REVIEWED
By Celena Shrum at 12:01 pm, Feb 14, 2023

TS

2/13/2023

Worklist: 6244

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
P2023-0098	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2023-0129	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2023-0138	1	CBUK	AM 27 Blood THC Quant by LC-QQQ	
P2023-0162	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2023-0168	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2023-0176	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2023-0202	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2023-0215	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2023-0226	2	BCK	AM 27 Blood THC Quant by LC-QQQ	

TS

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

Extraction Date: 02/06/2023
Plate lot#: 230113
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: Lampire 23A52593
Column: UCT Selectra DA 100 x 2.1mm 3um

Analyst: Tamara Salazar
Plate Retest Date: 07/13/2023
Mobile phase B: 0.1% Formic acid in Acetonitrile
Blank Urine Lot: N/A
LCMS-QQQ ID: 069901

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.
☒ 3. Using a calibrated pipette, add **1000µl blood and urine (if applicable) (calibrated pipette)** into the appropriate wells of analytical (standards) plate. **Pipette ID:** *42*
☒ 4. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 5. Add **500µL of 0.1% formic acid in water to blood samples, and 500µL of saturated phosphate buffer to urine samples** in the wells of the analytical plate.
☒ 6. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 7. Transfer **700-800µL of blood+acid or urine+acid** mixture to corresponding wells of SLE+ plate. Amount transferred: *750µL*
☒ 8. 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)***
☒ 9. Wait 5 minutes.
☒ 10. Add **2.25mL MTBE. (*Add in 3 increments of 750uL)***
☒ 11. Wait 5 minutes.
☒ 12. Apply positive pressure for approx. 15 seconds. ***(10-15 PSI- Selector to the left).***
☒ 13. Add **2.25mL Hexane. (*Add in 3 increments of 750uL)***
☒ 14. Wait 5 minutes.
☒ 15. Apply positive pressure for approx. 15 seconds. ***(10-15 PSI- Selector to the left).***
☒ 16. Remove plate containing eluate. Place on SPE Dry and evaporate to dryness at approx. 35°C.
☒ 17. 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. Did all QCs pass for each analyte? (if not, describe in comments section)
☒ 5. Enter QCs into control charting.
☒ 6. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: THC 3-100 – calibrator one dropped due to S/N

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	1	2	3	4	5	6
A	IS + Cal. 1	IS + QC_1	P2023-0202-1	IS + Sample	IS + Sample	IS + QC_1
B	IS + Cal. 2	Neg Blood	P2023-0215-2	IS + Sample	IS + Sample	IS + Cal. 7
C	IS + Cal. 3	P2023-0098-1	P2023-0226-2	IS + Sample	IS + Sample	IS + Cal. 6
D	IS + Cal. 4	P2023-0129-2	IS + Sample	IS + Sample	IS + Sample	IS + Cal. 5
E	IS + Cal. 5	P2023-0138-1	IS + Sample	IS + Sample	IS + Sample	IS + Cal. 4
F	IS + Cal. 6	P2023-0162-2	IS + Sample	IS + Sample	IS + Sample	IS + Cal. 3
G	IS + Cal. 7	P2023-0168-1	IS + Sample	IS + Sample	IS + Sample	IS + Cal. 2
H	IS + QC_1	P2023-0176-1	IS + Sample	IS + Sample	IS + QC_1	IS + Cal. 1

All wells to contain 100 µl of residual DMSO

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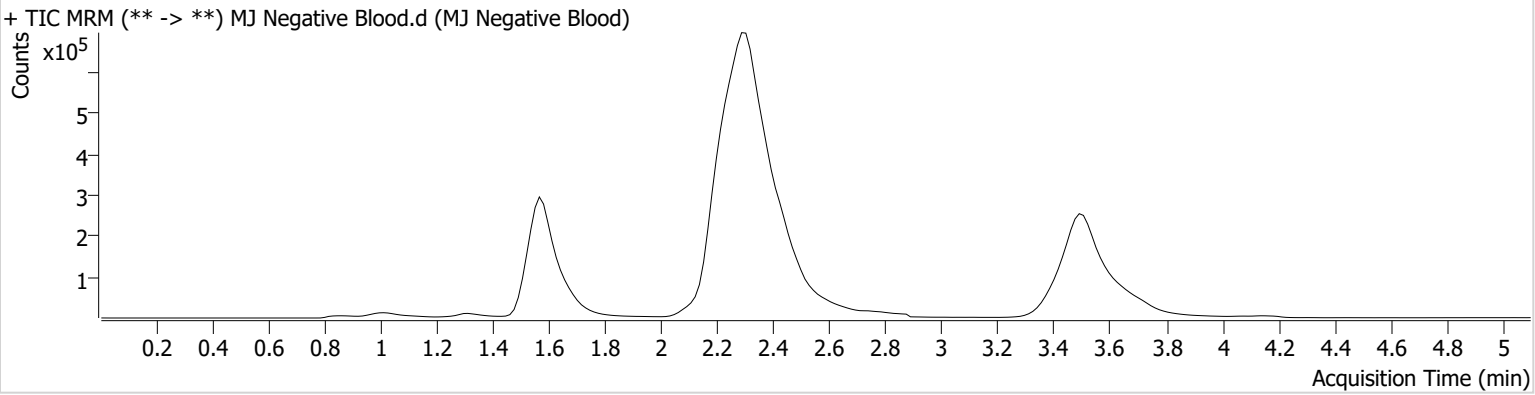
AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	MJ Negative Blood.d
Type	Sample	**Sample**	MJ Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-B2	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 2:05:12 PM		
Sample Info.			

Sample Chromatogram



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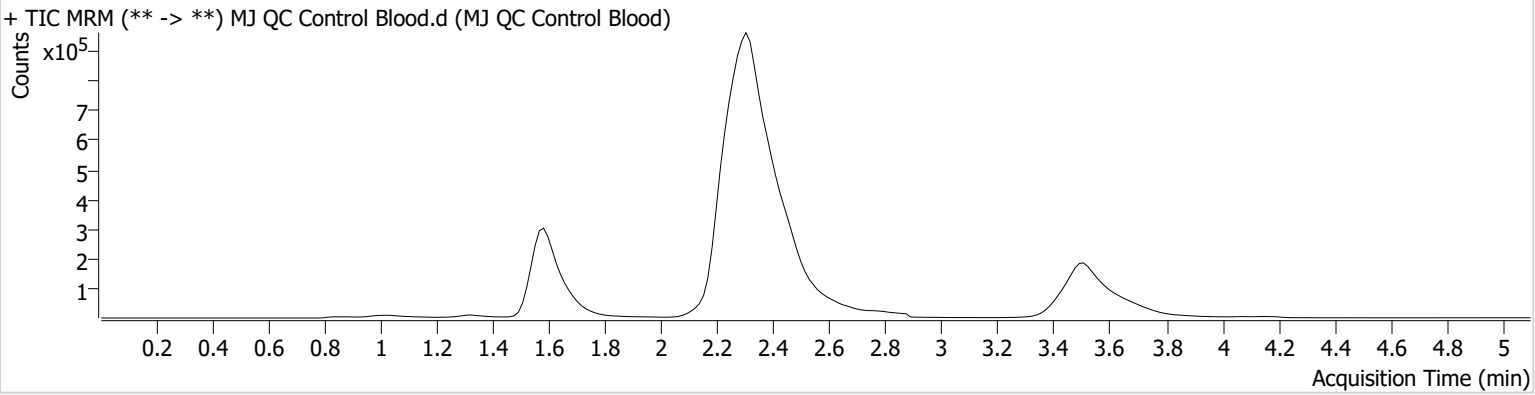
AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	MJ QC Control Blood.d
Type	QC	**Sample**	MJ QC Control Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 1:49:58 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.588	118512	∞	12.9	∞	1468159	4.5235	ng/ml
THC-COOH	1.614	30267	362.45	254.2	149.61	369232	14.9180	ng/ml
THC	3.510	95772	∞	24.5	119.85	2057324	5.3449	ng/ml

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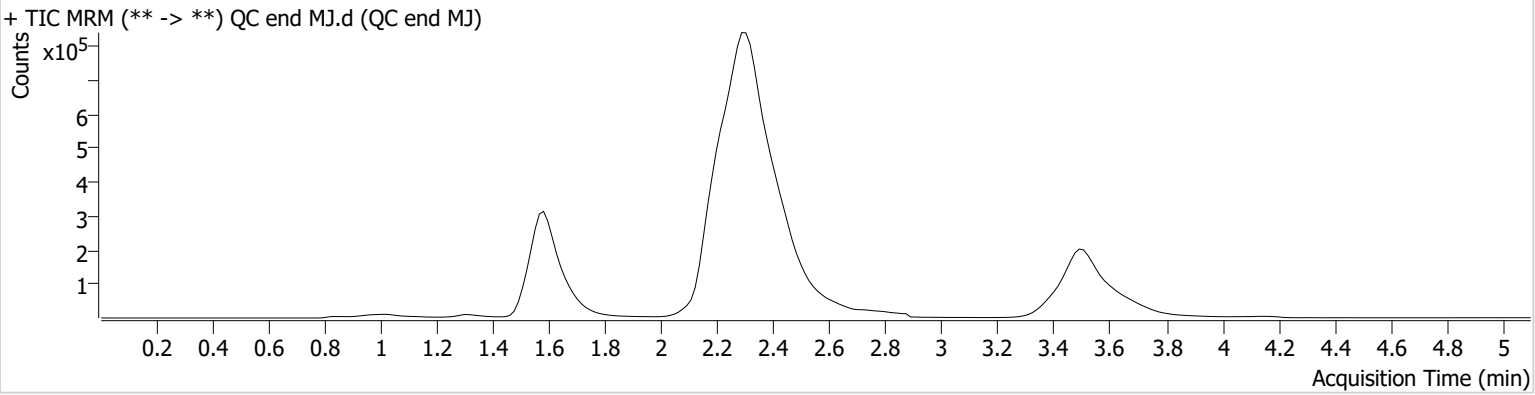
AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	QC end MJ.d
Type	QC	**Sample**	QC end MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 4:37:32 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.588	127485	∞	13.1	∞	1571290	4.5460	ng/ml
THC-COOH	1.614	34064	699.32	256.0	1547.45	424130	14.6365	ng/ml
THC	3.510	104167	∞	24.4	∞	2285228	5.2377	ng/ml

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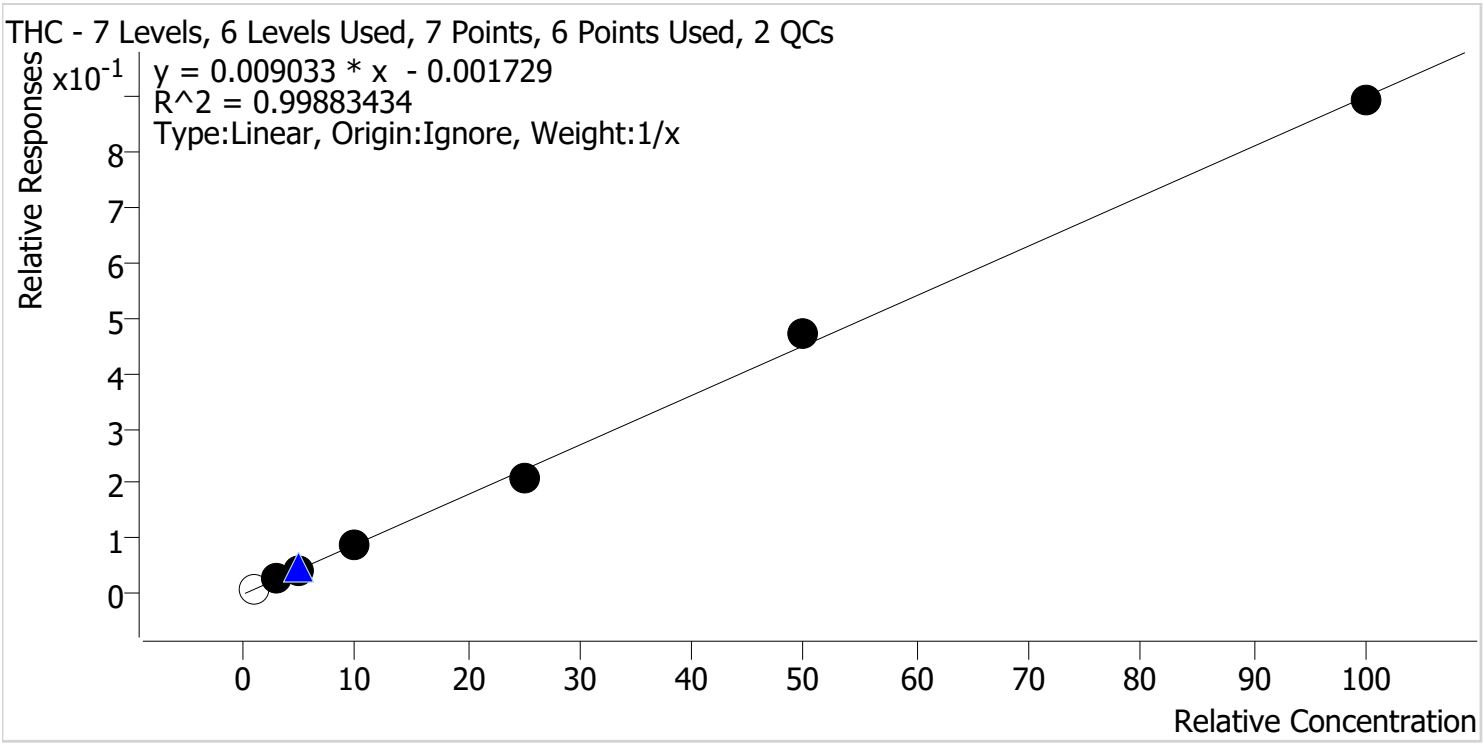
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	2/6/2023 4:53 PM
Analyst Name	ISP\tsalazar
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✗	1.0	1.1	114.2
Cal 2 MJ	2	✔	3.0	3.0	101.2
Cal 3 MJ	3	✔	5.0	5.0	100.4
Cal 4 MJ	4	✔	10.0	10.0	100.4
Cal 5 MJ	5	✔	25.0	23.7	94.6
Cal 6 MJ	6	✔	50.0	52.1	104.1
Cal 7 MJ	7	✔	100.0	99.2	99.2

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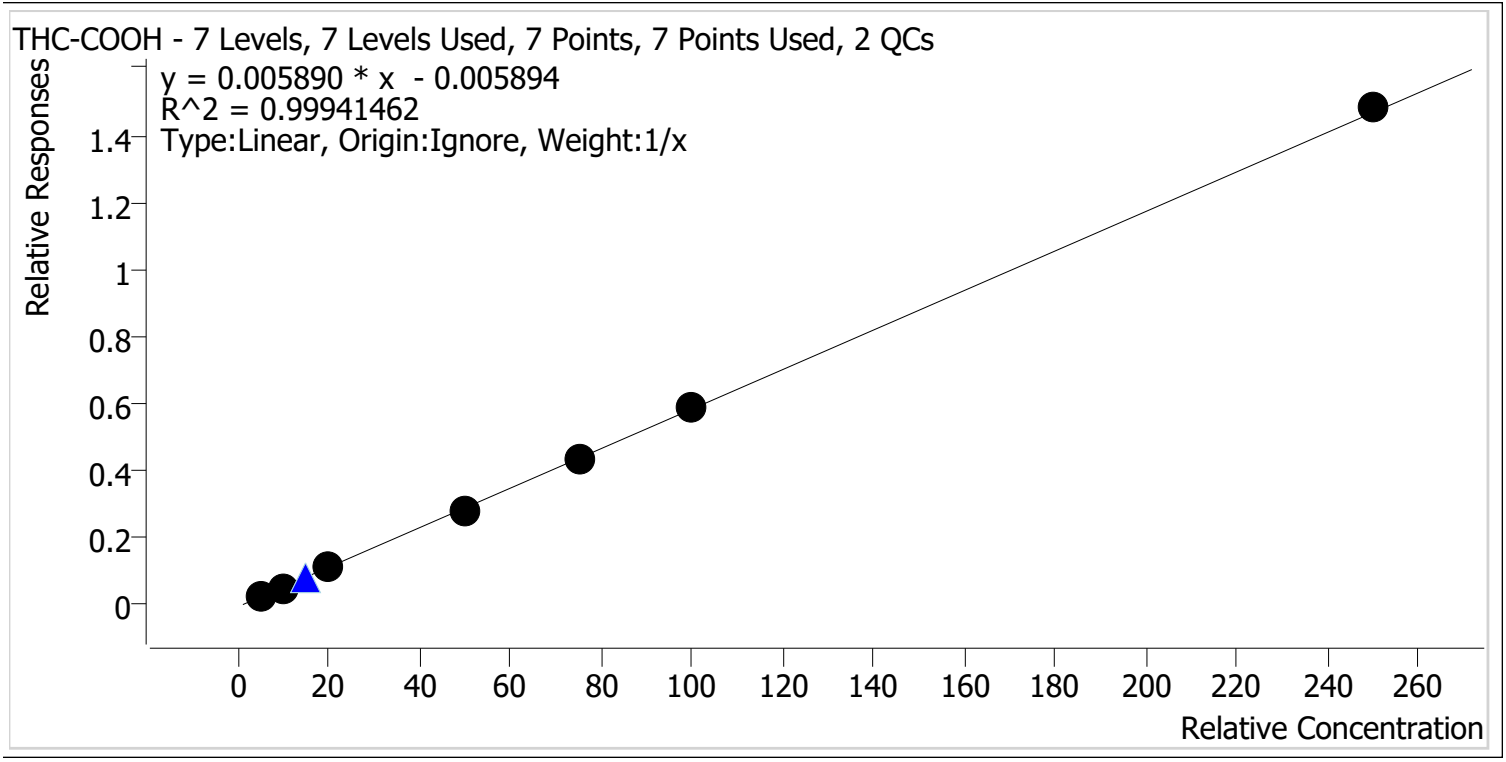
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	2/6/2023 4:53 PM
Analyst Name	ISP\tsalazar
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	5.0	5.5	110.0
Cal 2 MJ	2	✔	10.0	9.6	96.3
Cal 3 MJ	3	✔	20.0	19.4	97.0
Cal 4 MJ	4	✔	50.0	47.6	95.2
Cal 5 MJ	5	✔	75.0	75.5	100.6
Cal 6 MJ	6	✔	100.0	100.0	100.0
Cal 7 MJ	7	✔	250.0	252.5	101.0

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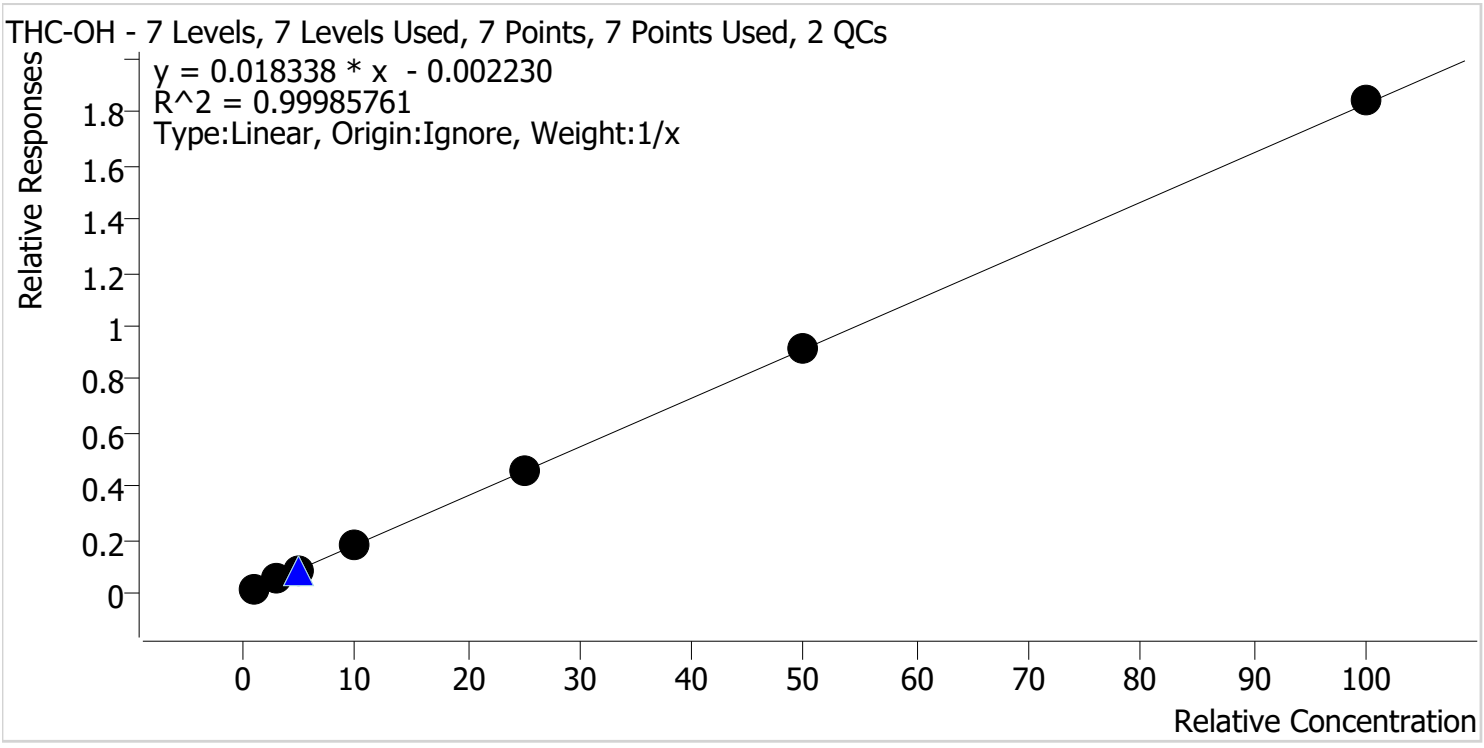
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	2/6/2023 4:53 PM
Analyst Name	ISP\tsalazar
Analyte	THC-OH
Internal Standard	THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	1.0	1.1	107.7
Cal 2 MJ	2	✔	3.0	2.9	98.2
Cal 3 MJ	3	✔	5.0	4.8	96.3
Cal 4 MJ	4	✔	10.0	9.8	98.0
Cal 5 MJ	5	✔	25.0	24.9	99.5
Cal 6 MJ	6	✔	50.0	49.8	99.6
Cal 7 MJ	7	✔	100.0	100.7	100.7

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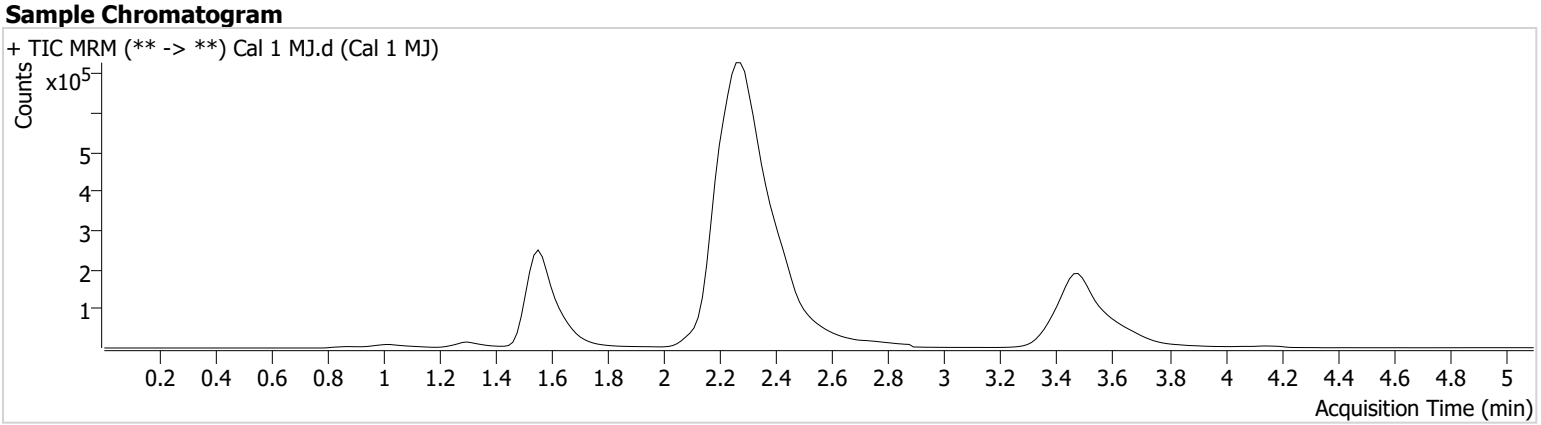
AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	Cal 1 MJ.d
Type	Cal	**Sample**	Cal 1 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 12:49:00 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.558	23458	∞	13.6	25.73	1339287	1.0768	ng/ml **Low**
THC-COOH	1.584	9349	159.33	256.1	∞	352674	5.5015	ng/ml
THC	3.495	19155	∞	22.9	6.64 **Low**	2230474	1.1421	ng/ml

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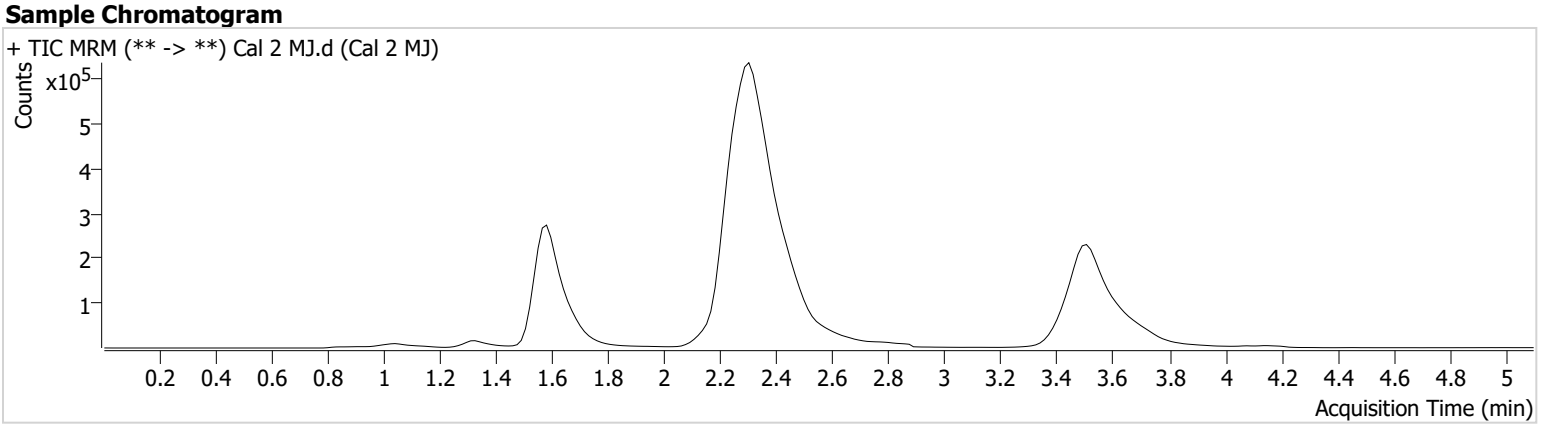
AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	Cal 2 MJ.d
Type	Cal	**Sample**	Cal 2 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 12:56:45 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.588	70145	∞	13.4	29.81	1353838	2.9470	ng/ml Low
THC-COOH	1.614	18538	95.16	255.9	∞	364878	9.6267	ng/ml
THC	3.510	64568	∞	25.3	40.48	2511583	3.0374	ng/ml

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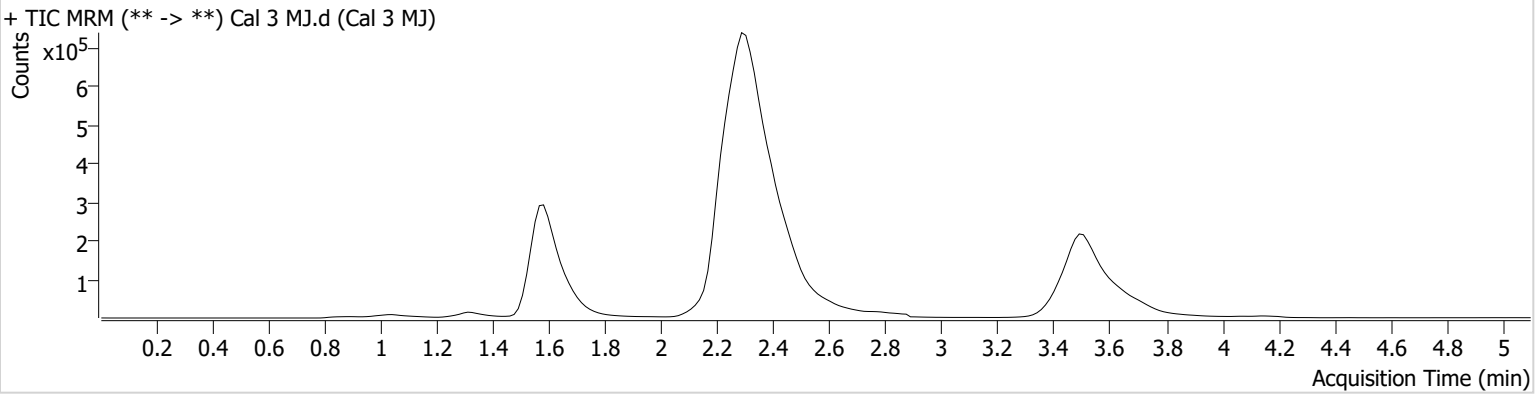
AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	Cal 3 MJ.d
Type	Cal	**Sample**	Cal 3 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 1:04:22 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.588	118306	∞	13.0	176.43	1375248	4.8128	ng/ml
THC-COOH	1.599	39088	735.45	251.0	∞	360755	19.3966	ng/ml
THC	3.510	103338	∞	24.8	∞	2368945	5.0206	ng/ml

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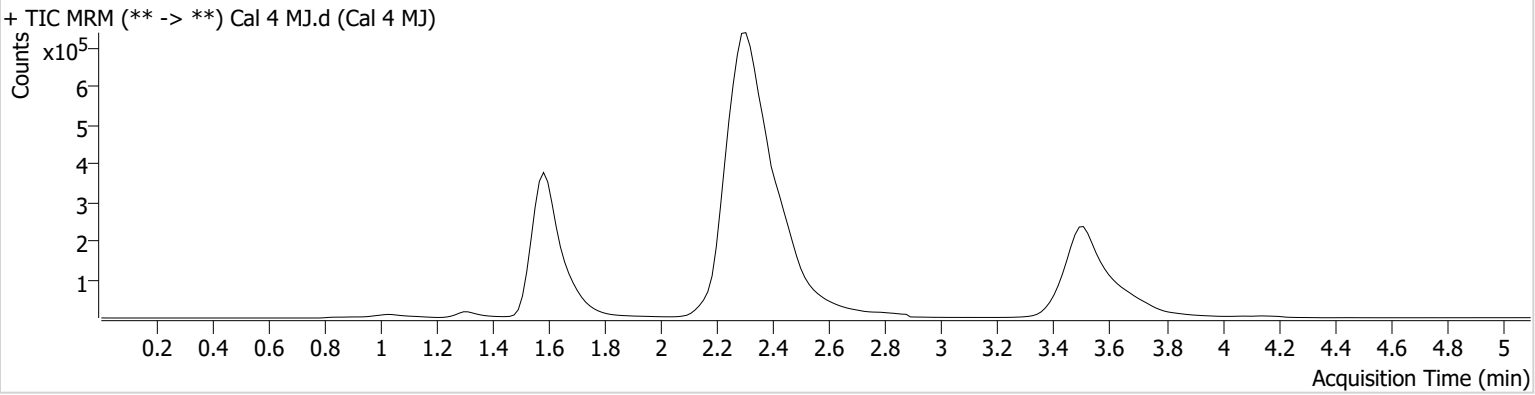
AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	Cal 4 MJ.d
Type	Cal	**Sample**	Cal 4 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 1:11:58 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.588	242289	∞	13.0	264.25	1364476	9.8048	ng/ml
THC-COOH	1.599	97441	∞	252.0	1174.56	355131	47.5854	ng/ml
THC	3.510	209240	∞	23.7	116.15	2352428	10.0383	ng/ml

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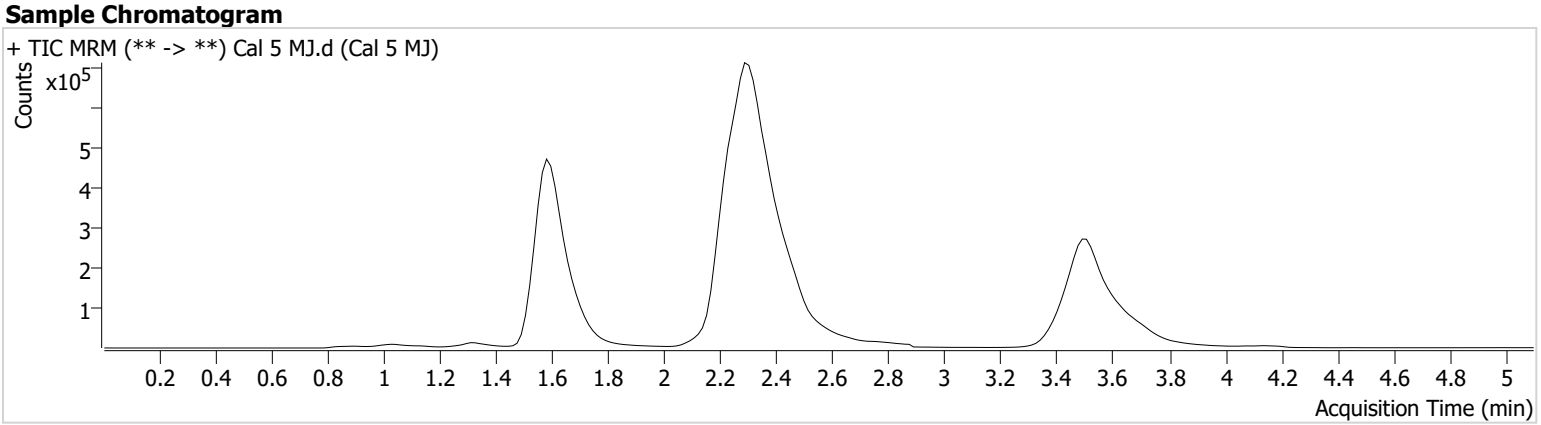
AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	Cal 5 MJ.d
Type	Cal	**Sample**	Cal 5 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 1:19:33 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.588	649858	∞	13.1	2341.48	1431936	24.8700	ng/ml
THC-COOH	1.614	158892	1754.32	242.0	6108.74	362348	75.4507	ng/ml
THC	3.510	560176	∞	23.2	∞	2642470	23.6598	ng/ml

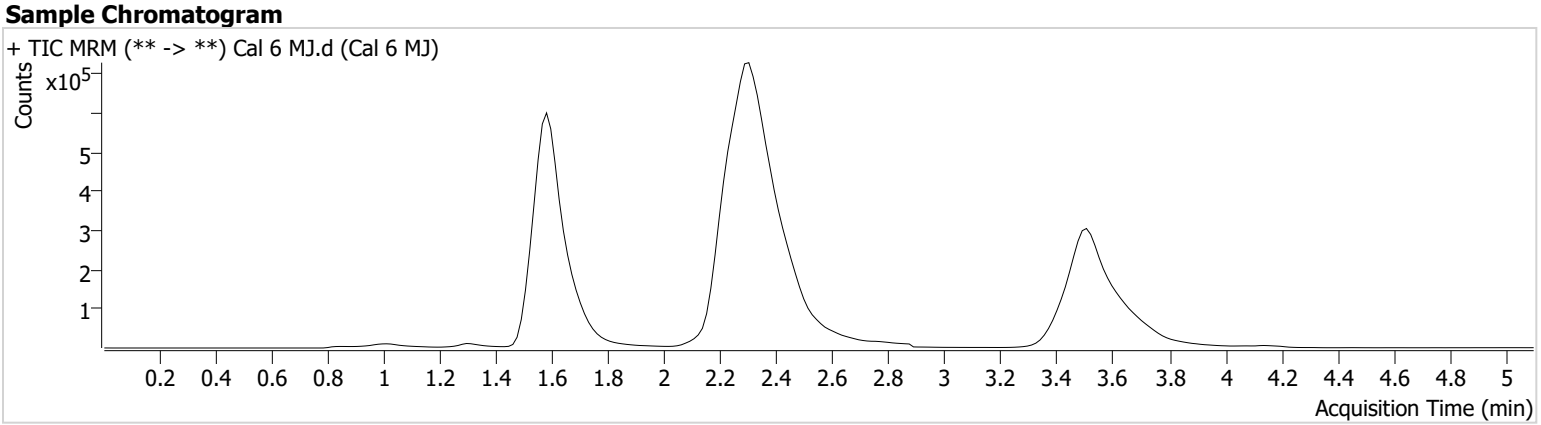
TS

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	Cal 6 MJ.d
Type	Cal	**Sample**	Cal 6 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 1:27:10 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.588	1284908	∞	13.2	∞	1409998	49.8158	ng/ml
THC-COOH	1.599	199887	603.68	240.0	∞	342961	99.9540	ng/ml
THC	3.510	1067788	∞	22.5	∞	2278394	52.0744	ng/ml

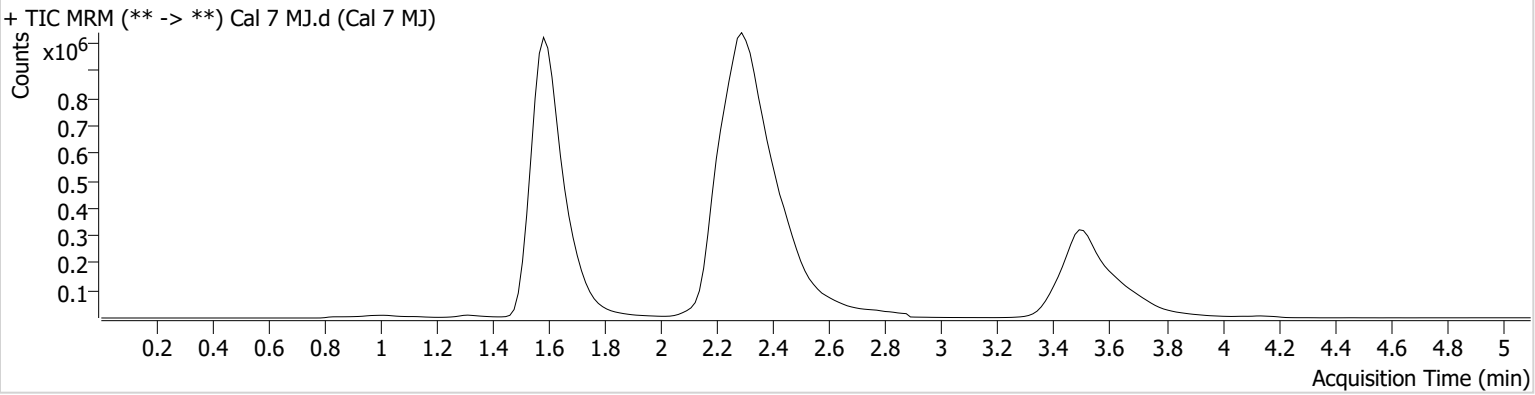
TS

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2023\AM 27 28\020623 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 2/6/2023 4:53:02 PM

Instrument	Falco (069901)	**Data File**	Cal 7 MJ.d
Type	Cal	**Sample**	Cal 7 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/6/2023 1:34:46 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.573	2567404	∞	13.4	∞	1392383	100.6728	ng/ml
THC-COOH	1.599	505807	13777.29	241.8	5538.05	341479	252.4852	ng/ml
THC	3.510	1766981	∞	22.4	∞	1976339	99.1695	ng/ml