REVIEWED
By Tamara Salazar at 2:00 pm, Mar 10, 2023

3/9/2023

Worklist: 6266

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2022-4969	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2023-0053	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2023-0201	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2023-0462	3	BCK	AM 27 Blood THC Quant by LC-QQQ
M2023-0556	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2023-0557	4	BCK	AM 27 Blood THC Quant by LC-QQQ
P2023-0335	1 *	BCK	AM 27 Blood THC Quant by LC-QQQ
P2023-0377	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2023-0406	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2023-0407	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2023-0413	1	BCK	AM 27 Blood THC Quant by LC-QQQ

*This sample was inadvertently included in the run. There was no indication of cannabinoids in the preliminary testing (AM 26).

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

Extraction Date: 03/06/2023
Analyst: Celena Shrum
Plate lot#: 220802
Plate Retest Date: 07/23/2023
Mobile phase A: 0.1% Formic Acid in LCMS Water
Mobile phase B: 0.1% Formic acid in Acetonitrile
Blank Blood Lot: Lampire 23A52593
Column: Phenomenex Phenyl Hexyl (4.6x50mm, 2.6um)
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 (if applicable): add 1.5mL urine to blank plate, add 250µl 1N KOH. Shake and incubate at 40 degrees for 15 minutes. - N/A, blood only
☒ 3. Using a calibrated pipette, add **1000µl blood or 1000µl hydrolyzed urine** into the appropriate wells of the analytical (standards) plate. **Pipette ID: #27**
☒ 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 or 500µl of saturated phosphate buffer to urine samples** to the appropriate wells of the analytical plate.
☒ 6. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 7. Transfer **800µL of blood+acid mixture or urine+acid** to corresponding wells of SLE+ plate.
☒ 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)*** Manifold ID: 067104
☒ 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. *SPE Dry ID: 067103*
☒ 16. Reconstitute in **100µL 100% MeOH** and heat seal plate with foil. Place in autosampler and run worklist.

Post-Analytic

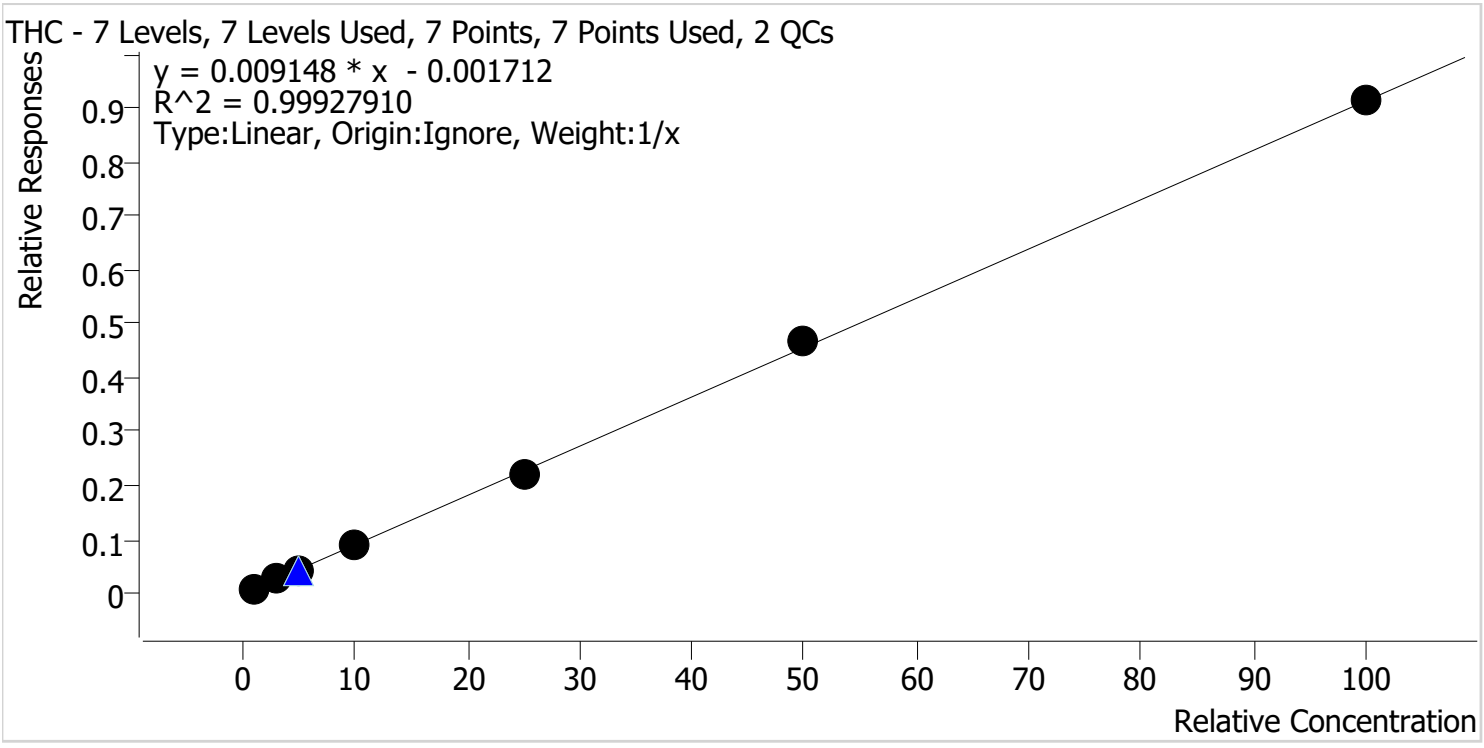
☒ 1. Create batch and process data.
☒ 2. Make any necessary integration changes, Curve weighting of Linear 1/x with r^2 values ≥0.98 for each analyte
☒ 3. RT +/- 3% or 0.100 min, whichever is greater, +/- 20% Accuracy for greater than (+/- 30% for 10ng/ml or less). Ion ratios must be within +/- 20% of the averaged calibrators
☒ 4. Case sample response for THC 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:

	1	2	3	4	5	6
A	IS + Cal. 1	QC2	P2023-0335-1			
B	IS + Cal. 2	NEG Blood	P2023-0377-1			
C	IS + Cal. 3	M2022-4969-1	P2023-0406-1			
D	IS + Cal. 4	M2023-0053-1	P2023-0407-1			
E	IS + Cal. 5	M2023-0201-1	P2023-0413-1			
F	IS + Cal. 6	M2023-0462-3				
G	IS + Cal. 7	M2023-0556-2				
H	QC1	M2023-0557-4				

AM #27 Cannabinoids Quant. Calibration Curve Report

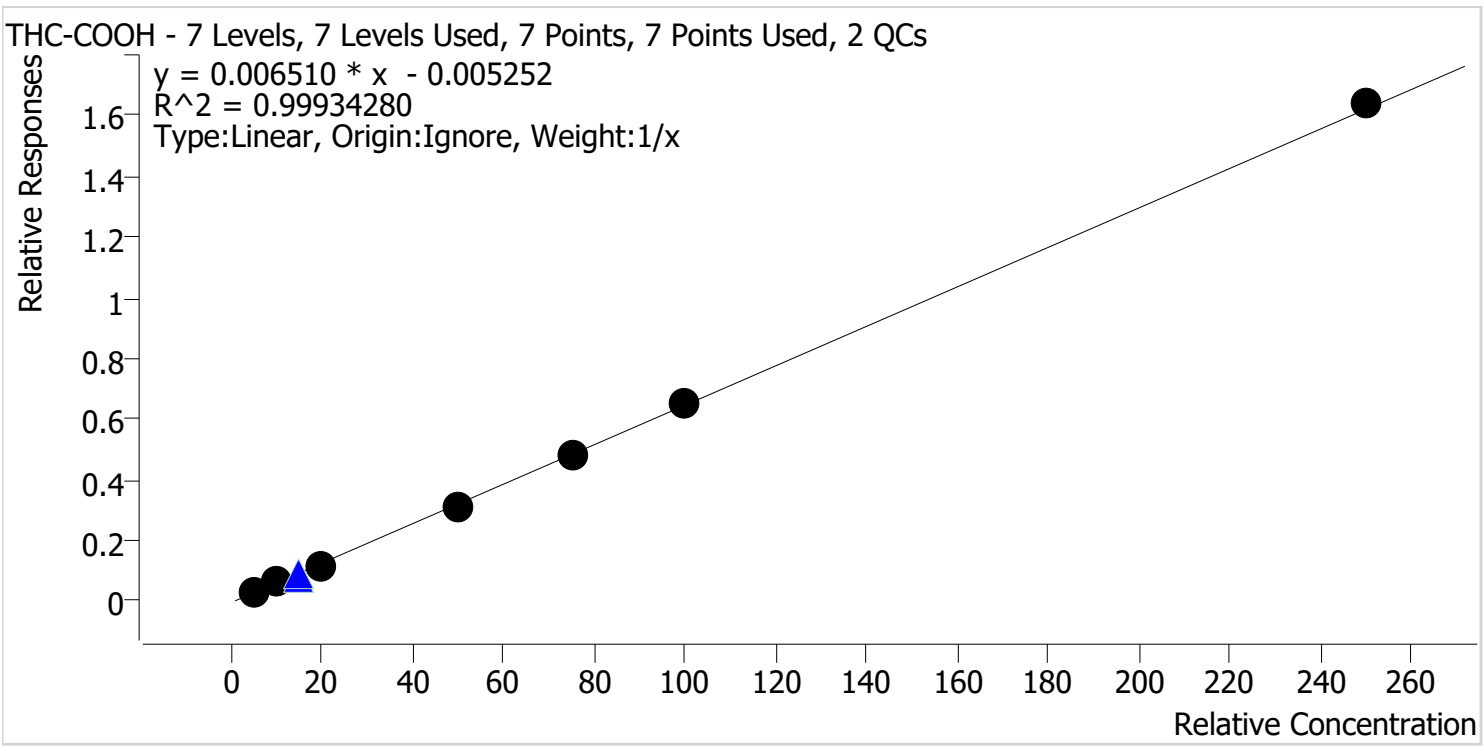
Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update	3/7/2023 7:30 AM
Analyst Name	ISP\Datastor
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	1.0	1.1	110.5
Cal 2 MJ	2	✔	3.0	3.0	99.9
Cal 3 MJ	3	✔	5.0	4.6	92.9
Cal 4 MJ	4	✔	10.0	9.8	97.8
Cal 5 MJ	5	✔	25.0	24.0	96.1
Cal 6 MJ	6	✔	50.0	51.4	102.8
Cal 7 MJ	7	✔	100.0	100.0	100.0

AM #27 Cannabinoids Quant. Calibration Curve Report

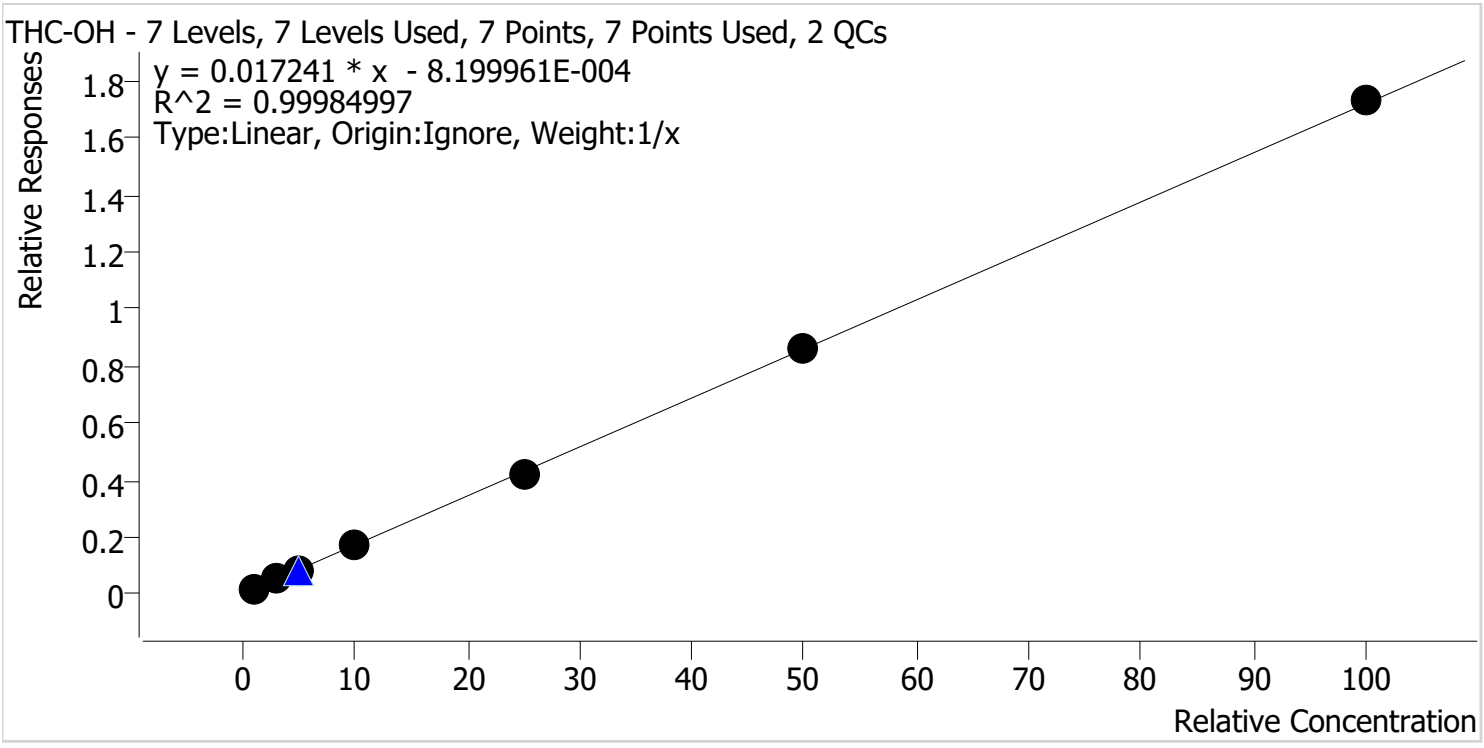
Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update	3/7/2023 7:30 AM
Analyst Name	ISP\Datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	5.0	5.5	109.0
Cal 2 MJ	2	✔	10.0	10.0	99.7
Cal 3 MJ	3	✔	20.0	19.0	95.0
Cal 4 MJ	4	✔	50.0	47.5	95.0
Cal 5 MJ	5	✔	75.0	74.6	99.4
Cal 6 MJ	6	✔	100.0	100.7	100.7
Cal 7 MJ	7	✔	250.0	252.8	101.1

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update	3/7/2023 7:30 AM
Analyst Name	ISP\Datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



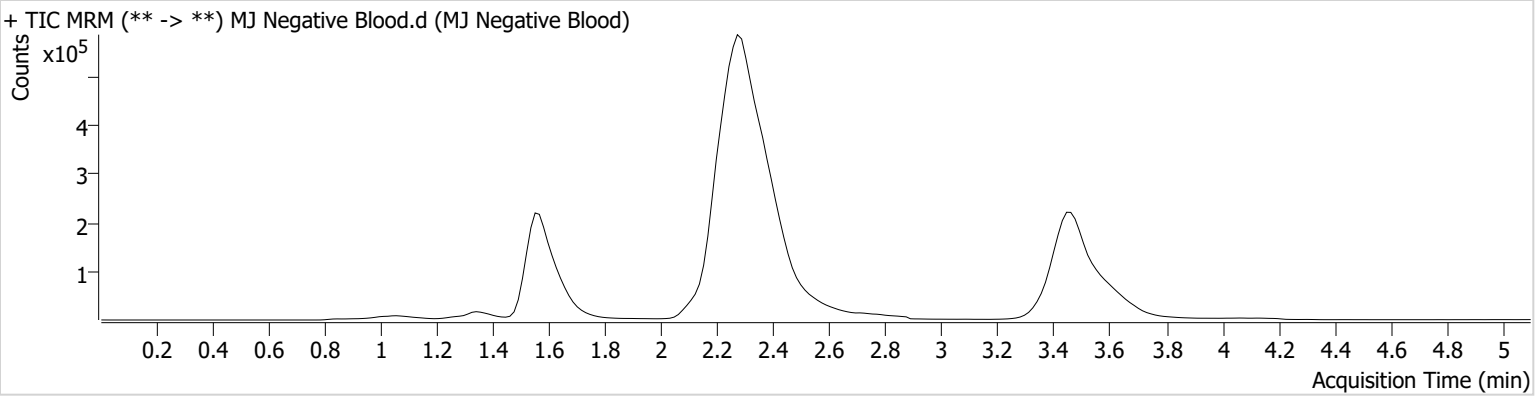
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	1.0	1.1	107.4
Cal 2 MJ	2	✔	3.0	3.0	98.5
Cal 3 MJ	3	✔	5.0	4.8	96.7
Cal 4 MJ	4	✔	10.0	9.8	98.1
Cal 5 MJ	5	✔	25.0	24.6	98.5
Cal 6 MJ	6	✔	50.0	50.1	100.2
Cal 7 MJ	7	✔	100.0	100.6	100.6

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	3/7/2023 7:30:36 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	P1-B2	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 3:56:57 PM		
Sample Info.			

Sample Chromatogram

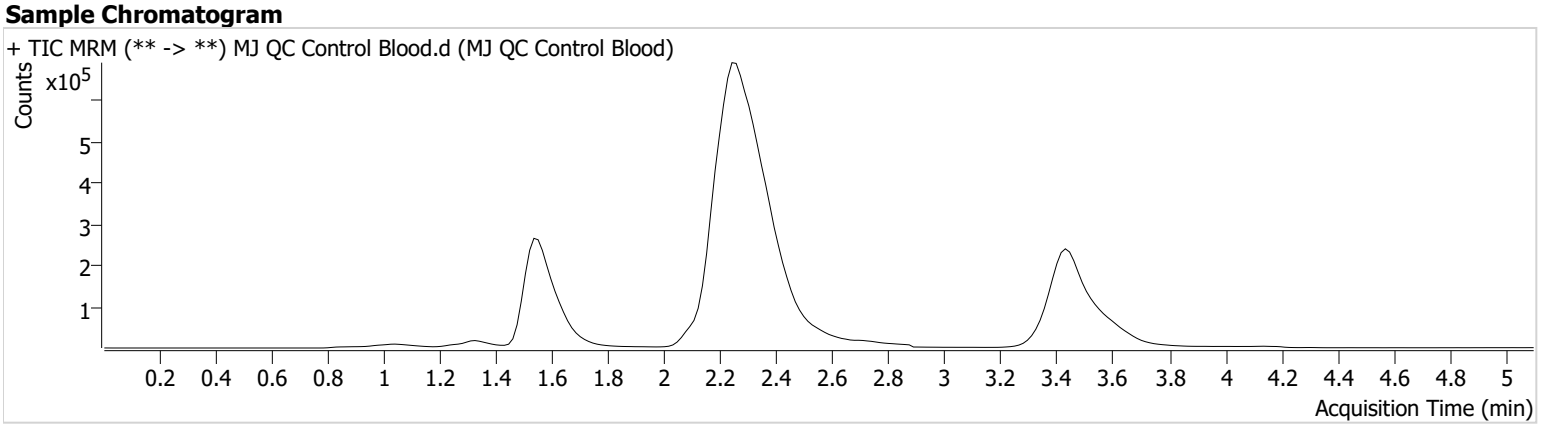


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/7/2023 7:30:36 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	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 3:41:33 PM		
Sample Info.			

Sample Chromatogram



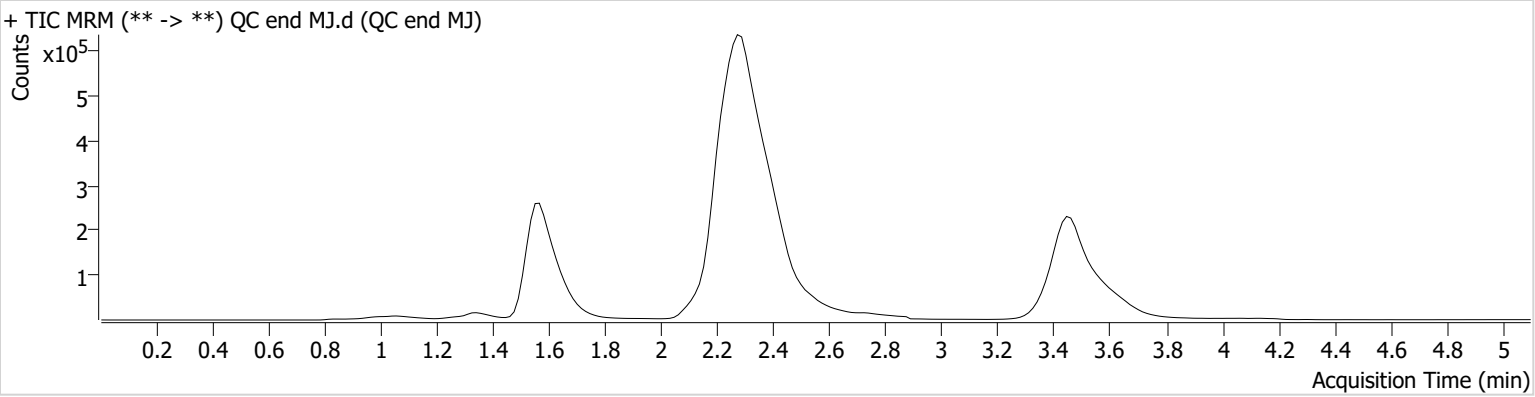
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.543	100144	378.29	12.7	248.71	1337724	4.3897	ng/ml
THC-COOH	1.569	29461	460.30	236.9	∞	373631	12.9198	ng/ml
THC	3.435	111355	∞	23.1	84.53	2476900	5.1018	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	3/7/2023 7:30:36 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	P1-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 6:59:43 PM		
Sample Info.			

Sample Chromatogram



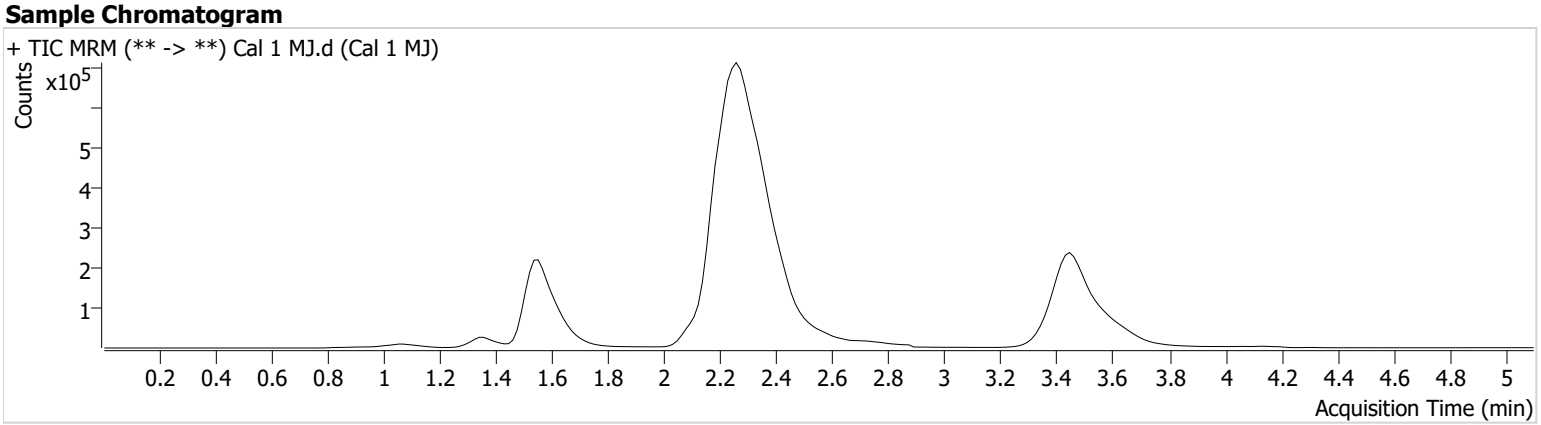
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.573	97690	∞	12.2	206.37	1297077	4.4160	ng/ml
THC-COOH	1.584	27928	407.78	240.0	691.65	313713	14.4824	ng/ml
THC	3.465	105169	∞	24.5	171.73	2370421	5.0373	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	3/7/2023 7:30:36 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	P1-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 1:36:33 PM		
Sample Info.			

Sample Chromatogram



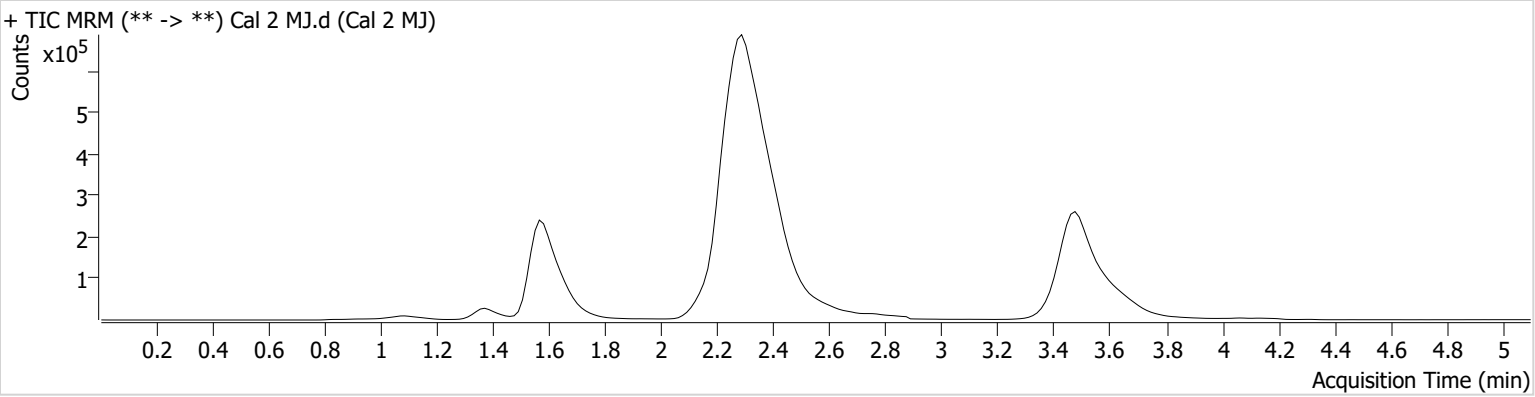
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.558	21749	220.32	12.2	12.50	1228782	1.0742	ng/ml Low
THC-COOH	1.584	9223	158.57	232.7	241.24	304998	5.4521	ng/ml
THC	3.465	23059	482.17	27.1	∞	2747243	1.1047	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	3/7/2023 7:30:36 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	P1-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 1:44:19 PM		
Sample Info.			

Sample Chromatogram



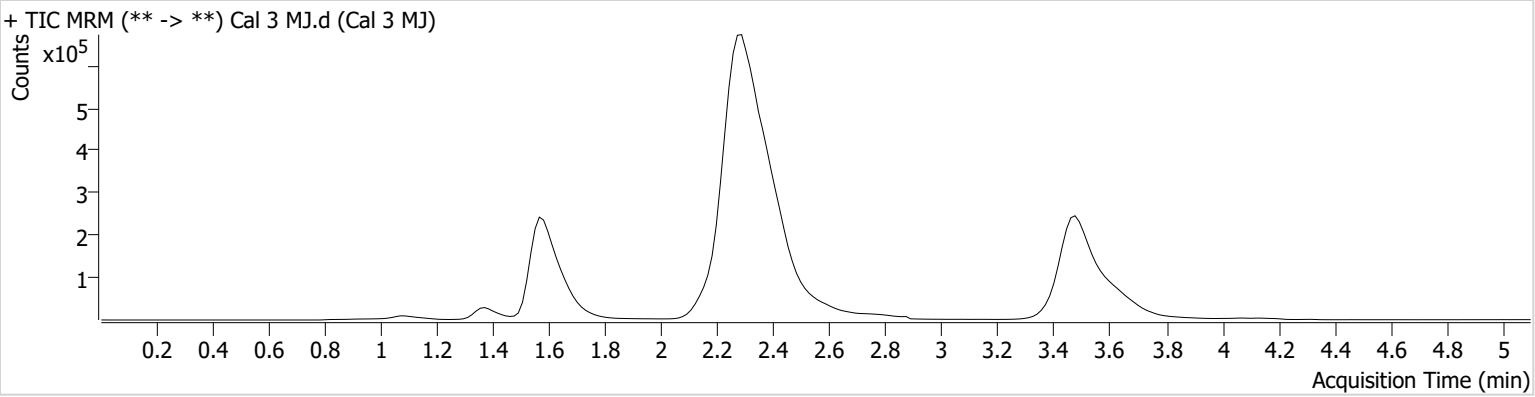
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.573	60376	∞	12.6	∞	1204660	2.9545	ng/ml Low
THC-COOH	1.599	18806	332.81	223.6	424.91	315195	9.9726	ng/ml
THC	3.480	67883	313.84	24.5	∞	2639974	2.9981	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/7/2023 7:30:36 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	P1-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 1:51:55 PM		
Sample Info.			

Sample Chromatogram



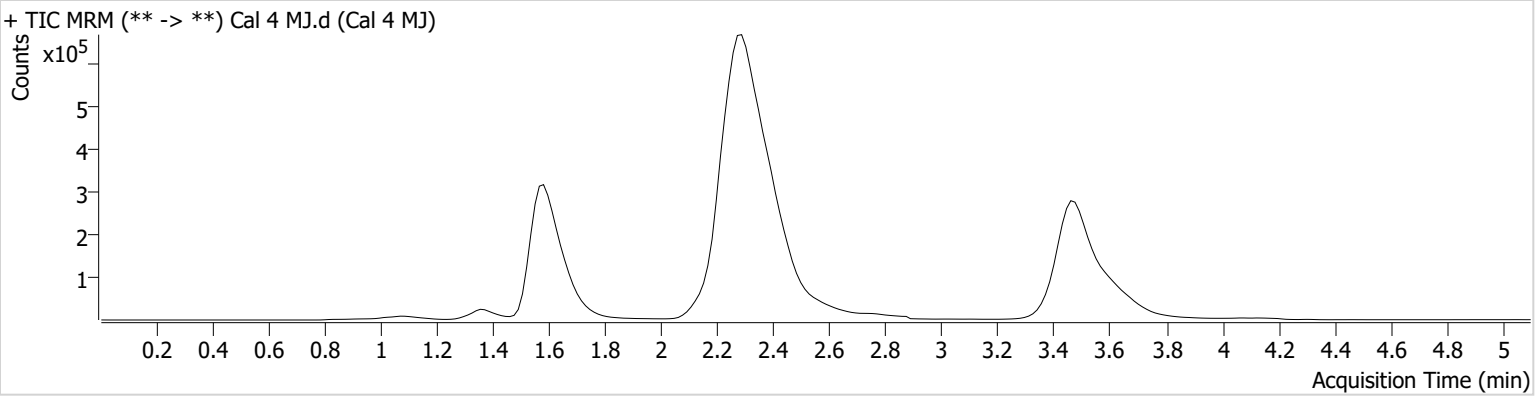
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.573	91154	∞	12.3	209.57	1104116	4.8361	ng/ml
THC-COOH	1.599	34327	587.65	228.4	1096.25	289934	18.9948	ng/ml
THC	3.480	103678	897.96	24.0	∞	2543309	4.6435	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/7/2023 7:30:36 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	P1-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 1:59:31 PM		
Sample Info.			

Sample Chromatogram



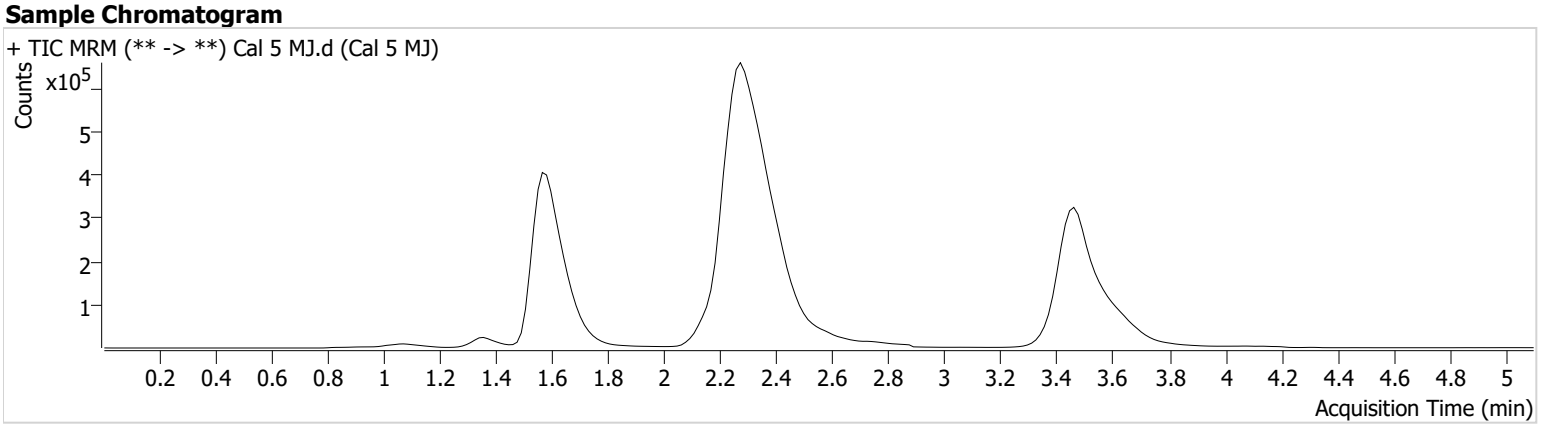
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.573	203421	∞	12.4	390.48	1208738	9.8088	ng/ml
THC-COOH	1.599	95011	1056.71	230.3	∞	312528	47.5083	ng/ml
THC	3.480	237303	∞	22.3	213.86	2705454	9.7758	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	3/7/2023 7:30:36 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	P1-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 2:07:09 PM		
Sample Info.			

Sample Chromatogram



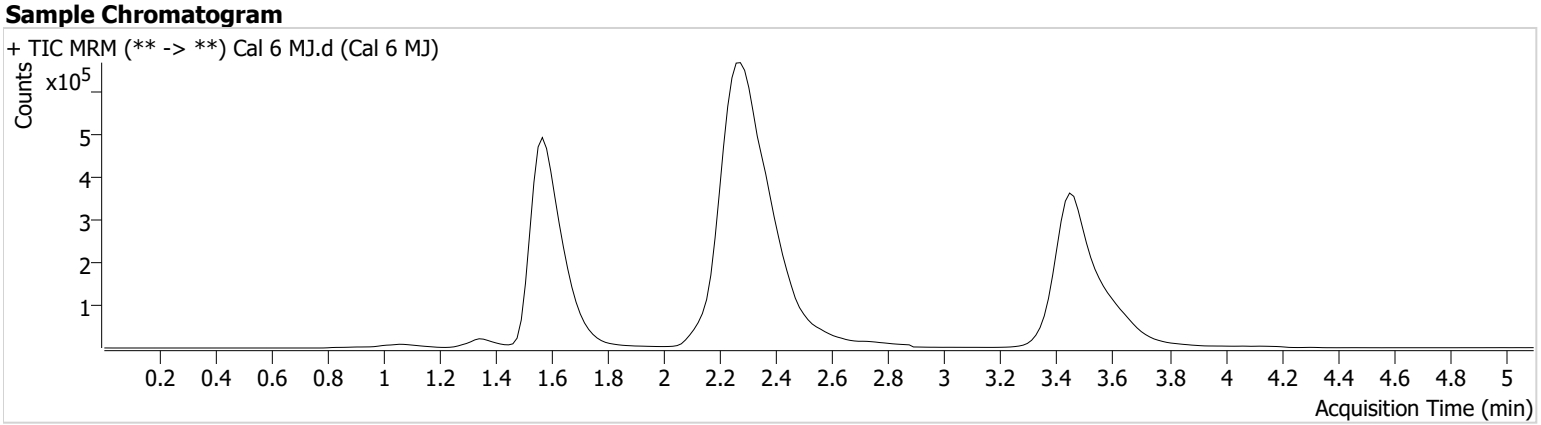
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.573	510972	∞	12.0	2409.87	1205843	24.6258	ng/ml
THC-COOH	1.599	146818	2759.50	223.5	6049.67	305717	74.5810	ng/ml
THC	3.465	602623	∞	22.9	∞	2763485	24.0260	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	3/7/2023 7:30:36 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	P1-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 2:14:47 PM		
Sample Info.			

Sample Chromatogram



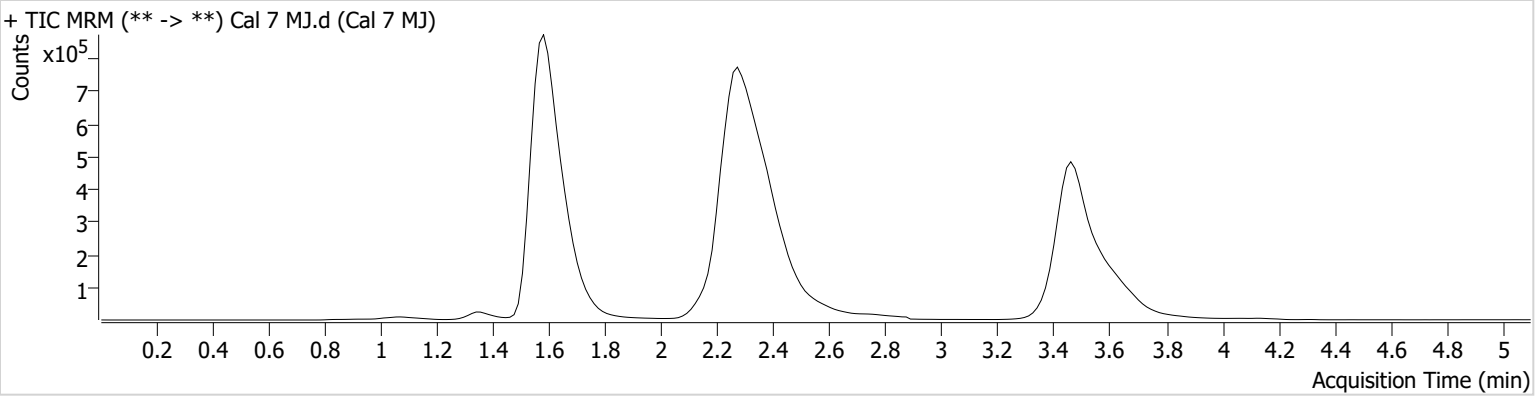
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.558	988102	∞	12.5	1576.09	1145490	50.0803	ng/ml
THC-COOH	1.584	186901	3277.12	220.0	1772.11	287505	100.6715	ng/ml
THC	3.465	1163070	∞	22.1	∞	2482144	51.4114	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2023\AM 27 28\030623 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/7/2023 7:30:36 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	P1-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/6/2023 2:22:23 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.573	1973098	∞	12.3	4636.25	1137924	100.6201	ng/ml
THC-COOH	1.599	450449	∞	224.4	∞	274580	252.8196	ng/ml
THC	3.465	2233590	∞	22.2	∞	2445327	100.0406	ng/ml