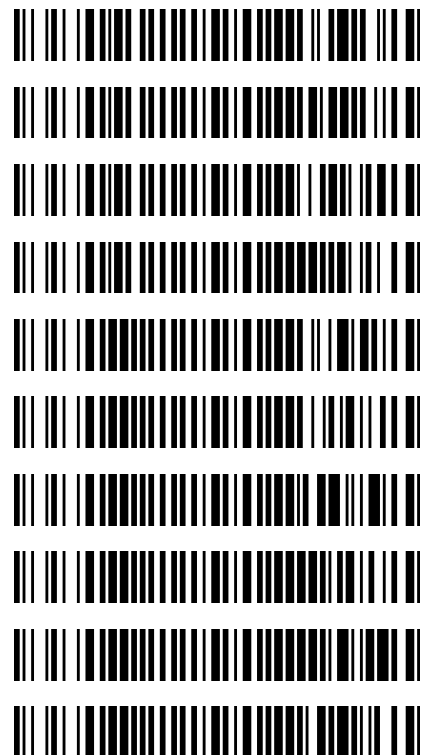


Worklist: 4095

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>
M2020-0494	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2020-0752	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2020-0854	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2020-1014	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0789	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0792	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0826	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0845	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0848	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0852	1	BCK	AM 27 Blood THC Quant by LC-QQQ



**Idaho State Police
Forensic Services
Toxicology Discipline**

Request for Departure from an Analytical Method

Date of Request

01/13/2020

Forensic Scientist

Celena Shrum

Analytical Methods

Toxicology AM #25, Toxicology AM #26/27, and AM #28

Deviation

The expiration dates listed for the current batch of PinPoint ToxBox extraction plates are as follows:

- *MDS (batch IDP-107-190725)- Expiration is 1/25/2020
- *THC (batch IDP-108-190716)- Expiration is 1/16/2020
- *MDQ P1 (batch IDP-111-190729)- Expiration is 1/29/2020
- *MDQ P2 (batch IDP-112-190730)- Expiration is 1/30/2020

I am issuing a deviation to allow for the use of the remaining plates of these batches. The controls will be used to evaluate if the plate is working as intended. In addition, at least one external control must be included for each run.

Celena Shrum

Date: 01/13/2020

Celena Shrum

Toxicology Discipline Lead

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

Extraction Date: 03/19/20
 Plate lot#: IDP-108-190716

Analyst: Sarah Pickle
 Plate Expiration: 01/16/20

Mobile phase A: 0.1% Formic Acid in LCMS Water
 MTBE LCMS Methanol

Mobile phase B: 0.1% Formic acid in Acetonitrile
 Hexane

Blank Blood Lot: Hemostat 445283-3

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. Pipette **1000 µL blood (calibrated pipette)** in wells of analytical (standards) plate. **Pipette ID: 3382167**
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 067105*
- 4. Pipette **500 µL 0.1% formic acid in LCMS water** in wells of analytical plate.
- 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 6. Transfer **800 µL of blood+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 85-95 PSI- Selector to the right) Manifold ID: 067104
- 8. Wait 5 minutes.
- 9. Add **2.25 mL MTBE. (Add in 3 increments of 750 µL)**
- 10. Wait 5 minutes.
- 11. Apply positive pressure for approx. 15 seconds. *(12-15 PSI- Selector to the left).*
- 12. Add **2.25 mL Hexane. (Add in 3 increments of 750 µL)**
- 13. Wait 5 minutes.
- 14. Apply positive pressure for approx. 15 seconds. *(12-15 PSI- Selector to the left).*
- 15. Remove plate containing eluate. Place on SPE Dry and evaporate to dryness at approx. 35°C.
SPE Dry ID: 067103
- 16. Reconstitute in **100 µL 100% MeOH** and heat seal plate with foil. Place in autosampler and run worklist.

Post-Analytic

- 1. Create batch and process data.
 Worklist path: D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP Batch Name: AM 27
- 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 and OH-THC 3ng/mL (quantitative), Carboxy-THC: 10ng/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? Y / N
- 6. Enter QCs into control charting.
- 7. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: *Curves limited: THC 3-100, COOH 10-250, THC-OH 5-100 (reported qualitatively)*



Idaho State Police Forensic Services

AM #26 Blood THC and Metabolites Screen by LCMS-QQQ and AM #27 Quantitative Analysis of THC and Metabolites in Blood by LCMS-QQQ

Methanol External Control Solution (Lot: WS011620)

*10 μ L of 1mg/mL THC, 100 μ L of 100 μ g/mL THC-OH, C-THC in 9790 μ L MeOH
Approximate concentration 1 μ g/mL.*

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>	<i>Expiration Date</i>
Methanol (LCMS)	Fisher	193941	
THC	Cerilliant	FE09101501	11/30/2020
C-THC	Cerilliant	FE07171501	09/30/2020
THC-OH	Cerilliant	FE07221601	07/31/2021
Prepared:	01/16/2020		
Prepared By:	Tamara Salazar		
Expires:	09/30/2020		

Blood External Control Solution (Lot: 031820)

*200 μ L of methanol external control solution was added to 9800 μ L of blood.
Approximately 20 ng/mL of each compound.*

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Negative Blood	Hemostat	445283-3
Methanol External Control Solution	-	WS011620
Prepared:	03/18/20	
Prepared by:	Sarah Pickle	
Expires:	09/30/2020	

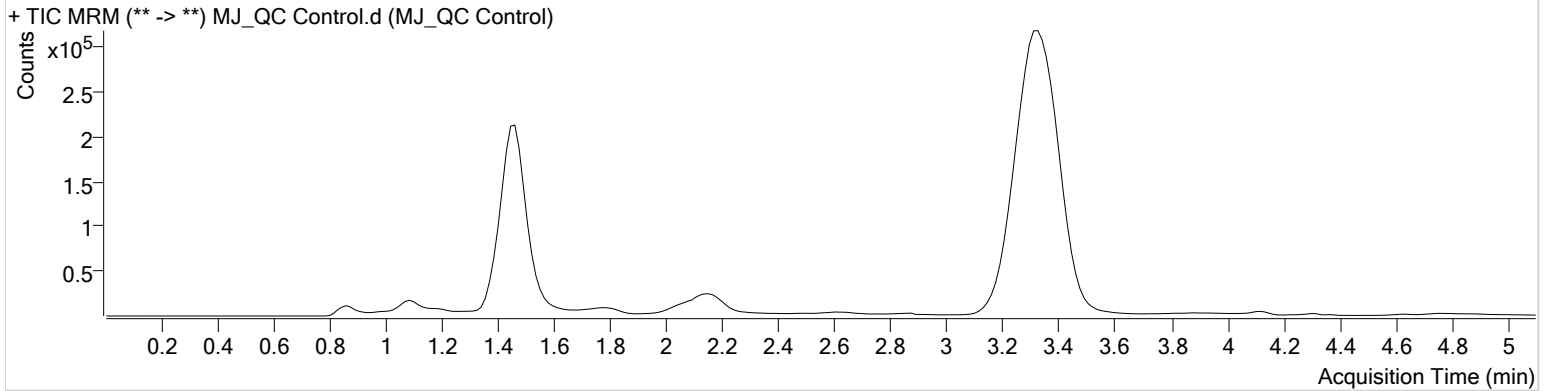
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_QC Control.d
Type	Sample	Sample	MJ_QC Control
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-A6	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 11:35:53 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	112976	∞	9.1	83.06	865327	4.3101 ng/ml
THC-COOH	1.489	101742	∞	47.5	164.62	254341	14.1975 ng/ml
THC	3.345	104044	391.14	28.5	73.61	3302796	4.3274 ng/ml

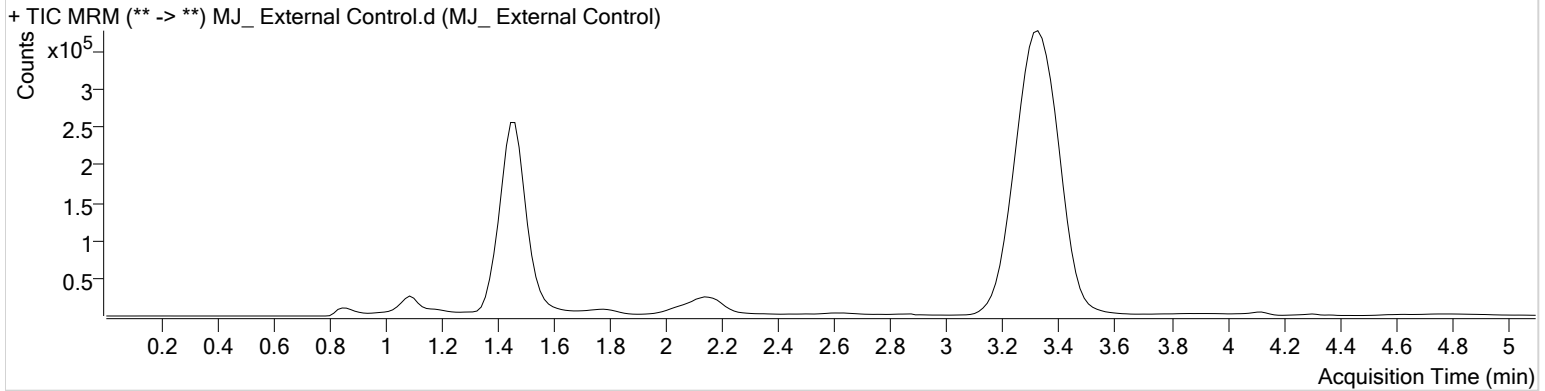
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 11:26:22 AM

Instrument	Falco	Data File	MJ_ External Control.d
Type	Sample	Sample	MJ_ External Control
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-G5	Comment	
Injection Volume	10		
Acq. Date-Time	3/20/2020 12:06:16 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	283567	∞	11.1	358.58	920050	17.0200 ng/ml
THC-COOH	1.489	142435	∞	42.9	895.60	261977	19.9688 ng/ml
THC	3.330	461529	∞	25.1	371.66	3571570	16.6414 ng/ml

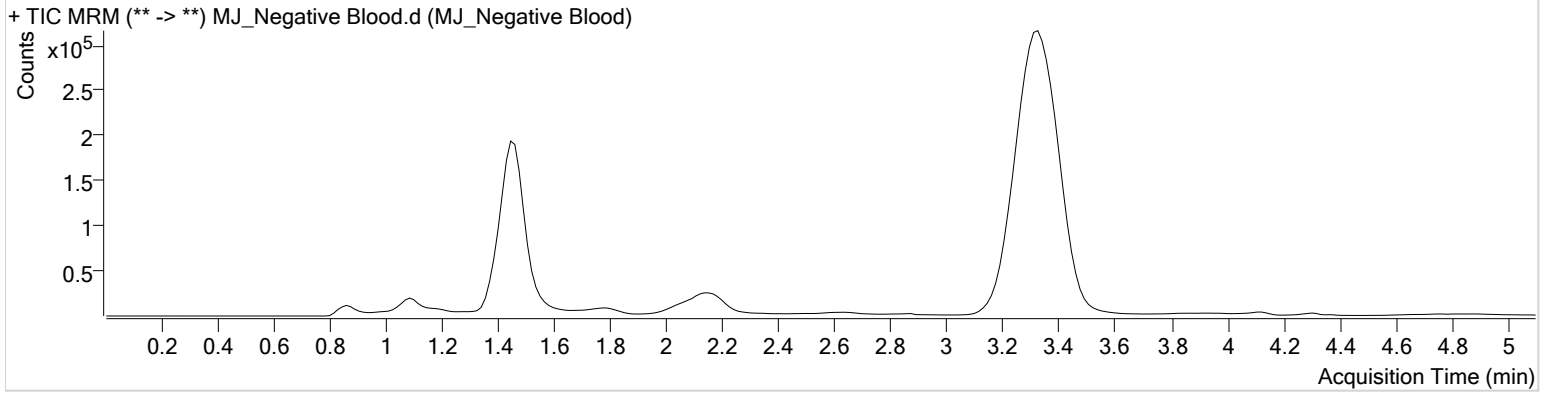
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_Negative Blood.d
Type	Sample	Sample	MJ_Negative Blood
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-H5	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 11:51:05 PM		
Sample Info.			

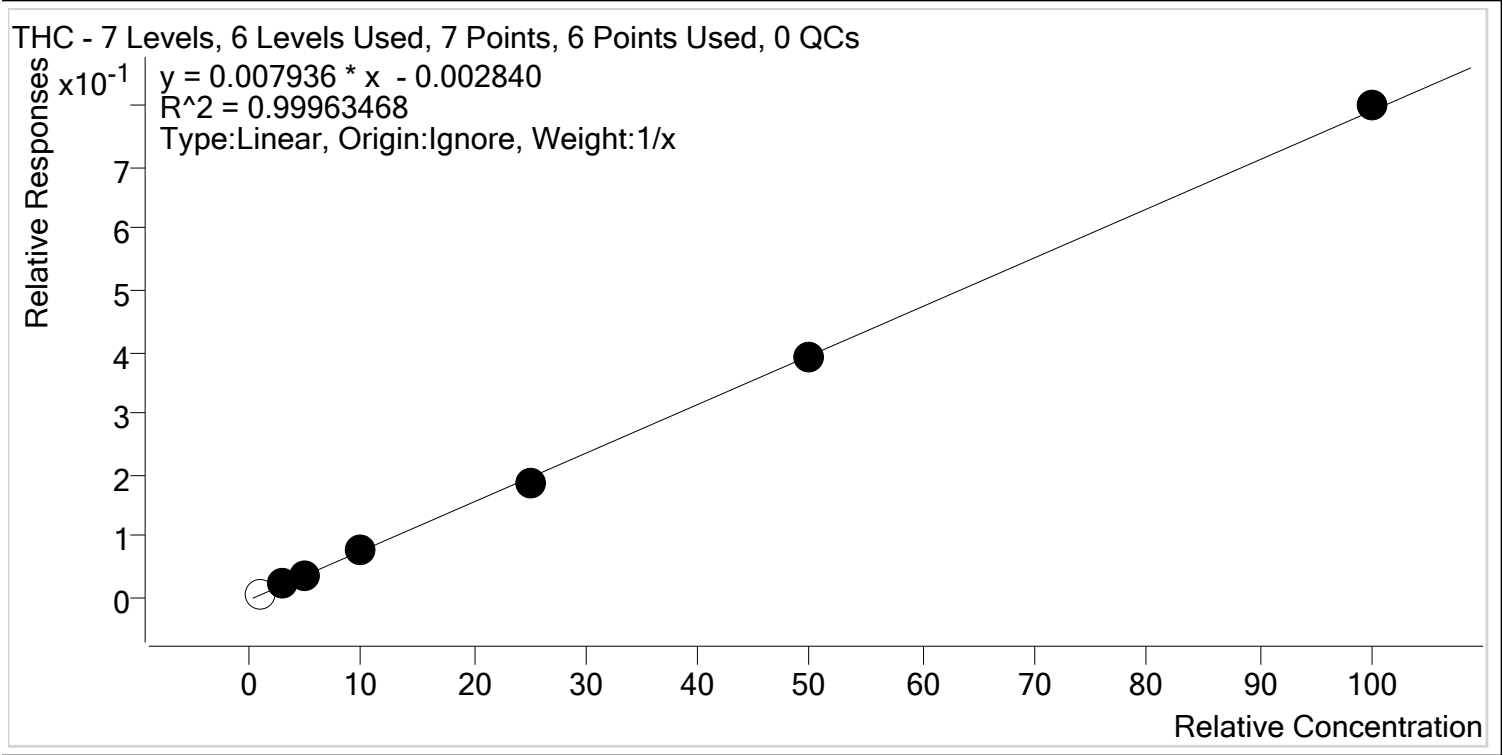
Sample Chromatogram





AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Last Cal. Update 3/20/2020 10:03 AM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-D3

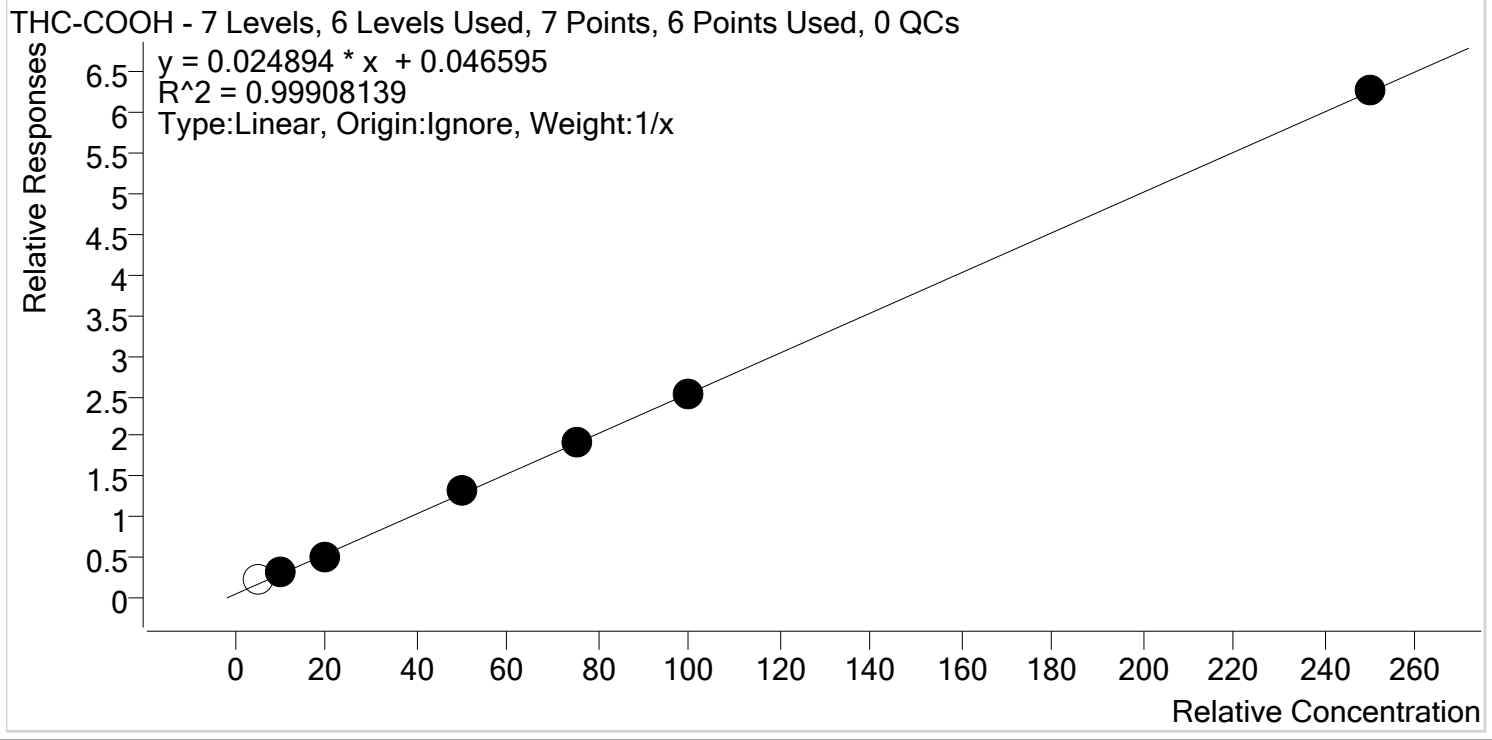


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	x	1.0	1.3	128.0
MJ_Cal 2	2	✓	3.0	3.1	104.3
MJ_Cal 3	3	✓	5.0	5.0	99.2
MJ_Cal 4	4	✓	10.0	9.9	99.5
MJ_Cal 5	5	✓	25.0	24.1	96.3
MJ_Cal 6	6	✓	50.0	49.8	99.6
MJ_Cal 7	7	✓	100.0	101.1	101.1



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Last Cal. Update 3/20/2020 10:03 AM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9



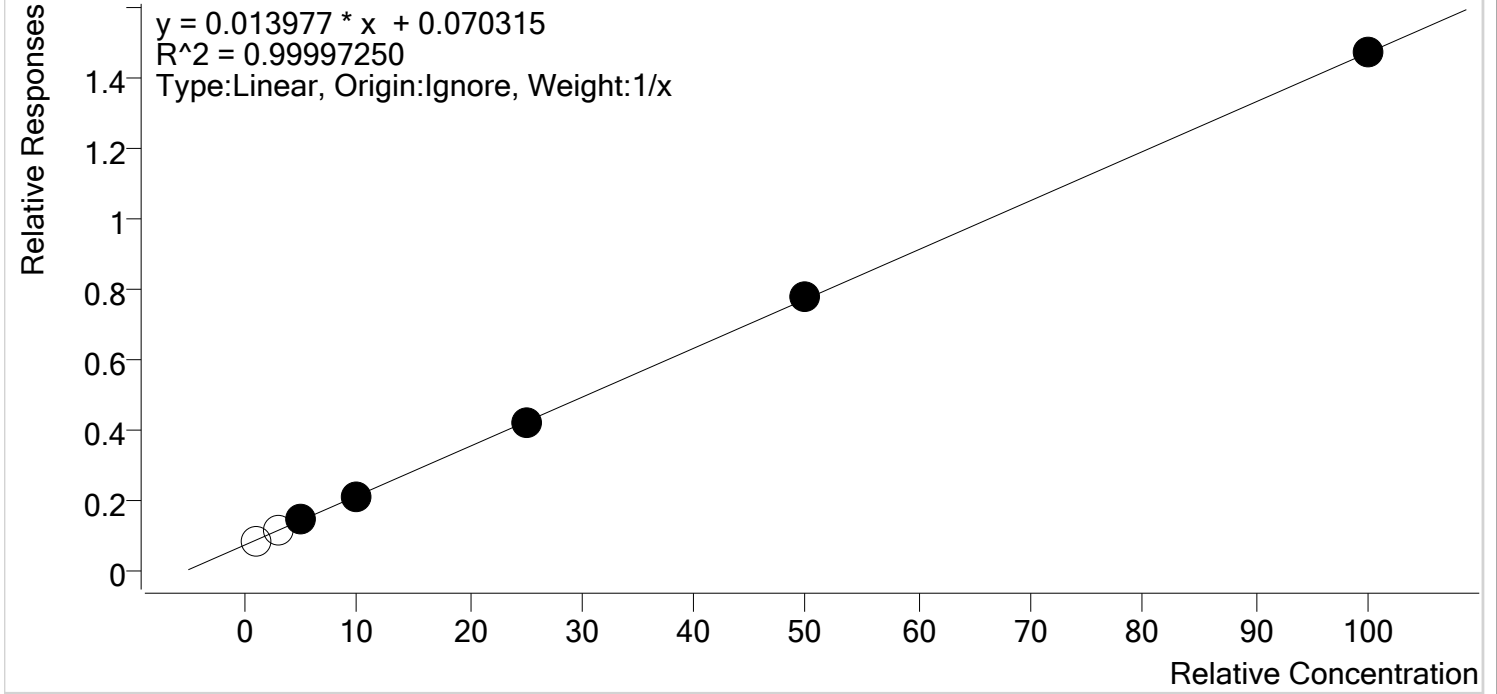
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	x	5.0	7.7	154.3
MJ_Cal 2	2	✓	10.0	10.7	107.4
MJ_Cal 3	3	✓	20.0	18.1	90.3
MJ_Cal 4	4	✓	50.0	51.5	103.1
MJ_Cal 5	5	✓	75.0	74.8	99.7
MJ_Cal 6	6	✓	100.0	99.2	99.2
MJ_Cal 7	7	✓	250.0	250.6	100.2



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Last Cal. Update 3/20/2020 10:03 AM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-D3

THC-OH - 7 Levels, 5 Levels Used, 7 Points, 5 Points Used, 0 QCs



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	x	1.0	0.9	87.4
MJ_Cal 2	2	x	3.0	2.9	96.7
MJ_Cal 3	3	✓	5.0	5.1	101.4
MJ_Cal 4	4	✓	10.0	9.9	98.6
MJ_Cal 5	5	✓	25.0	24.9	99.7
MJ_Cal 6	6	✓	50.0	50.1	100.2
MJ_Cal 7	7	✓	100.0	100.0	100.0

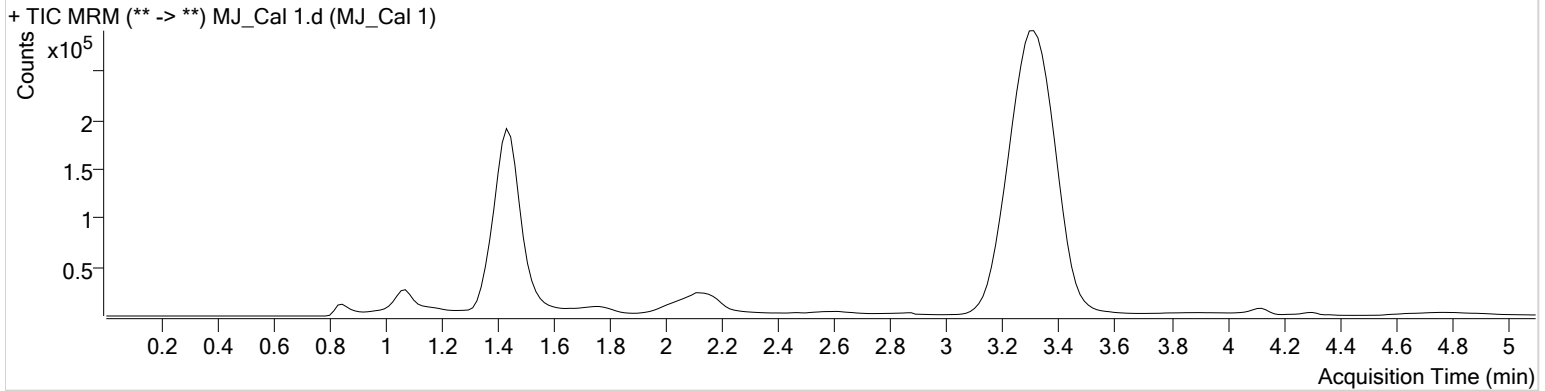
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_Cal 1.d
Type	Cal	Sample	MJ_Cal 1
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-B6	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 10:35:07 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.498	73037	∞	3.9 Low	17.82	885008	0.8737 ng/ml Low
THC-COOH	1.474	61269	∞	27.4 Low	∞	256729	7.7152 ng/ml Low
THC	3.315	24443	36.47	24.8	4.58 Low	3338927	1.2803 ng/ml Low

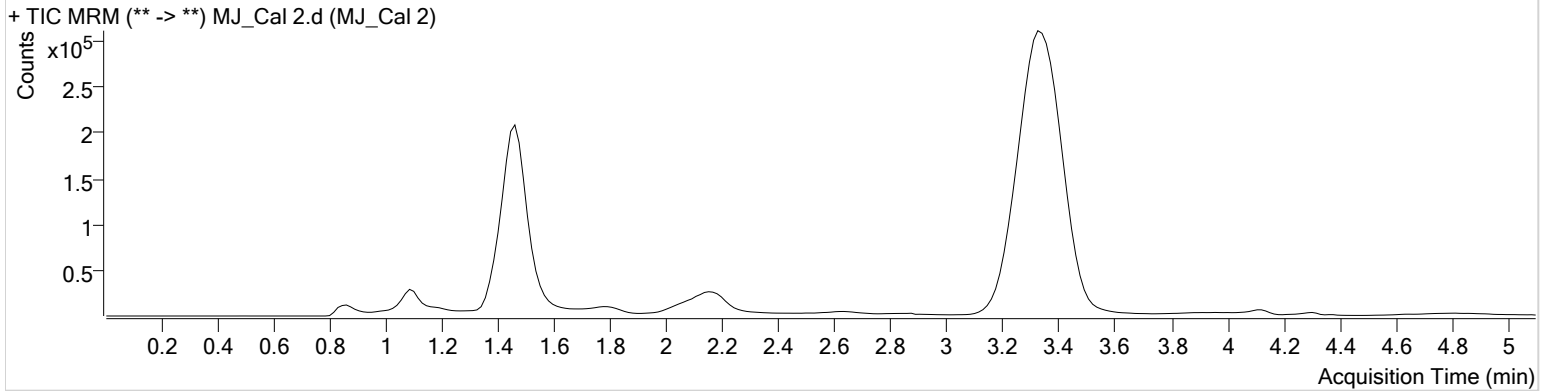
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_Cal 2.d
Type	Cal	Sample	MJ_Cal 2
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-C6	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 10:42:51 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.513	99602	∞	7.1 Low	27.91	898513	2.9002 ng/ml Low
THC-COOH	1.504	80288	∞	44.7	286.25	255816	10.7359 ng/ml
THC	3.360	73601	∞	28.8	14.81	3346112	3.1296 ng/ml

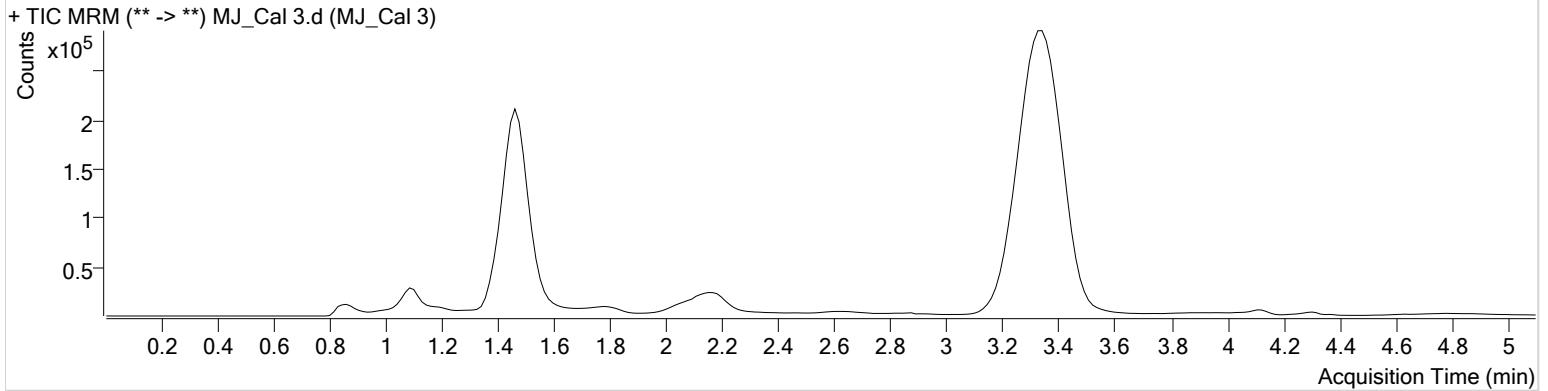
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_Cal 3.d
Type	Cal	Sample	MJ_Cal 3
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-D6	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 10:50:26 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.483	117488	45.89	9.3	∞	832181	5.0700 ng/ml
THC-COOH	1.504	123238	∞	52.8	337.12	248277	18.0680 ng/ml
THC	3.345	111069	1016.77	27.0	405.33	3042401	4.9581 ng/ml

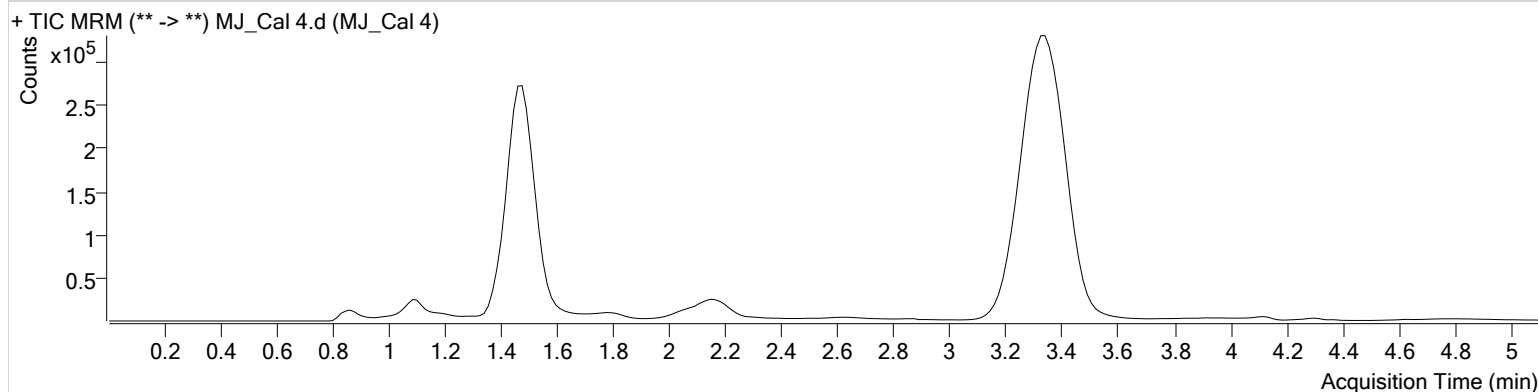


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_Cal 4.d
Type	Cal	Sample	MJ_Cal 4
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-E6	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 10:58:00 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	187015	∞	10.1	290.25	898341	9.8634 ng/ml
THC-COOH	1.504	341623	948.42	53.5	1080.87	256895	51.5479 ng/ml
THC	3.360	253251	2129.54	26.6	141.99	3327312	9.9489 ng/ml

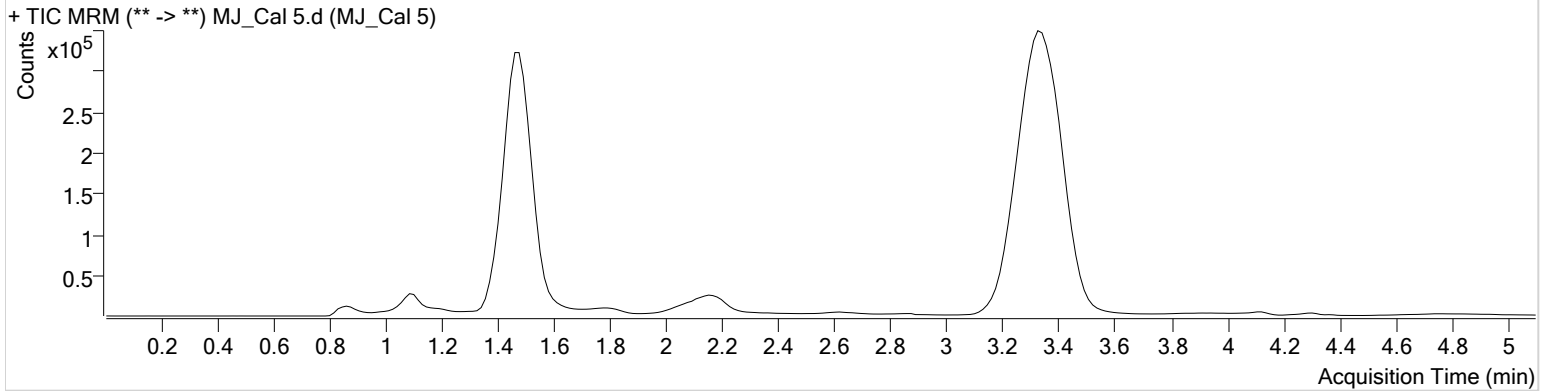
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_Cal 5.d
Type	Cal	Sample	MJ_Cal 5
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-F6	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 11:05:34 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	359059	∞	11.6	9226.08	857534	24.9258 ng/ml
THC-COOH	1.504	465256	1027.77	55.4	1347.97	243807	74.7862 ng/ml
THC	3.345	588957	∞	27.8	378.74	3127953	24.0843 ng/ml

8

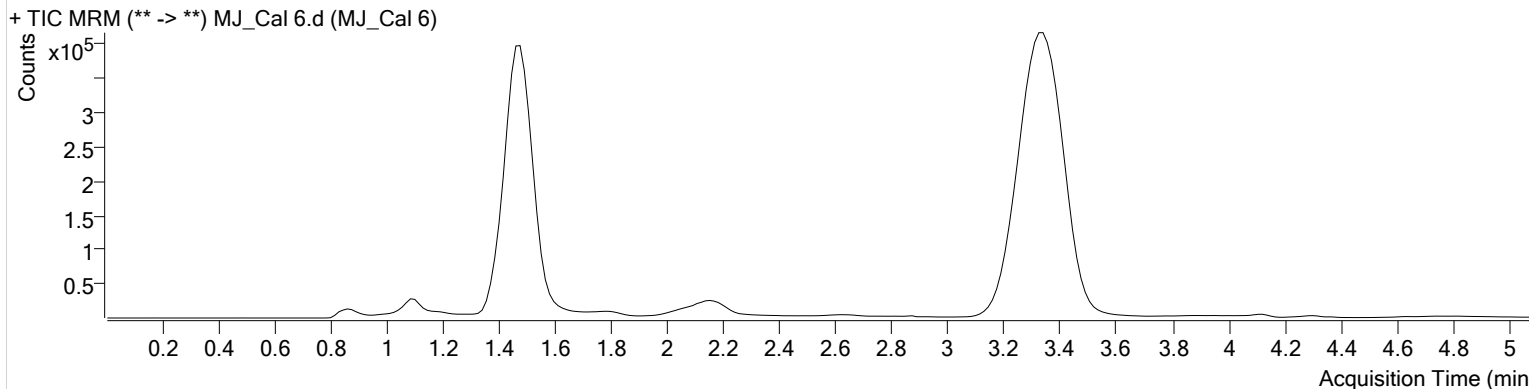


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_Cal 6.d
Type	Cal	Sample	MJ_Cal 6
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-G6	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 11:13:08 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	645429	∞	12.2	867.18	837263	50.1216 ng/ml
THC-COOH	1.504	591182	3495.89	56.6	3554.17	234866	99.2423 ng/ml
THC	3.345	1225303	5275.78	25.8	204.85	3122090	49.8125 ng/ml

AM #27 Cannabinoid Quant. Results

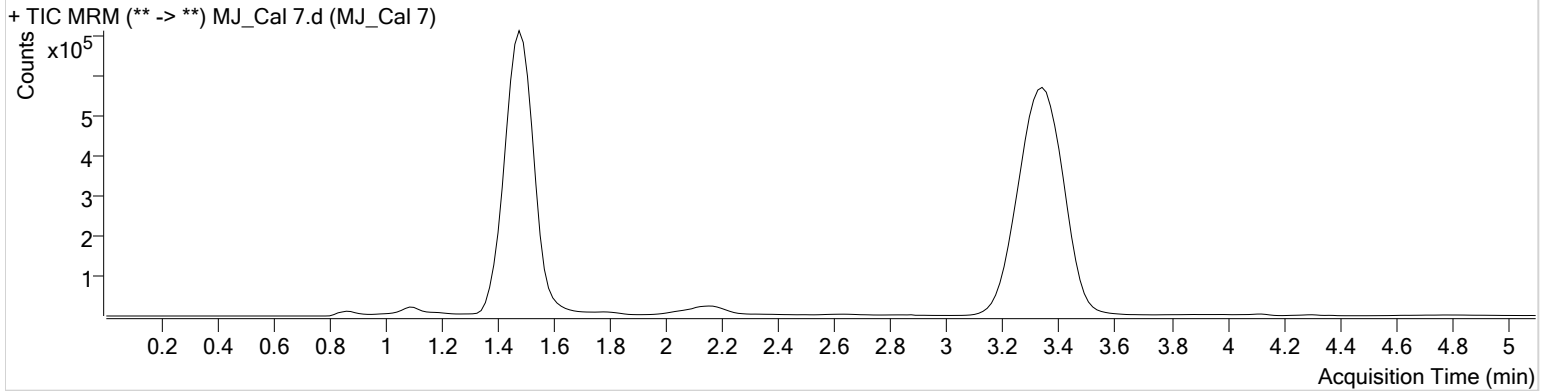


Batch results D:\MassHunter\Data\2020\AM 27-28\031920 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/20/2020 10:03:29 AM

Instrument	Falco	Data File	MJ_Cal 7.d
Type	Cal	Sample	MJ_Cal 7
Acq. Method	AM 27 THC quant.m	Operator	Sarah Pickle
Sample Position	P3-H6	Comment	
Injection Volume	10		
Acq. Date-Time	3/19/2020 11:20:42 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	1268743	∞	12.9	2631.18	864082	100.0192 ng/ml
THC-COOH	1.504	1429056	1705.78	58.3	∞	227360	250.6197 ng/ml
THC	3.345	2573238	7481.23	25.8	∞	3219750	101.0666 ng/ml