REVIEWED
By Anne Nord at 9:34 am, Sep 01, 2022

8/31/2022

Worklist: 6080

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2022-3083	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2022-3106	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2022-3219	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2022-3328	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2022-3402	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2022-3403	3	BCK	AM 27 Blood THC Quant by LC-QQQ
P2022-2491	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2022-2496	2	BCK	AM 27 Blood THC Quant by LC-QQQ
P2022-2523	3	BCK	AM 27 Blood THC Quant by LC-QQQ
P2022-2528	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2022-2532	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2022-2579	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2022-2581	1	BLOOD	AM 27 Blood THC Quant by LC-QQQ
P2022-2606	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2022-2634	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: 08/25/2022
Plate lot#: 220309
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: Lampire 22B52015-1
LCMS-QQQ ID: 069901

Analyst: Celena Shrum
Plate Retest Date: 09/09/2022
Mobile phase B: 0.1% Formic acid in Acetonitrile
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. 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** 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)***
☒ 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.
☒ 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 1ng/mL 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: There was a leak in the instrument, so the run stopped prior to finishing. New mobile phase had to be made so the run was restarted from the beginning on 082622.

THC curve range: 3-100, THC-OH curve range: 3-100.

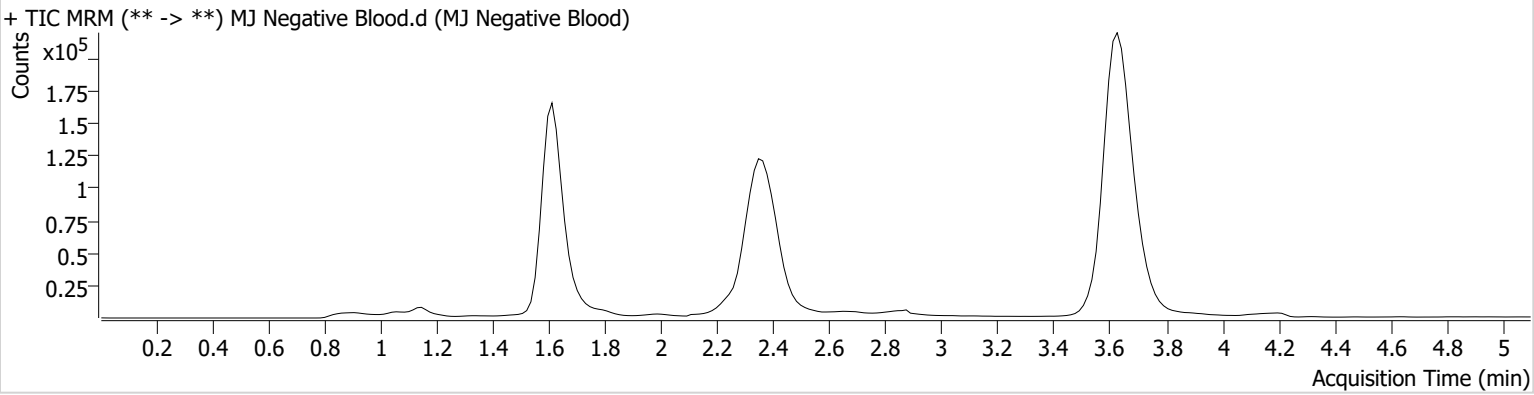
	1	2	3	4	5	6
a				P2022-2606-1	M2022-3403-3	QC 1
b				P2022-2581-1	M2022-3402-2	cal 100 ng
c				P2022-2579-1	M2022-3328-1	cal 50 ng
d				P2022-2532-1	M2022-3219-1	cal 25 ng
e				P2022-2528-1	M2022-3106-2	cal 10ng
f				P2022-2523-3	M2022-3083-1	cal 5 ng
g				P2022-2496-2	NEG Blood	cal 3 ng
h			P2022-2634-1	P2022-2491-1	QC 2	cal 1ng

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update	9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	MJ Negative Blood.d
Type	Sample	**Sample**	MJ Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-G5	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 2:08:13 PM		
Sample Info.			

Sample Chromatogram

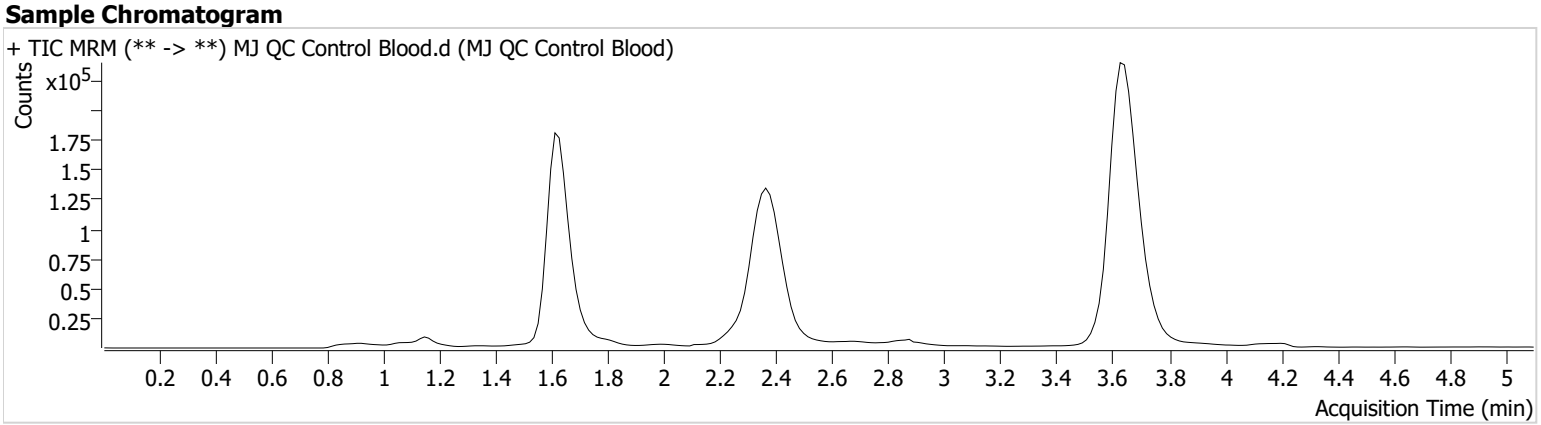


AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update	9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	MJ QC Control Blood.d
Type	QC	**Sample**	MJ QC Control Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 1:52:59 PM		
Sample Info.			

Sample Chromatogram



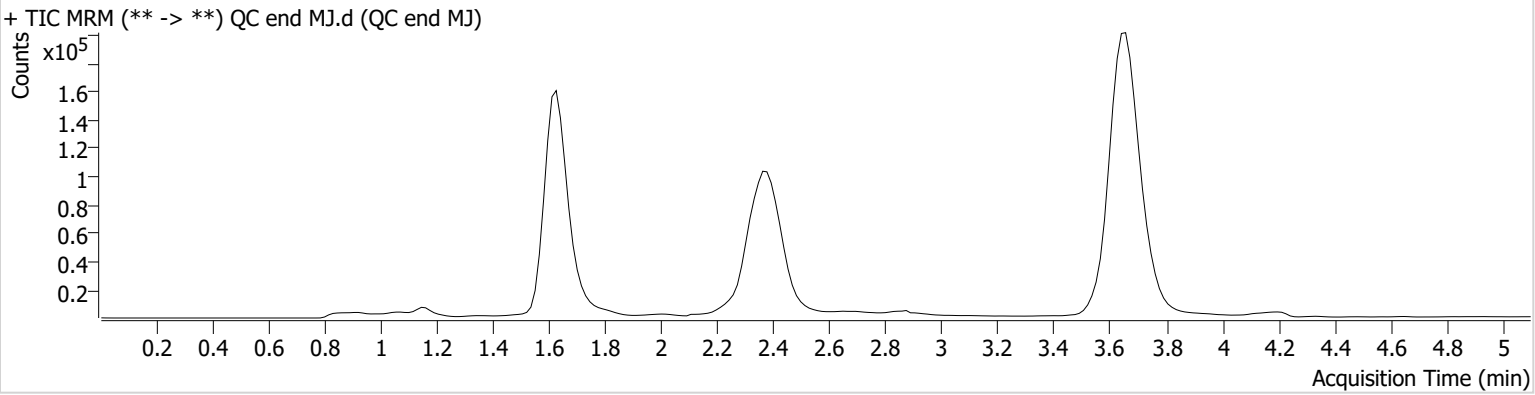
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.633	56254	∞	12.0	98.98	732249	4.6599	ng/ml
THC-COOH	1.655	48020	∞	56.1	515.37	146346	14.4190	ng/ml
THC	3.646	68674	135.74	32.7	∞	1716282	4.6653	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update 9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	QC end MJ.d
Type	QC	**Sample**	QC end MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 6:11:53 PM		
Sample Info.			

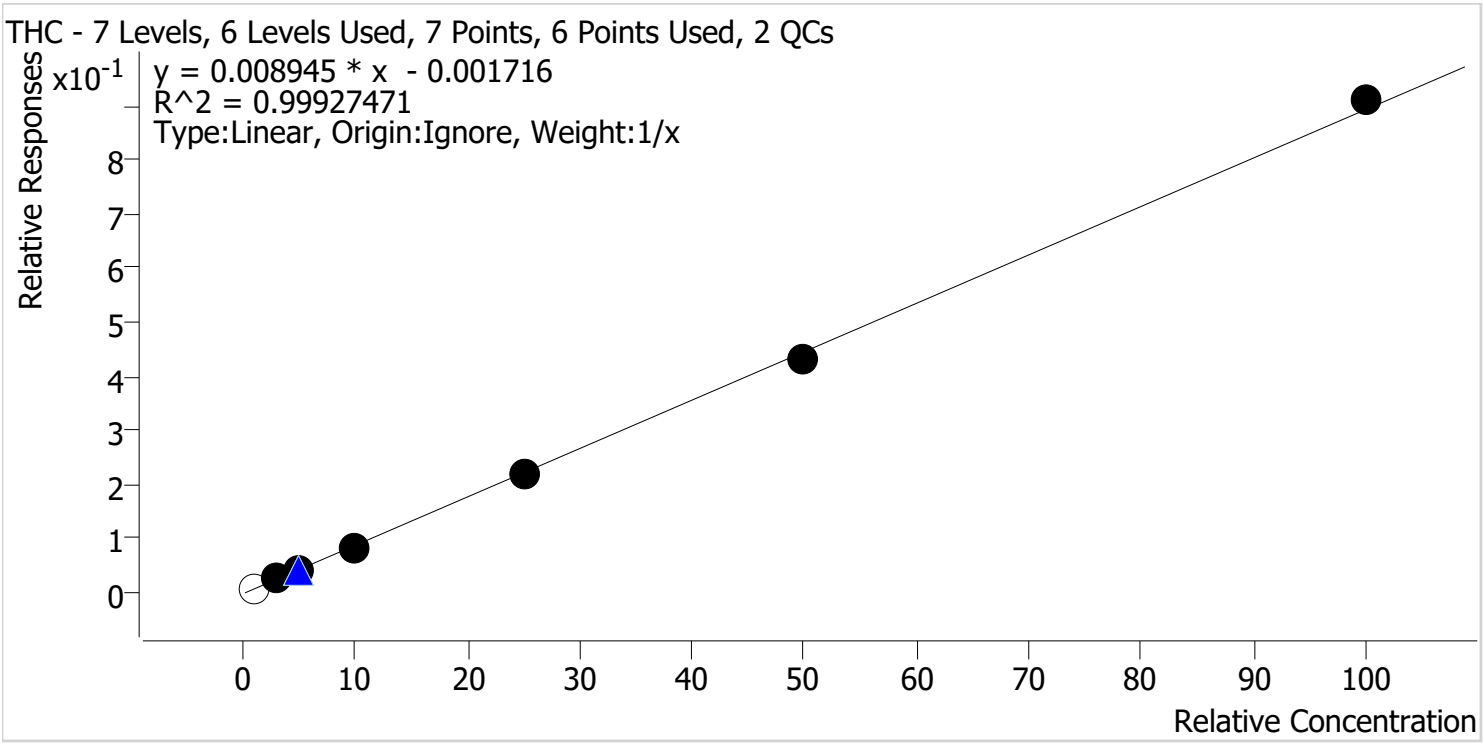
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.633	53071	∞	11.7	115.17	677318	4.7684	ng/ml
THC-COOH	1.655	43476	256.52	57.8	193.41	134186	14.2432	ng/ml
THC	3.661	62532	171.28	32.2	129.96	1507726	4.8287	ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Last Cal. Update	9/1/2022 9:52 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3

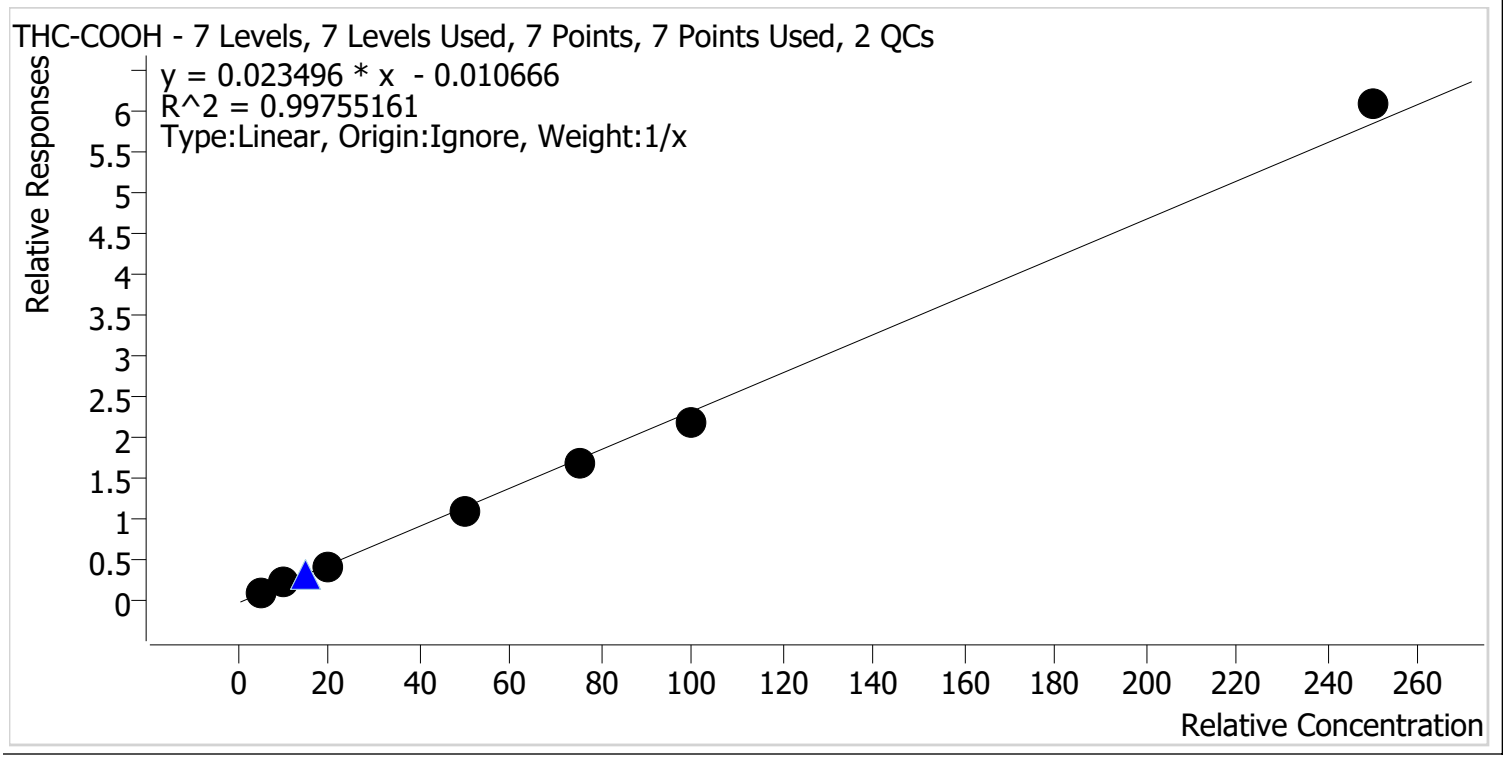


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✗	1.0	1.1	112.6
Cal 2 MJ	2	✔	3.0	3.2	107.0
Cal 3 MJ	3	✔	5.0	5.0	99.3
Cal 4 MJ	4	✔	10.0	9.6	96.2
Cal 5 MJ	5	✔	25.0	24.5	98.0
Cal 6 MJ	6	✔	50.0	48.8	97.6
Cal 7 MJ	7	✔	100.0	101.9	101.9

Calibrator 1 dropped due to not meeting ratio requirement.

AM #27 Cannabinoids Quant. Calibration Curve Report

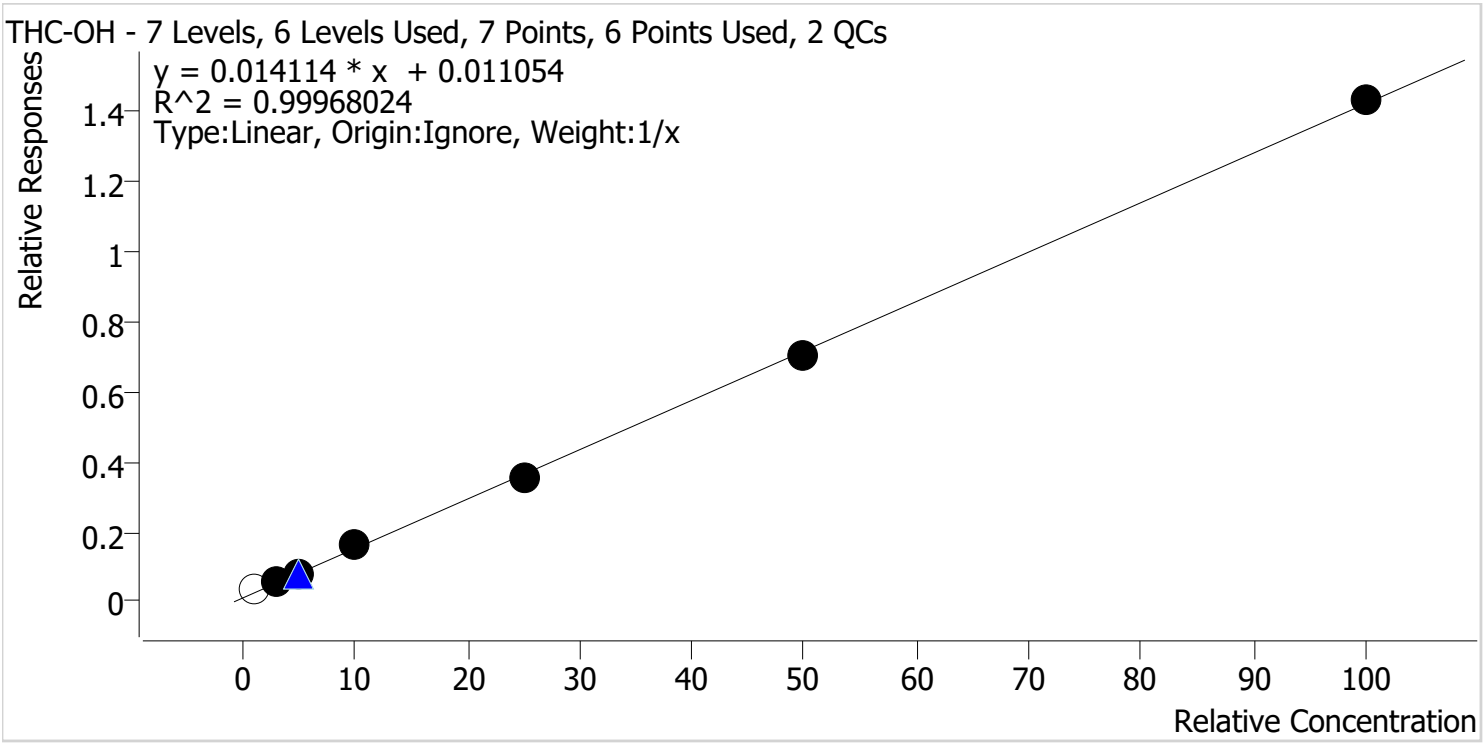
Batch results	D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Last Cal. Update	9/1/2022 9:52 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	5.0	5.5	110.2
Cal 2 MJ	2	✔	10.0	10.3	103.3
Cal 3 MJ	3	✔	20.0	19.0	95.2
Cal 4 MJ	4	✔	50.0	48.1	96.2
Cal 5 MJ	5	✔	75.0	72.2	96.3
Cal 6 MJ	6	✔	100.0	94.8	94.8
Cal 7 MJ	7	✔	250.0	260.0	104.0

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Last Cal. Update	9/1/2022 9:52 AM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✗	1.0	1.9	194.1
Cal 2 MJ	2	✔	3.0	3.0	100.6
Cal 3 MJ	3	✔	5.0	4.8	96.8
Cal 4 MJ	4	✔	10.0	10.5	104.7
Cal 5 MJ	5	✔	25.0	24.6	98.3
Cal 6 MJ	6	✔	50.0	49.4	98.8
Cal 7 MJ	7	✔	100.0	100.7	100.7

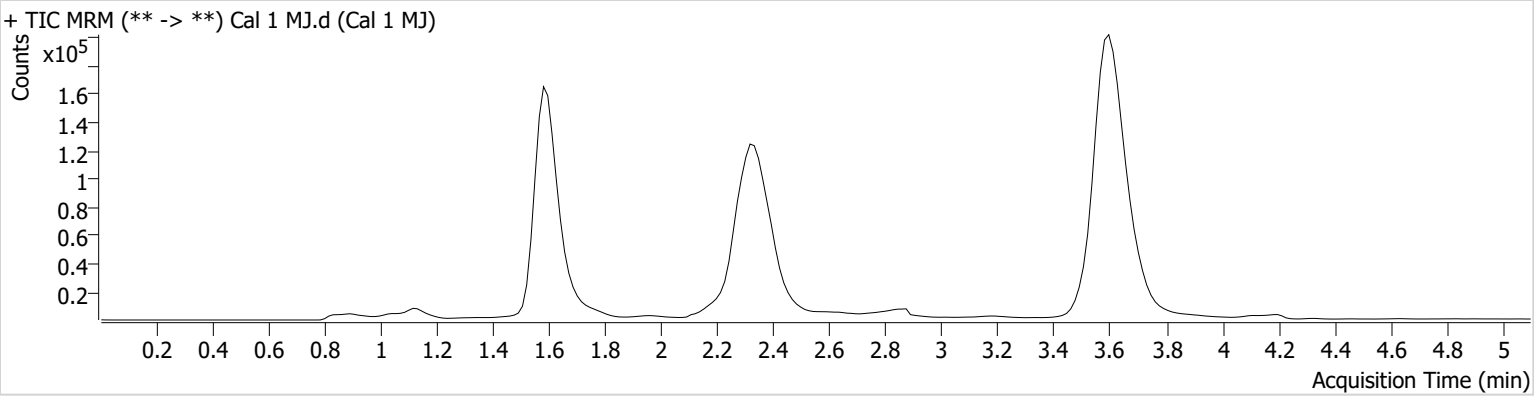
Calibrator 1 dropped due to not meeting ratio requirement.

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update	9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	Cal 1 MJ.d
Type	Cal	**Sample**	Cal 1 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 12:52:00 PM		
Sample Info.			

Sample Chromatogram



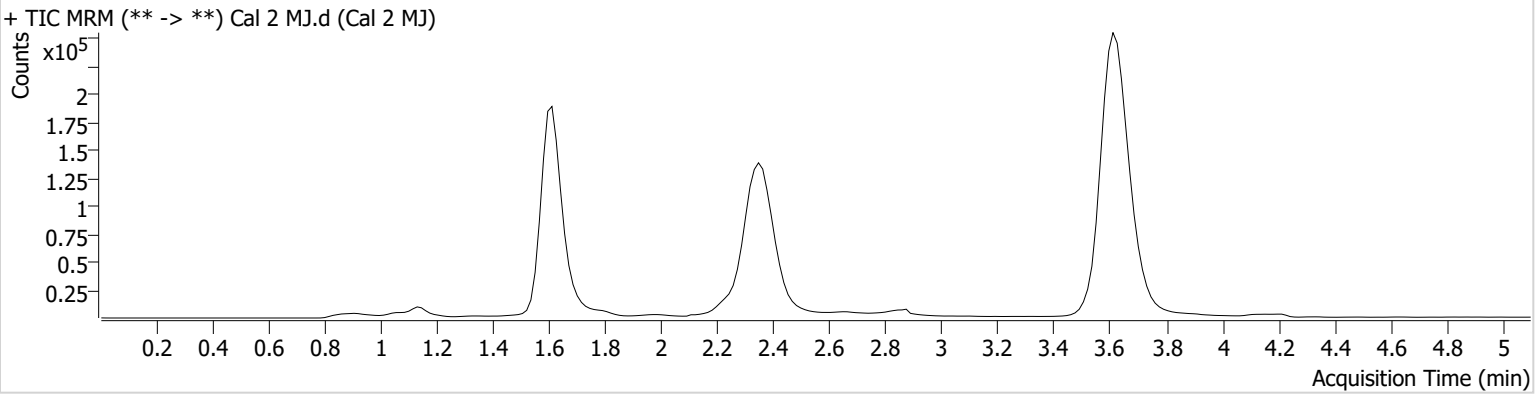
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.648	30154	∞	7.1 **Low**	14.34	784333	1.9407	ng/ml **Low**
THC-COOH	1.625	18130	54.61	48.0	49.07	152633	5.5093	ng/ml
THC	3.601	13785	23.23	49.5 **High**	∞	1649629	1.1261	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update 9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	Cal 2 MJ.d
Type	Cal	**Sample**	Cal 2 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 12:59:47 PM		
Sample Info.			

Sample Chromatogram



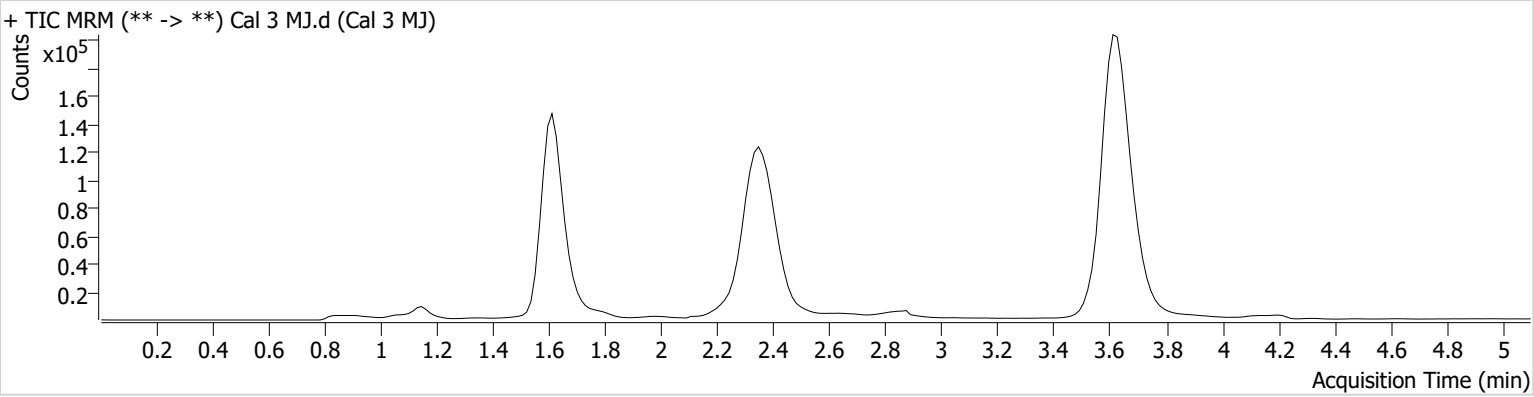
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.618	41118	∞	10.8	15.93	766235	3.0189	ng/ml
THC-COOH	1.640	33950	∞	52.6	260.88	146265	10.3325	ng/ml
THC	3.631	48047	310.36	29.5	94.59	1780657	3.2085	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update	9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	Cal 3 MJ.d
Type	Cal	**Sample**	Cal 3 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 1:07:22 PM		
Sample Info.			

Sample Chromatogram



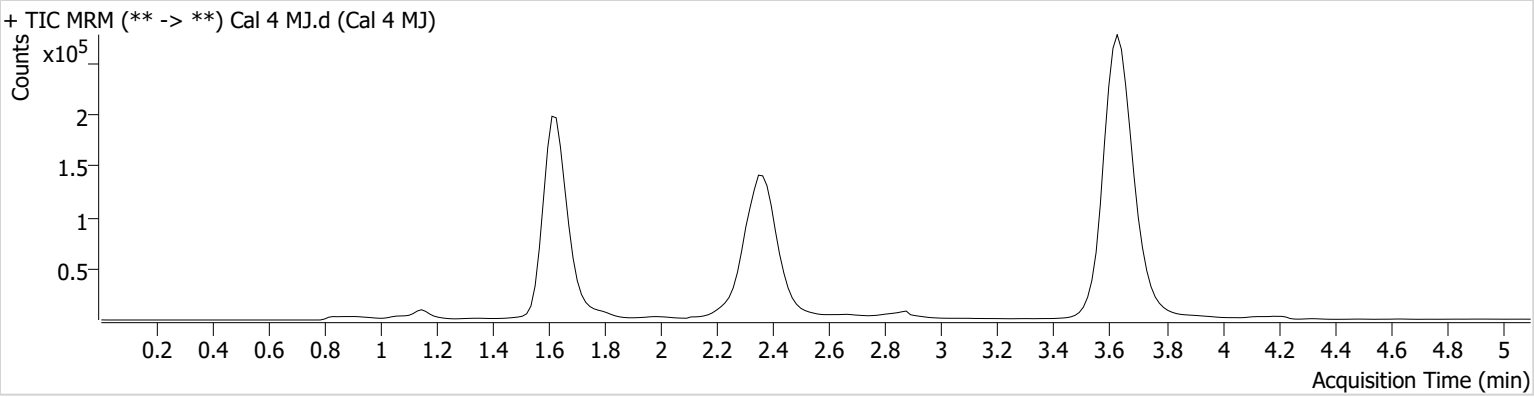
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.618	46731	∞	12.3	∞	588768	4.8404	ng/ml
THC-COOH	1.640	48360	520.42	54.7	271.04	110749	19.0384	ng/ml
THC	3.631	62960	179.97	27.8	17.68	1473942	4.9675	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update	9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	Cal 4 MJ.d
Type	Cal	**Sample**	Cal 4 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-E6	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 1:14:58 PM		
Sample Info.			

Sample Chromatogram



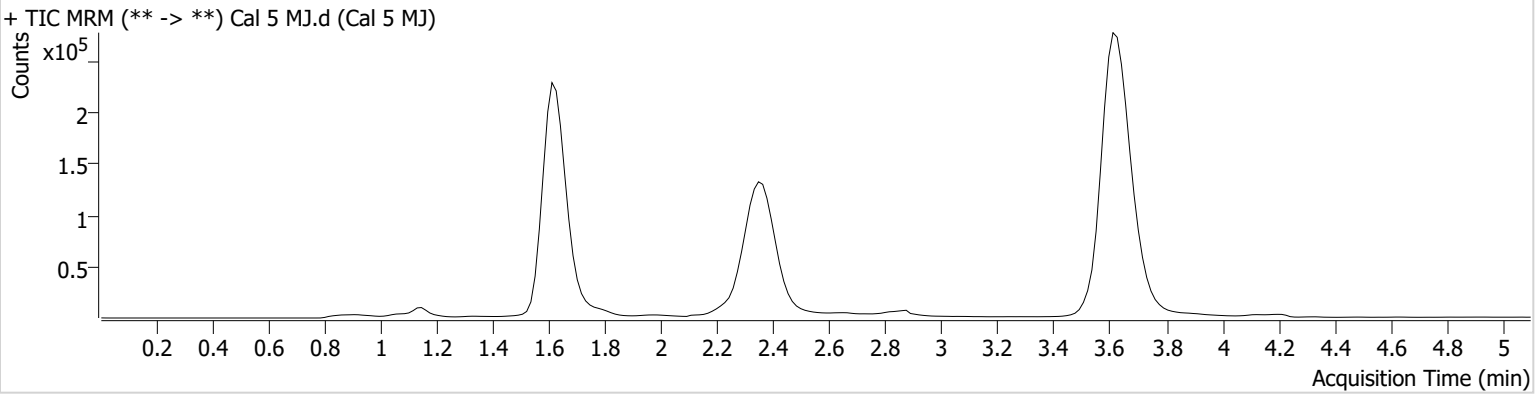
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.618	107125	278.34	11.2	162.19	674395	10.4714	ng/ml
THC-COOH	1.655	151768	590.41	56.5	1721.69	135616	48.0829	ng/ml
THC	3.631	160721	418.85	27.8	191.59	1906188	9.6183	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update 9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	Cal 5 MJ.d
Type	Cal	**Sample**	Cal 5 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 1:22:35 PM		
Sample Info.			

Sample Chromatogram



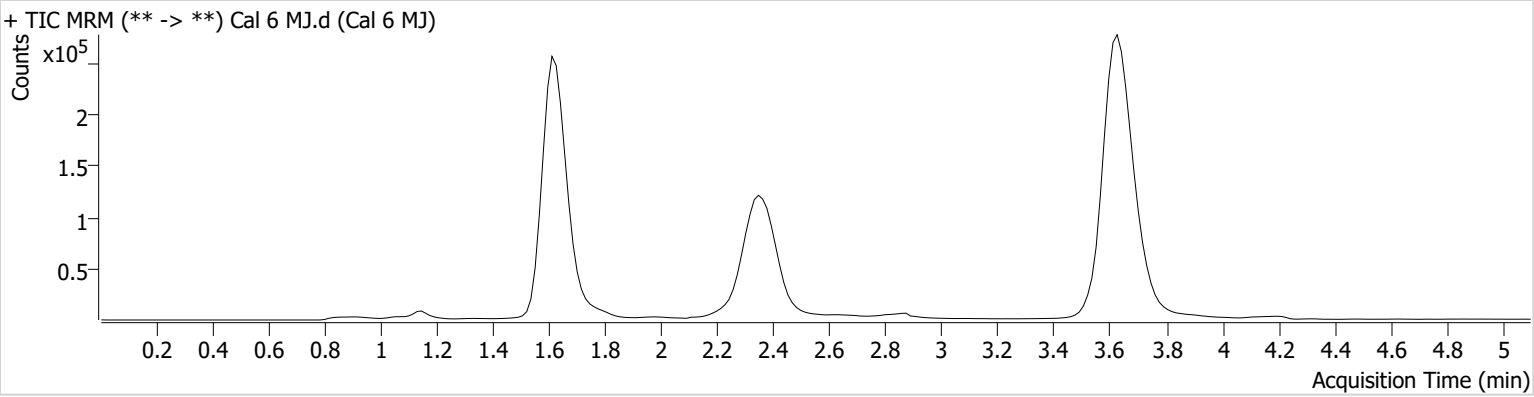
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.618	220181	883.44	13.2	1007.54	614913	24.5867	ng/ml
THC-COOH	1.640	199383	3243.65	55.3	∞	118231	72.2265	ng/ml
THC	3.631	358049	451.97	28.0	398.19	1646967	24.4972	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update 9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	Cal 6 MJ.d
Type	Cal	**Sample**	Cal 6 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 1:30:11 PM		
Sample Info.			

Sample Chromatogram



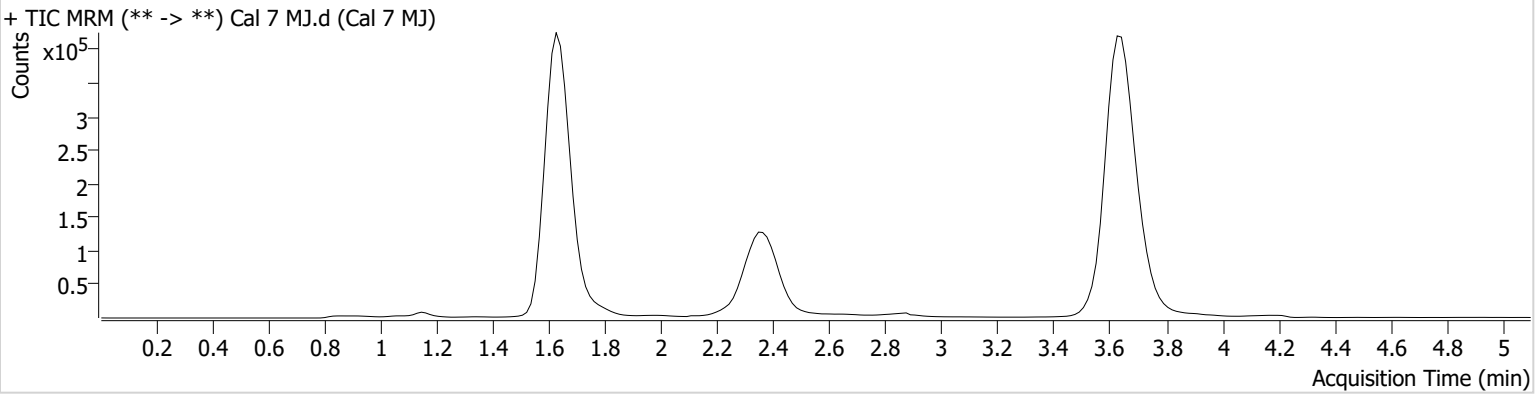
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.618	401561	∞	13.1	1826.03	566717	49.4206	ng/ml
THC-COOH	1.640	234034	1733.15	58.2	683.44	105529	94.8404	ng/ml
THC	3.631	604946	3439.22	27.6	1227.05	1390837	48.8196	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\082522 AM 27 28 CS TS\082622 Injections\QuantResults\AM 27.batch.bin
Calibration Last Update 9/1/2022 9:52:47 AM

Instrument	Falco (069901)	**Data File**	Cal 7 MJ.d
Type	Cal	**Sample**	Cal 7 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/26/2022 1:37:47 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.618	796233	∞	13.3	2755.00	556111	100.6620	ng/ml
THC-COOH	1.655	600862	∞	59.6	4058.66	98540	259.9700	ng/ml
THC	3.646	1406353	∞	27.6	3321.44	1546070	101.8889	ng/ml