REVIEWED
By Tamara Salazar at 8:15 am, Jun 08, 2021

6/4/2021

Worklist: 5017

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2021-2244	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2021-2271	2	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1662	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1690	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1691	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1697	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1701	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1702	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1705	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1706	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1725	1	BLOOD	AM 27 Blood THC Quant by LC-QQQ
P2021-1735	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1736	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1761	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1764	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1800	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1801	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1848	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1850	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: 06/03/21
Plate lot#: IDP-108-2-210412
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: Lampire 20L20724
LCMS-QQQ ID: 069901

Analyst: Sarah Collins
Plate Expiration: 10/12/2021
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. 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:** 3382167
☒ 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: 800 uL
☒ 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: Curve range limited: THC-OH 3-100

	1	2	3	4	5	6
A	IS + Cal. 1	negative blood	p2021-1702-1*	p2021-1800-1		
B	IS + Cal. 2	m2021-2244-2	p2021-1705-1	p2021-1801-1		
C	IS + Cal. 3	m2021-2271-2	p2021-1706-1	p2021-1848-1		
D	IS + Cal. 4	p2021-1662-1	p2021-1725-1	p2021-1850-1		
E	IS + Cal. 5	p2021-1690-1	p2021-1735-1	p2021-1702-1		
F	IS + Cal. 6	p2021-1691-1	p2021-1736-1			
G	IS + Cal. 7	p2021-1697-1	p2021-1761-1			
H	IS + QC_1	p2021-1701-1	p2021-1764-1			

All wells to contain 100 µl of residual DMSO

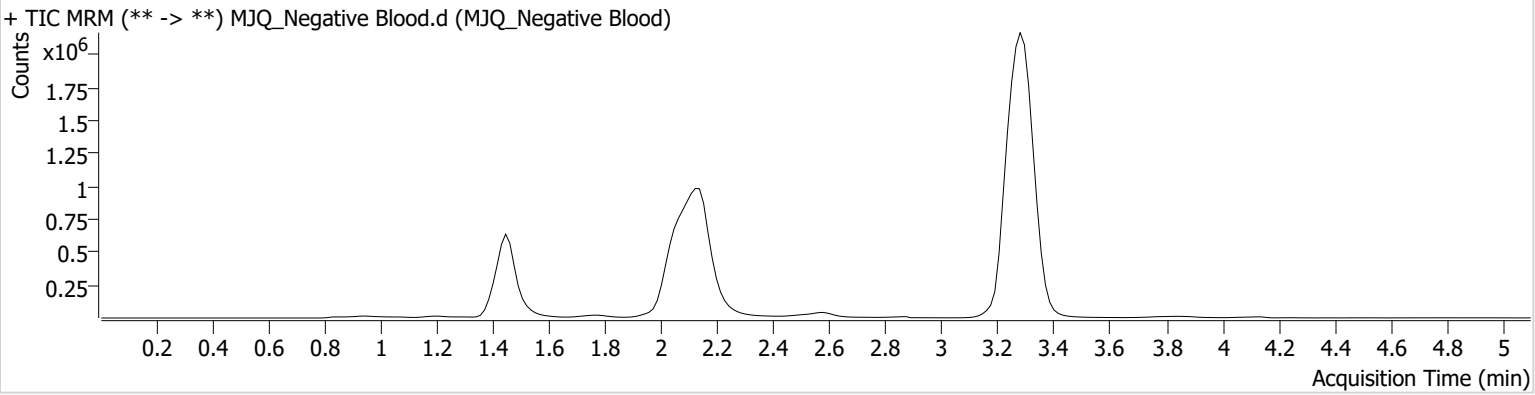
*Sample moved during analytical step 6 due to blood clot

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update	6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_Negative Blood.d
Type	Sample	**Sample**	MJQ_Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 3:53:09 PM		
Sample Info.			

Sample Chromatogram

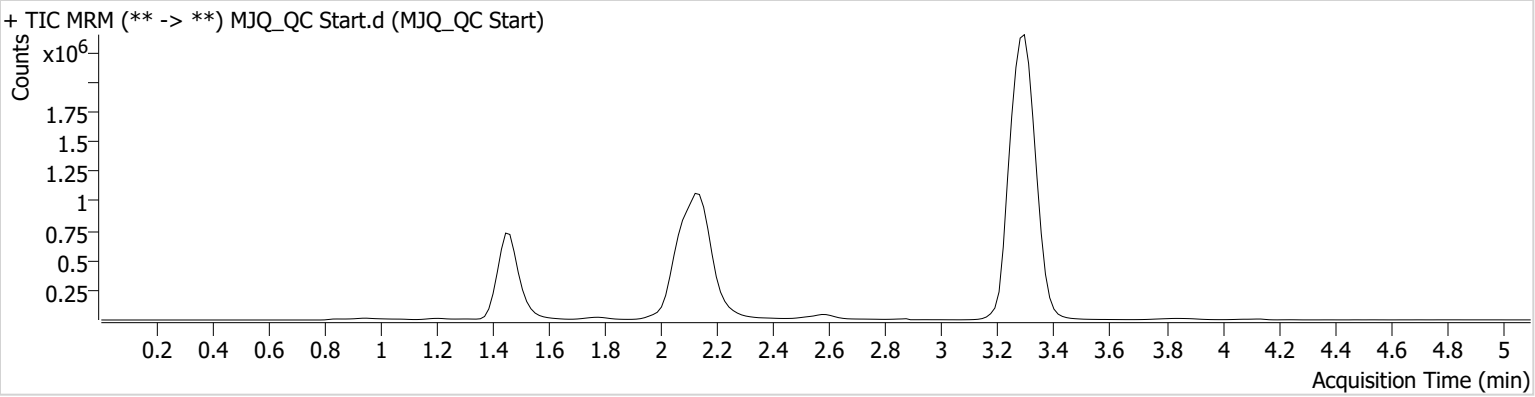


AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update	6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_QC Start.d
Type	Sample	**Sample**	MJQ_QC Start
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 4:08:22 PM		
Sample Info.			

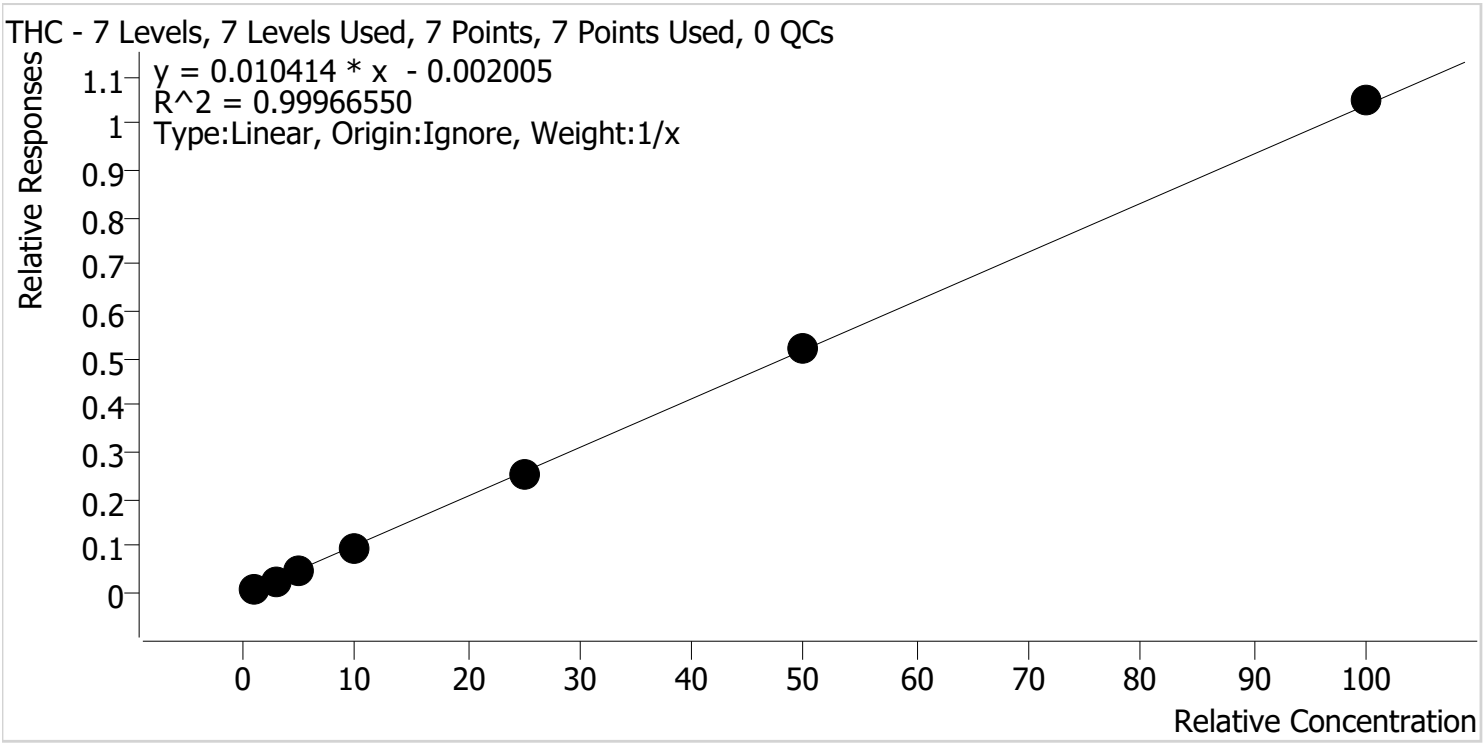
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	315306	∞	7.6	260.97	2326669	4.3578	ng/ml
THC-COOH	1.489	286471	∞	49.4	490.16	722527	14.1835	ng/ml
THC	3.300	682772	33955.42	26.8	∞	14837921	4.6110	ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

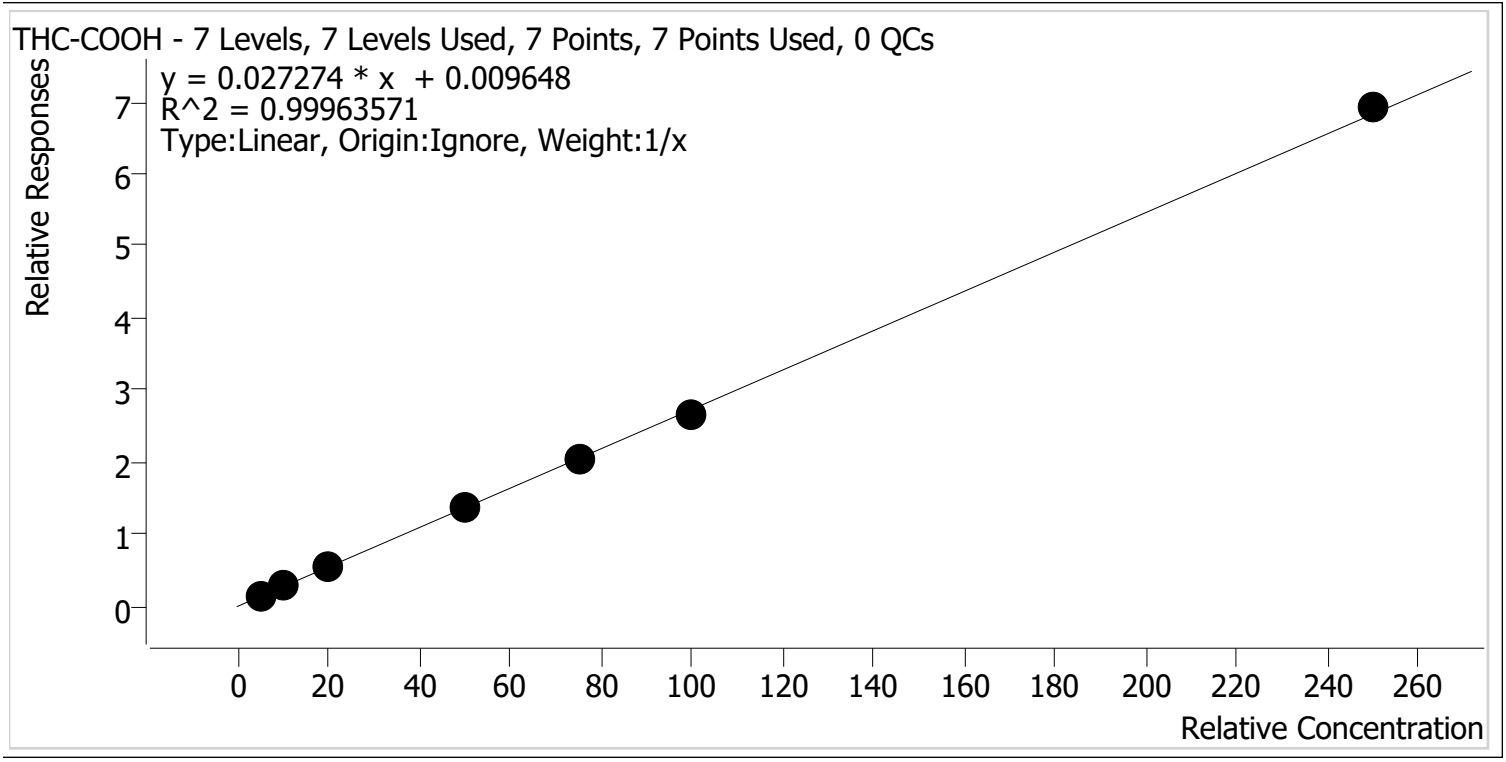
Batch results	D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update	6/4/2021 12:16 PM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✔	1.0	1.1	111.7
MJQ_Cal 2	2	✔	3.0	2.9	95.9
MJQ_Cal 3	3	✔	5.0	4.8	97.0
MJQ_Cal 4	4	✔	10.0	9.6	96.3
MJQ_Cal 5	5	✔	25.0	24.5	98.1
MJQ_Cal 6	6	✔	50.0	50.1	100.1
MJQ_Cal 7	7	✔	100.0	100.9	100.9

AM #27 Cannabinoids Quant. Calibration Curve Report

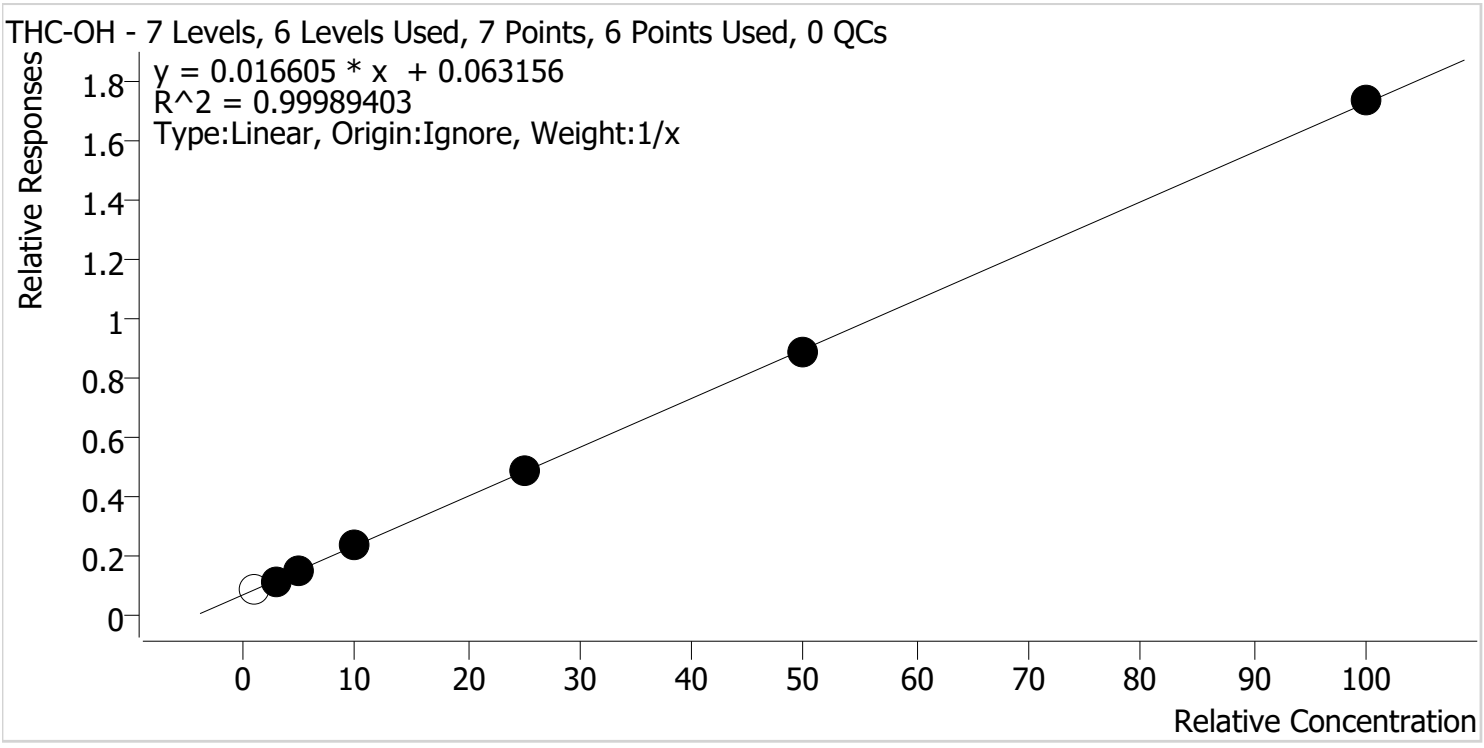
Batch results	D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update	6/4/2021 12:16 PM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✔	5.0	5.2	104.9
MJQ_Cal 2	2	✔	10.0	10.1	100.7
MJQ_Cal 3	3	✔	20.0	19.3	96.3
MJQ_Cal 4	4	✔	50.0	49.7	99.4
MJQ_Cal 5	5	✔	75.0	74.8	99.8
MJQ_Cal 6	6	✔	100.0	97.6	97.6
MJQ_Cal 7	7	✔	250.0	253.3	101.3

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update	6/4/2021 12:16 PM
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	1.3	126.2
MJQ_Cal 2	2	✔	3.0	3.0	100.6
MJQ_Cal 3	3	✔	5.0	4.9	98.3
MJQ_Cal 4	4	✔	10.0	10.2	102.1
MJQ_Cal 5	5	✔	25.0	24.9	99.7
MJQ_Cal 6	6	✔	50.0	49.4	98.9
MJQ_Cal 7	7	✔	100.0	100.5	100.5

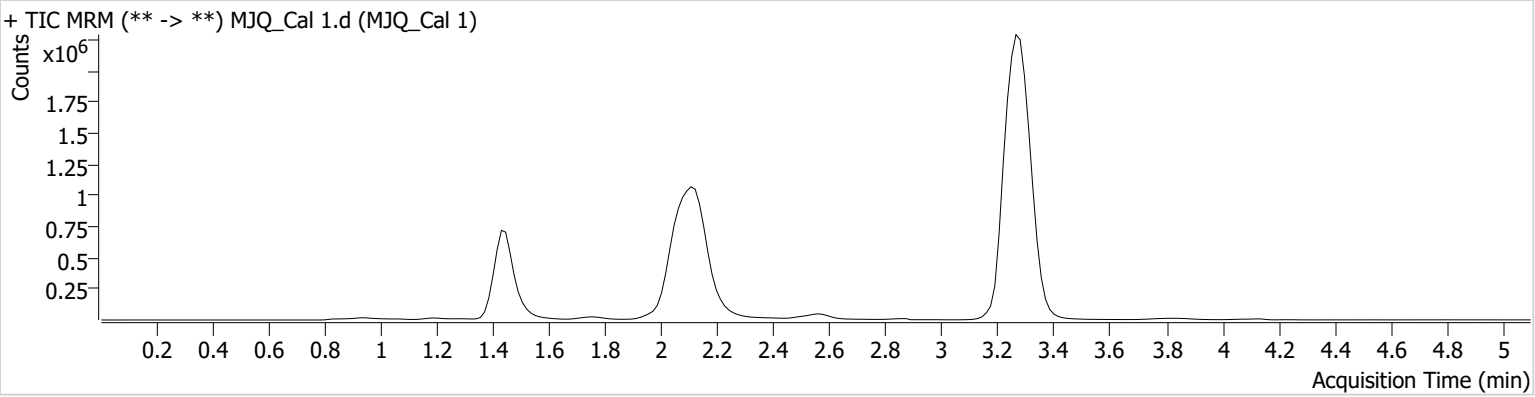
Calibrator 1 dropped due to not meeting ratio requirement

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 1.d
Type	Cal	**Sample**	MJQ_Cal 1
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 2:52:17 PM		
Sample Info.			

Sample Chromatogram



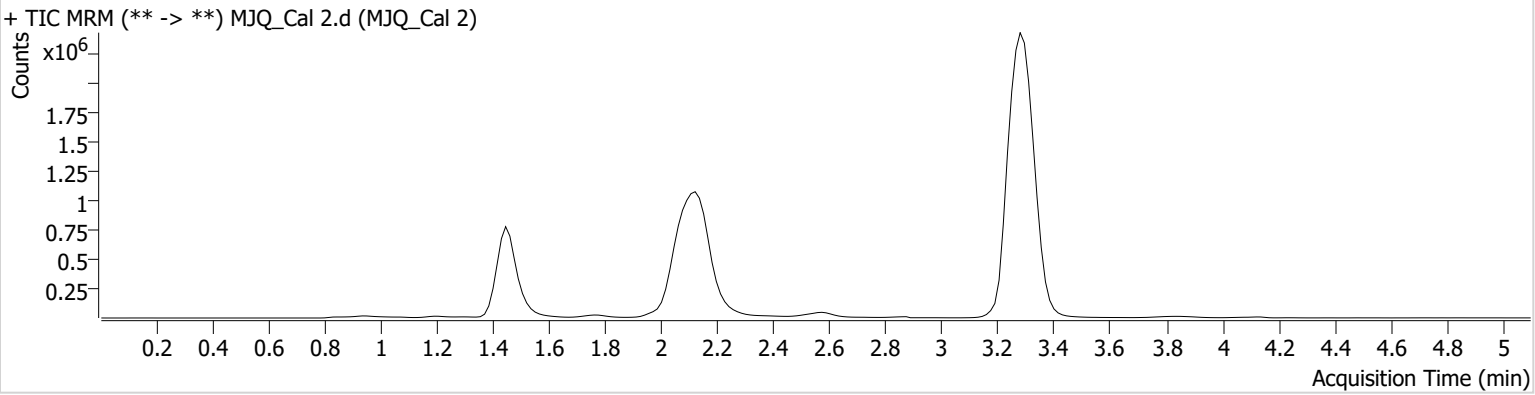
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.498	207806	∞	4.5 **Low**	∞	2470488	1.2622	ng/ml **Low**
THC-COOH	1.474	115025	∞	43.8	∞	753321	5.2447	ng/ml
THC	3.285	144209	1260.26	28.1	63.40	14977733	1.1170	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update	6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 2.d
Type	Cal	**Sample**	MJQ_Cal 2
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 2:59:53 PM		
Sample Info.			

Sample Chromatogram



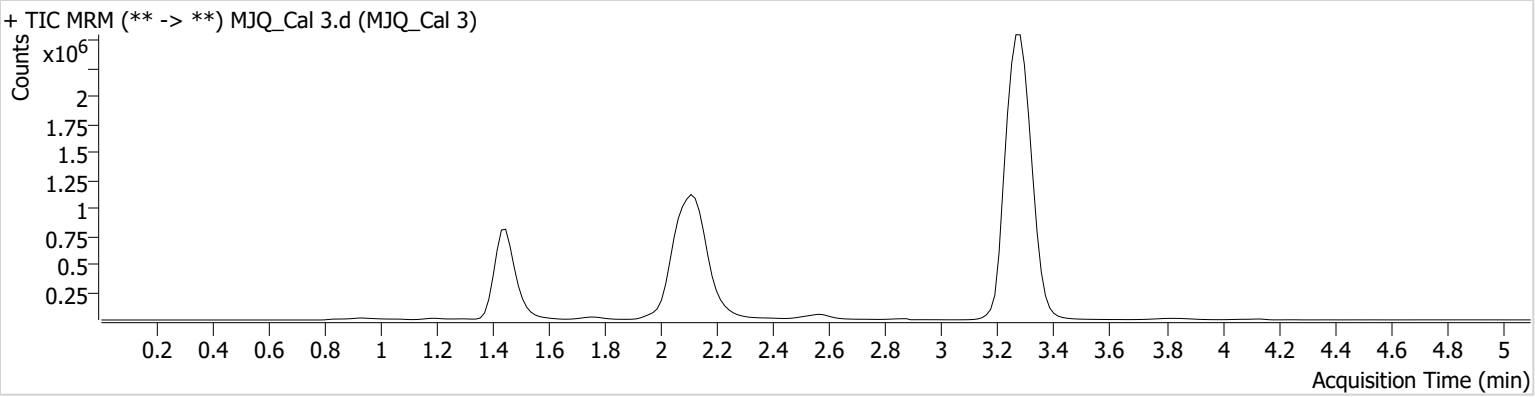
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.498	277382	∞	7.2	∞	2448978	3.0176	ng/ml
THC-COOH	1.474	215080	117.06	46.9	∞	756536	10.0700	ng/ml
THC	3.300	426392	∞	29.1	352.90	15250973	2.8771	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update	6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 3.d
Type	Cal	**Sample**	MJQ_Cal 3
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 3:07:32 PM		
Sample Info.			

Sample Chromatogram



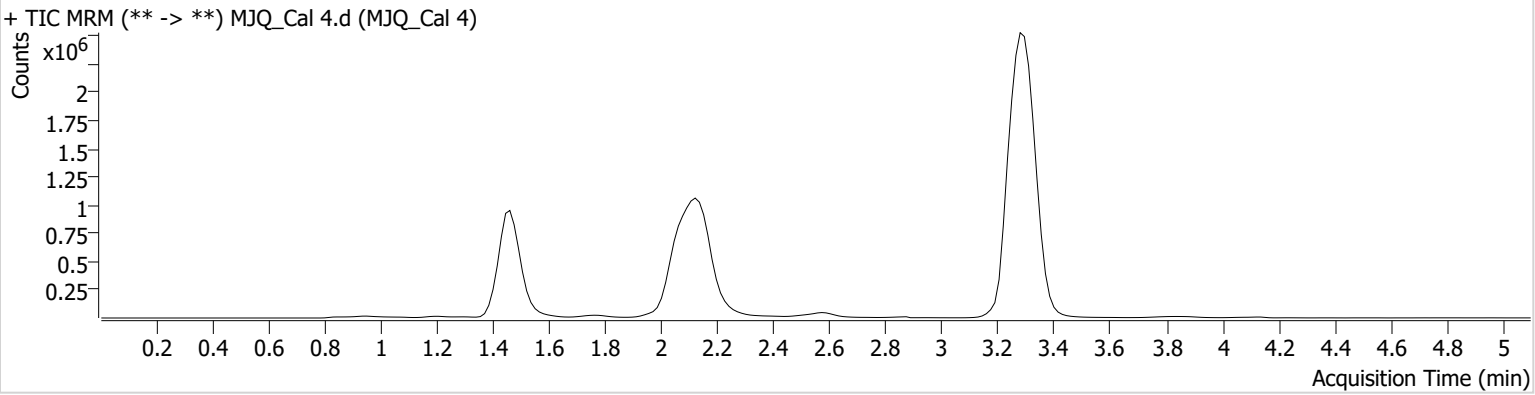
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	353281	∞	7.4	529.66	2440619	4.9138	ng/ml
THC-COOH	1.474	401624	∞	49.6	∞	750892	19.2571	ng/ml
THC	3.285	763907	3808.58	28.1	1109.38	15756458	4.8479	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 4.d
Type	Cal	**Sample**	MJQ_Cal 4
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 3:15:08 PM		
Sample Info.			

Sample Chromatogram



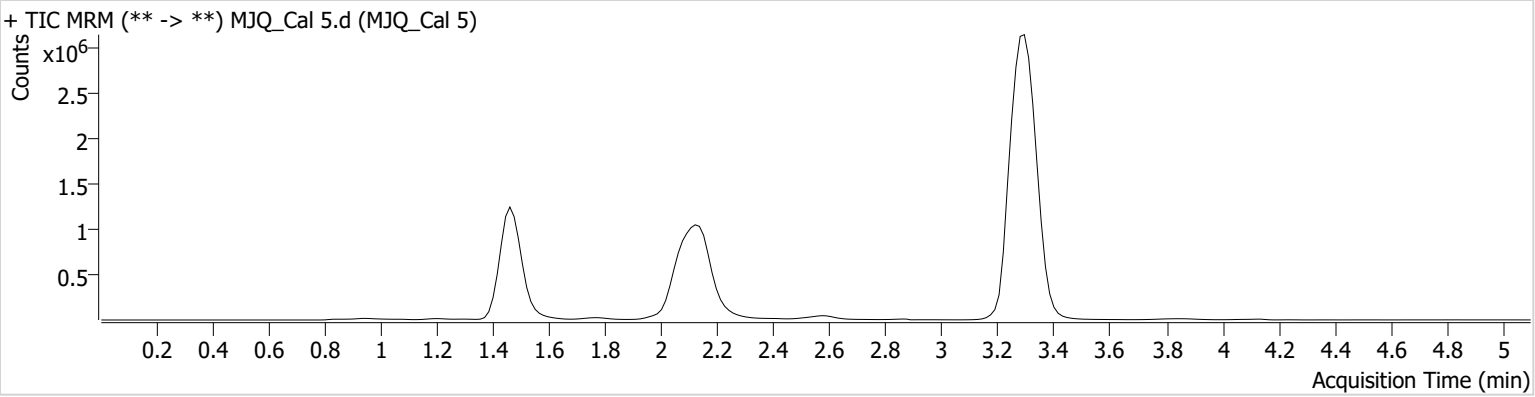
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	523678	∞	8.4	∞	2251160	10.2058	ng/ml
THC-COOH	1.474	971419	∞	52.7	∞	711400	49.7128	ng/ml
THC	3.300	1488022	6375.76	27.6	1502.99	15146099	9.6262	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 5.d
Type	Cal	**Sample**	MJQ_Cal 5
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 3:22:43 PM		
Sample Info.			

Sample Chromatogram



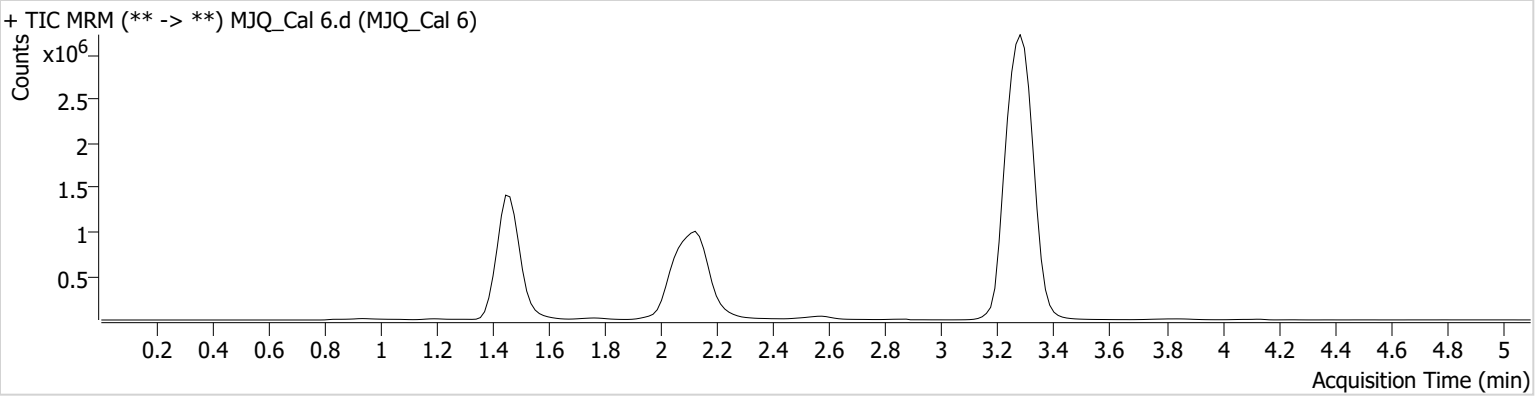
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	1091572	∞	9.7	∞	2287922	24.9285	ng/ml
THC-COOH	1.489	1444065	∞	53.9	∞	704361	74.8165	ng/ml
THC	3.300	4012378	87720.08	27.9	∞	15834713	24.5239	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 6.d
Type	Cal	**Sample**	MJQ_Cal 6
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 3:30:20 PM		
Sample Info.			

Sample Chromatogram



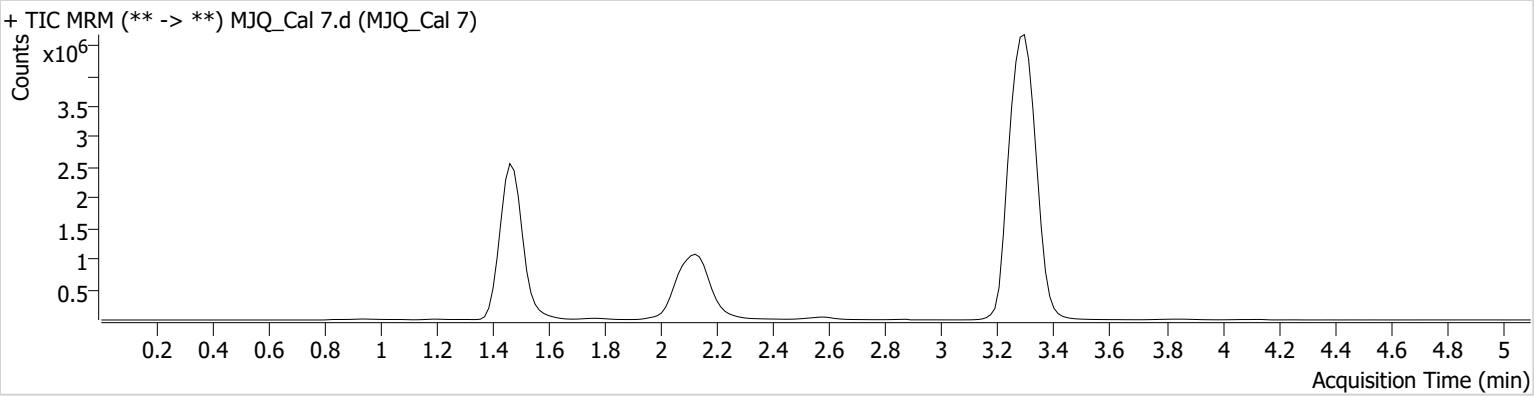
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	1925605	320.01	10.5	780.95	2178335	49.4313	ng/ml
THC-COOH	1.474	1765065	∞	54.3	∞	660468	97.6321	ng/ml
THC	3.285	6998526	∞	26.9	∞	13473395	50.0700	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\060321 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 6/4/2021 12:16:23 PM

Instrument	Falco (069901)	**Data File**	MJQ_Cal 7.d
Type	Cal	**Sample**	MJQ_Cal 7
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2021 3:37:56 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	3678484	∞	10.5	∞	2123778	100.5031	ng/ml
THC-COOH	1.474	4242491	∞	54.2	∞	613325	253.2667	ng/ml
THC	3.300	14121112	∞	27.5	∞	13459174	100.9379	ng/ml