REVIEWED
By Celena Shrum at 2:15 pm, Jun 15, 2021

TS 6/10/2021

Worklist: 5039

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2021-1933	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1592	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1605	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1614	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1833	3	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1866	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1870	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1902	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1911	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1927	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1928	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1930	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1948	5	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: 06/10/2021
Plate lot#: IDP-108-2-210412
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: Lampire 20L20723
LCMS-QQQ ID: 069901

Analyst: Tamara Salazar
Plate Re-test Date: 10/12/21
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, 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 **700-800µL of blood+acid or urine+acid** mixture to corresponding wells of SLE+ plate. Amount transferred: 800uL
☒ 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 samples with calculated concentrations for THC at 1ng/mL or greater and OH-THC at 3ng/mL or greater may be reported quantitatively (blood only). Calculated concentrations for carboxy-THC of 5ng/mL may be reported qualitatively. 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: *THC-OH not evaluated due to a possible interfering compound.*

TS

	1	2	3	4	5	6
A	IS + Cal. 1	Neg Blood	P2021-1902-1			IS + QC_1
B	IS + Cal. 2	Sample moved during step 6 to position G3	P2021-1911-1			IS + Cal. 7
C	IS + Cal. 3	Sample moved during step 6 to position H3	P2021-1927-1			IS + Cal. 6
D	IS + Cal. 4	P2021-1605-1	P2021-1928-1			IS + Cal. 5
E	IS + Cal. 5	P2021-1614-1	P2021-1930-1			IS + Cal. 4
F	IS + Cal. 6	P2021-1833-3	P2021-1948-5			IS + Cal. 3
G	IS + Cal. 7	P2021-1866-1	M2021-1933-1			IS + Cal. 2
H	IS + QC_1	P2021-1870-1	P2021-1592-1			IS + Cal. 1

All wells to contain 100 µl of residual DMSO

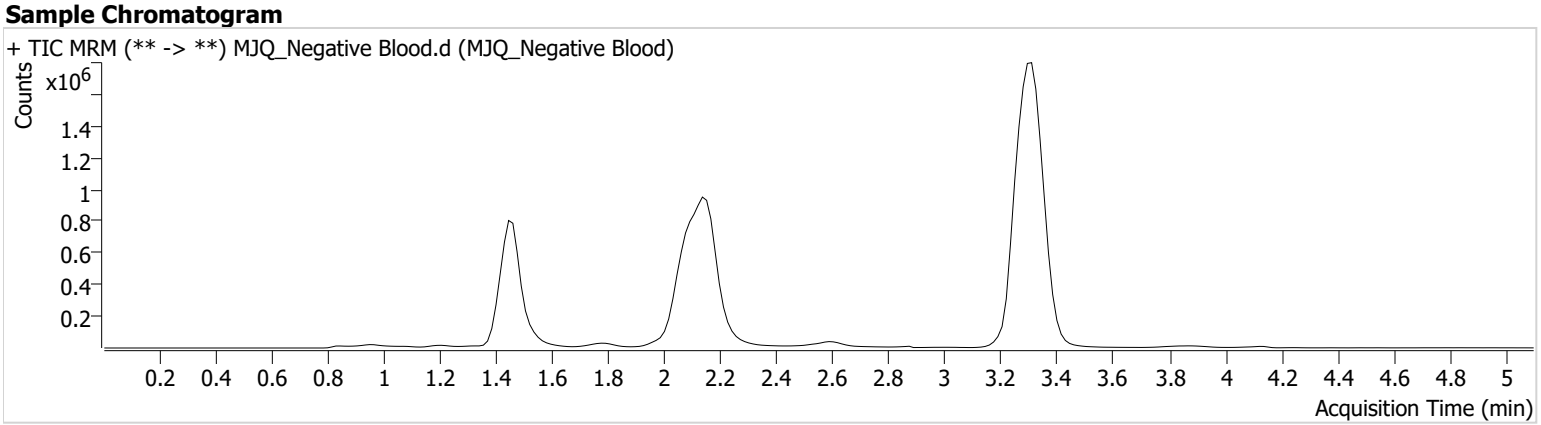
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_Negative Blood.d
Type	Sample	**Sample**	MJQ_Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 6:36:18 PM		
Sample Info.			

Sample Chromatogram



	Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
*	THC-OH	1.528	217905	∞	2.6 **Low**	227.24	3142554	4.0737	ng/ml

*Compound not evaluated.

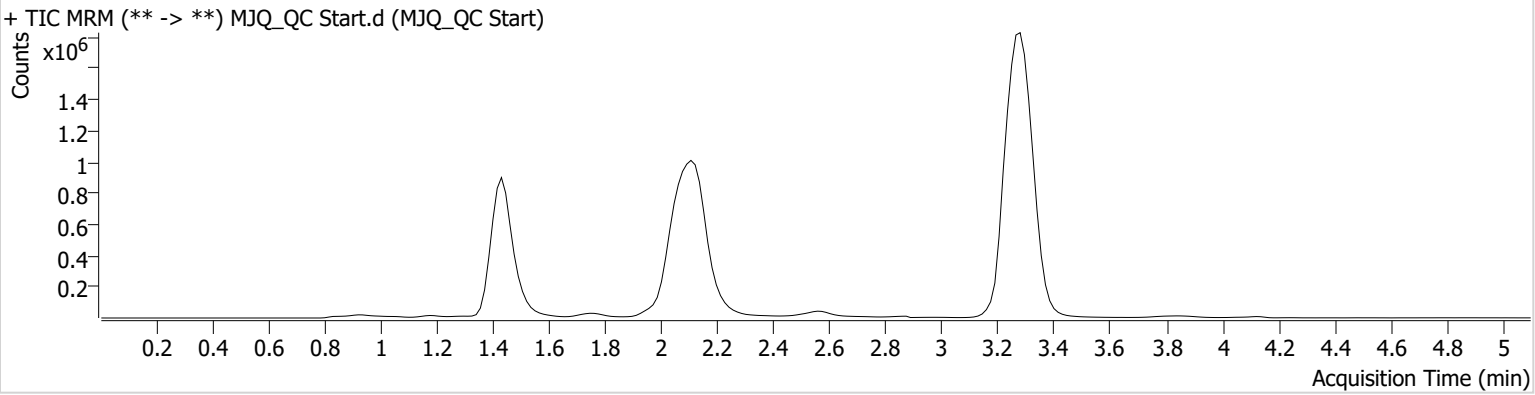
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_QC Start.d
Type	Sample	**Sample**	MJQ_QC Start
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 6:20:56 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453 Low	402768	∞	7.0 Low	∞	3095880	8.0095	ng/ml
THC-COOH	1.459 Low	288052	∞	55.8	842.45	824780	14.1234	ng/ml
THC	3.285	512294	∞	27.7	∞	12155099	4.2544	ng/ml

THC-COOH retention time shifted with the internal standard. Data is ok as per the analytical method.

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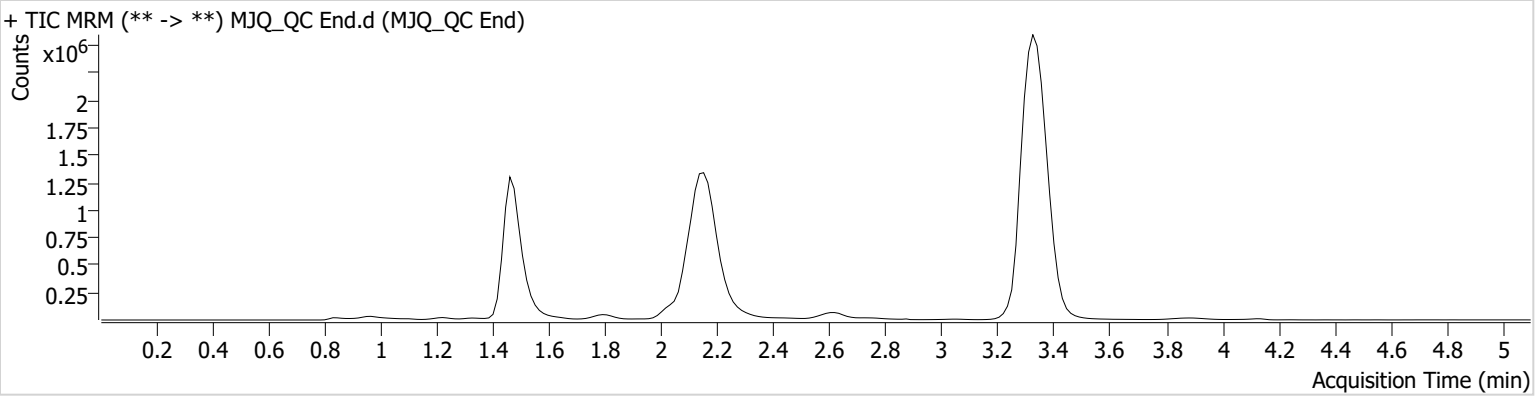
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_QC End.d
Type	Sample	**Sample**	MJQ_QC End
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 10:09:12 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	477545	∞	6.8 **Low**	∞	3886574	7.5413	ng/ml
THC-COOH	1.504	337102	∞	54.9	∞	958778	14.2198	ng/ml
THC	3.345	741615	∞	24.3	1233.28	15861366	4.6961	ng/ml

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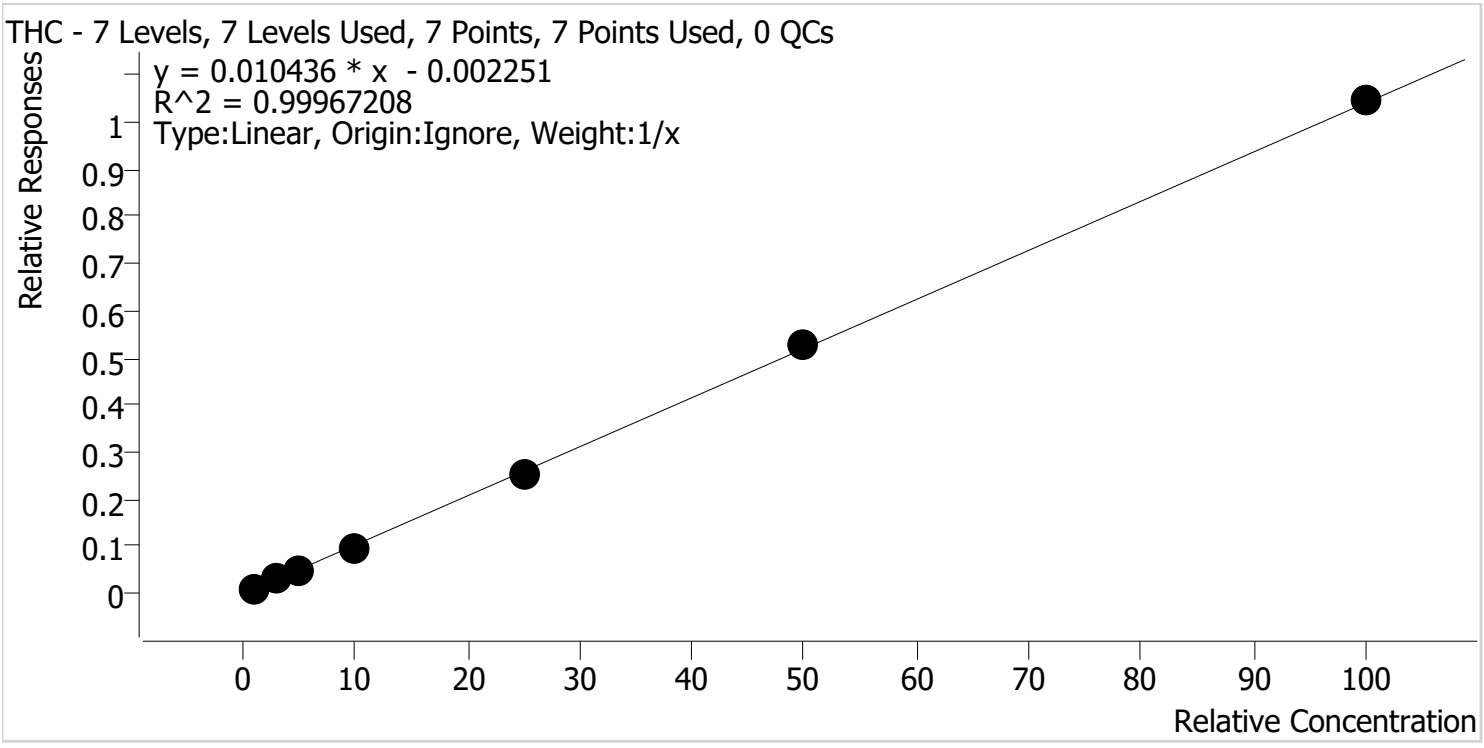
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	6/14/2021 11:07 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✔	1.0	1.1	110.7
MJQ_Cal 2	2	✔	3.0	2.9	97.6
MJQ_Cal 3	3	✔	5.0	4.8	96.3
MJQ_Cal 4	4	✔	10.0	9.5	95.1
MJQ_Cal 5	5	✔	25.0	24.7	98.9
MJQ_Cal 6	6	✔	50.0	50.5	101.0
MJQ_Cal 7	7	✔	100.0	100.4	100.4

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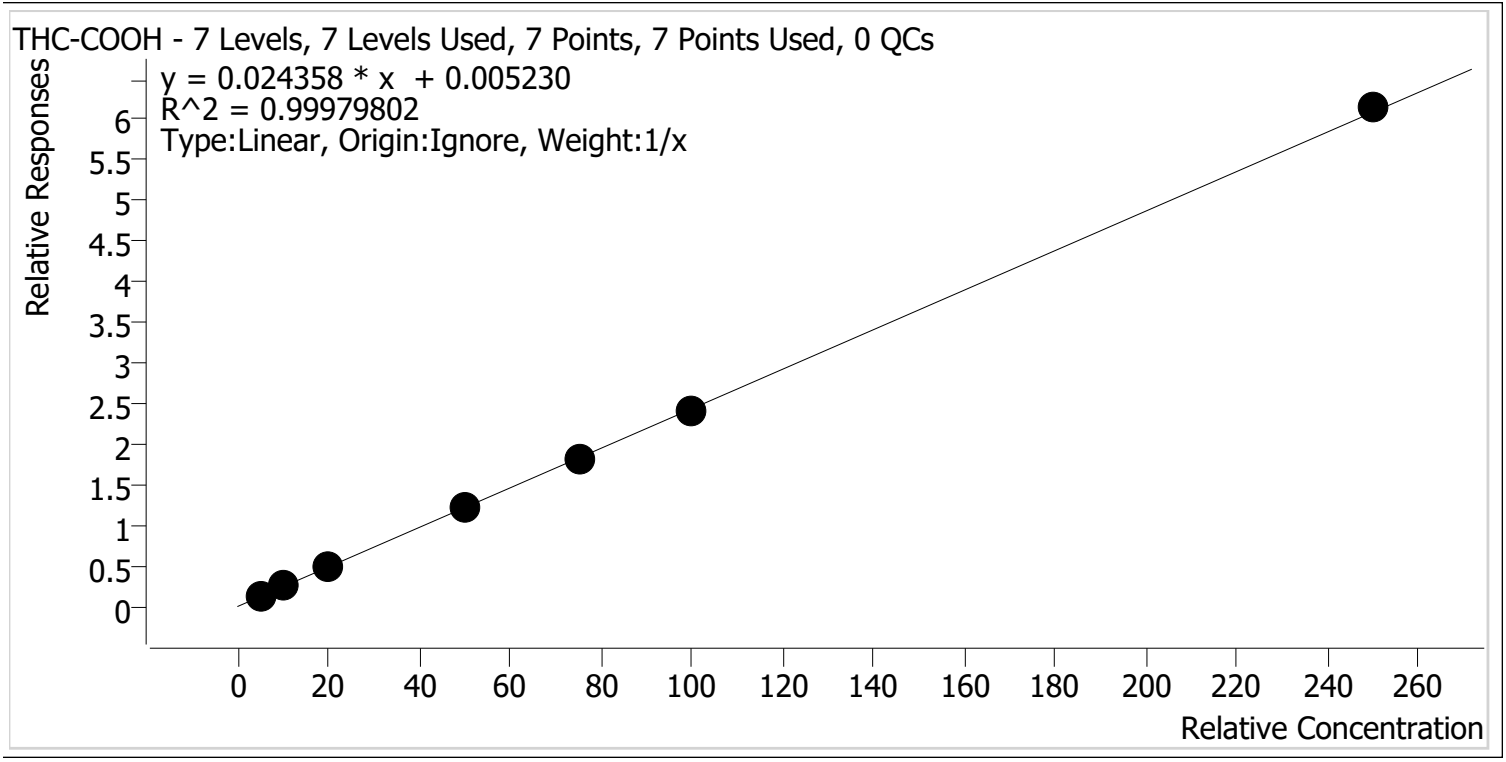
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	6/14/2021 11:07 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✔	5.0	5.3	106.0
MJQ_Cal 2	2	✔	10.0	9.7	97.3
MJQ_Cal 3	3	✔	20.0	19.7	98.6
MJQ_Cal 4	4	✔	50.0	49.6	99.3
MJQ_Cal 5	5	✔	75.0	73.7	98.2
MJQ_Cal 6	6	✔	100.0	99.7	99.7
MJQ_Cal 7	7	✔	250.0	252.3	100.9

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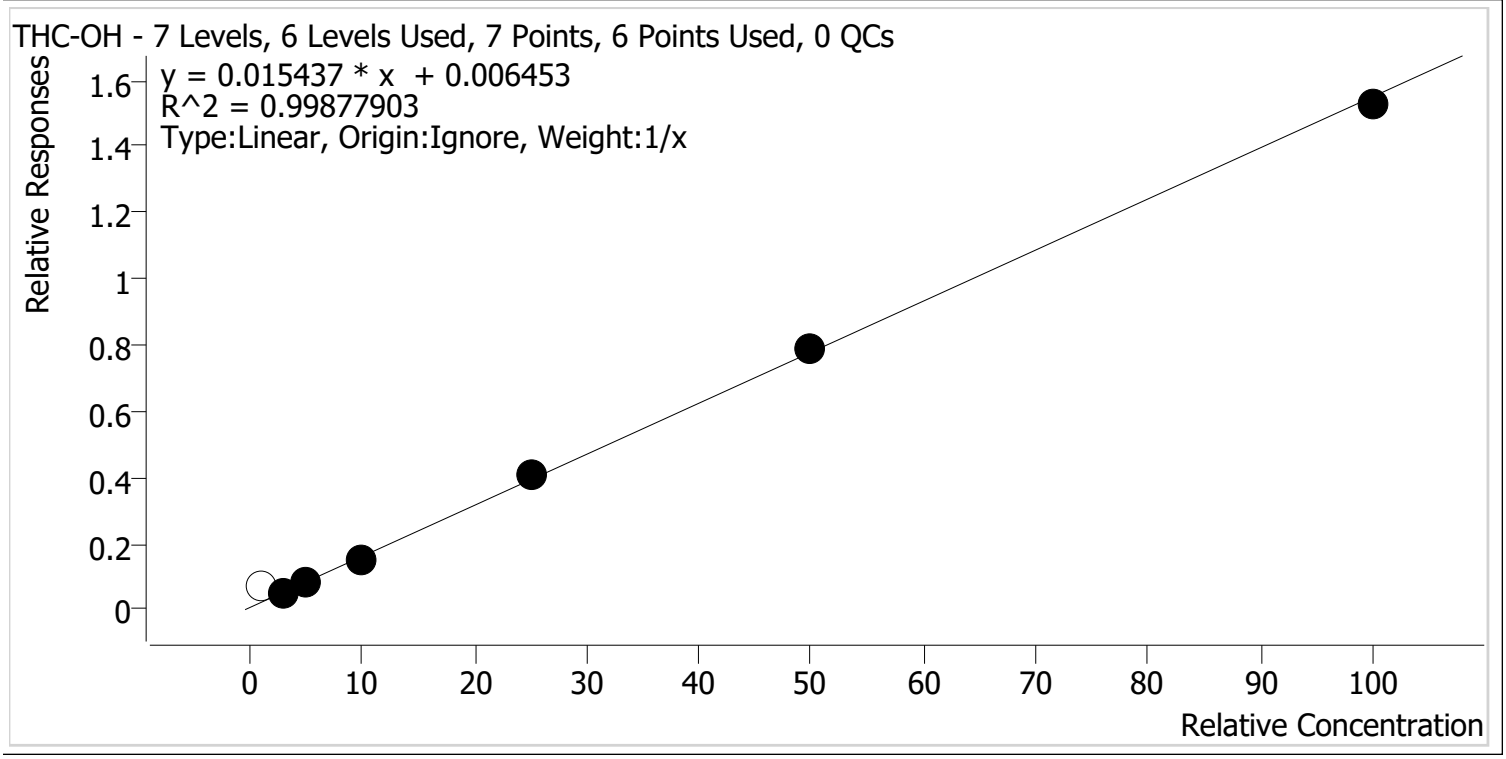
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	6/14/2021 11:07 AM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✗	1.0	4.6	457.9
MJQ_Cal 2	2	✔	3.0	2.9	96.7
MJQ_Cal 3	3	✔	5.0	5.3	105.6
MJQ_Cal 4	4	✔	10.0	9.3	92.6
MJQ_Cal 5	5	✔	25.0	26.2	104.8
MJQ_Cal 6	6	✔	50.0	50.9	101.9
MJQ_Cal 7	7	✔	100.0	98.4	98.4

THC-OH not evaluated due to possible interfering compound.

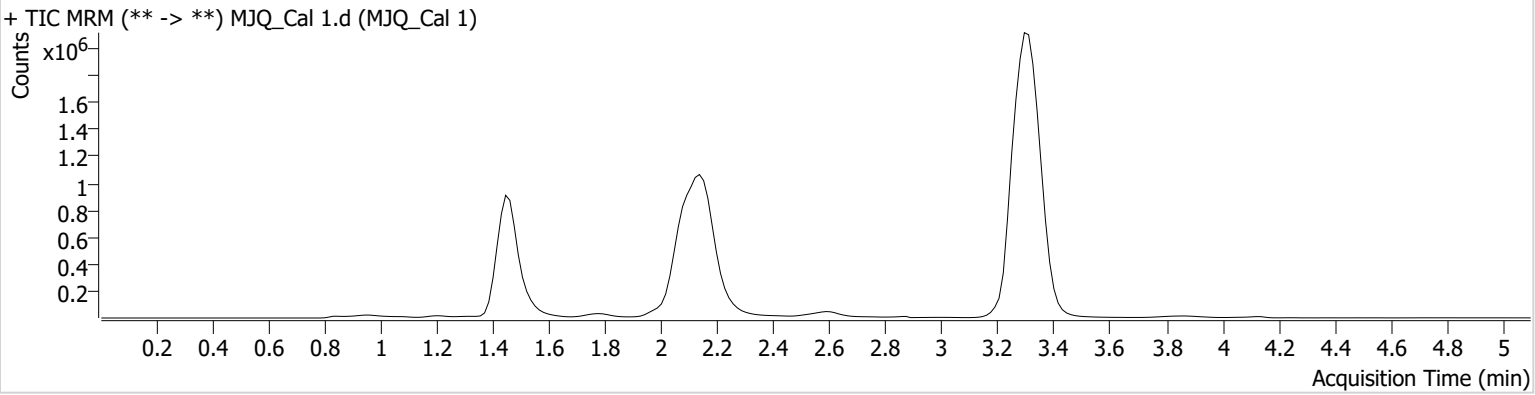
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 1.d
Type	Cal	**Sample**	MJQ_Cal 1
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 4:30:01 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.528	268971	∞	4.1 **Low**	∞	3487056	4.5786	ng/ml
THC-COOH	1.489	124873	∞	47.7	446.65	929445	5.3010	ng/ml
THC	3.315	136883	∞	31.3	337.05	14715487	1.1070	ng/ml

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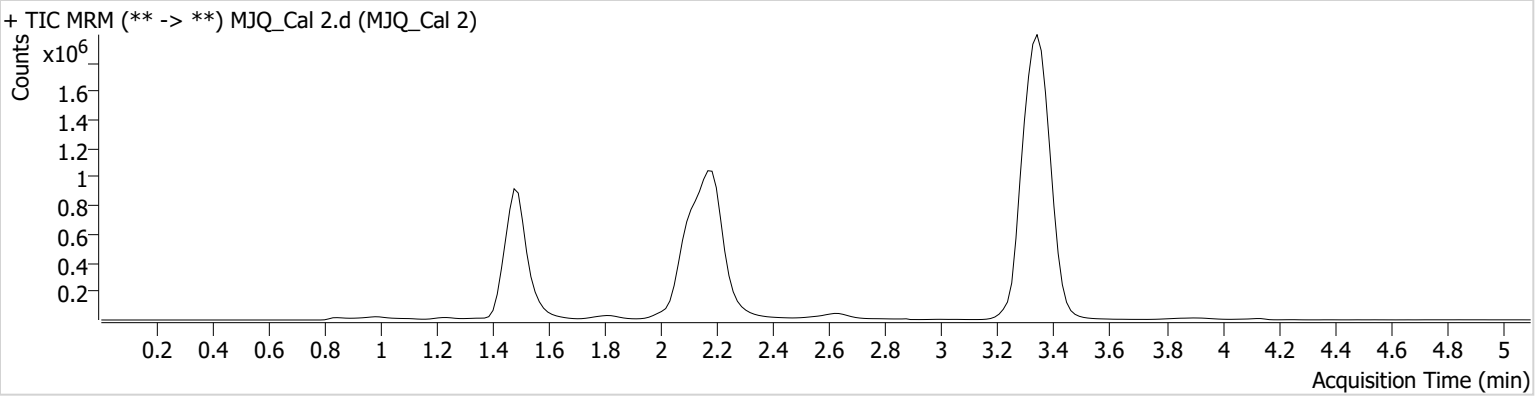
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 2.d
Type	Cal	**Sample**	MJQ_Cal 2
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 4:37:47 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.498	173375	∞	11.4	250.22	3384235	2.9006	ng/ml Low
THC-COOH	1.519	219920	∞	54.0	688.70	907534	9.7339	ng/ml
THC	3.360	385125	2161.79	27.9	579.92	13601017	2.9290	ng/ml

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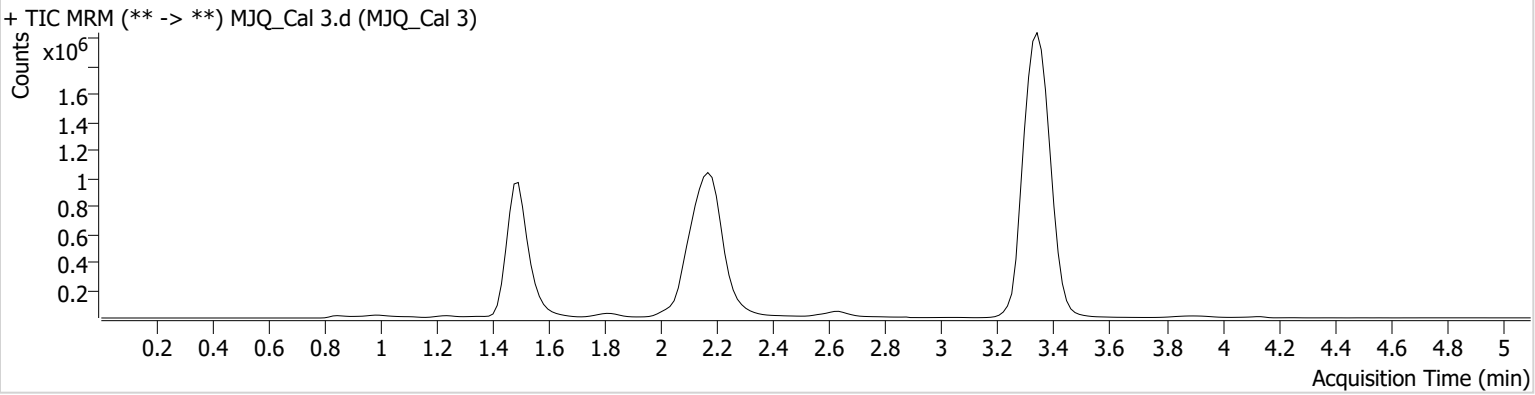
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 3.d
Type	Cal	**Sample**	MJQ_Cal 3
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 4:45:23 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.498	280950	∞	9.9	∞	3193686	5.2806	ng/ml
THC-COOH	1.519	413964	∞	54.6	2081.89	852723	19.7155	ng/ml
THC	3.360	625621	∞	25.9	∞	13030485	4.8165	ng/ml

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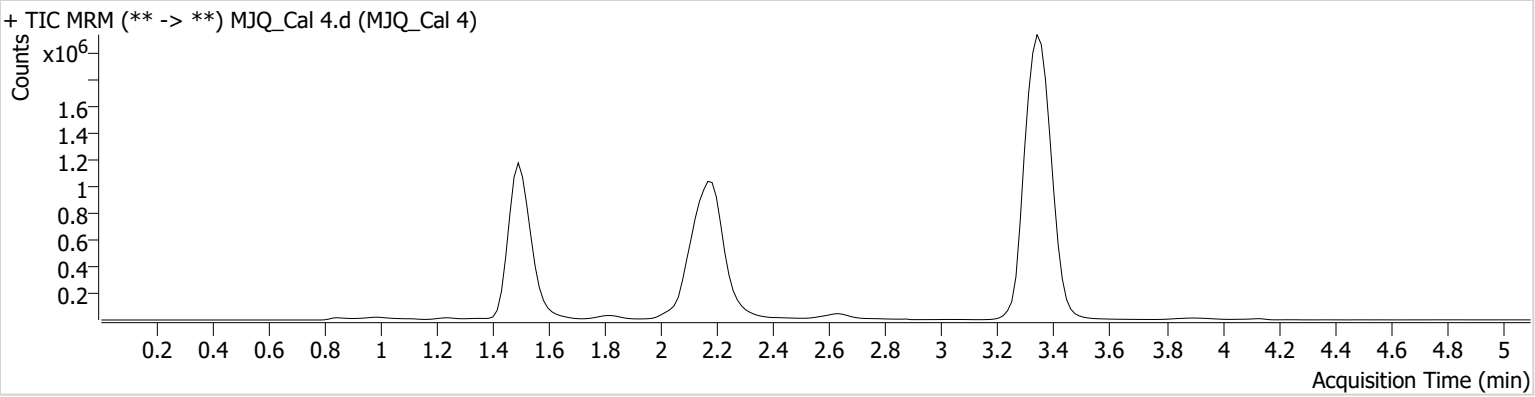
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 4.d
Type	Cal	**Sample**	MJQ_Cal 4
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 4:52:59 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.498	475047	∞	11.7	∞	3179014	9.2619	ng/ml
THC-COOH	1.519	1016959	∞	55.5	5220.65	837489	49.6373	ng/ml
THC	3.360	1234605	∞	26.1	573.16	12734704	9.5058	ng/ml

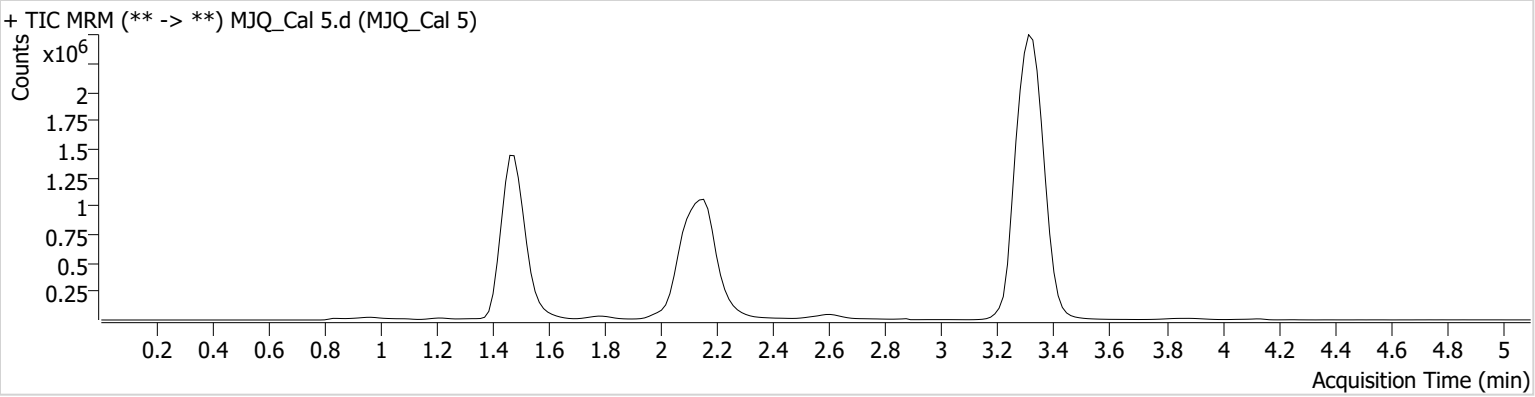
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 5.d
Type	Cal	**Sample**	MJQ_Cal 5
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 5:24:51 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	1366642	∞	9.9	∞	3326604	26.1943	ng/ml
THC-COOH	1.489	1583398	∞	57.8	4003.64	879987	73.6559	ng/ml
THC	3.330	3429860	∞	25.7	∞	13415092	24.7156	ng/ml

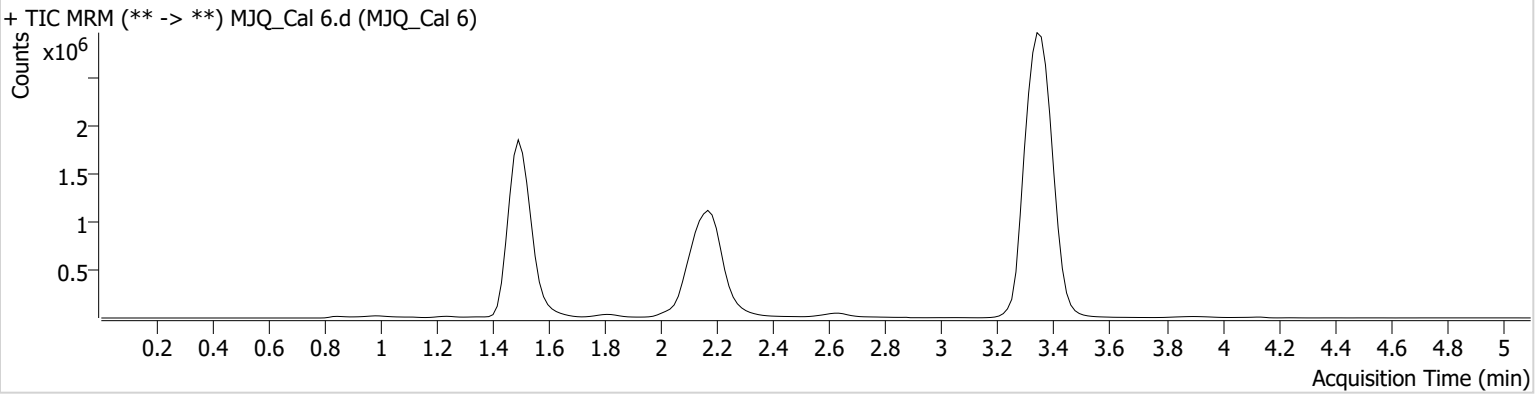
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 6.d
Type	Cal	**Sample**	MJQ_Cal 6
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 5:32:39 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.483	2527711	∞	10.9	∞	3188000	50.9435	ng/ml
THC-COOH	1.519	2000984	6367.48	56.5	3347.99	822485	99.6641	ng/ml
THC	3.360	6523738	∞	25.7	∞	12435395	50.4868	ng/ml

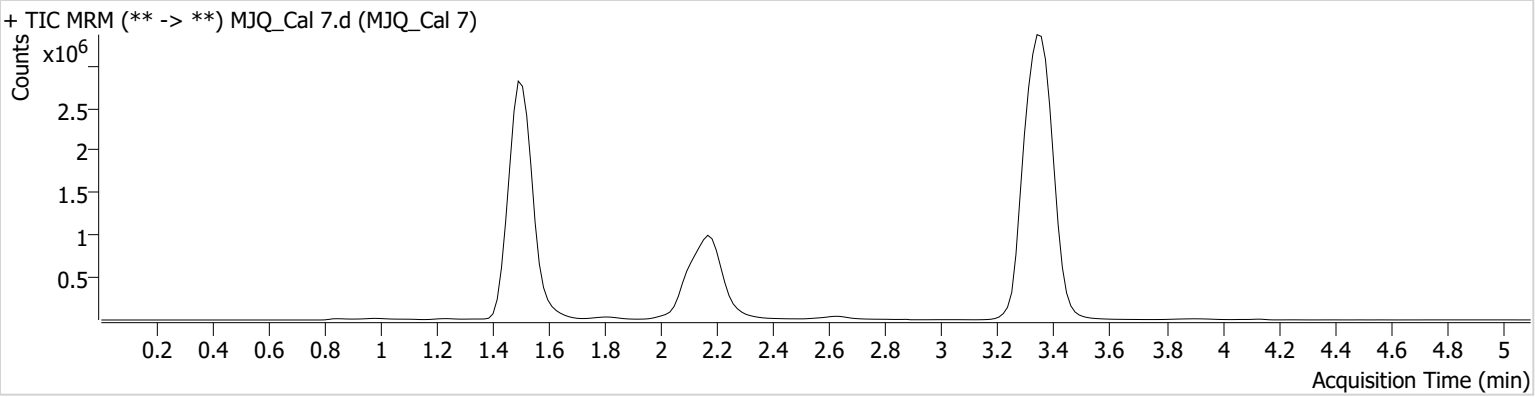
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\061021 AM 27 28 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/14/2021 11:07:54 AM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 7.d
Type	Cal	**Sample**	MJQ_Cal 7
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P1-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/10/2021 5:40:15 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.483	4506017	∞	11.3	∞	2953260	98.4191	ng/ml
THC-COOH	1.519	4515091	3646.07	56.7	54937.45	734093	252.2923	ng/ml
THC	3.360	11243921	∞	25.6	∞	10750525	100.4393	ng/ml