

Worklist: 4863

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
M2021-0777	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-0921	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-1064	3	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0673	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0705	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0706	1	BLOOD	AM 27 Blood THC Quant by LC-QQQ	
P2021-0729	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0752	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0759	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0762	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0779	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0780	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0782	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0783	1	BCK	AM 27 Blood THC Quant by LC-QQQ	

P2021-0377-1 and P2021-0391-1 were also included in this run.

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

Extraction Date: 03/25/2021

Analyst: Celena Shrum

Plate lot#: IDP-108-2-201206

Plate Expiration: 06/06/2021

Mobile phase A: 0.1% Formic Acid in LCMS Water

Mobile phase B: 0.1% Formic acid in Acetonitrile

Blank Blood Lot: Lampire 20L20724

Column: UCT Selectra DA 100 x 2.1mm 3um

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. 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: Curve Limitations: THC 3-100, c-THC 5-250, THC-OH 3-100. THC-OH will be reported qualitatively due the end QC not meeting accuracy requirement.

Samples were injected on 3/25/21 but the instrument stopped about half way through the run and the solvent kept running so the solvent ran out. As such, the entire run was reinjected on 3/29/21 with no issues.

**Idaho State Police
Forensic Services
Toxicology Discipline**

Request for Departure from an Analytical Method

Date of Request

02/24/2021

Forensic Scientist

Anne Nord

Analytical Method

Toxicology AM #27: Quantitative Analysis of THC and Metabolites in Blood and Urine by LCMS-QQQ

Request

The method currently reads:

4.3.2.5 If any points are dropped from the approved quantitative range of the curve, the compound will be reported qualitatively. For calibrators and controls 10 ng and below, the accuracy must be within 30%, for calibrators and controls greater than 10 ng/mL the accuracy must be within 20%. If a control falls outside the accuracy range, at the analyst's discretion, the compound may be reported qualitatively.

I would like to add in the following exception:

If the 1ng/ml point is dropped for THC, the quantitative range will be 3-50 ng/ml.

Discipline Leader Review

Departure approved

Comments: This deviation is approved and will remain in effect until it is changed in the actual method.

Departure Not Approved

Comments:

Celena Shrum

Toxicology Discipline Lead

Date: 02/24/2021

	1	2	3	4	5	6
a				P2021-0377-1	P2021-0729-1	QC 1
b				P2021-0783-1	P2021-0706-1	cal 100 ng
c				P2021-0782-1	P2021-0705-1	cal 50 ng
d				P2021-0780-1	P2021-0673-1	cal 25 ng
e				P2021-0779-1	M2021-1064-3	cal 10ng
f				P2021-0762-1	M2021-0921-1	cal 5 ng
g				P2021-0759-1	M2021-0777-1	cal 3 ng
h			P2021-0391-1	P2021-0752-2	Blood NC	cal 1ng

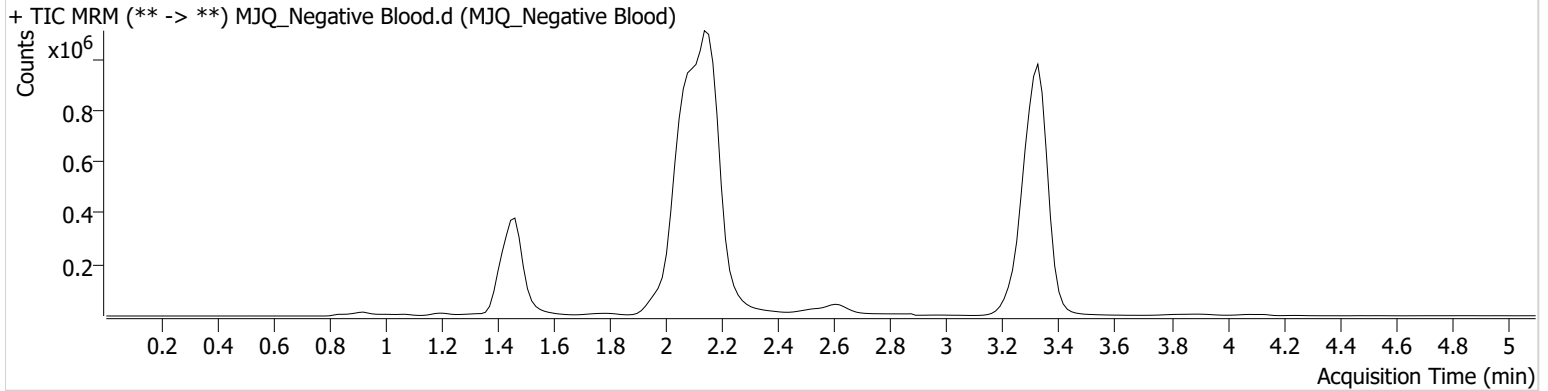
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument	Instrument 1	Data File	MJQ_Negative Blood.d
Type	Sample	Sample	MJQ_Negative Blood
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-H5	Comment	
Injection Volume	10		
Acq. Date-Time	3/29/2021 10:42:23 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.528 High	30329	∞	2.8 Low	4.23 Low	1503486	0.1468 ng/ml Low

. THC



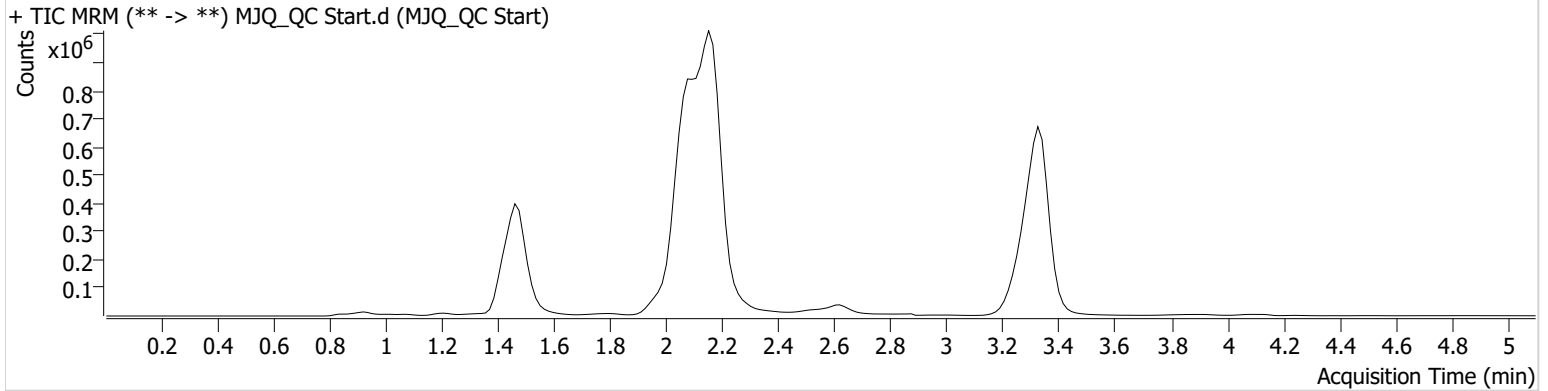
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument Instrument 1
Type Sample
Acq. Method AM 27 THCQ.m
Sample Position P1-A6
Injection Volume 10
Acq. Date-Time 3/29/2021 10:57:34 AM
Sample Info.

Data File MJQ_QC Start.d
Sample MJQ_QC Start
Operator Celena Shrum
Comment

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	88913	∞	8.9	∞	1362266	3.5270 ng/ml
THC-COOH	1.504	164052	∞	53.8	488.91	497199	12.0425 ng/ml
THC	3.345	129522	∞	30.9	∞	4149396	3.5827 ng/ml

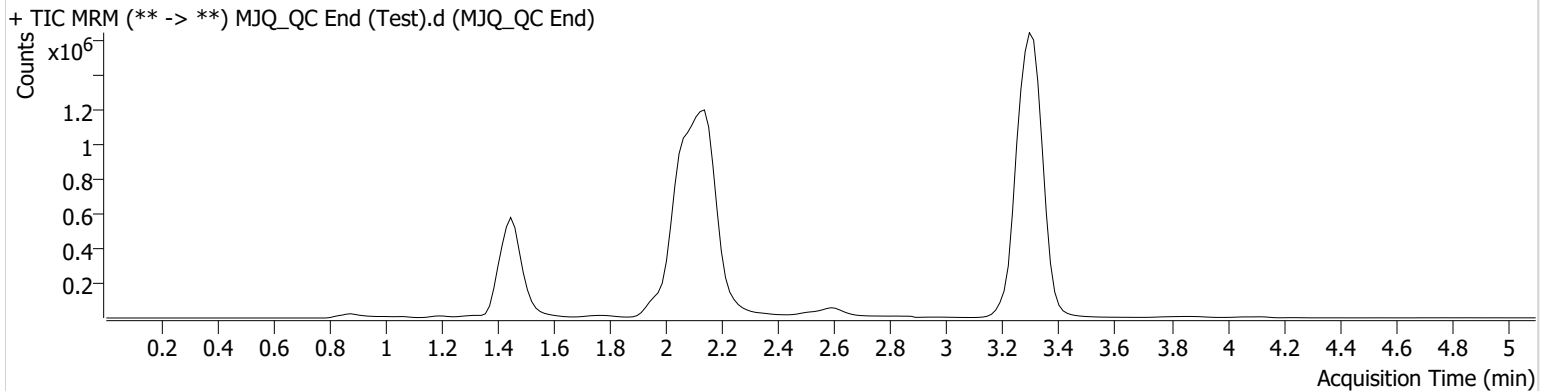
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 8:32:58 AM

Instrument	Instrument 1	Data File	MJQ_QC End (Test).d
Type	Sample	Sample	MJQ_QC End
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-A6	Comment	
Injection Volume	10		
Acq. Date-Time	3/29/2021 3:08:19 PM		
Sample Info.			

Sample Chromatogram

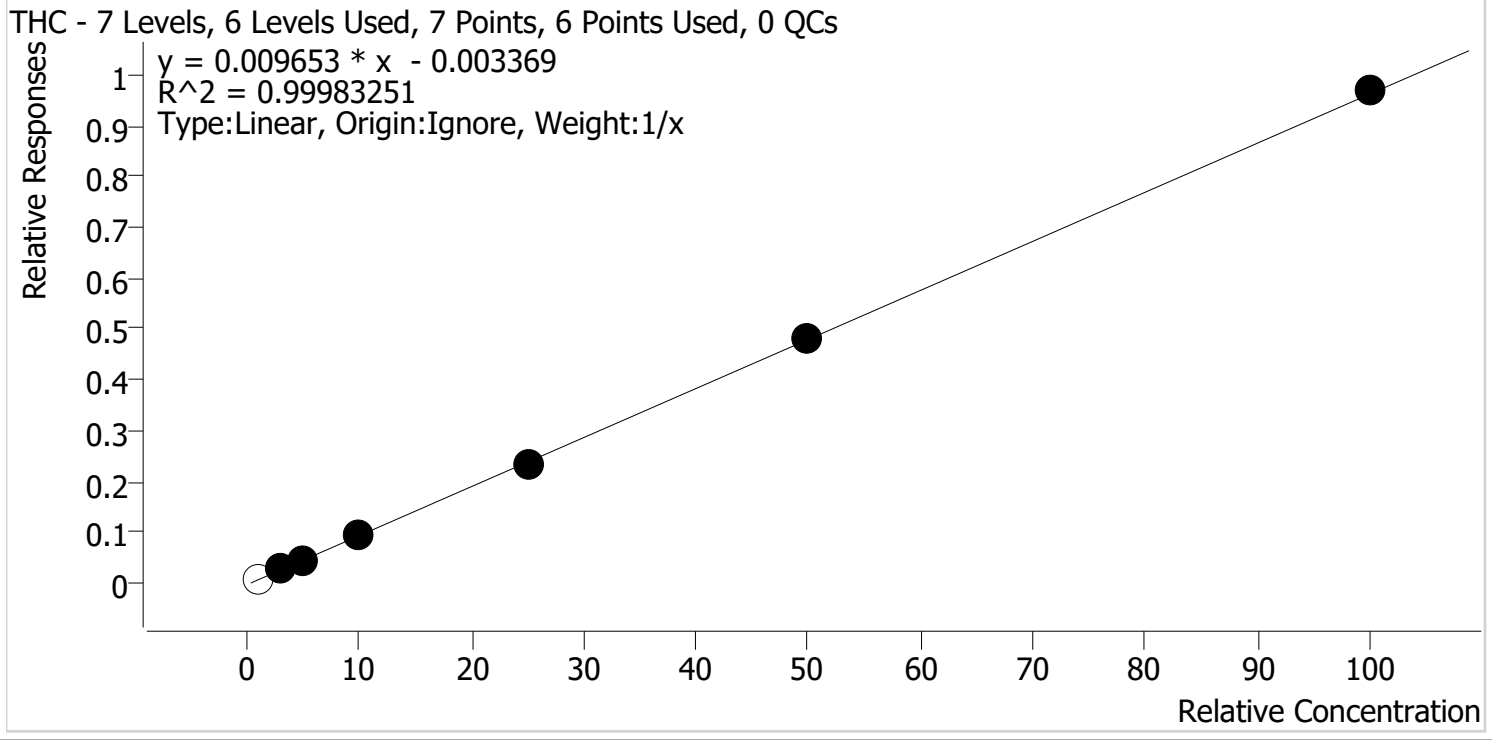


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	126096	∞	9.0	88.92	2067452	3.2063 ng/ml
THC-COOH	1.489	214728	∞	55.0	∞	621900	12.6373 ng/ml
THC	3.315	328564	∞	27.9	271.24	10341284	3.6405 ng/ml



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Last Cal. Update 4/2/2021 7:32 AM
Analyst Name ISP\Datastor
Analyte THC **Internal Standard** THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	x	1.0	1.2	124.0
MJQ_Cal 2	2	✓	3.0	3.0	101.2
MJQ_Cal 3	3	✓	5.0	5.1	102.1
MJQ_Cal 4	4	✓	10.0	9.9	98.6
MJQ_Cal 5	5	✓	25.0	24.4	97.6
MJQ_Cal 6	6	✓	50.0	49.9	99.9
MJQ_Cal 7	7	✓	100.0	100.7	100.7

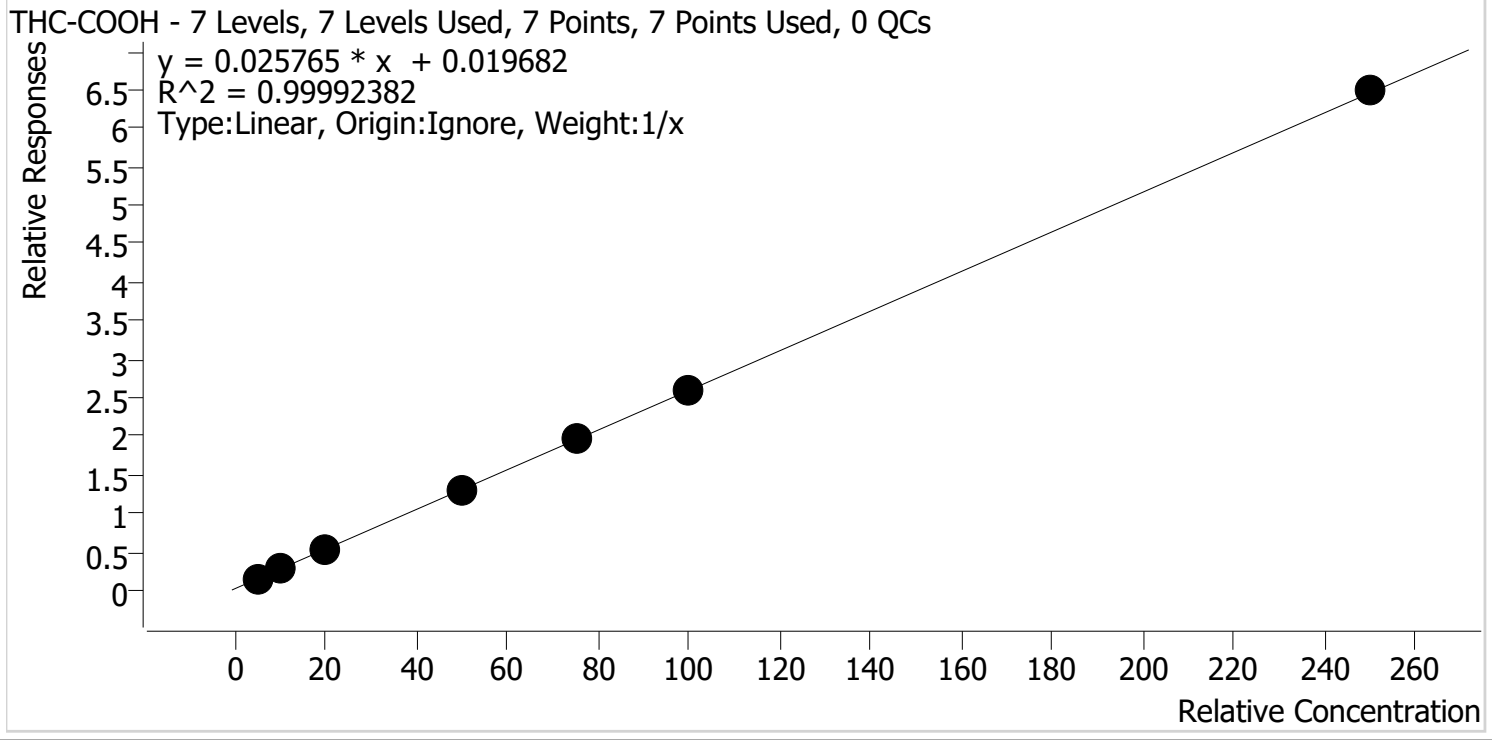
Calibrator 1 dropped due to poor peak shape/response.

cg



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Last Cal. Update 4/2/2021 7:32 AM
Analyst Name ISP\Datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9



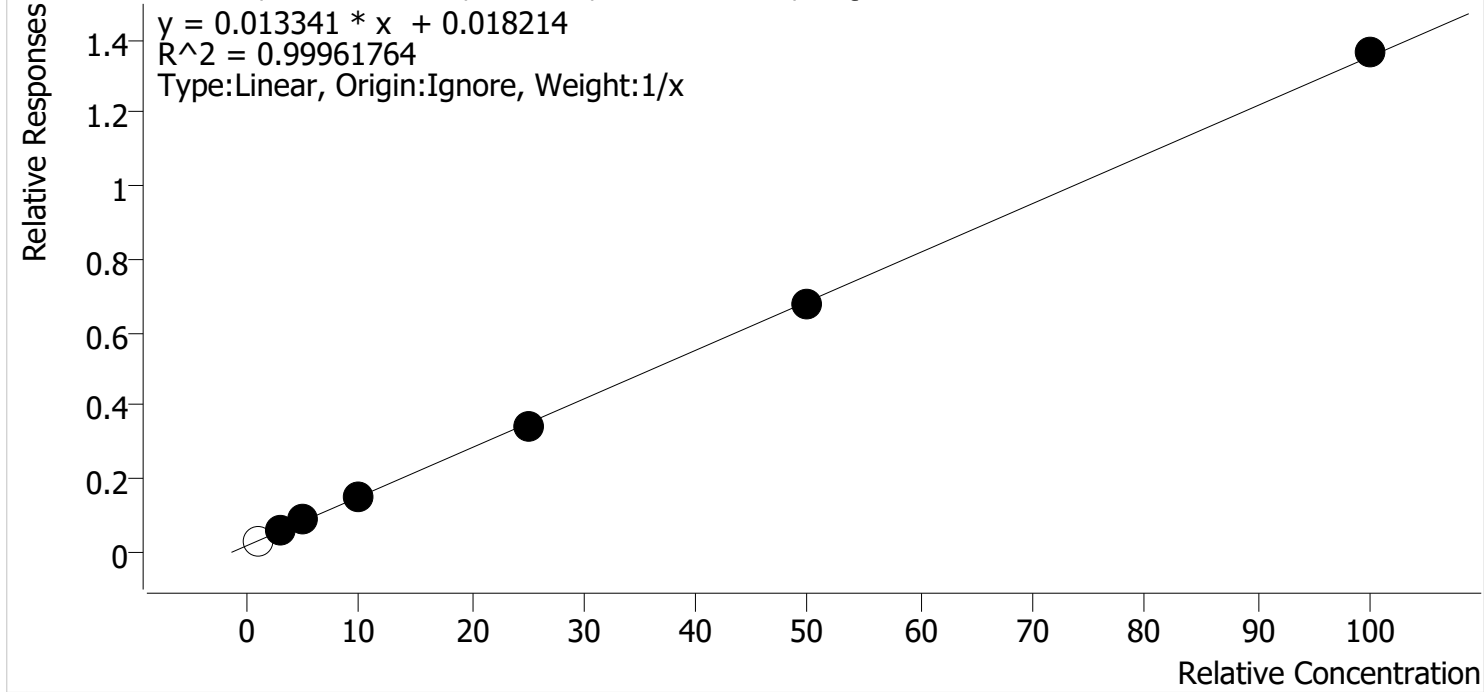
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✓	5.0	4.9	98.9
MJQ_Cal 2	2	✓	10.0	10.3	102.8
MJQ_Cal 3	3	✓	20.0	20.0	100.0
MJQ_Cal 4	4	✓	50.0	49.3	98.5
MJQ_Cal 5	5	✓	75.0	75.0	100.0
MJQ_Cal 6	6	✓	100.0	99.3	99.3
MJQ_Cal 7	7	✓	250.0	251.2	100.5



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Last Cal. Update 4/2/2021 7:32 AM
Analyst Name ISP\Datastor
Analyte THC-OH **Internal Standard** THC-OH-D3

THC-OH - 7 Levels, 6 Levels Used, 7 Points, 6 Points Used, 0 QCs



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	x	1.0	1.0	102.0
MJQ_Cal 2	2	✓	3.0	2.9	95.7
MJQ_Cal 3	3	✓	5.0	5.4	107.5
MJQ_Cal 4	4	✓	10.0	9.9	98.6
MJQ_Cal 5	5	✓	25.0	24.6	98.5
MJQ_Cal 6	6	✓	50.0	49.4	98.9
MJQ_Cal 7	7	✓	100.0	100.8	100.8

Calibrator 1 dropped due to poor peak shape/response.



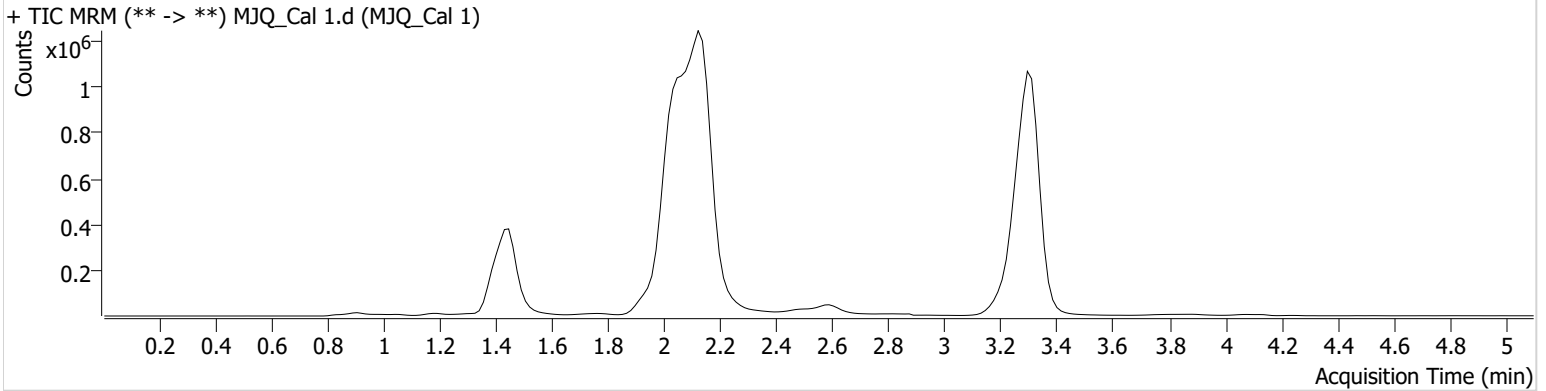
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument Instrument 1
Type Cal
Acq. Method AM 27 THCQ.m
Sample Position P1-H6
Injection Volume 10
Acq. Date-Time 3/29/2021 9:41:24 AM
Sample Info.

Data File MJQ_Cal 1.d
Sample MJQ_Cal 1
Operator Celena Shrum
Comment

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.498	48191	∞	5.7 Low	8.48 Low	1514364	1.0200 ng/ml Low
THC-COOH	1.474	78446	79.26	48.5	799.19	533215	4.9462 ng/ml Low
THC	3.315	57119	283.38	44.3 High	∞	6637915	1.2405 ng/ml



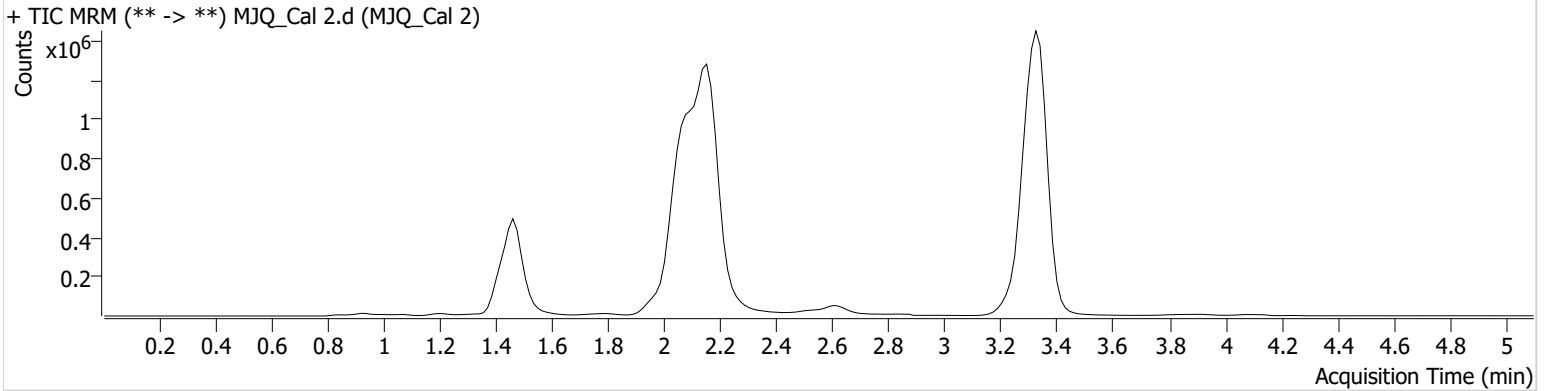
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument Instrument 1
Type Cal
Acq. Method AM 27 THCQ.m
Sample Position P1-G6
Injection Volume 10
Acq. Date-Time 3/29/2021 9:49:09 AM
Sample Info.

Data File MJQ_Cal 2.d
Sample MJQ_Cal 2
Operator Celena Shrum
Comment

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	96665	∞	8.6	145.29	1710437	2.8708 ng/ml Low
THC-COOH	1.489	171540	223.37	49.9	250.59	603018	10.2772 ng/ml
THC	3.345	225517	717.54	27.3	186.60	8691726	3.0369 ng/ml



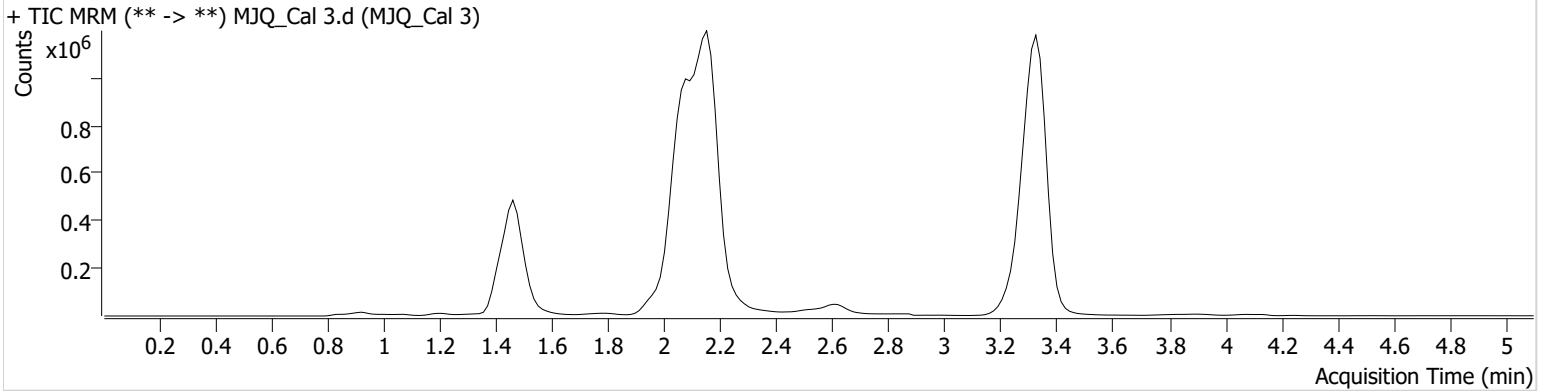
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument Instrument 1
Type Cal
Acq. Method AM 27 THCQ.m
Sample Position P1-F6
Injection Volume 10
Acq. Date-Time 3/29/2021 9:56:45 AM
Sample Info.

Data File MJQ_Cal 3.d
Sample MJQ_Cal 3
Operator Celena Shrum
Comment

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	139807	77.73	8.4	381.55	1554524	5.3759 ng/ml
THC-COOH	1.489	296739	514.78	55.1	∞	554884	19.9923 ng/ml
THC	3.345	318003	1203.40	26.9	272.78	6929372	5.1032 ng/ml



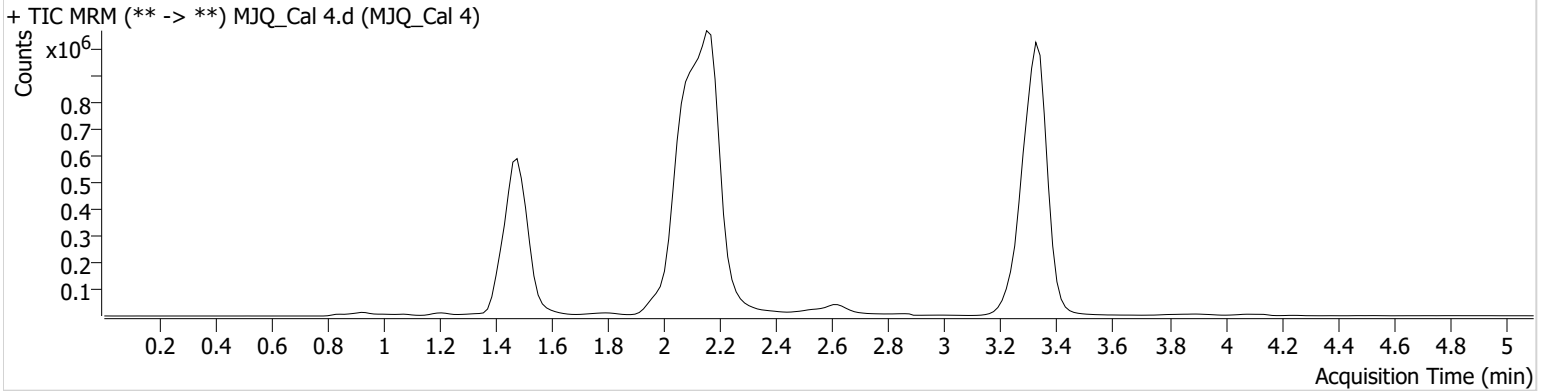
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument Instrument 1
Type Cal
Acq. Method AM 27 THCQ.m
Sample Position P1-E6
Injection Volume 10
Acq. Date-Time 3/29/2021 10:04:20 AM
Sample Info.

Data File MJQ_Cal 4.d
Sample MJQ_Cal 4
Operator Celena Shrum
Comment

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	223841	∞	10.3	210.48	1495293	9.8553 ng/ml
THC-COOH	1.504	701682	∞	56.1	∞	544488	49.2543 ng/ml
THC	3.345	532115	1068.75	26.8	∞	5797120	9.8580 ng/ml

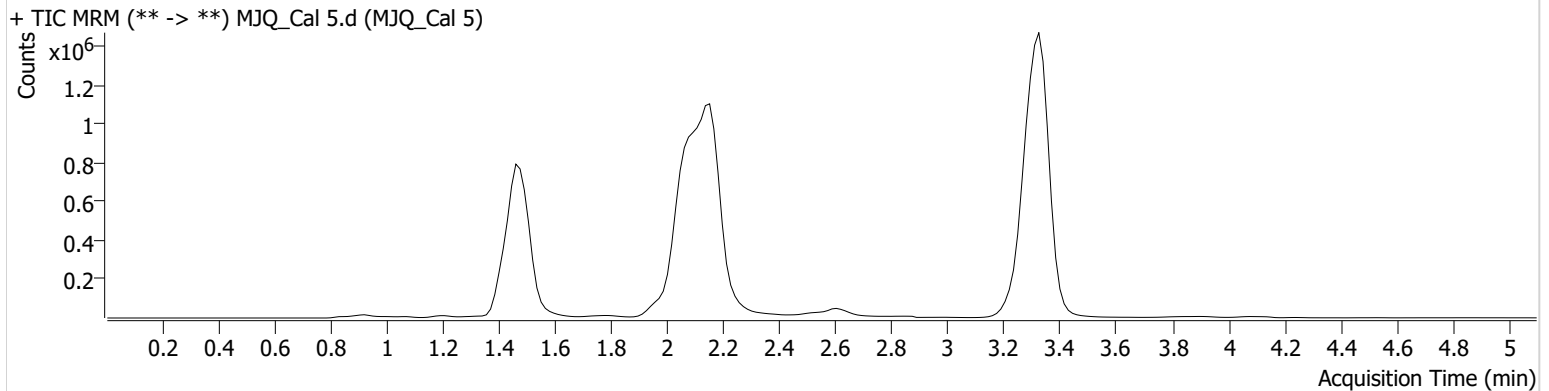
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument	Instrument 1	Data File	MJQ_Cal 5.d
Type	Cal	Sample	MJQ_Cal 5
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-D6	Comment	
Injection Volume	10		
Acq. Date-Time	3/29/2021 10:11:56 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	552763	∞	10.8	∞	1593832	24.6302 ng/ml
THC-COOH	1.489	1075754	∞	57.1	∞	550879	75.0297 ng/ml
THC	3.330	1672436	12506.78	25.4	460.47	7204142	24.3986 ng/ml

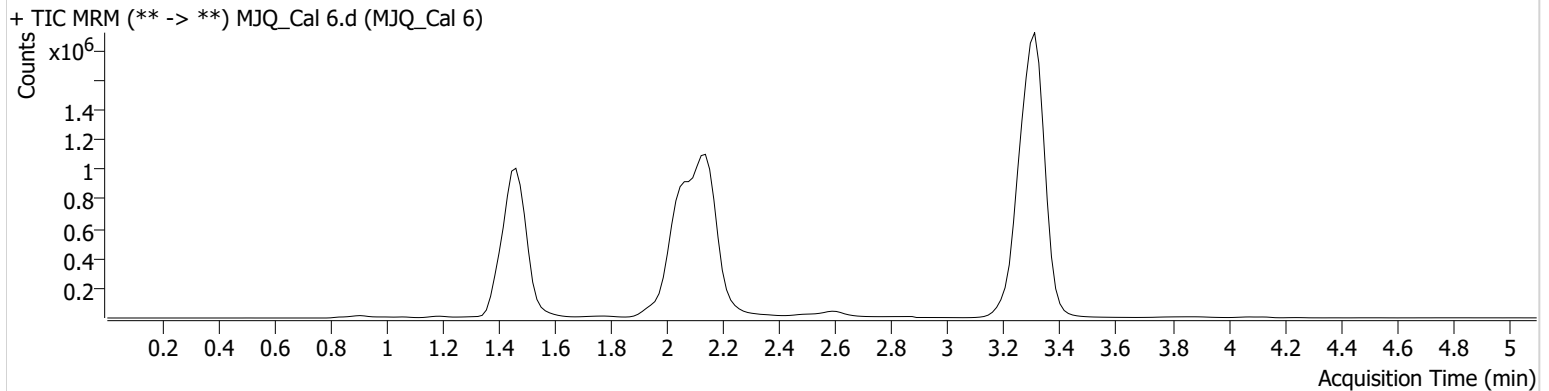
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument	Instrument 1	Data File	MJQ_Cal 6.d
Type	Cal	Sample	MJQ_Cal 6
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-C6	Comment	
Injection Volume	10		
Acq. Date-Time	3/29/2021 10:19:35 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	1126150	∞	11.5	776.65	1661231	49.4468 ng/ml
THC-COOH	1.474	1472051	3223.66	57.7	1545.19	570833	99.3259 ng/ml
THC	3.315	3712508	13646.36	25.5	866.64	7757511	49.9265 ng/ml

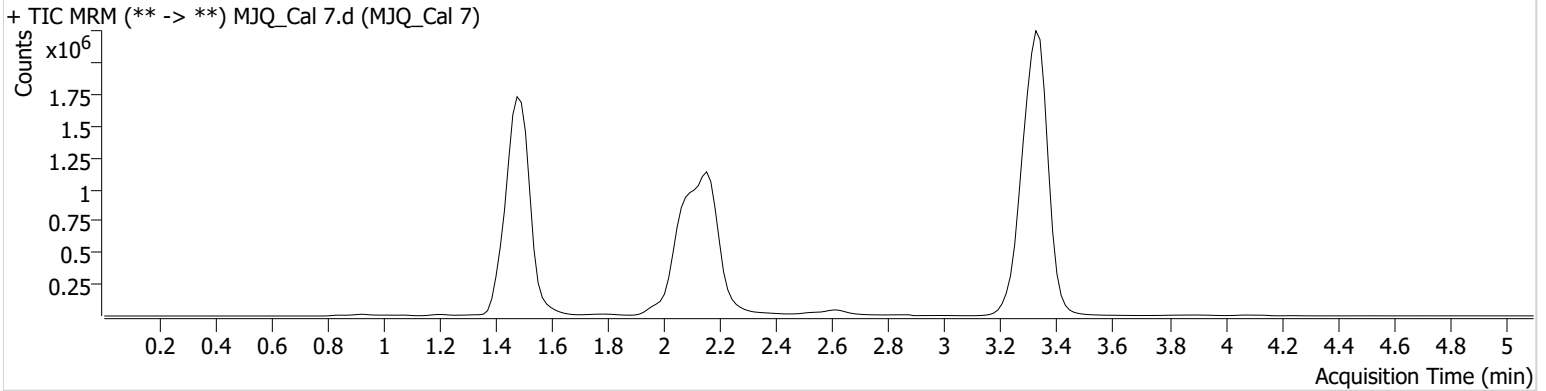
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\032521 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 4/2/2021 7:32:41 AM

Instrument	Instrument 1	Data File	MJQ_Cal 7.d
Type	Cal	Sample	MJQ_Cal 7
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-B6	Comment	
Injection Volume	10		
Acq. Date-Time	3/29/2021 10:27:11 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	2106963	∞	11.7	1647.48	1545484	100.8210 ng/ml
THC-COOH	1.504	3332336	∞	58.2	16229.4	513370	251.1745 ng/ml
THC	3.345	6383677	14964.95	25.2	∞	6591587	100.6767 ng/ml