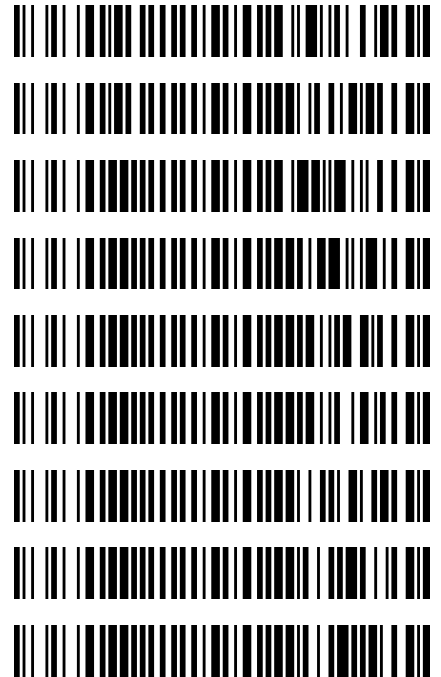


Worklist: 4078

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>
M2020-0645	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2020-0798	2	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0497	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0667	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0673	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0674	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0749	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0755	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0755	2	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: 03/05/20
 Plate lot#: IDP-108-190716

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

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

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\030520 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 3-100*

Due to differences in the AM 26 (worklist 3998) screen and AM 27 (worklist 4020) confirmation results, M2020-0260 was re-extracted and ran with this worklist.



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 μL of 1mg/mL THC, 100 μL of 100 $\mu\text{g/mL}$ THC-OH, C-THC in 9790 μL MeOH
Approximate concentration 1 $\mu\text{g/mL}$.

Component	Source	Source Lot Number	Expiration Date
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: 021320)

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

Component	Source	Source Lot Number
Negative Blood	Hemostat	445283-3
Methanol External Control Solution	-	WS011620
Prepared:	02/13/2020	
Prepared by:	Celena Shrum	
Expires:	09/30/2020	

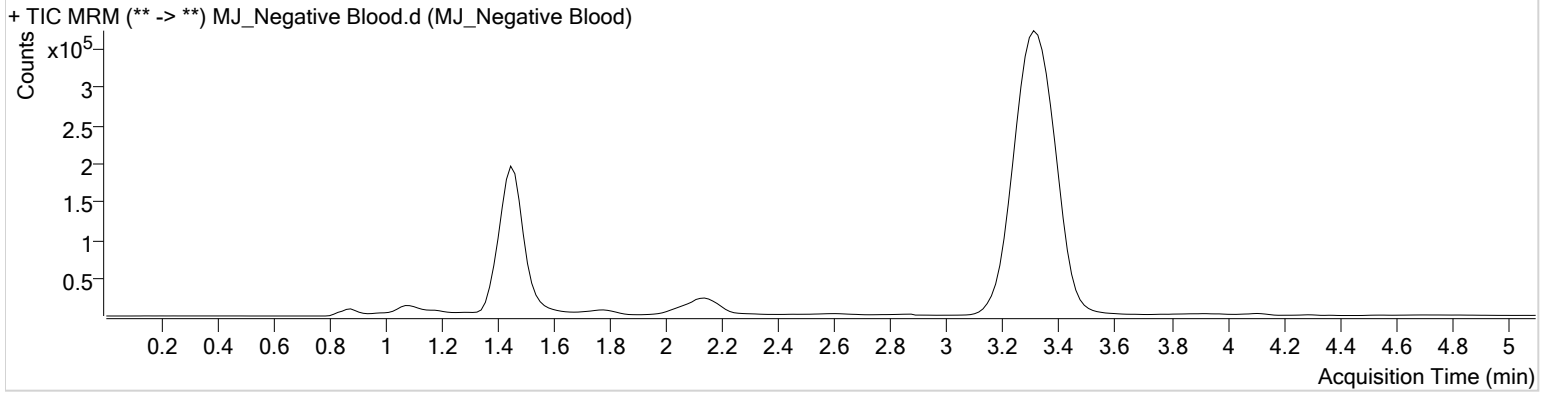
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/5/2020 4:29:06 PM

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	3/5/2020 1:05:47 PM		
Sample Info.			

Sample Chromatogram



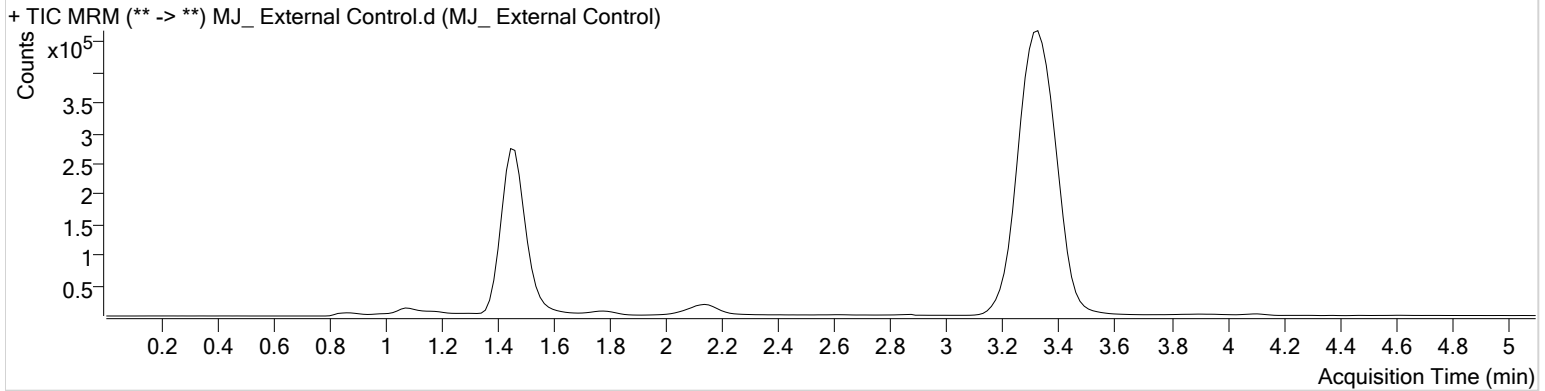


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2020 11:34:20 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	3/5/2020 1:20:58 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	274882	∞	11.1	∞	904757	17.9540 ng/ml
THC-COOH	1.489	135939	220.31	47.6	743.02	258773	19.9343 ng/ml
THC	3.330	493569	3273.44	25.6	321.32	3853743	16.3356 ng/ml

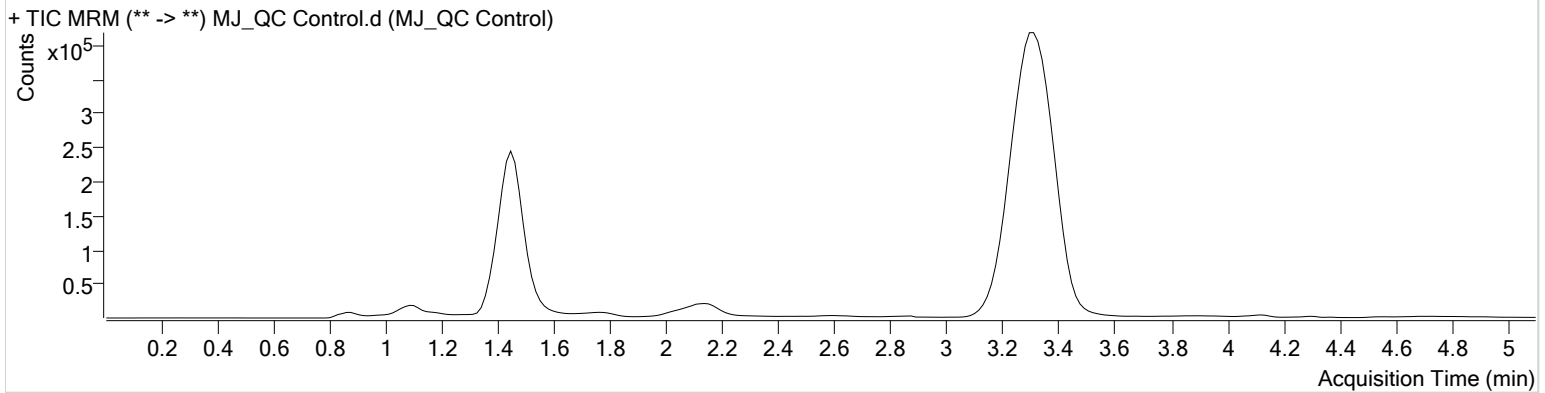
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2020 12:45:19 PM

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	3/5/2020 12:50:34 PM		
Sample Info.			

Sample Chromatogram

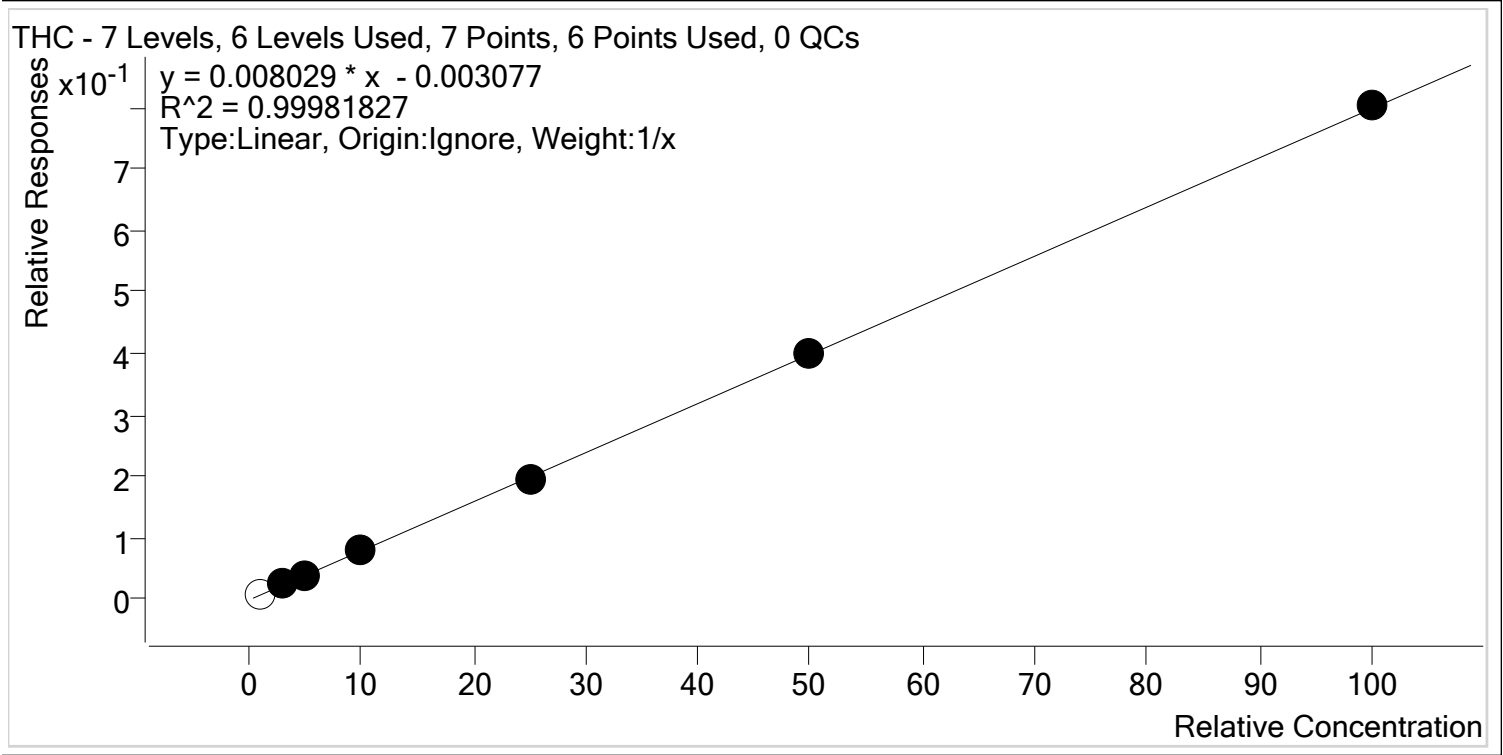


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	115278	∞	8.9	56.58	1017614	3.9395 ng/ml
THC-COOH	1.489	109682	∞	49.0	822.27	279286	14.4508 ng/ml
THC	3.330	140674	237.01	28.1	168.09	4356377	4.4053 ng/ml



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Last Cal. Update 3/5/2020 4:29 PM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-D3

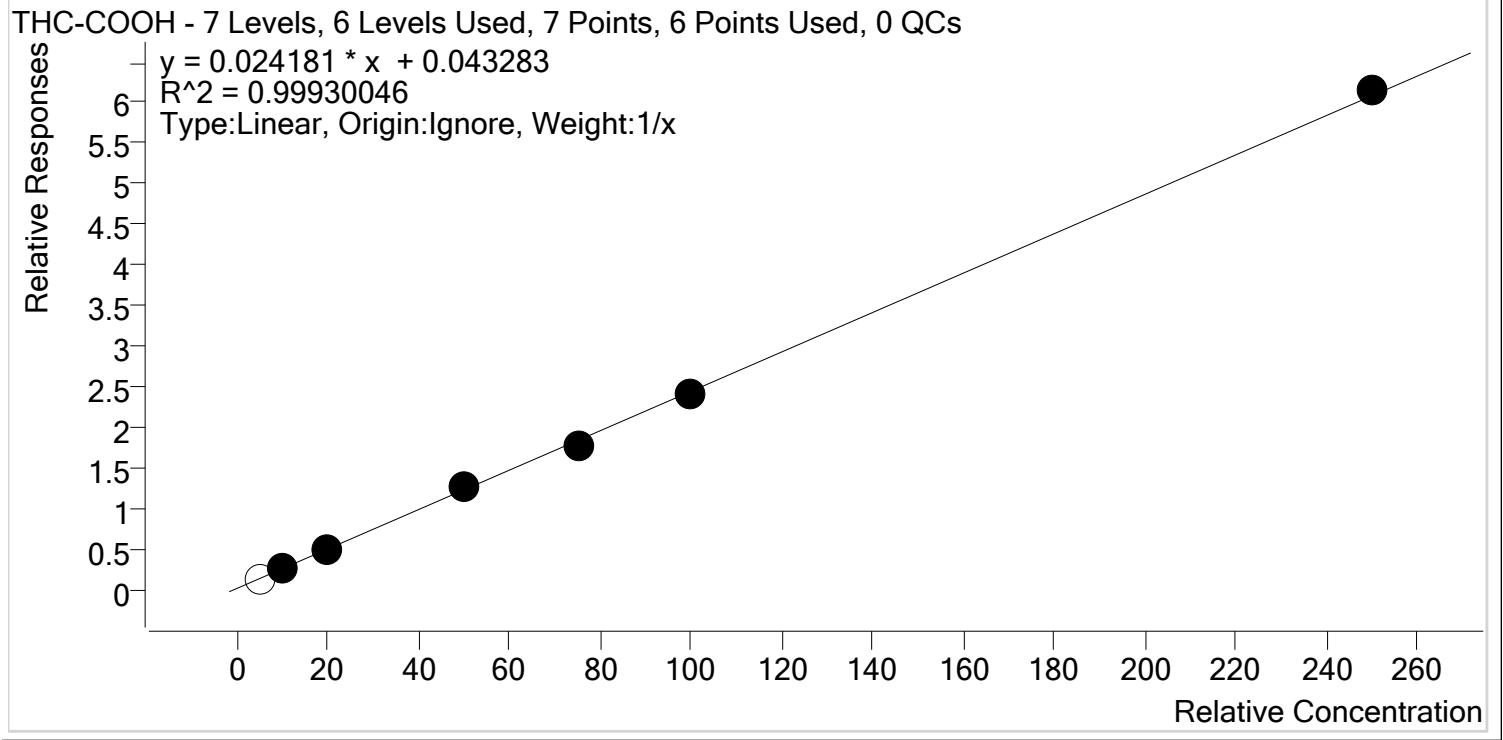


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	x	1.0	1.3	130.1
MJ_Cal 2	2	✓	3.0	3.1	102.8
MJ_Cal 3	3	✓	5.0	5.0	99.0
MJ_Cal 4	4	✓	10.0	10.0	100.2
MJ_Cal 5	5	✓	25.0	24.3	97.2
MJ_Cal 6	6	✓	50.0	50.0	100.1
MJ_Cal 7	7	✓	100.0	100.6	100.6



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Last Cal. Update 3/5/2020 4:29 PM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9

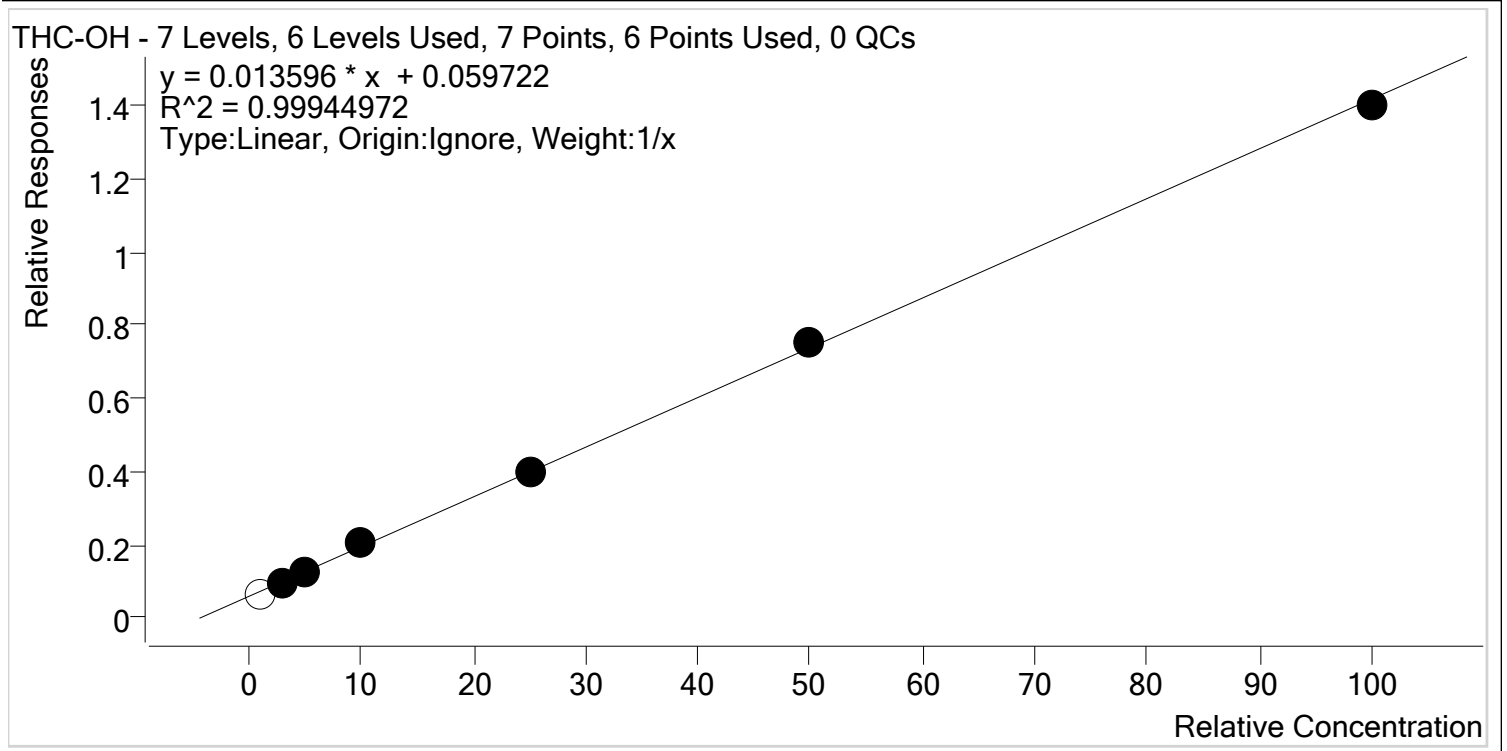


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	x	5.0	4.8	96.7
MJ_Cal 2	2	✓	10.0	10.6	106.3
MJ_Cal 3	3	✓	20.0	18.8	94.2
MJ_Cal 4	4	✓	50.0	51.2	102.4
MJ_Cal 5	5	✓	75.0	73.2	97.6
MJ_Cal 6	6	✓	100.0	98.6	98.6
MJ_Cal 7	7	✓	250.0	252.6	101.0



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Last Cal. Update 3/5/2020 4:29 PM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	x	1.0	0.5	54.2
MJ_Cal 2	2	✓	3.0	2.9	96.0
MJ_Cal 3	3	✓	5.0	4.9	97.9
MJ_Cal 4	4	✓	10.0	10.5	105.5
MJ_Cal 5	5	✓	25.0	25.0	99.8
MJ_Cal 6	6	✓	50.0	51.1	102.1
MJ_Cal 7	7	✓	100.0	98.6	98.6

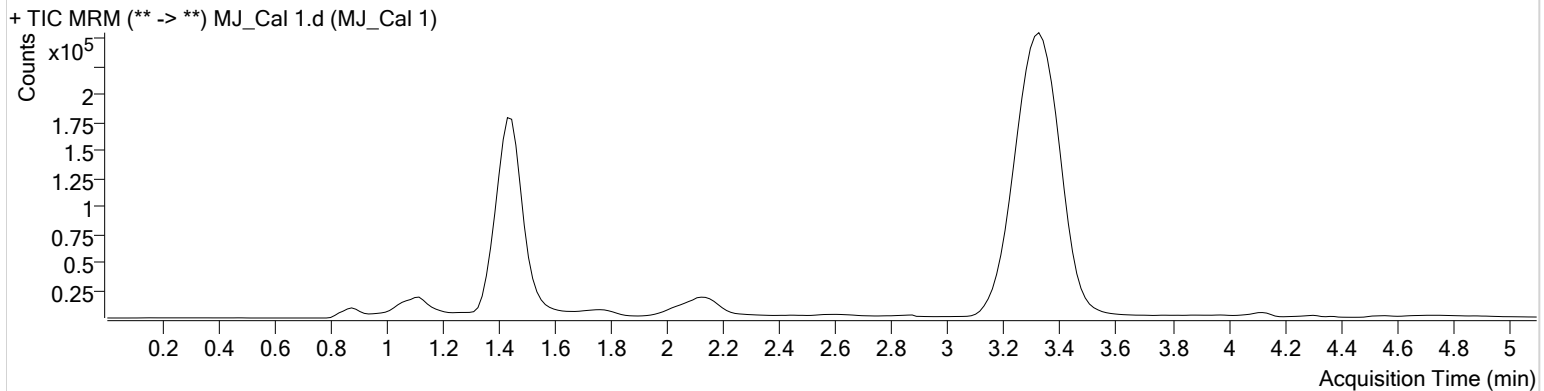
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/5/2020 4:29:06 PM

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	3/5/2020 11:49:40 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.498	58266	∞	5.6 Low	19.46	868498	0.5418 ng/ml Low
THC-COOH	1.474	34312	66.18	42.4 Low	87.94	214161	4.8357 ng/ml Low
THC	3.330	20833	66.33	31.2	6.69 Low	2827031	1.3012 ng/ml Low

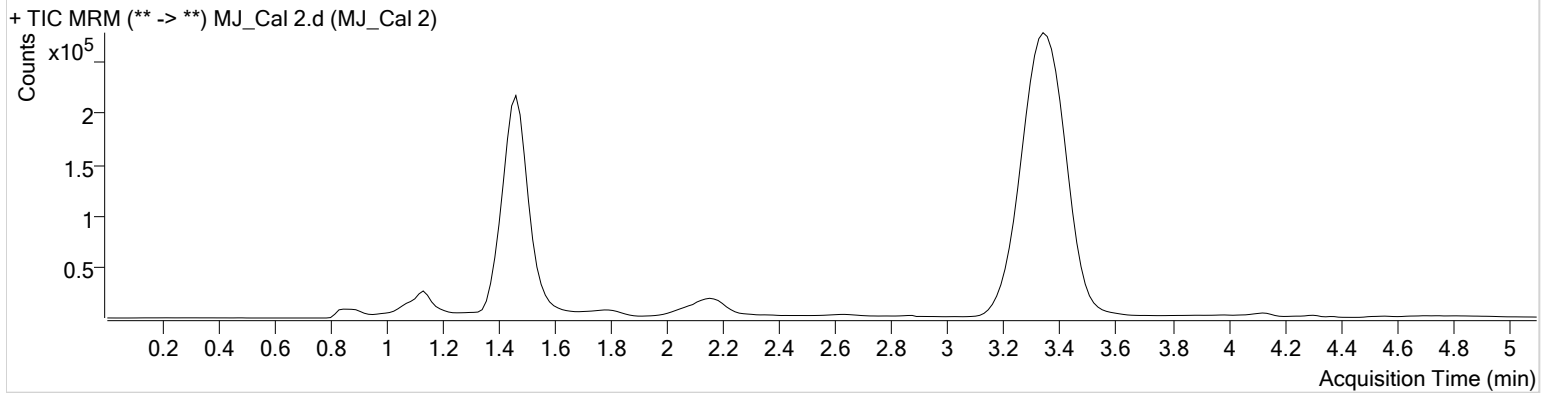
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/5/2020 4:29:06 PM

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	3/5/2020 11:57:24 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.498	98217	∞	9.0	54.30	993396	2.8795 ng/ml Low
THC-COOH	1.504	68619	112.27	44.4	∞	228548	10.6263 ng/ml
THC	3.375	65258	145.36	29.4	∞	3008915	3.0846 ng/ml

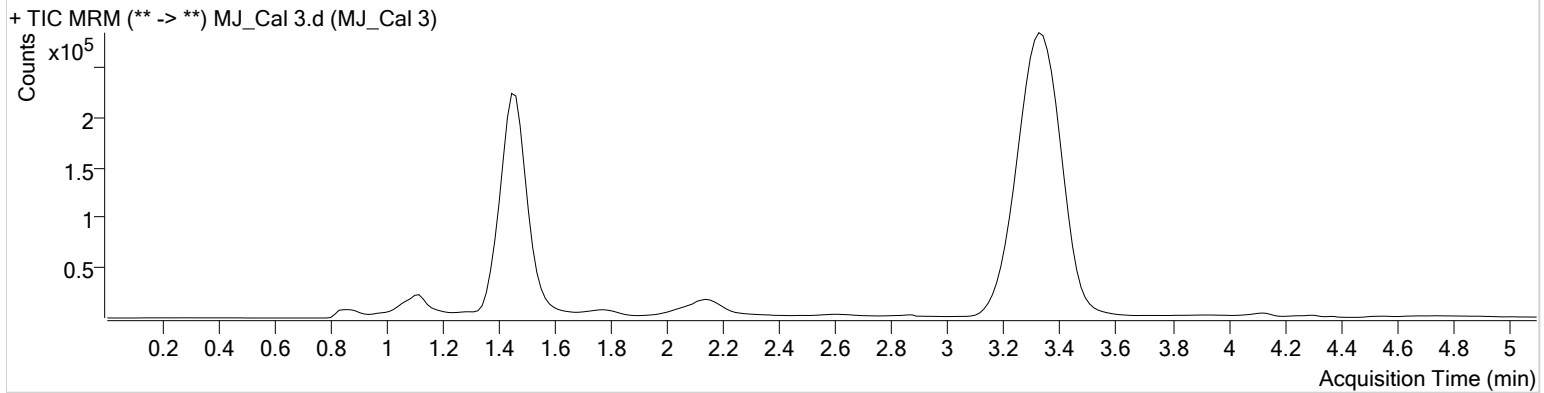
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/5/2020 4:29:06 PM

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	3/5/2020 12:05:00 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	119092	∞	9.4	∞	943011	4.8962 ng/ml
THC-COOH	1.489	112552	282.84	52.5	448.04	225666	18.8358 ng/ml
THC	3.345	109449	101.92	28.3	∞	2983560	4.9525 ng/ml

AM #27 Cannabinoid Quant. Results

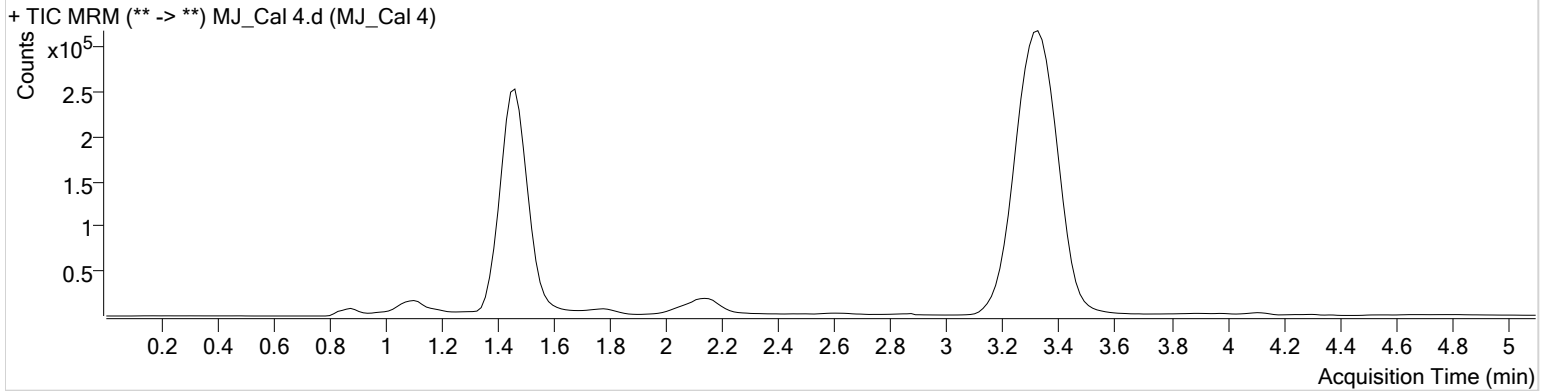


Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/5/2020 4:29:06 PM

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	3/5/2020 12:12:37 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	172269	∞	10.7	164.80	848202	10.5458 ng/ml
THC-COOH	1.489	272143	∞	54.6	1217.62	212464	51.1805 ng/ml
THC	3.345	237085	1131.94	27.3	∞	3063239	10.0234 ng/ml

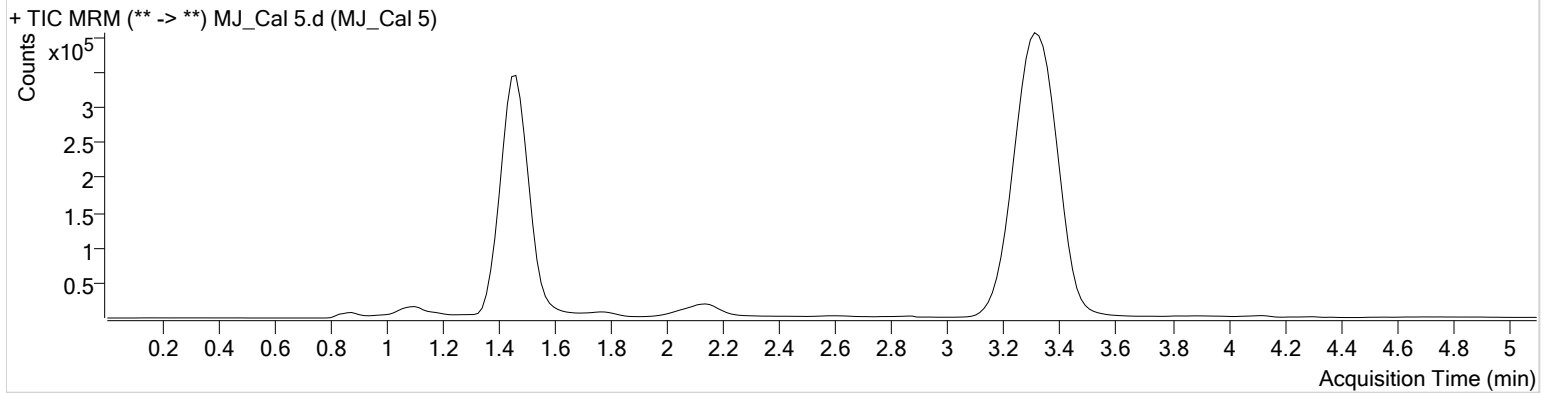
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/5/2020 4:29:06 PM

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	3/5/2020 12:20:14 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	383212	∞	11.6	350.17	960179	24.9625 ng/ml
THC-COOH	1.489	439794	∞	57.5	1611.51	242594	73.1807 ng/ml
THC	3.330	677395	1018.03	25.9	∞	3527022	24.3050 ng/ml

\$

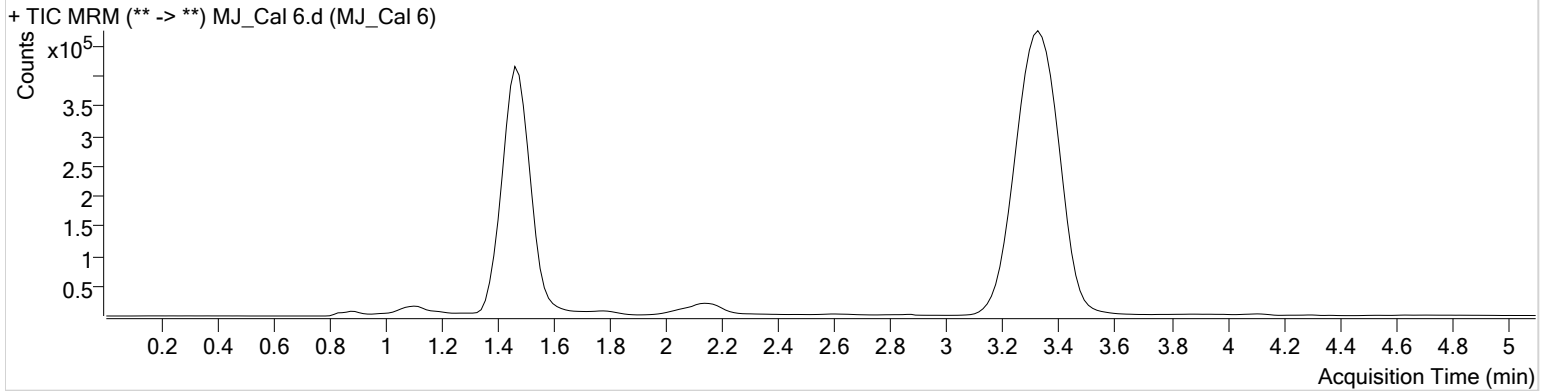


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/5/2020 4:29:06 PM

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	3/5/2020 12:27:48 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	674139	∞	12.6	3336.93	894015	51.0702 ng/ml
THC-COOH	1.489	553928	649.64	57.9	1428.89	228214	98.5865 ng/ml
THC	3.330	1373520	17612.09	25.9	535.88	3445190	50.0403 ng/ml

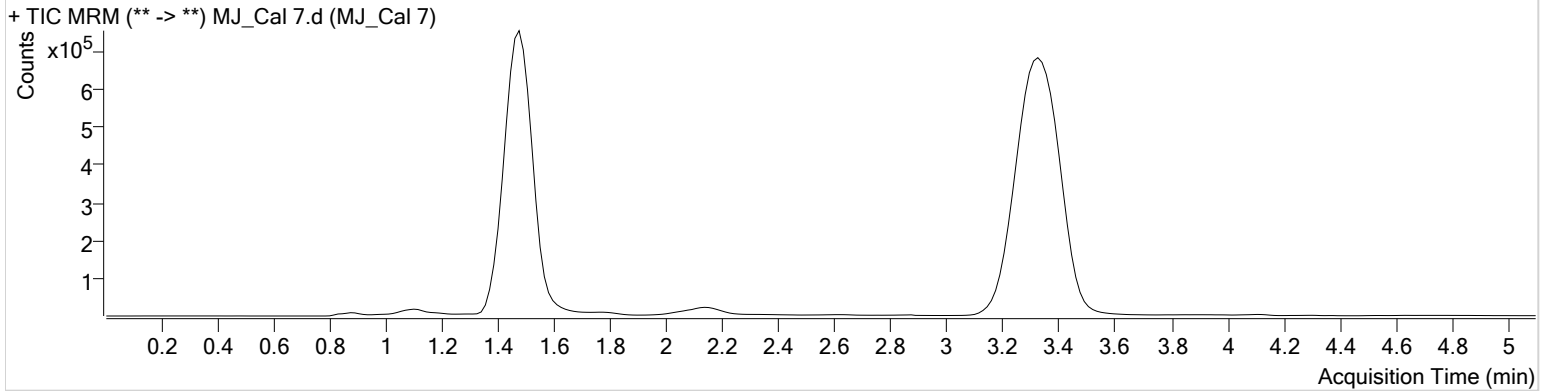
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\030520 AM 27 28 SP\QuantResults\AM 27.batch.bin
Calibration Last Update 3/5/2020 4:29:06 PM

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	3/5/2020 12:35:22 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.453	1299127	∞	13.1	∞	927362	98.6459 ng/ml
THC-COOH	1.489	1432053	3501.26	58.6	4852.50	232808	252.5902 ng/ml
THC	3.345	2996344	16704.75	26.2	2338.07	3724227	100.5941 ng/ml