

REVIEWED

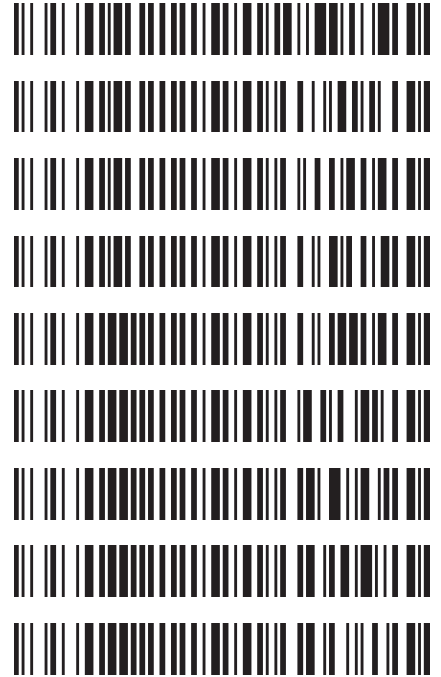
By Anne Nord at 12:36 pm, Mar 08, 2021

3/4/2021

CS

Worklist: 4820

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>
M2021-0297	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2021-0429	4	BCK	AM 27 Blood THC Quant by LC-QQQ
M2021-0436	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2021-0437	2	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0344	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0377	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0402	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0404	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0414	1	BCK	AM 27 Blood THC Quant by LC-QQQ



P2021-0391-1 and P2021-0402-1 were accidentally removed from the worklist and as such, another worklist was created and the results will be entered under that worklist (Worklist 4831).

03/09/2021 CS

cg

	1	2	3	4	5	6
A	IS + Cal. 1	NEG	P2021-0402-1			
B	IS + Cal. 2	M2021-0297-1	P2021-0404-1			
C	IS + Cal. 3	M2021-0429-4	P2021-0414-1			
D	IS + Cal. 4	M2021-0436-1				
E	IS + Cal. 5	M2021-0437-2				
F	IS + Cal. 6	P2021-0344-1				
G	IS + Cal. 7	P2021-0377-1				
H	IS + QC_1	P2021-0391-1 3/18/21 es				

*There was blood in this well so it was not used.

03/18/21 es

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

Extraction Date: 03/02/2021

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: 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 (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** 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 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 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 limits: THC 3-100, Carboxy-THC: 10-250, Hydroxy-THC: 3-100 (Qualitative only)

**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

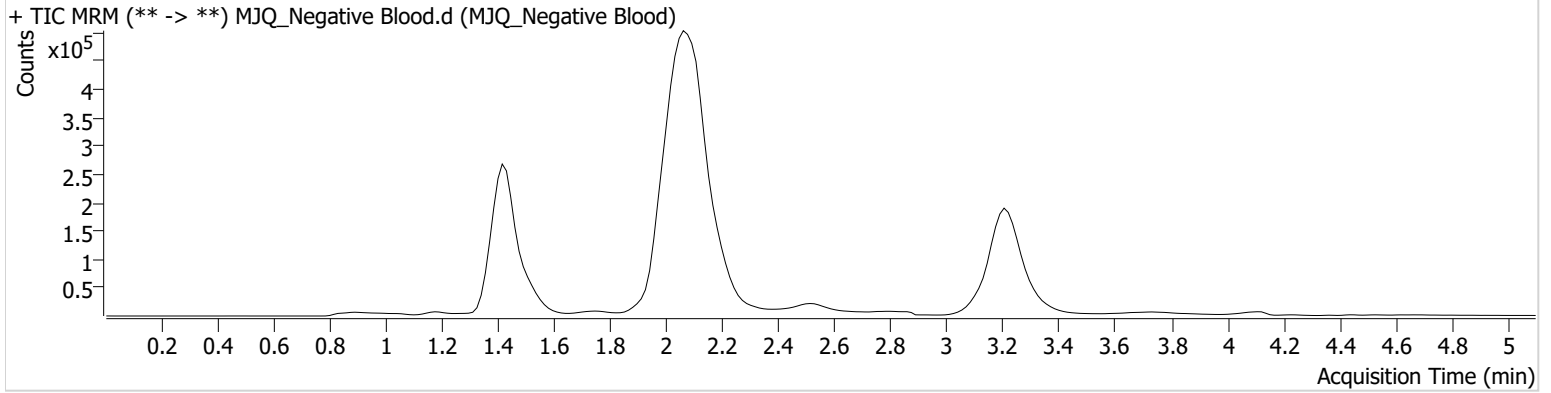


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 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-A2	Comment	
Injection Volume	10		
Acq. Date-Time	3/2/2021 5:15:19 PM		
Sample Info.			

Sample Chromatogram



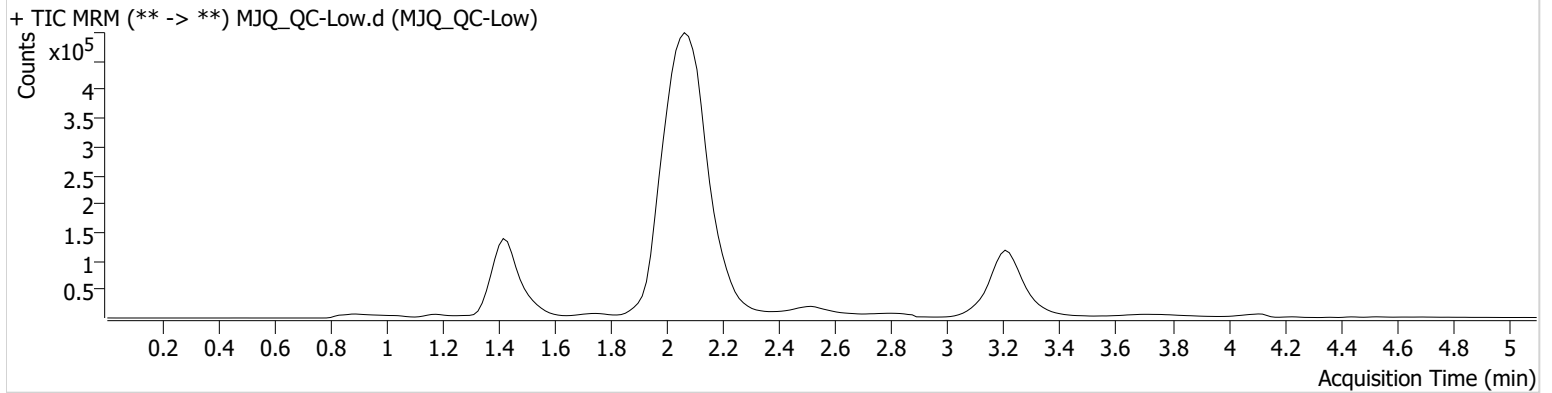
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 AM

Instrument	Instrument 1	Data File	MJQ_QC-Low.d
Type	Sample	Sample	MJQ_QC-Low
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-H1	Comment	
Injection Volume	10		
Acq. Date-Time	3/2/2021 8:17:36 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.438	51391	∞	9.4	26.83	620950	5.3945 ng/ml
THC-COOH	1.444	65421	∞	49.7	118.98	160925	15.3135 ng/ml
THC	3.224	39166	136.49	37.5	64.87	980613	4.0241 ng/ml

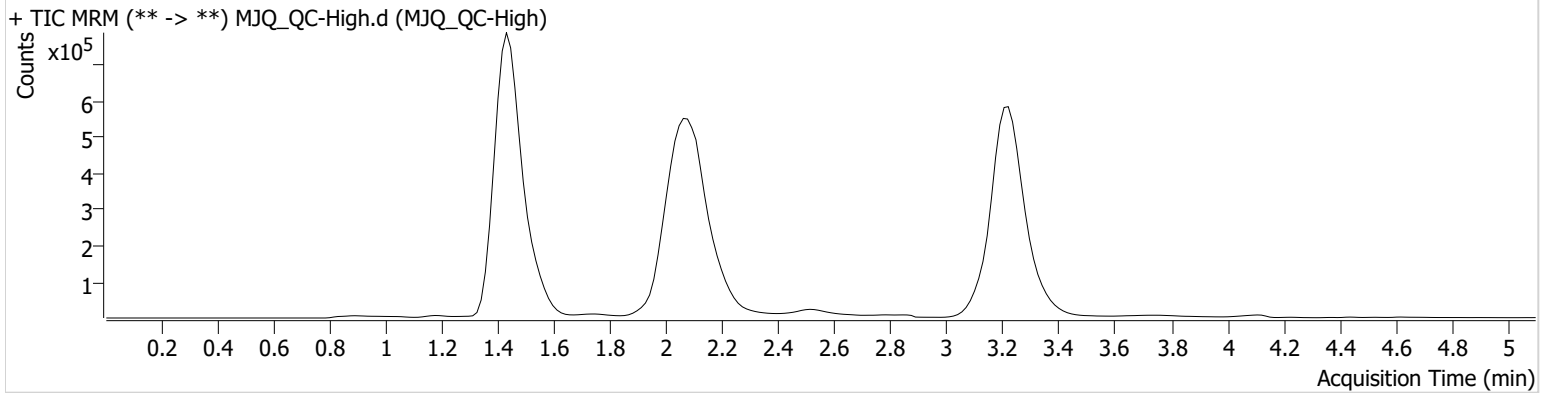
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 AM

Instrument	Instrument 1	Data File	MJQ_QC-High.d
Type	Sample	Sample	MJQ_QC-High
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-F1	Comment	
Injection Volume	10		
Acq. Date-Time	3/2/2021 5:30:31 PM		
Sample Info.			

Sample Chromatogram

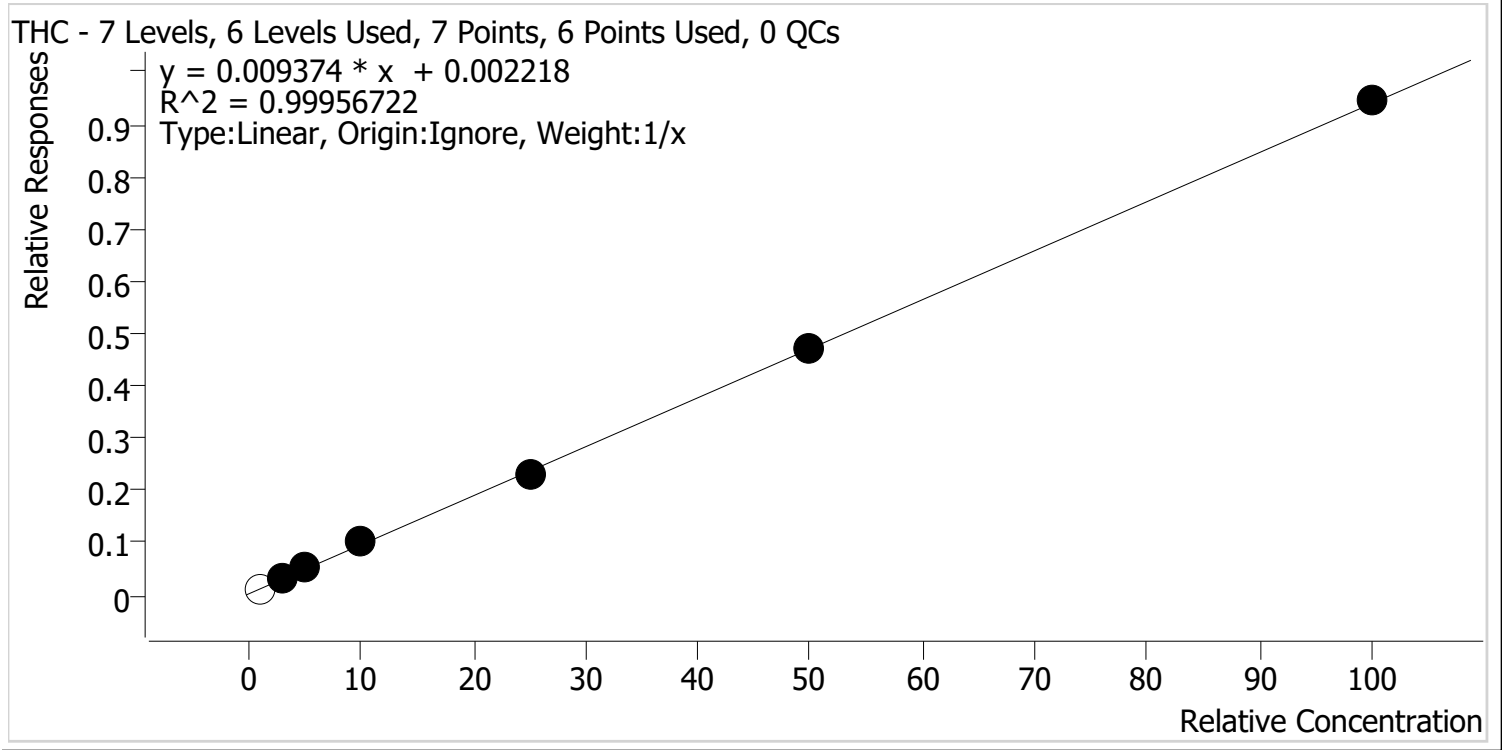


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.423	1184156	∞	11.5	2056.45	1786227	55.8973 ng/ml
THC-COOH	1.459	1115149	∞	57.0	∞	441884	122.2855 ng/ml
THC	3.224	1503070	∞	25.8	∞	2951787	54.0849 ng/ml



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update 3/8/2021 9:19 AM
Analyst Name ISP\Datastor
Analyte THC **Internal Standard** THC-D3



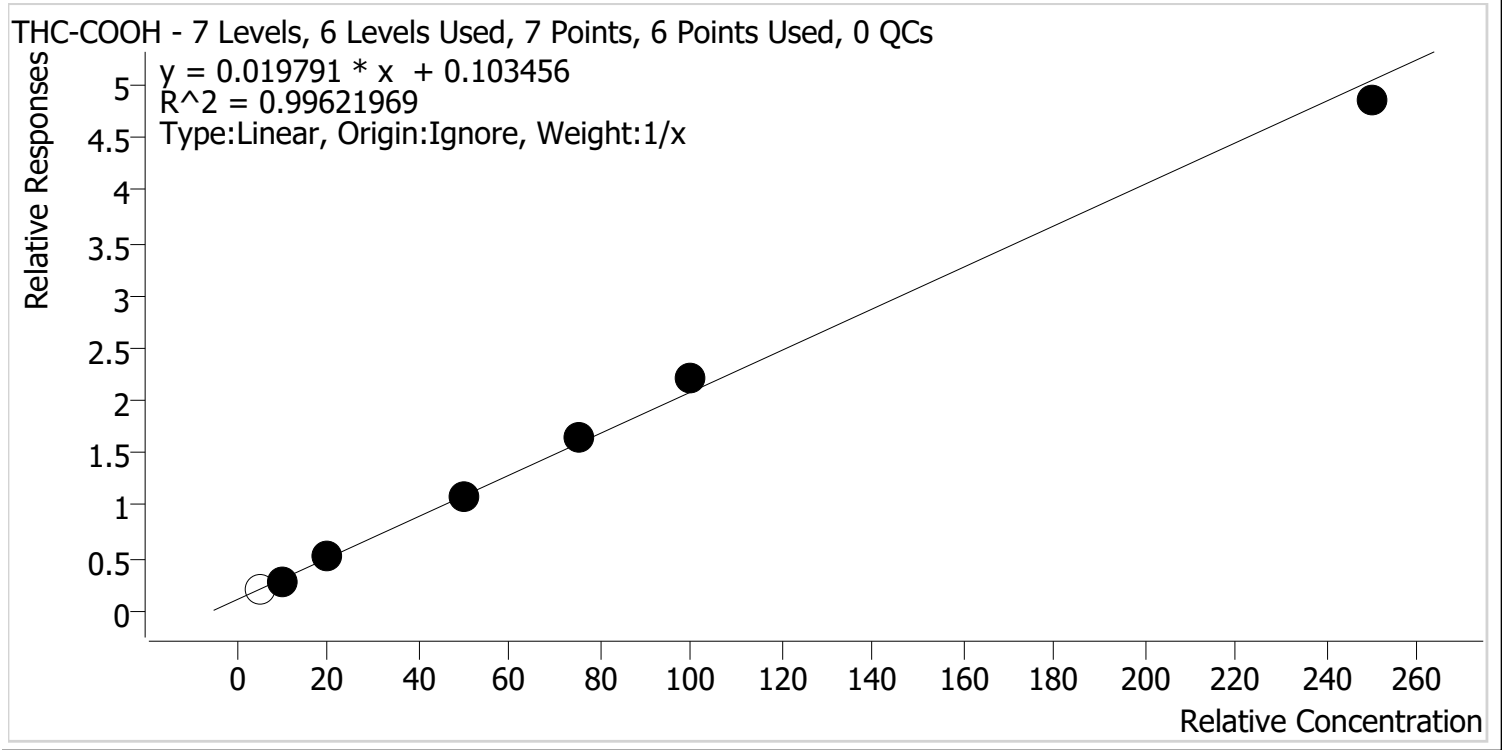
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	x	1.0	1.0	104.3
MJQ_Cal 2	2	✓	3.0	2.9	96.6
MJQ_Cal 3	3	✓	5.0	5.1	102.4
MJQ_Cal 4	4	✓	10.0	10.4	104.3
MJQ_Cal 5	5	✓	25.0	24.1	96.3
MJQ_Cal 6	6	✓	50.0	49.9	99.8
MJQ_Cal 7	7	✓	100.0	100.6	100.6

Cal 1 dropped due to poor secondary peak shape.



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update 3/8/2021 9:19 AM
Analyst Name ISP\Datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9



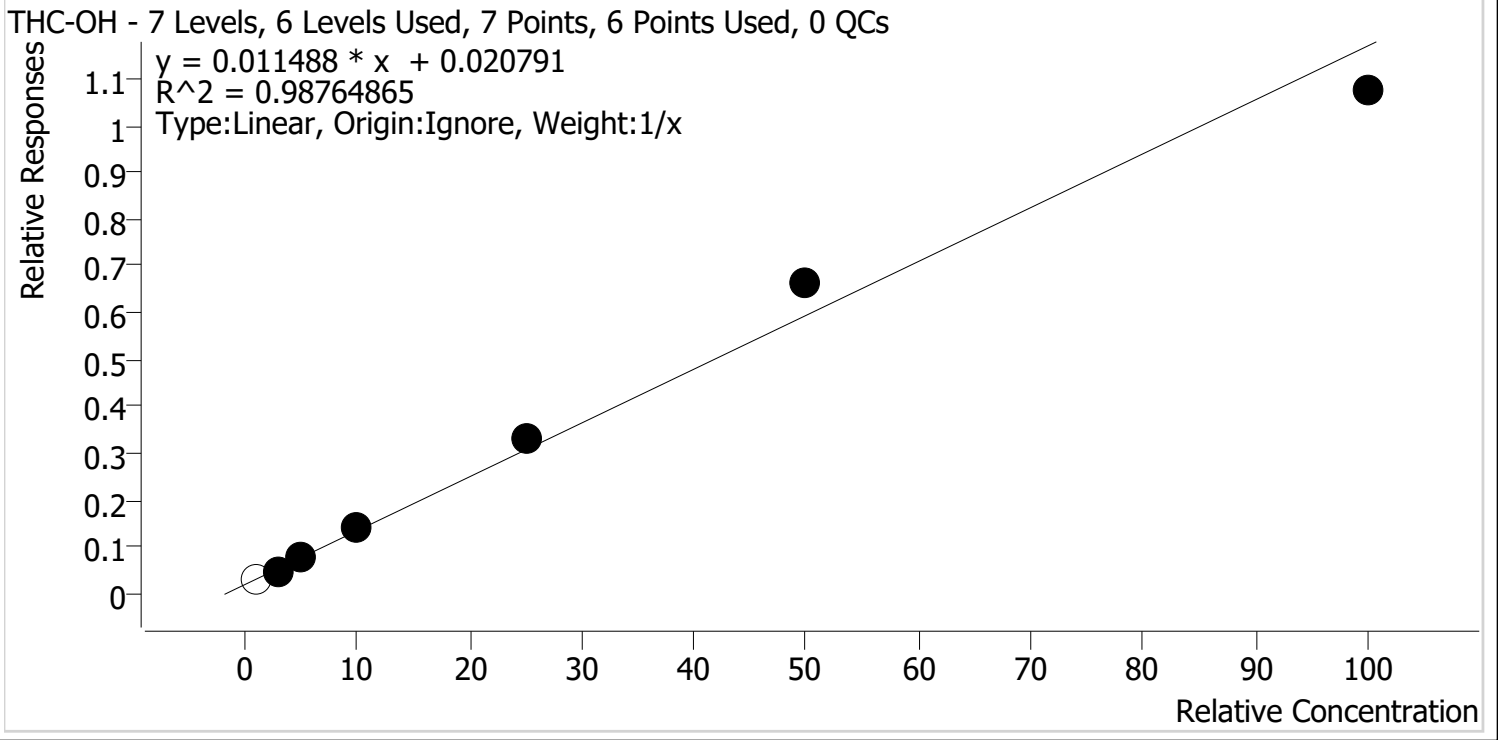
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	x	5.0	5.2	104.0
MJQ_Cal 2	2	✓	10.0	8.6	86.2
MJQ_Cal 3	3	✓	20.0	21.3	106.6
MJQ_Cal 4	4	✓	50.0	50.4	100.8
MJQ_Cal 5	5	✓	75.0	77.6	103.5
MJQ_Cal 6	6	✓	100.0	106.8	106.8
MJQ_Cal 7	7	✓	250.0	240.2	96.1

Cal 1 dropped due to ratio.



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update 3/8/2021 9:19 AM
Analyst Name ISP\Datastor
Analyte THC-OH **Internal Standard** THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	x	1.0	1.1	109.7
MJQ_Cal 2	2	✓	3.0	2.4	80.7
MJQ_Cal 3	3	✓	5.0	5.0	100.4
MJQ_Cal 4	4	✓	10.0	10.7	106.6
MJQ_Cal 5	5	✓	25.0	27.1	108.4
MJQ_Cal 6	6	✓	50.0	56.1	112.2
MJQ_Cal 7	7	✓	100.0	91.7	91.7

Cal 1 dropped due to ratio.

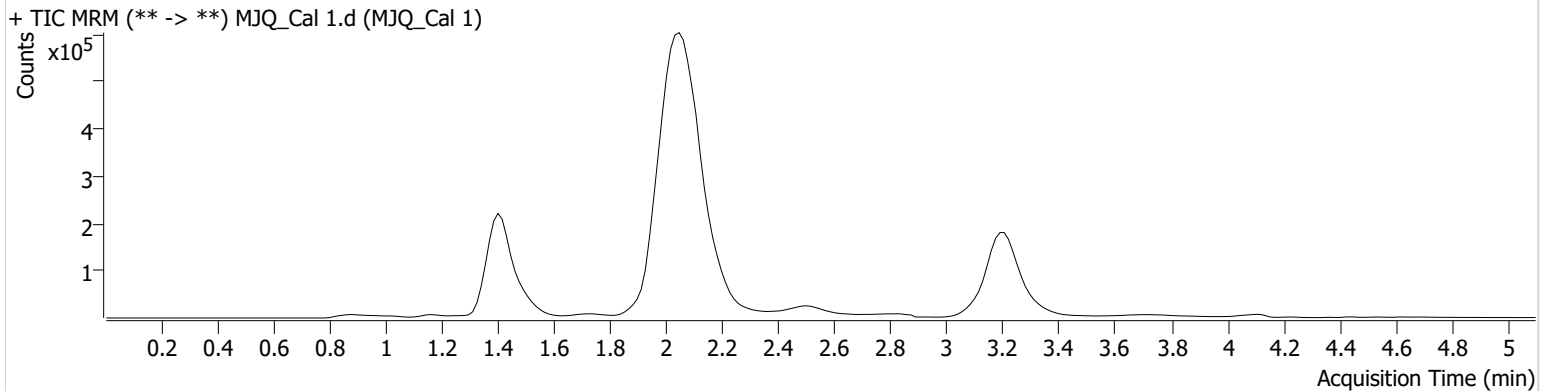
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 AM

Instrument	Instrument 1	Data File	MJQ_Cal 1.d
Type	Cal	Sample	MJQ_Cal 1
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-A1	Comment	
Injection Volume	10		
Acq. Date-Time	3/2/2021 4:14:22 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.468	35826	∞	7.5 Low	11.82	1072919	1.0968 ng/ml Low
THC-COOH	1.444	57513	∞	33.0 Low	∞	278629	5.2023 ng/ml
THC	3.194	17276	4.49 Low	31.8	29.53	1440046	1.0431 ng/ml



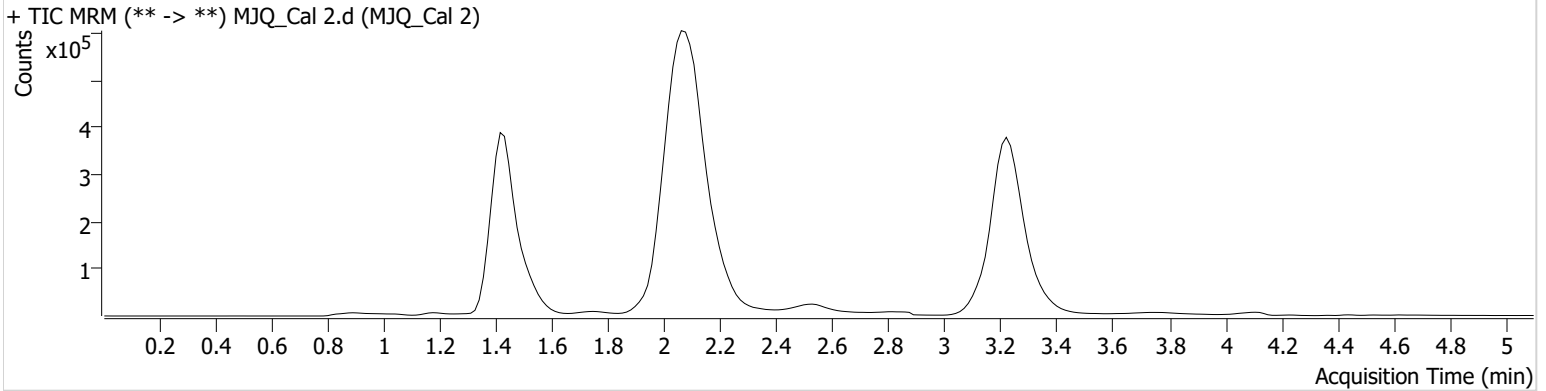
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 AM

Instrument Instrument 1
Type Cal
Acq. Method AM 27 THCQ.m
Sample Position P1-B1
Injection Volume 10
Acq. Date-Time 3/2/2021 4:22:07 PM
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	1.438	87597	∞	10.0	156.08	1802671	2.4201 ng/ml Low
THC-COOH	1.459	124966	∞	49.1	∞	455976	8.6203 ng/ml
THC	3.224	84678	∞	34.7	∞	2881886	2.8979 ng/ml



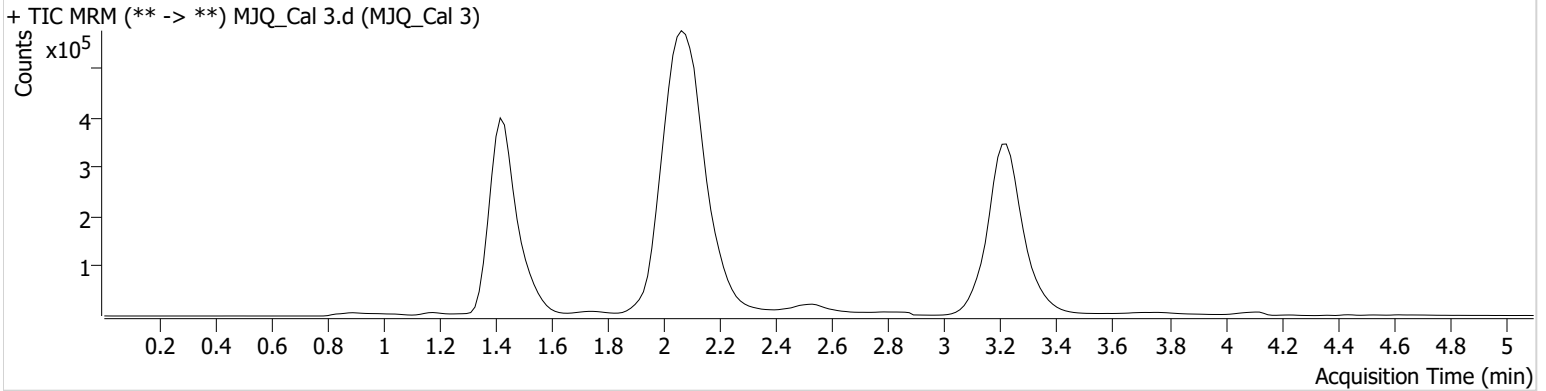
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 AM

Instrument Instrument 1
Type Cal
Acq. Method AM 27 THCQ.m
Sample Position P1-C1
Injection Volume 10
Acq. Date-Time 3/2/2021 4:29:43 PM
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	1.423	134923	∞	10.6	∞	1719554	5.0203 ng/ml
THC-COOH	1.459	232700	∞	47.6	1164.35	442924	21.3185 ng/ml
THC	3.224	133714	1065.80	36.2	∞	2662812	5.1202 ng/ml

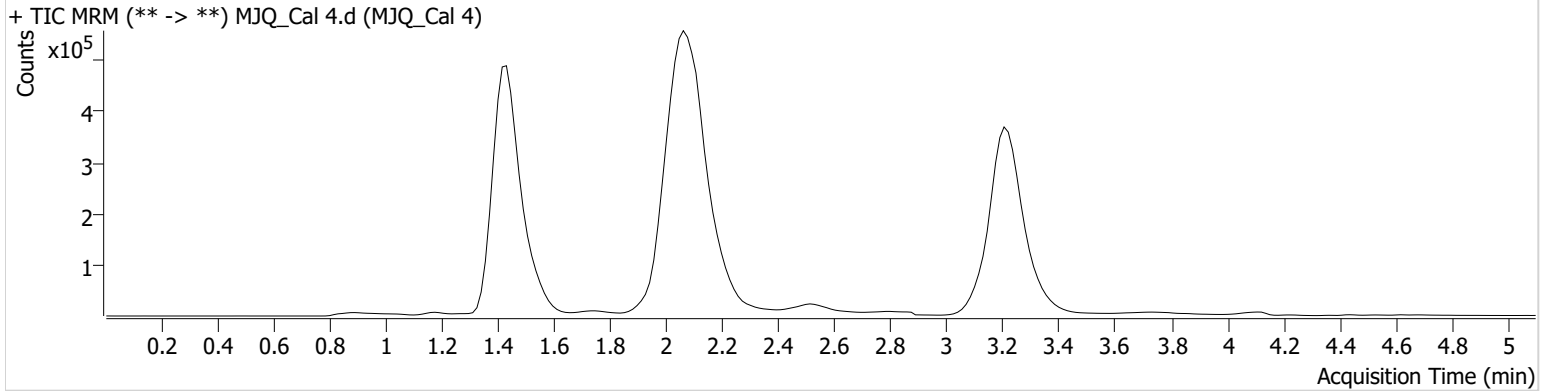
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 AM

Instrument	Instrument 1	Data File	MJQ_Cal 4.d
Type	Cal	Sample	MJQ_Cal 4
Acq. Method	AM 27 THCQ.m	Operator	Celena Shrum
Sample Position	P1-D1	Comment	
Injection Volume	10		
Acq. Date-Time	3/2/2021 4:37:19 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.423	251238	∞	11.0	∞	1753629	10.6613 ng/ml
THC-COOH	1.444	483641	∞	57.2	∞	439336	50.3957 ng/ml
THC	3.224	265088	∞	30.7	113.72	2651655	10.4281 ng/ml

AM #27 Cannabinoid Quant. Results

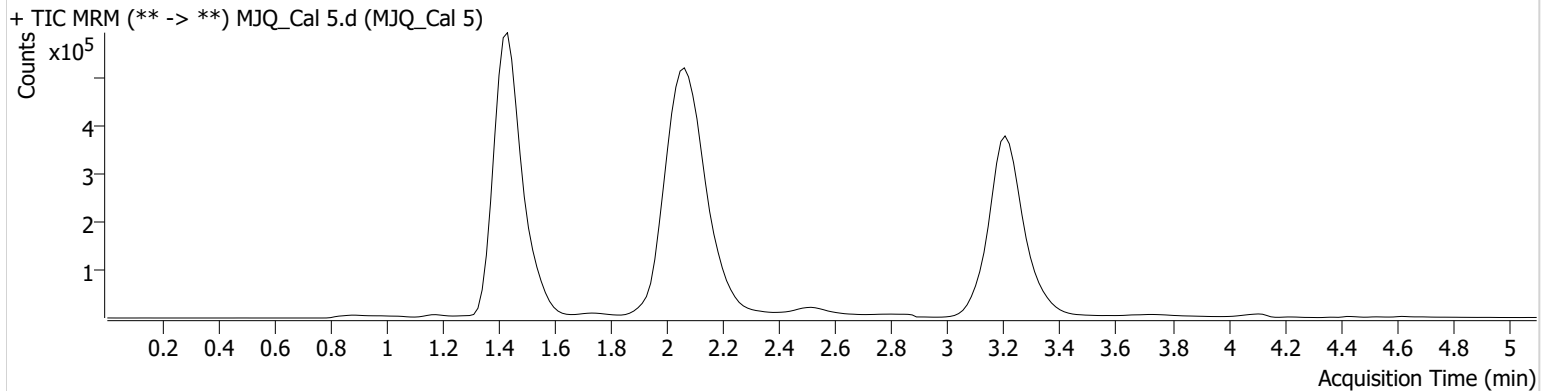


Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 AM

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-E1	Comment	
Injection Volume	10		
Acq. Date-Time	3/2/2021 4:44:56 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.423	572873	358.96	11.2	∞	1725078	27.0974 ng/ml
THC-COOH	1.444	691946	∞	63.6	1724.43	422047	77.6127 ng/ml
THC	3.209	589711	∞	26.8	484.97	2586390	24.0867 ng/ml



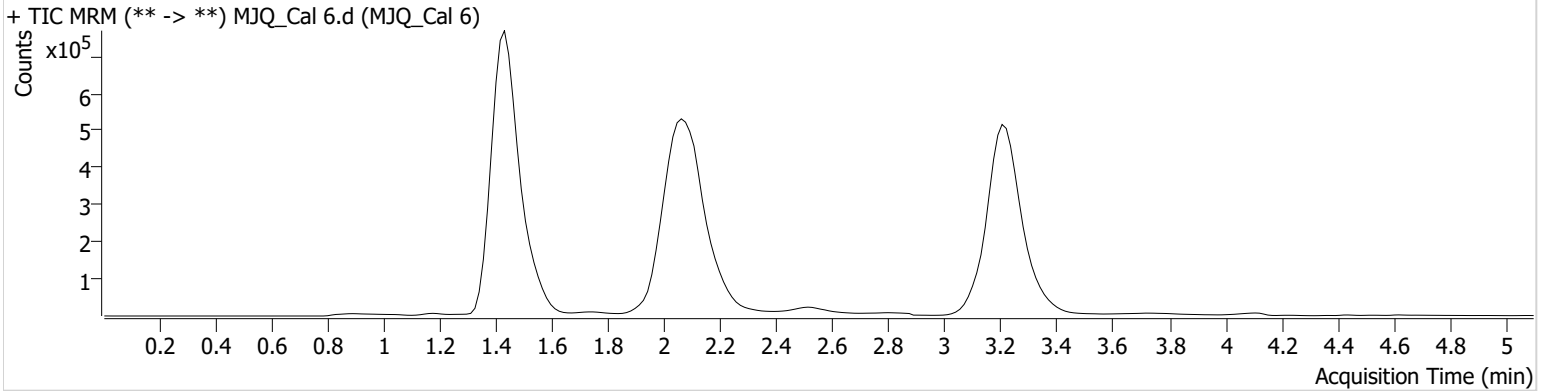
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 AM

Instrument Instrument 1
Type Cal
Acq. Method AM 27 THCQ.m
Sample Position P1-F1
Injection Volume 10
Acq. Date-Time 3/2/2021 4:52:32 PM
Sample Info.

Data File MJQ_Cal 6.d
Sample MJQ_Cal 6
Operator Celena Shrum
Comment

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.423	1181769	∞	11.5	697.49	1775741	56.1211 ng/ml
THC-COOH	1.444	938244	∞	57.6	∞	422997	106.8474 ng/ml
THC	3.224	1336544	∞	26.0	613.70	2843563	49.9050 ng/ml

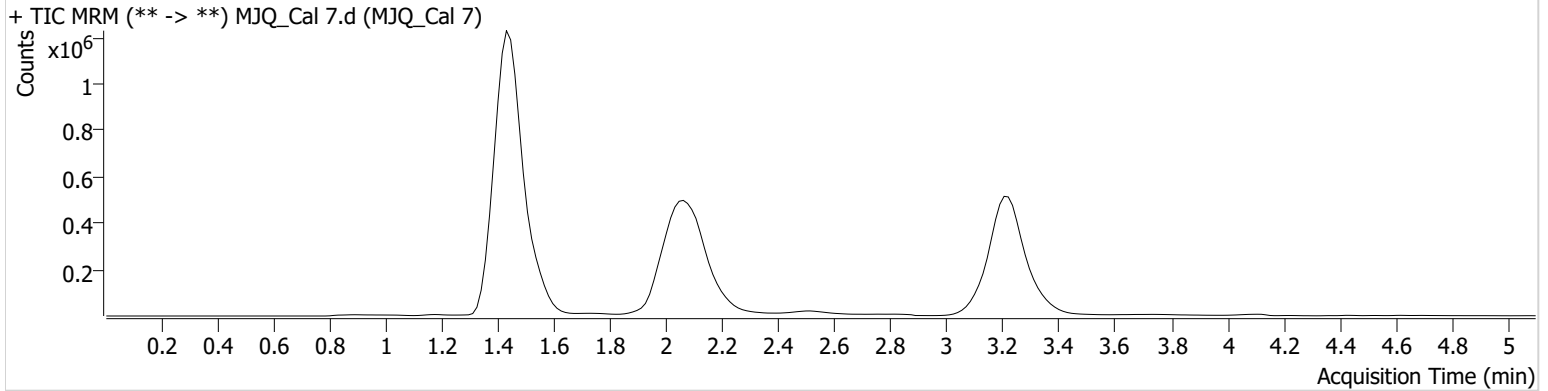
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2021\AM 27-28\030221 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 3/8/2021 9:19:06 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-G1	Comment	
Injection Volume	10		
Acq. Date-Time	3/2/2021 5:00:08 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.423	2110796	∞	11.7	∞	1965352	91.6798 ng/ml
THC-COOH	1.459	2289642	∞	58.4	∞	471372	240.2054 ng/ml
THC	3.224	2072625	∞	25.7	∞	2193528	100.5622 ng/ml