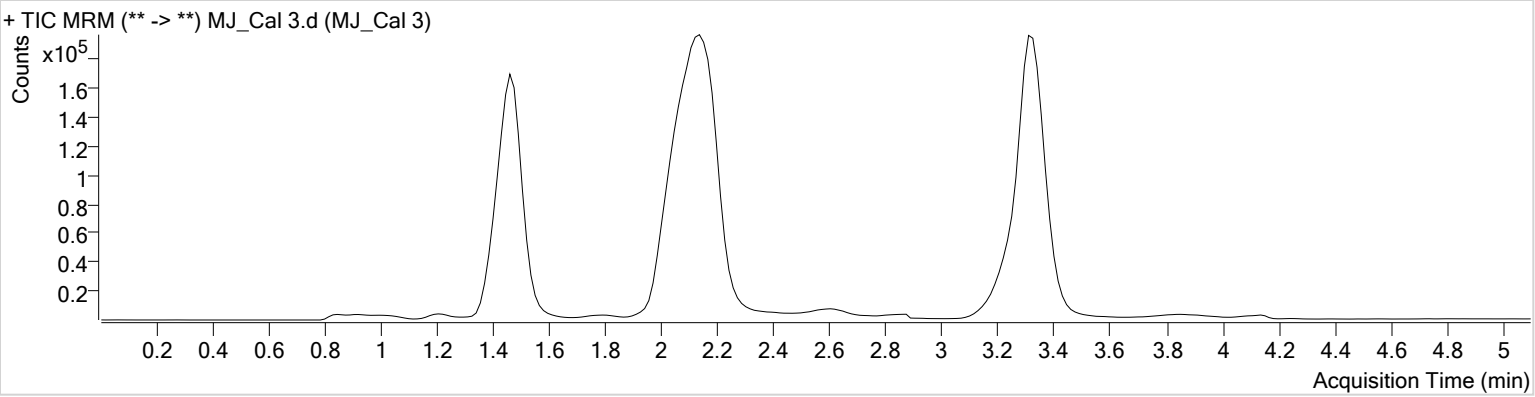
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.		
THC-OH	1.468	31722	∞	13.7	∞	647083	2.8784	ng/ml	Low
THC-COOH	1.489	59906	∞	56.1	∞	229875	9.6468	ng/ml	
THC	3.330	35845	∞	32.8	∞	1467500	2.9202	ng/ml	Low

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Cal 3.d
Type	Cal	**Sample**	MJ_Cal 3
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 3:55:48 PM		
Sample Info.			

Sample Chromatogram



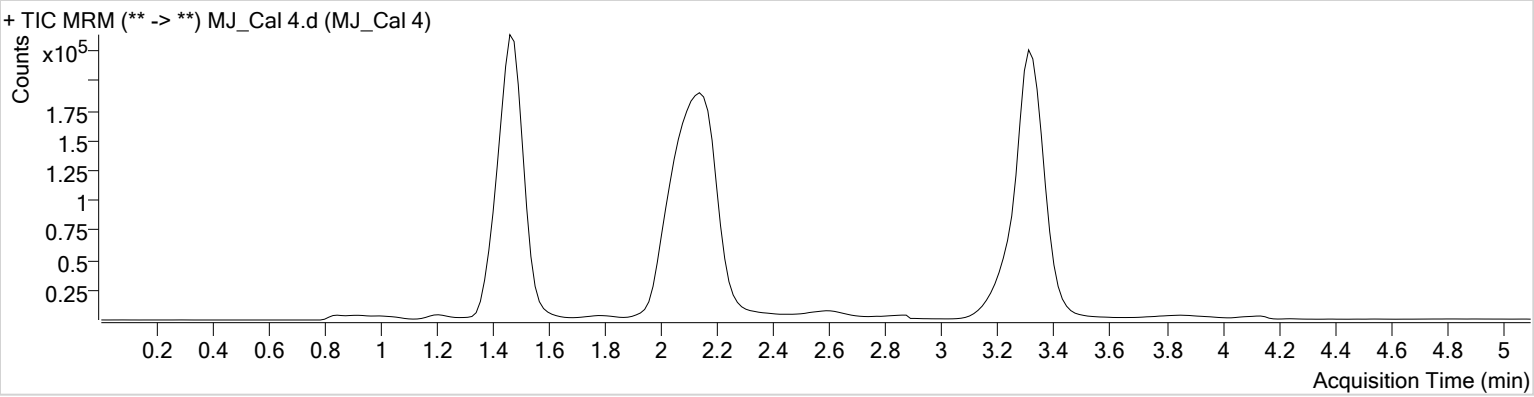
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	50507	∞	13.6	∞	597632	4.9802	ng/ml
THC-COOH	1.489	106226	∞	59.7	421.36	214681	19.3761	ng/ml
THC	3.330	59824	∞	30.9	∞	1408106	5.0477	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Cal 4.d
Type	Cal	**Sample**	MJ_Cal 4
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-E6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 4:03:26 PM		
Sample Info.			

Sample Chromatogram



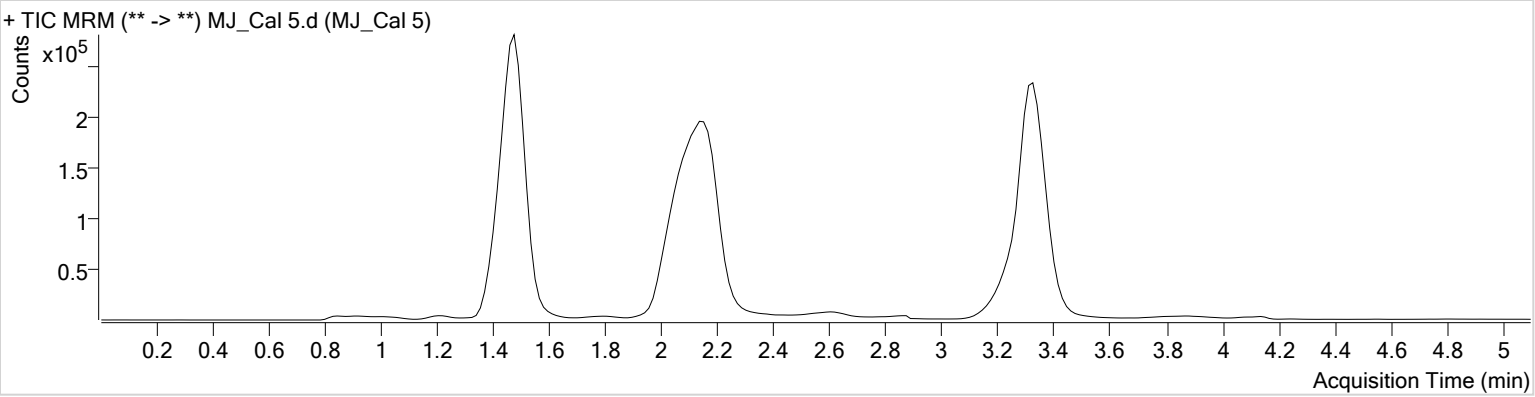
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	108462	∞	13.2	406.33	657662	9.7423	ng/ml
THC-COOH	1.489	280251	∞	62.3	∞	231834	49.0380	ng/ml
THC	3.330	125476	∞	29.4	∞	1541987	9.6287	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Cal 5.d
Type	Cal	**Sample**	MJ_Cal 5
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 4:11:02 PM		
Sample Info.			

Sample Chromatogram



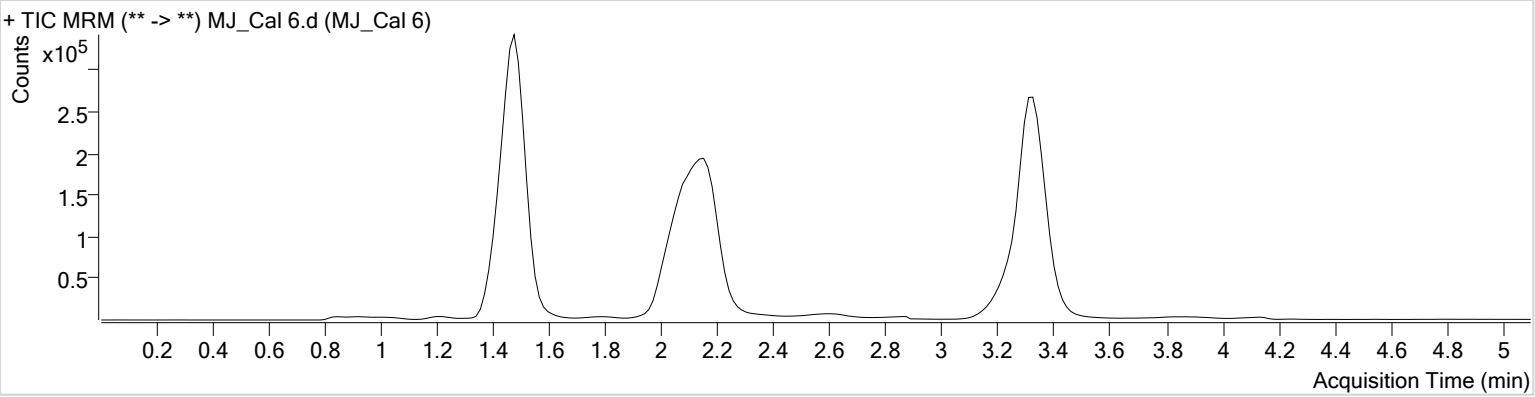
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	247040	∞	13.2	∞	586993	24.9001	ng/ml
THC-COOH	1.489	377471	∞	63.4	954.07	205752	75.0326	ng/ml
THC	3.330	279818	∞	29.2	∞	1363663	24.2154	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Cal 6.d
Type	Cal	**Sample**	MJ_Cal 6
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 4:18:39 PM		
Sample Info.			

Sample Chromatogram



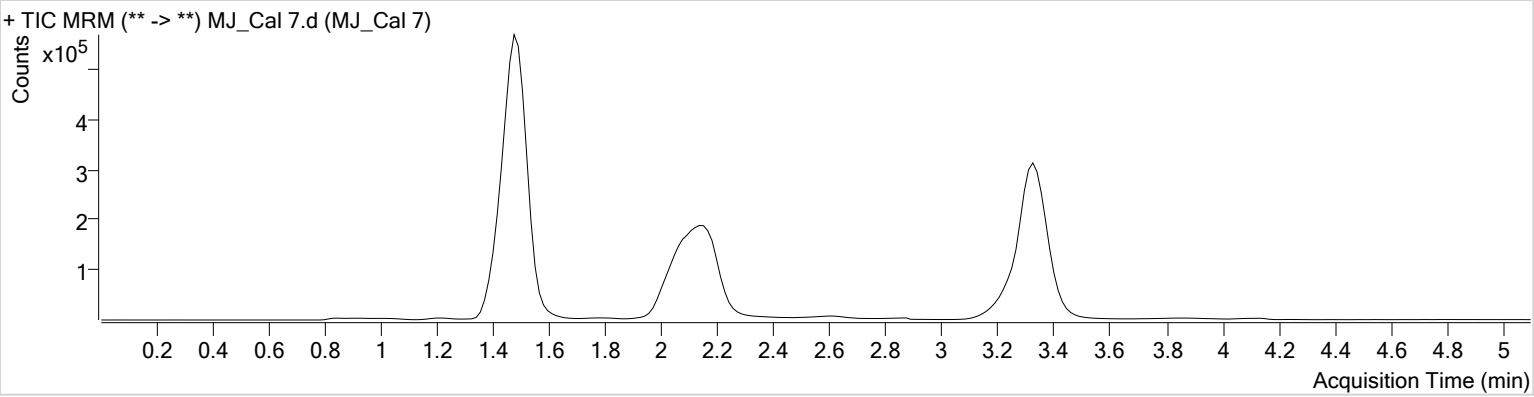
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	466657	∞	13.4	∞	555408	49.7358	ng/ml
THC-COOH	1.489	472453	∞	63.8	3801.34	195178	99.3774	ng/ml
THC	3.330	537384	∞	27.9	∞	1275818	49.6620	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 120320 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 12/7/2020 9:19:27 AM

Instrument	Instrument 1	**Data File**	MJ_Cal 7.d
Type	Cal	**Sample**	MJ_Cal 7
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/3/2020 4:26:15 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	860371	∞	13.3	∞	505932	100.6905	ng/ml
THC-COOH	1.489	1039699	∞	62.9	∞	170516	252.1136	ng/ml
THC	3.330	928695	∞	27.4	∞	1078863	101.4481	ng/ml