






REVIEWED
By Tamara Salazar at 10:09 am, Nov 12, 2020

11/4/2020

CS

Worklist: 4591

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
P2020-2673	1	CBUK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-2770	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-2787	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-2904	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2020-3104	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	

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

Extraction Date: 11/03/2020

Analyst: Celena Shrum

Plate lot#: IDP-108-2-200723

Plate Expiration: 01/23/2021

Mobile phase A: 0.1% Formic Acid in LCMS Water

Mobile phase B: 0.1% Formic acid in Acetonitrile

Blank Blood Lot: 445283-4

Column: UCT Selectra DA 100 x 2.1mm 3um

LCMS-QQQ ID: 069901

Blank Urine Lot: POC031319

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: #42**
- 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 **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)** Manifold ID: 067104
- 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. **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 r² 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: 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: THC-OH not evaluated. Calibrator 7 did not inject properly and had to be reinjected.



Idaho State Police Forensic Services

AM #26 Screening of THC and Metabolites and AM #27 Confirmation of THC and Metabolites Urine External Control Prep Sheet

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 1ug/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		

Urine External Control Solution (Lot: 110220)

200 ul of methanol external control solution was added to 9800 ul of urine.

Approximately 20ng/mL each

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Negative Urine	Pocatello Lab	POC031319
Methanol External Control Solution	-	WS011620
Prepared:	11/02/2020	
Prepared by:	Celena Shrum	

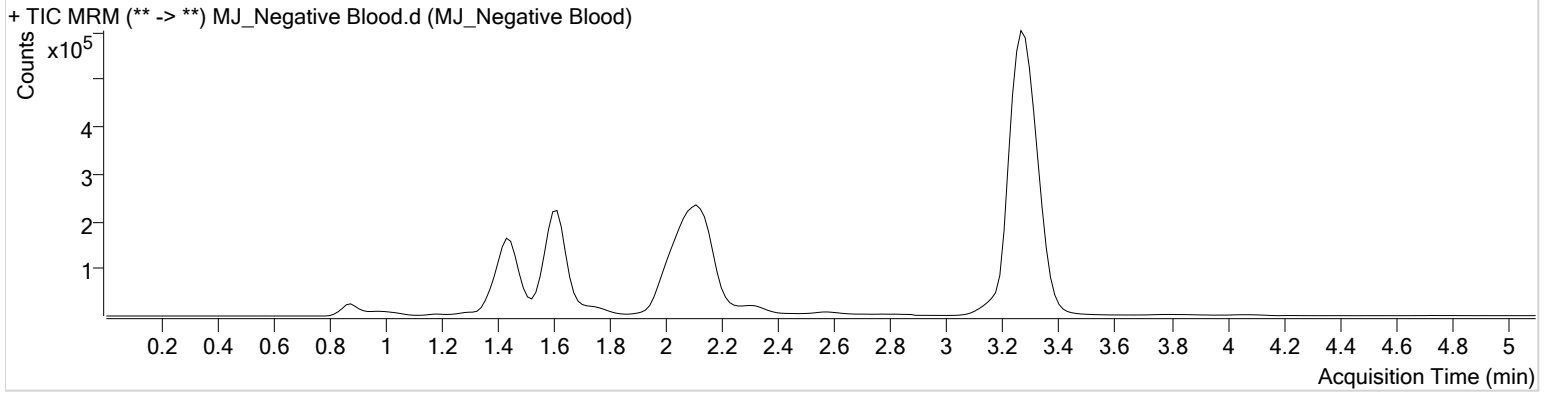
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_Negative Blood.d
Type	Sample	Sample	MJ_Negative Blood
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-H5	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 4:16:56 PM		
Sample Info.			

Sample Chromatogram



CS

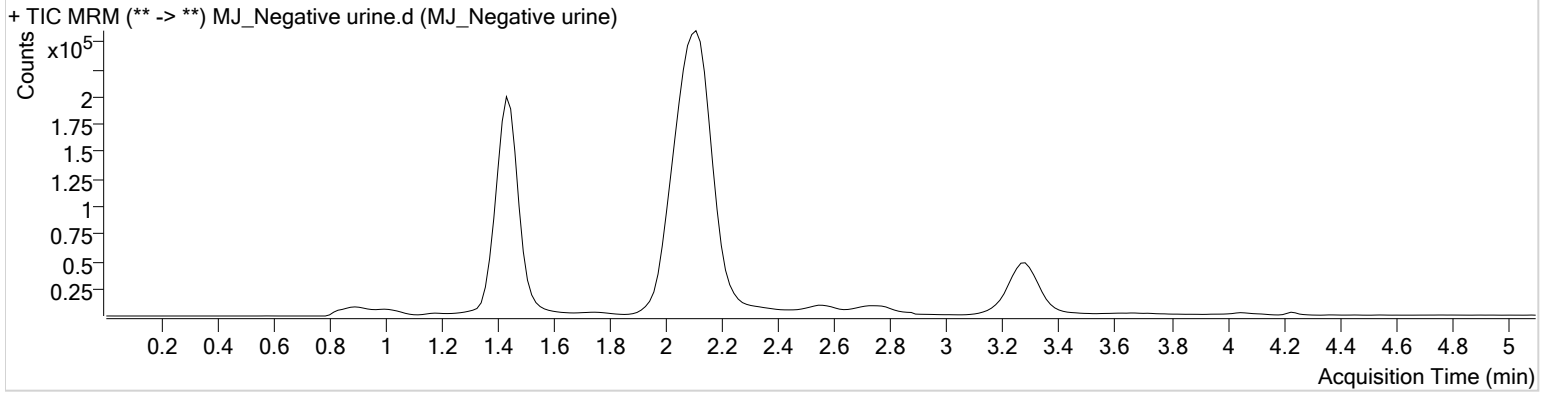


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_Negative urine.d
Type	Sample	Sample	MJ_Negative urine
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-G5	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 4:32:08 PM		
Sample Info.			

Sample Chromatogram



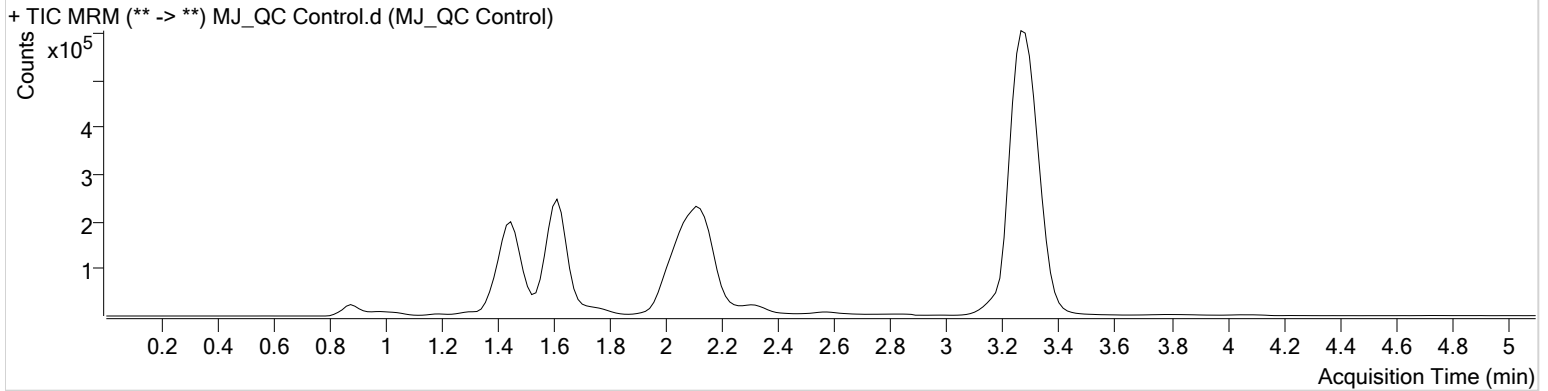
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_QC Control.d
Type	Sample	Sample	MJ_QC Control
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-A6	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 4:01:43 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	90388	∞	52.2	∞	170871	15.2483 ng/ml
THC-OH	1.483	121425	∞	8.7	∞	725344	4.1291 ng/ml
THC	3.285	186102	1170.65	27.5	∞	4172577	4.5410 ng/ml

AM #27 Cannabinoid Quant. Results

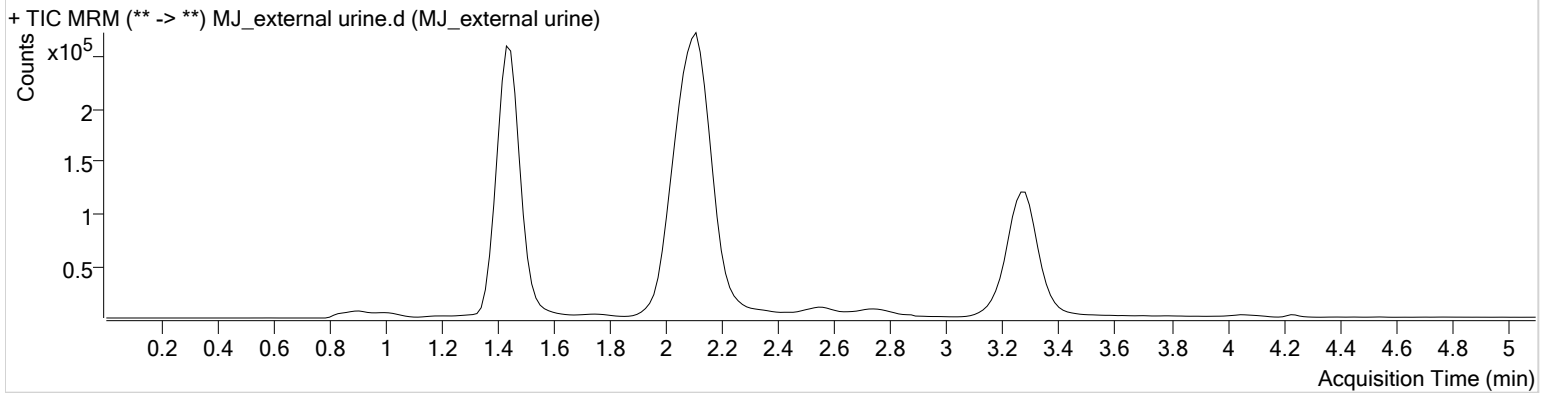


Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_external urine.d
Type	Sample	Sample	MJ_external urine
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-F5	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 4:47:18 PM		

Sample Info.

Sample Chromatogram

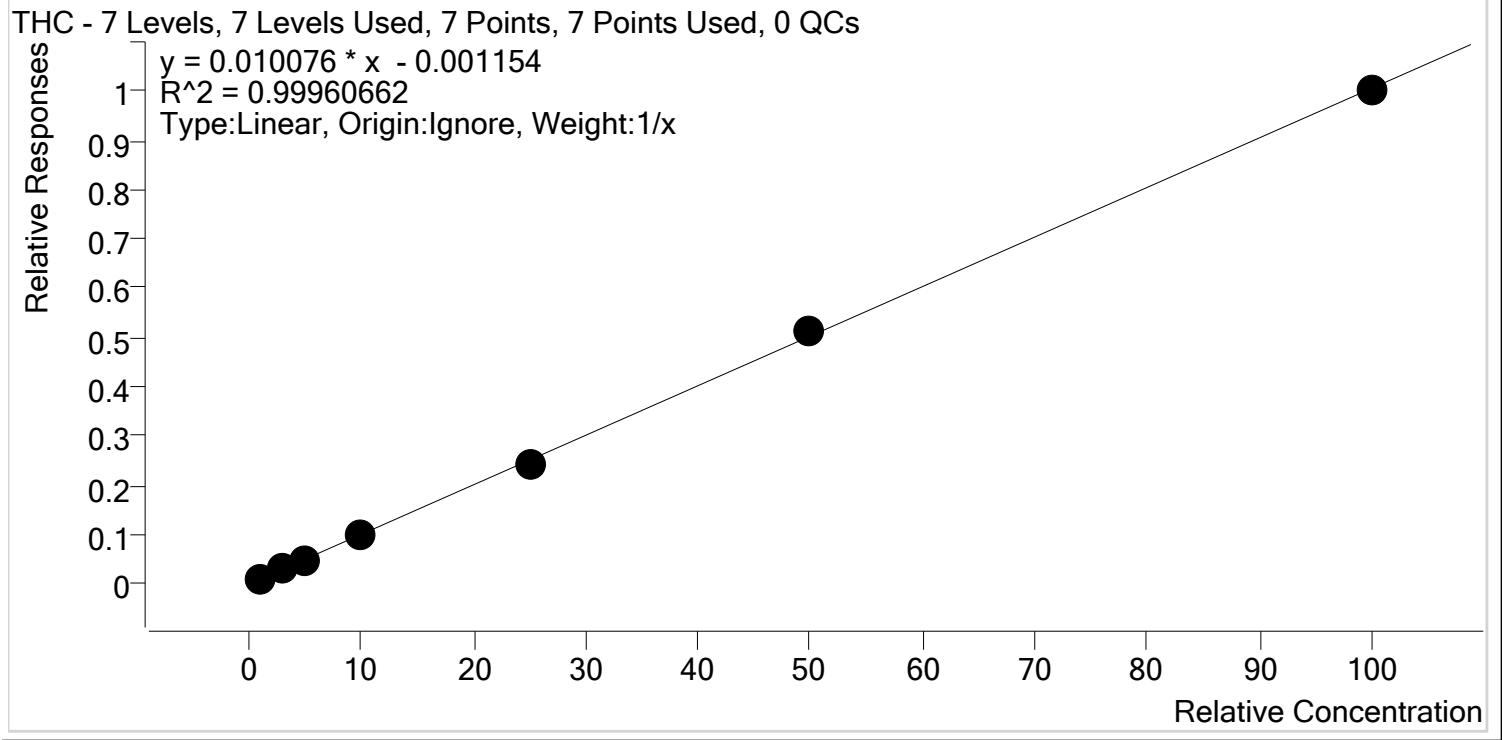


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	73902	∞	67.6	∞	225874	8.0339 ng/ml
THC-OH	1.438	118460	419.88	14.0 High	927.55	1001875	1.3414 ng/ml Low
THC	3.285	30297	112.15	32.9	26.09	945048	3.2962 ng/ml



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Last Cal. Update 11/3/2020 11:43 AM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-D3

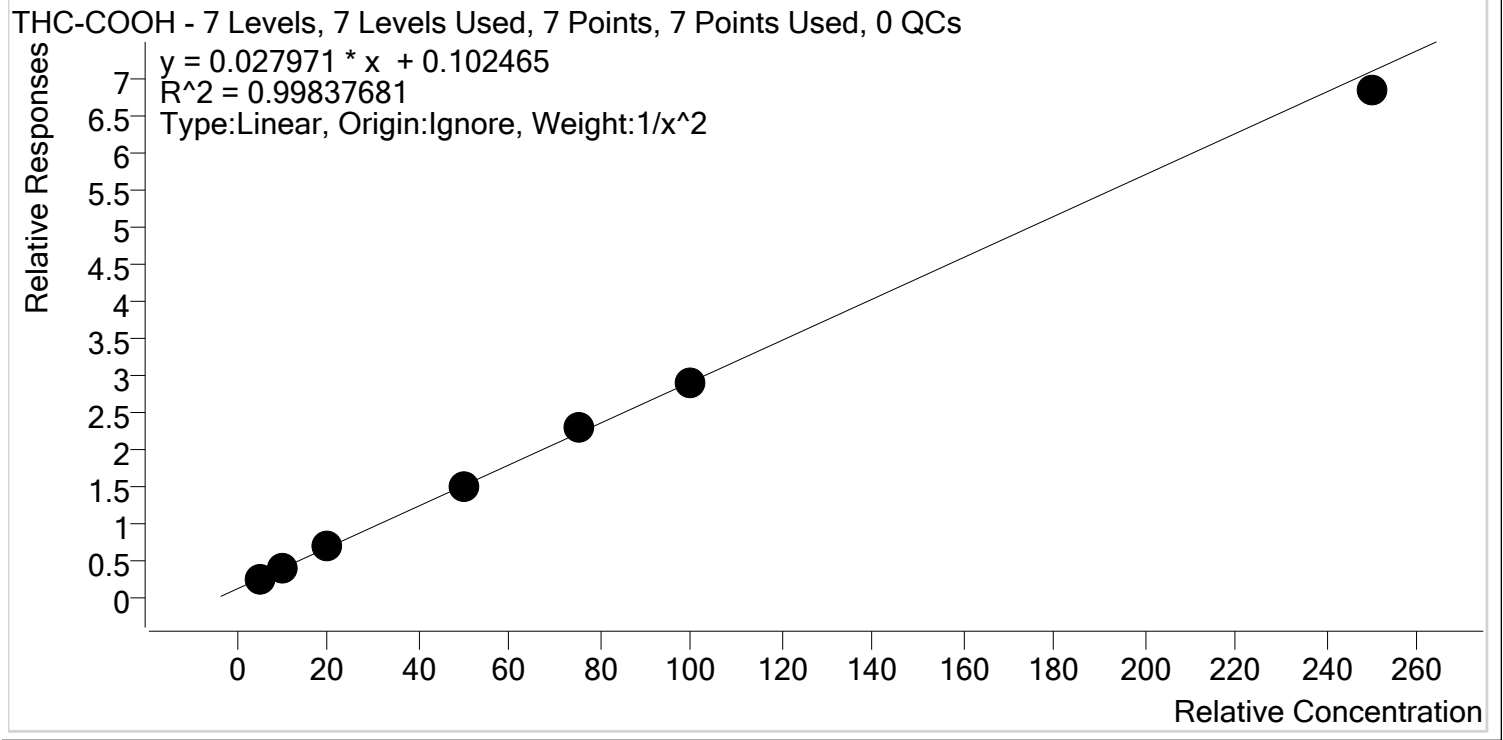


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	1.0	1.1	106.9
MJ_Cal 2	2	✓	3.0	2.9	97.9
MJ_Cal 3	3	✓	5.0	4.8	96.3
MJ_Cal 4	4	✓	10.0	9.9	98.8
MJ_Cal 5	5	✓	25.0	24.5	97.8
MJ_Cal 6	6	✓	50.0	51.3	102.7
MJ_Cal 7r	7	✓	100.0	99.5	99.5



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Last Cal. Update 11/3/2020 11:43 AM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9

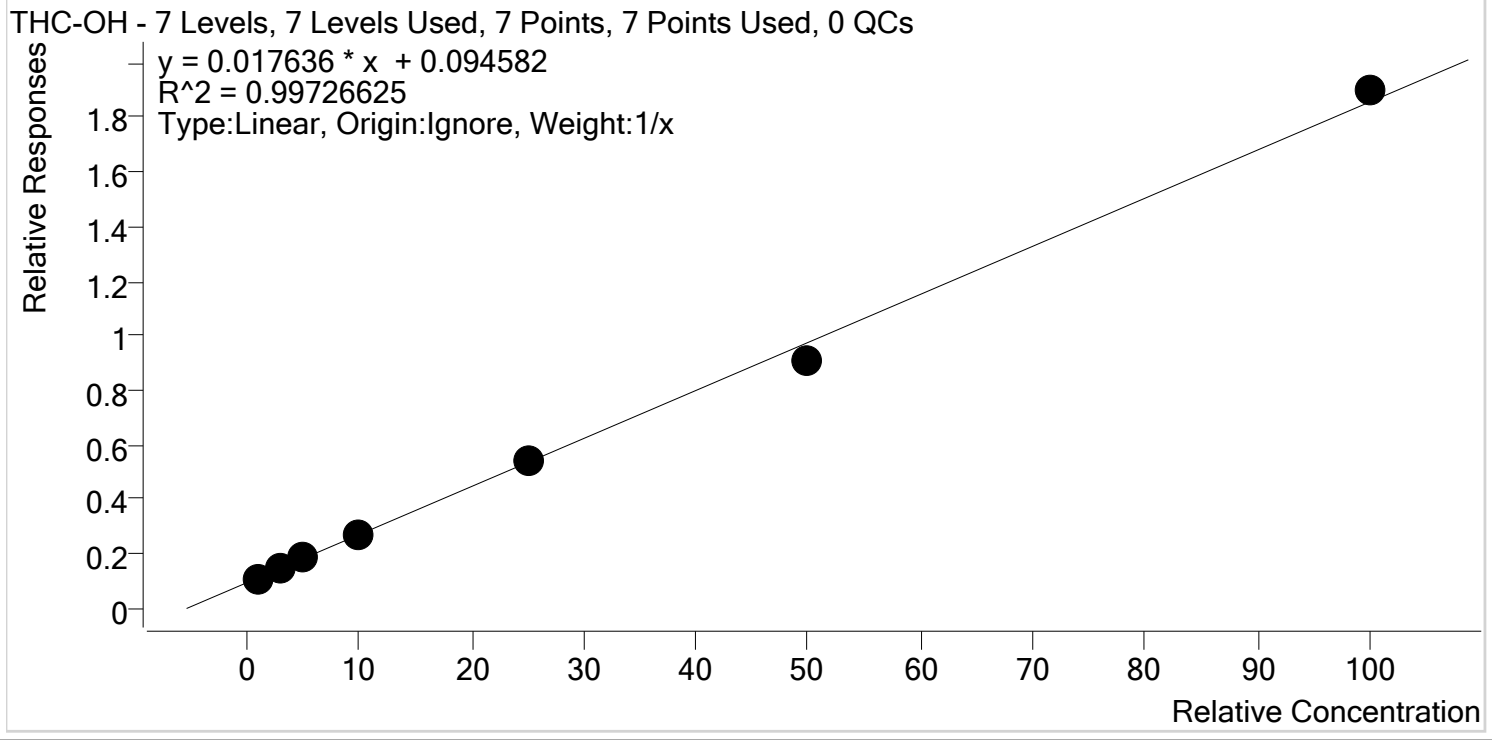


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	5.0	4.9	97.6
MJ_Cal 2	2	✓	10.0	10.5	104.7
MJ_Cal 3	3	✓	20.0	20.1	100.5
MJ_Cal 4	4	✓	50.0	48.5	96.9
MJ_Cal 5	5	✓	75.0	77.8	103.7
MJ_Cal 6	6	✓	100.0	100.3	100.3
MJ_Cal 7r	7	✓	250.0	240.6	96.2



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Last Cal. Update 11/3/2020 11:43 AM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	1.0	0.8	83.4
MJ_Cal 2	2	✓	3.0	3.1	102.7
MJ_Cal 3	3	✓	5.0	5.7	114.3
MJ_Cal 4	4	✓	10.0	10.3	103.4
MJ_Cal 5	5	✓	25.0	25.3	101.0
MJ_Cal 6	6	✓	50.0	46.4	92.9
MJ_Cal 7r	7	✓	100.0	102.3	102.3

cg

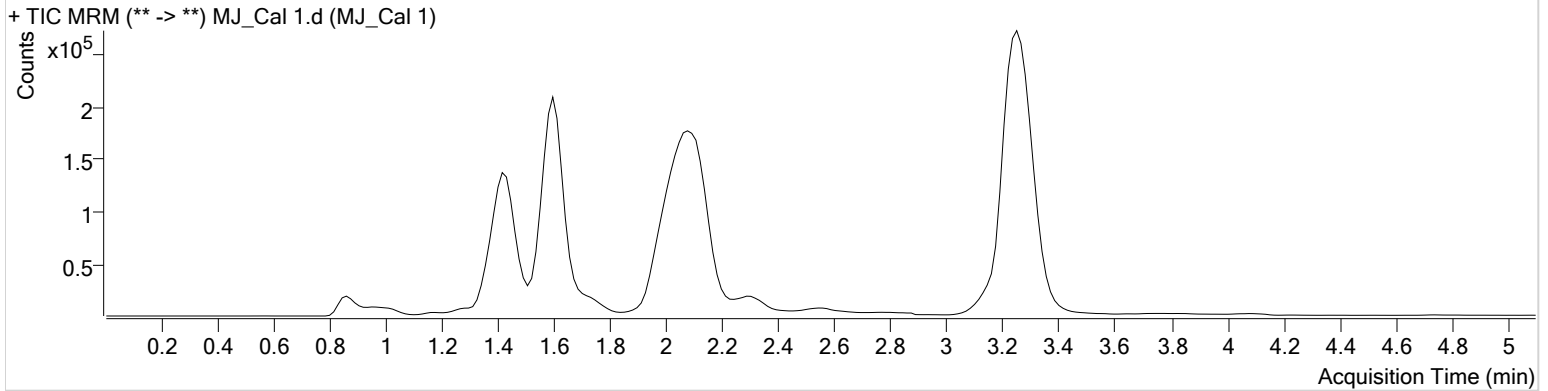


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_Cal 1.d
Type	Cal	Sample	MJ_Cal 1
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-H6	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 3:00:43 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.444	31541	∞	53.9	∞	131961	4.8820 ng/ml Low
THC-OH	1.483	64189	∞	5.2 Low	26.97	587334	0.8339 ng/ml Low
THC	3.254	20033	44.95	32.8	19.73	2082268	1.0693 ng/ml Low

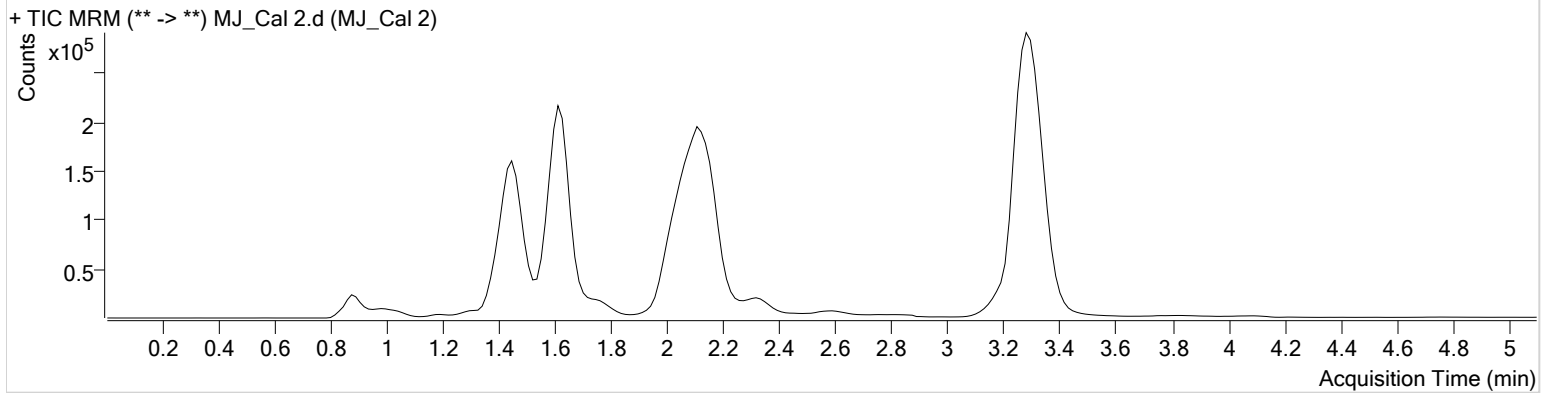
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_Cal 2.d
Type	Cal	Sample	MJ_Cal 2
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-G6	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 3:08:28 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	54276	∞	50.4	∞	137287	10.4709 ng/ml
THC-OH	1.498 High	94821	∞	7.3 Low	∞	636672	3.0818 ng/ml
THC	3.300	59680	209.76	29.3	35.36	2097659	2.9381 ng/ml Low

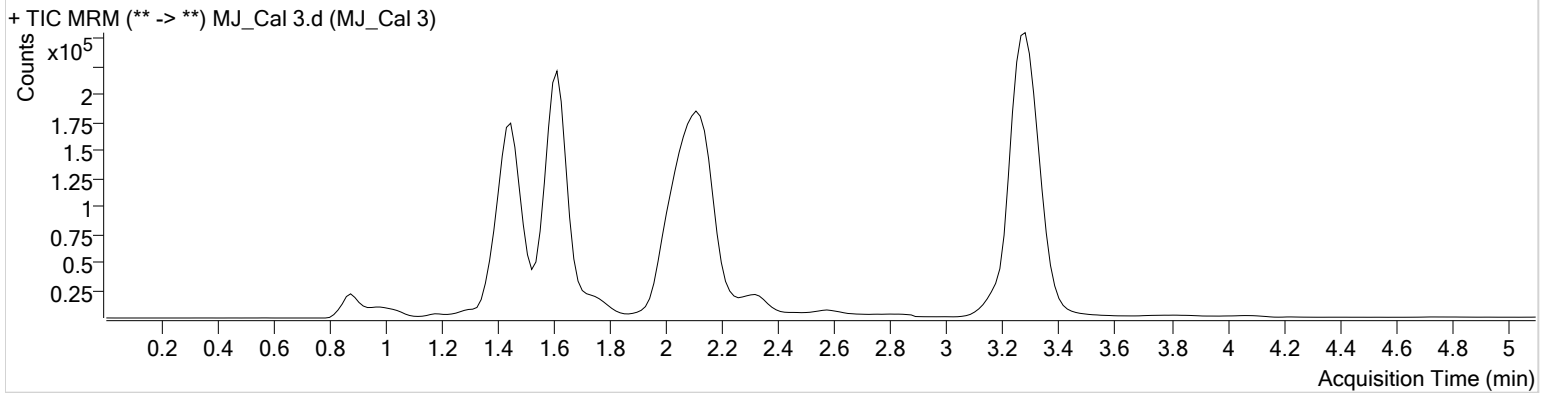
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_Cal 3.d
Type	Cal	Sample	MJ_Cal 3
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-F6	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 3:16:04 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	93701	∞	53.9	∞	140949	20.1034 ng/ml
THC-OH	1.468	123646	∞	8.0	∞	633015	5.7125 ng/ml
THC	3.285	86618	233.88	30.0	∞	1828512	4.8159 ng/ml

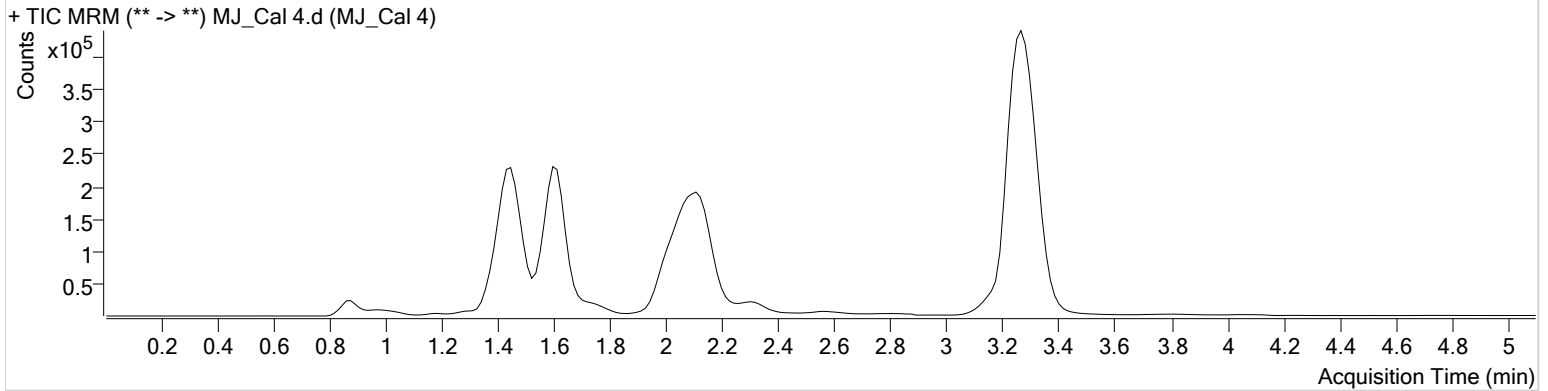


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument Falco **Data File** MJ_Cal 4.d
Type Cal **Sample** MJ_Cal 4
Acq. Method AM 27 THC quant.m **Operator** Celena Shrum
Sample Position P3-E6 **Comment**
Injection Volume 10
Acq. Date-Time 11/2/2020 3:23:41 PM
Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.459	220841	∞	57.5	∞	151487	48.4551 ng/ml
THC-OH	1.453	187210	∞	10.0	∞	675970	10.3405 ng/ml
THC	3.270	290789	2501.54	26.7	46.83	2955347	9.8797 ng/ml

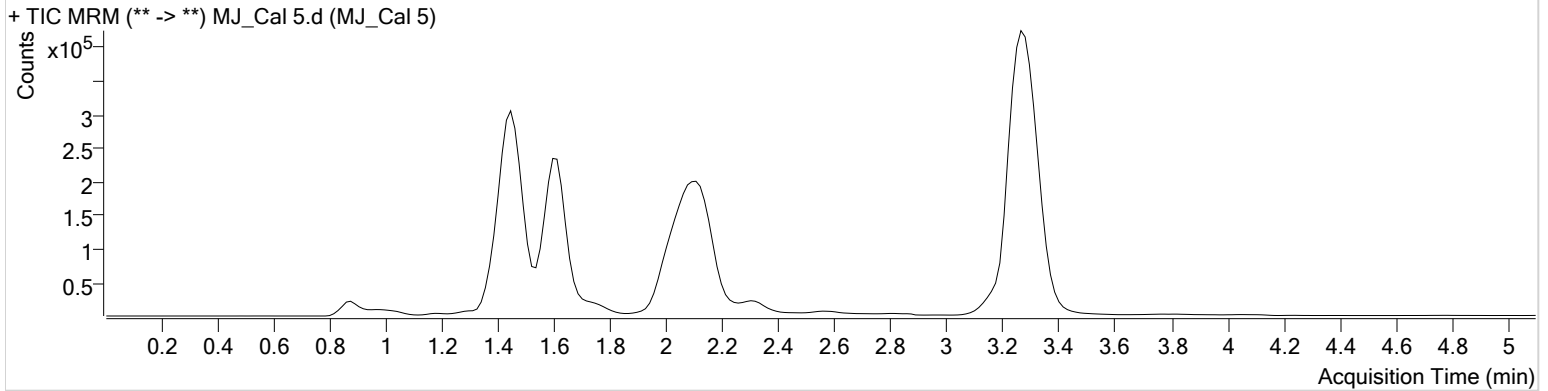
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_Cal 5.d
Type	Cal	Sample	MJ_Cal 5
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-D6	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 3:31:18 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.459	357501	∞	59.6	∞	156910	77.7905 ng/ml
THC-OH	1.438	365340	∞	11.8	∞	676665	25.2509 ng/ml
THC	3.285	597788	758.81	27.0	933.44	2437286	24.4563 ng/ml

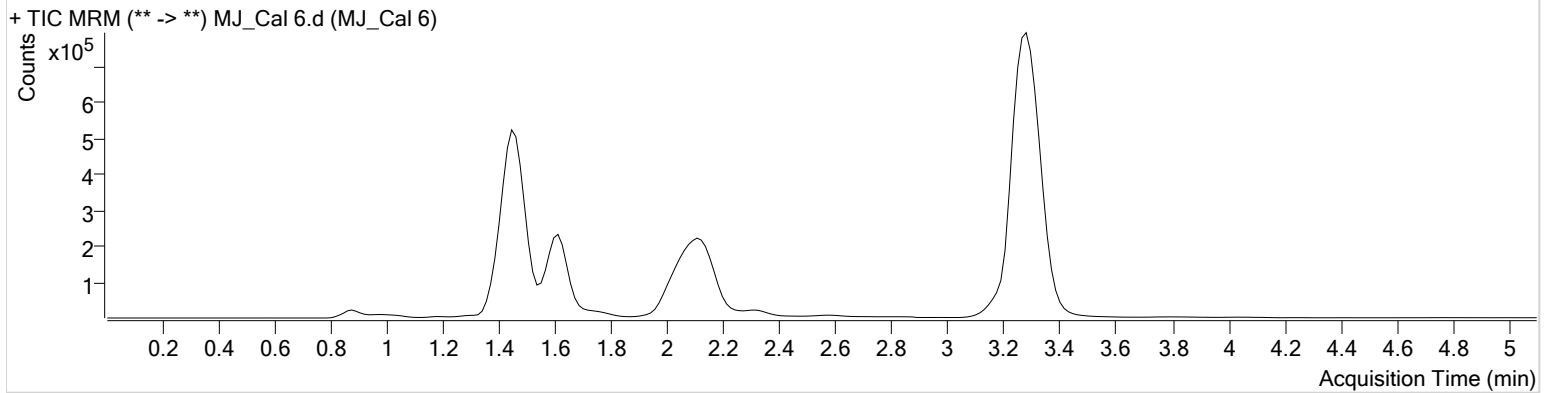
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_Cal 6.d
Type	Cal	Sample	MJ_Cal 6
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-C6	Comment	
Injection Volume	10		
Acq. Date-Time	11/2/2020 3:38:54 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.474	599489	∞	64.5	∞	206237	100.2571 ng/ml
THC-OH	1.438	813224	∞	13.3 High	∞	890054	46.4441 ng/ml
THC	3.285	1826212	27898.68	26.3	2252.17	3537635	51.3476 ng/ml

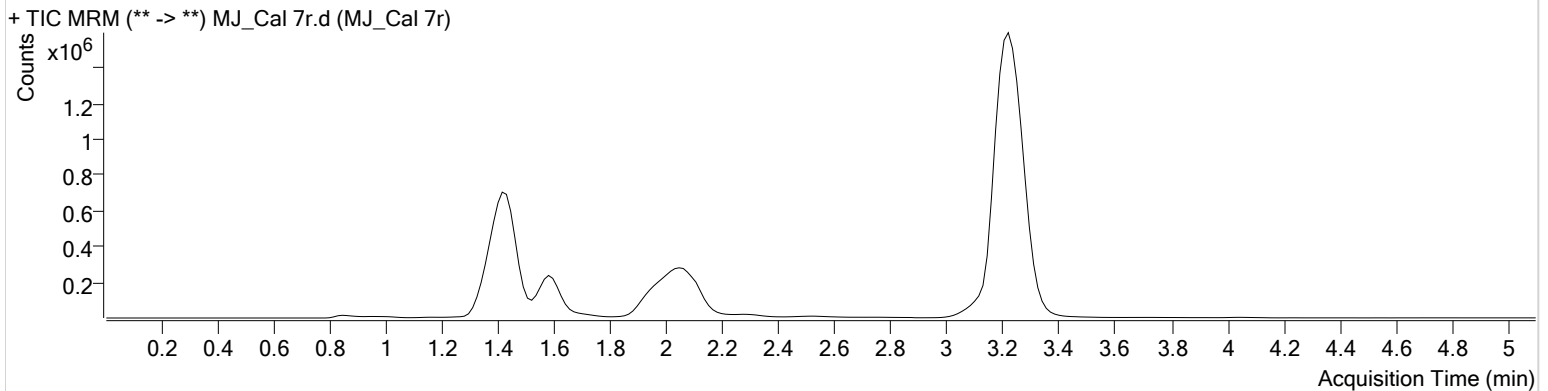
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\AM 27-28 110220 CS\QuantResults\AM 27 CS.batch.bin
Calibration Last Update 11/3/2020 11:43:17 AM

Instrument	Falco	Data File	MJ_Cal 7r.d
Type	Cal	Sample	MJ_Cal 7r
Acq. Method	AM 27 THC quant.m	Operator	Celena Shrum
Sample Position	P3-B6	Comment	
Injection Volume	10		
Acq. Date-Time	11/3/2020 11:18:22 AM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.444	1240348	∞	60.4	∞	181527	240.6166 ng/ml
THC-OH	1.408 Low	1356469	∞	14.0 High	∞	714154	102.3363 ng/ml
THC	3.224	5198880	43811.71	26.0	3291.96	5191933	99.4931 ng/ml