

REVIEWED

By Amber Gerheart at 9:54 am, Aug 04, 2022

TS 8/3/2022

Worklist: 6041

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
M2022-2004	4	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2022-2290	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2022-3003	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-1665	4	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-1665	5	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-1671	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-1711	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-1765	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-1794	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-1796	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-1892	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	

**Idaho State Police
Forensic Services**

Request for Departure from an Analytical Method or Quality Standard

Deviation Number (assigned by QM): TOX-22-02

Date of Request:
03/02/2022

Requestor/Discipline:
Celena Shrum/Toxicology

Analytical Method/Quality Standard, Revision #:
Toxicology AM #25, AM #26, and AM #27, Revision 13

Temporary or Permanent Deviation:
Permanent

Scope of Deviation (record specific information, e.g. affected programs, evidence types, expected end date; etc):

Deviation will remain in place until the change is made in the next method revision.

Deviation Request (Describe detailed instructions of the changes being made; include reference to specific section number(s) in the method manual):

Toxicology AM #25 3.3.1.1 Internal standards are prepared by the ToxBox plate manufacturer and contained on the 96 well plate. If the run contains urine samples, a positive external urine control must also be run.

Toxicology AM #26 3.3.2 A negative control will be run with each extraction. If the run contains urine samples, a negative urine control and external positive urine control must also be included.

Toxicology AM #27 3.3.2 A negative control will be run with each extraction. If the run contains urine samples, a negative urine control and positive external urine control will also be included in the run.

The deviation is to include the option of using an internal urine control in lieu of an external urine control.

Technical Justification for Analytical Method Deviations:

Internal controls serve the same purpose as external controls but also helps to avoid the possible issues that can occur with using external controls (incorrect spiking, incorrect preparation, evaporation of compounds, etc.). If these errors occur, runs need to be repeated and this wastes time, sample, and supplies.

Technical Review

Departure approved
Comments:

Departure Not Approved
Comments:



Approver: Rachel Cutler
Title: Lab Manager

Date: 3/2/22

Quality Review

Quality Approver: Jason Crowe
Title: Quality Manager
Date: 3/2/2022



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

Extraction Date: 07/27/2022

Analyst: Tamara Salazar

Plate lot#: 220309

Plate Retest Date: 09/09/2022

Mobile phase A: 0.1% Formic Acid in LCMS Water

Mobile phase B: 0.1% Formic acid in Acetonitrile

Blank Blood Lot: Lampire 22B52015-1

Blank Urine Lot: POC021022

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. Urine hydrolysis: add 1.5mL urine to blank plate, add 250µl 1N KOH. Shake and incubate at 40 degrees for 15 minutes.
- 3. Using a calibrated pipette, add 1000µl blood and urine (if applicable) (calibrated pipette) into the appropriate wells of analytical (standards) plate. **Pipette ID: 42**
- 4. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 5. Add 500µL of 0.1% formic acid in water to blood samples, and 500µL of saturated phosphate buffer to urine samples in the wells of the analytical plate.
- 6. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 7. Transfer 700-800µL of blood+acid or urine+acid mixture to corresponding wells of SLE+ plate. Amount transferred: 750µL
- 8. 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*)
- 9. Wait 5 minutes.
- 10. Add 2.25mL MTBE. (*Add in 3 increments of 750uL*)
- 11. Wait 5 minutes.
- 12. Apply positive pressure for approx. 15 seconds. (*10-15 PSI- Selector to the left*).
- 13. Add 2.25mL Hexane. (*Add in 3 increments of 750uL*)
- 14. Wait 5 minutes.
- 15. Apply positive pressure for approx. 15 seconds. (*10-15 PSI- Selector to the left*).
- 16. Remove plate containing eluate. Place on SPE Dry and evaporate to dryness at approx. 35°C.
- 17. 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 samples with calculated concentrations for THC at 1ng/mL or greater and OH-THC at 3ng/mL or greater may be reported quantitatively (blood only). Calculated concentrations for carboxy-THC of 5ng/mL may be reported qualitatively. 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 3-100

TS

	1	2	3	4	5	6
A	IS + Cal. 1	IS + QC_1	IS + Sample	IS + Sample	P2022-1665-5	IS + QC_1
B	IS + Cal. 2	IS + Sample	IS + Sample	IS + Sample	P2022-1665-4	IS + Cal. 7
C	IS + Cal. 3	IS + Sample	IS + Sample	P2022-1892-1	M2022-3003-3	IS + Cal. 6
D	IS + Cal. 4	IS + Sample	IS + Sample	P2022-1796-1	M2022-2290-3	IS + Cal. 5
E	IS + Cal. 5	IS + Sample	IS + Sample	P2022-1794-1	M2022-2004-4	IS + Cal. 4
F	IS + Cal. 6	IS + Sample	IS + Sample	P2022-1765-1	Neg Urine	IS + Cal. 3
G	IS + Cal. 7	IS + Sample	IS + Sample	P2022-1711-1	Neg Blood	IS + Cal. 2
H	IS + QC_1	IS + Sample	IS + Sample	P2022-1671-1	IS + QC_1	IS + Cal. 1

All wells to contain 100 µl of residual DMSO

TS

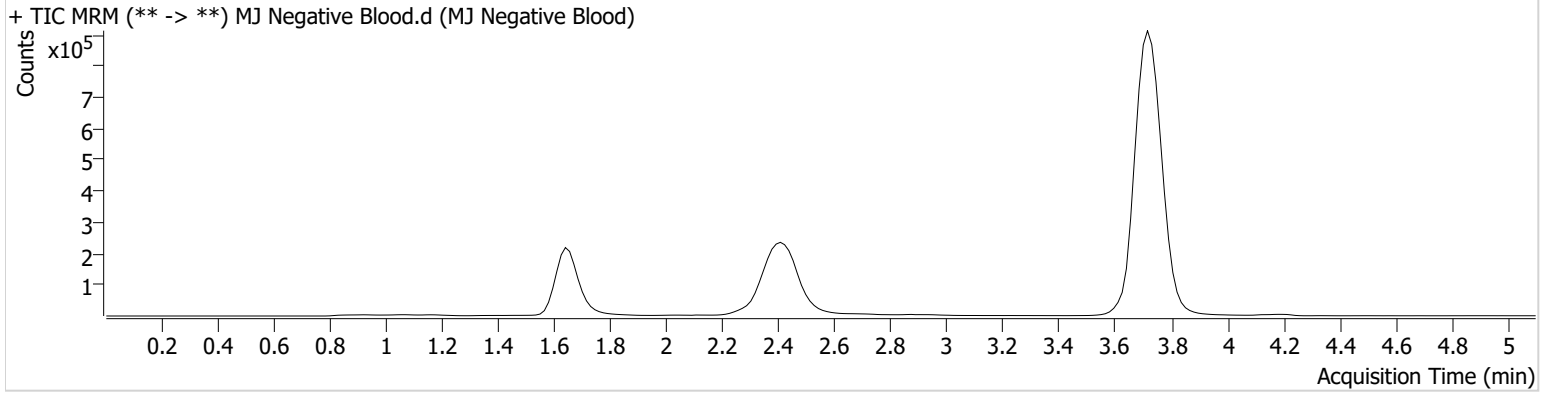


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	MJ Negative Blood.d
Type	Sample	Sample	MJ Negative Blood
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-G5	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 4:15:10 PM		
Sample Info.			

Sample Chromatogram



TS

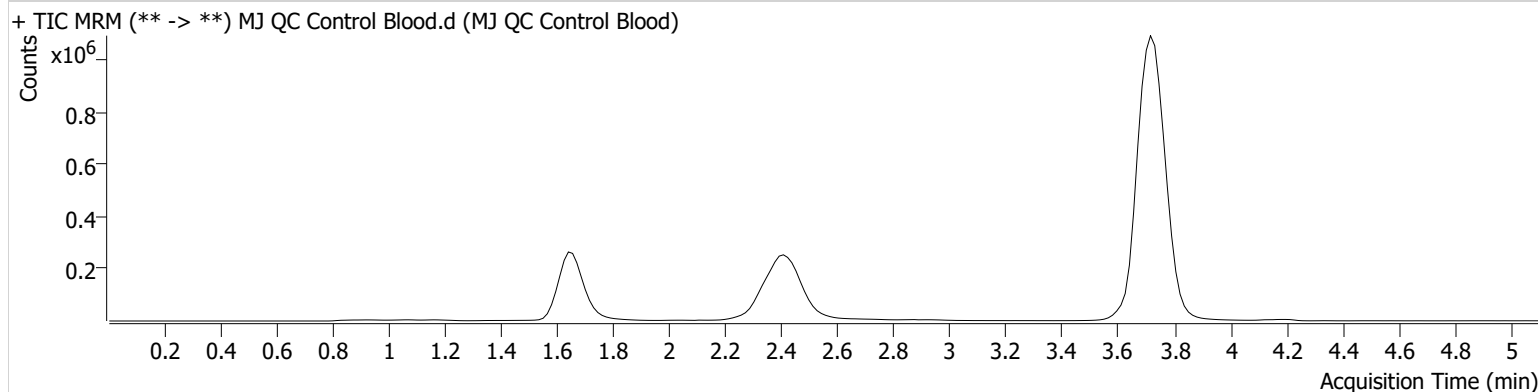


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	MJ QC Control Blood.d
Type	QC	Sample	MJ QC Control Blood
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-A6	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 3:59:53 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.648	73193	∞	13.6	108.72	1082581	4.6652 ng/ml
THC-COOH	1.685	109528	106.04	52.7	293.91	311243	14.6538 ng/ml
THC	3.736	296196	2790.00	28.5	137.49	7325571	4.8088 ng/ml

TS

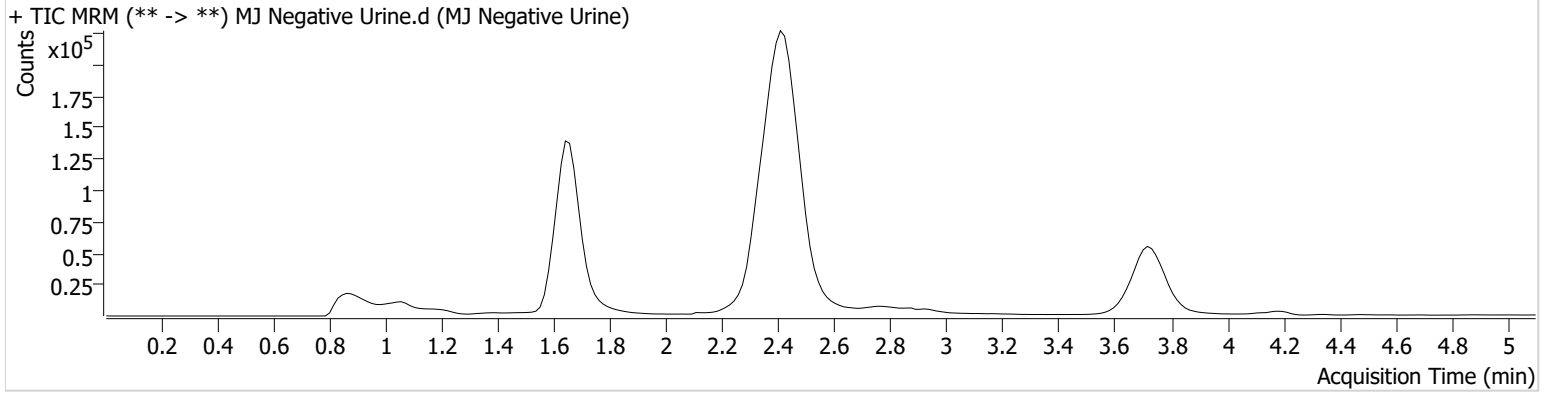


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	MJ Negative Urine.d
Type	Sample	Sample	MJ Negative Urine
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-F5	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 4:22:46 PM		
Sample Info.			

Sample Chromatogram



TS



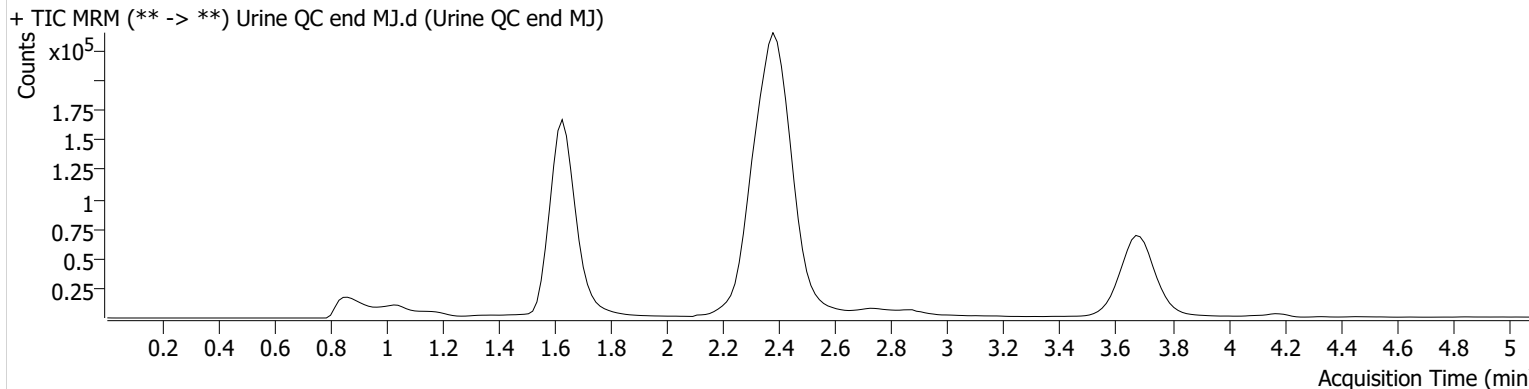
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	Urine QC end MJ.d
Type	QC	Sample	Urine QC end MJ
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-H5	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 7:25:48 PM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.633	46061	∞	14.0	92.42	748450	4.2530 ng/ml
THC-COOH	1.670	60141	256.11	53.1	333.52	169974	14.7370 ng/ml
THC	3.706	24064	109.37	33.3	26.83	573546	4.9818 ng/ml

TS



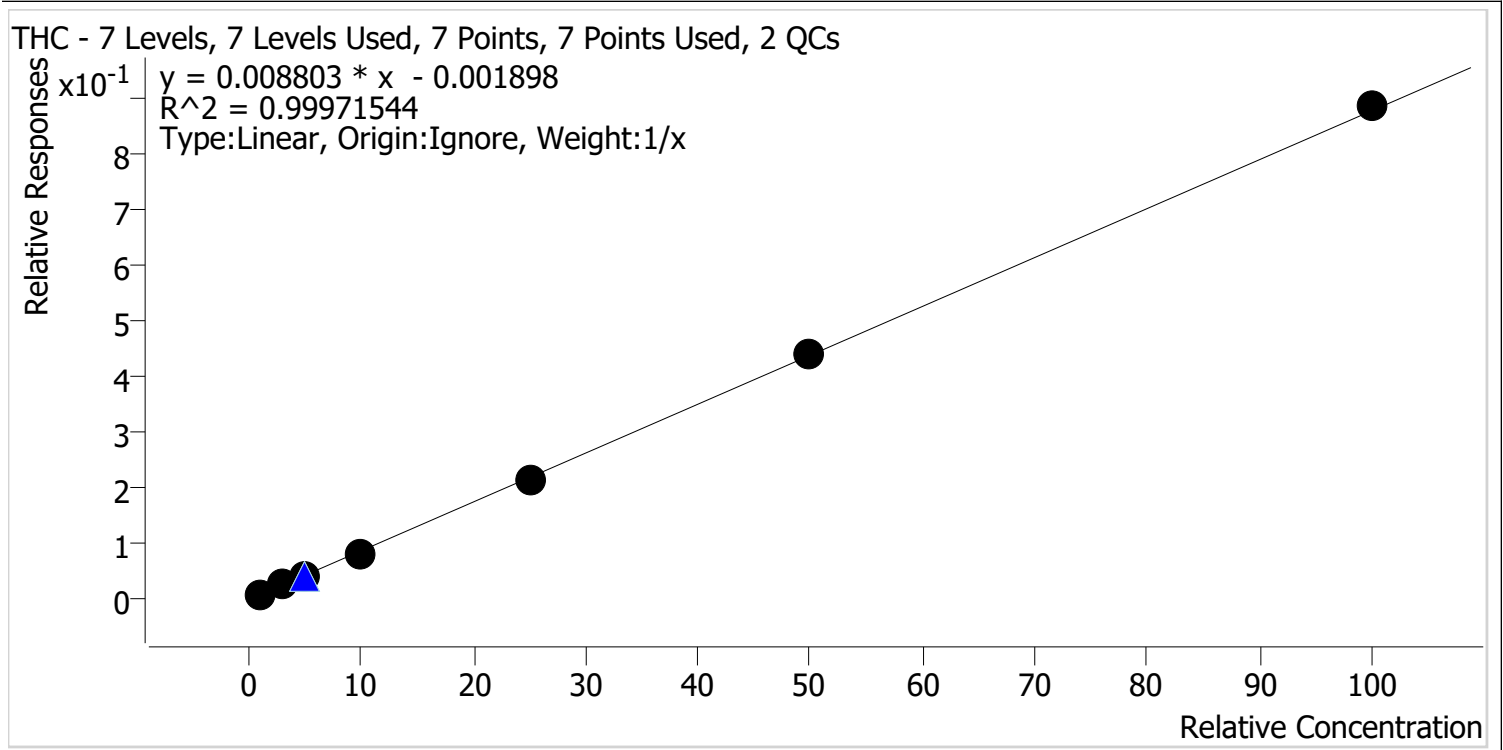
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin

Last Cal. Update 8/2/2022 12:51 PM

Analyst Name ISP\datastor

Analyte THC **Internal Standard** THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✓	1.0	1.1	109.9
Cal 2 MJ	2	✓	3.0	3.0	98.5
Cal 3 MJ	3	✓	5.0	4.8	96.2
Cal 4 MJ	4	✓	10.0	9.6	95.7
Cal 5 MJ	5	✓	25.0	24.7	99.0
Cal 6 MJ	6	✓	50.0	49.9	99.9
Cal 7 MJ	7	✓	100.0	100.9	100.9

TS



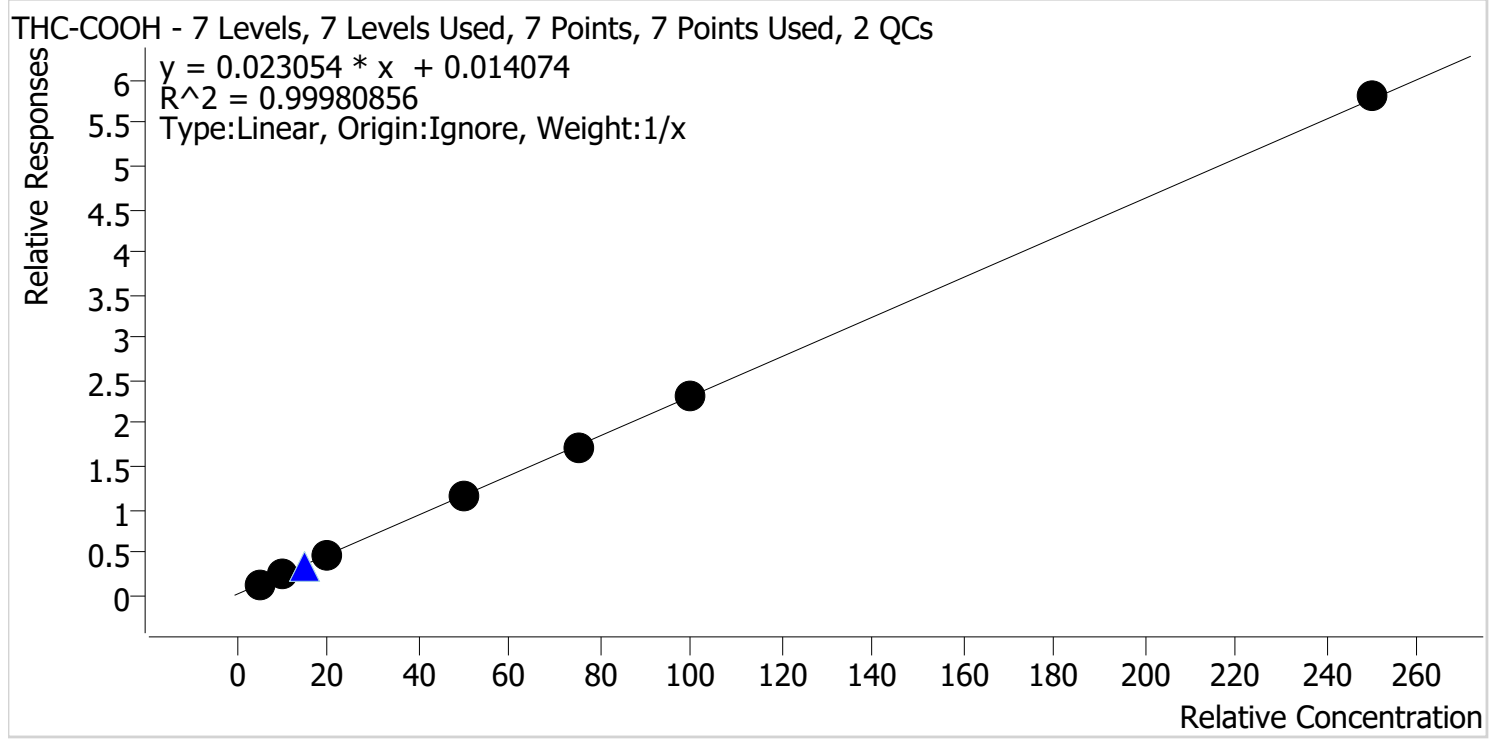
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin

Last Cal. Update 8/2/2022 12:51 PM

Analyst Name ISP\datastor

Analyte THC-COOH **Internal Standard** THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✓	5.0	4.9	98.3
Cal 2 MJ	2	✓	10.0	10.3	103.4
Cal 3 MJ	3	✓	20.0	20.1	100.6
Cal 4 MJ	4	✓	50.0	49.6	99.3
Cal 5 MJ	5	✓	75.0	73.1	97.5
Cal 6 MJ	6	✓	100.0	100.4	100.4
Cal 7 MJ	7	✓	250.0	251.5	100.6

TS



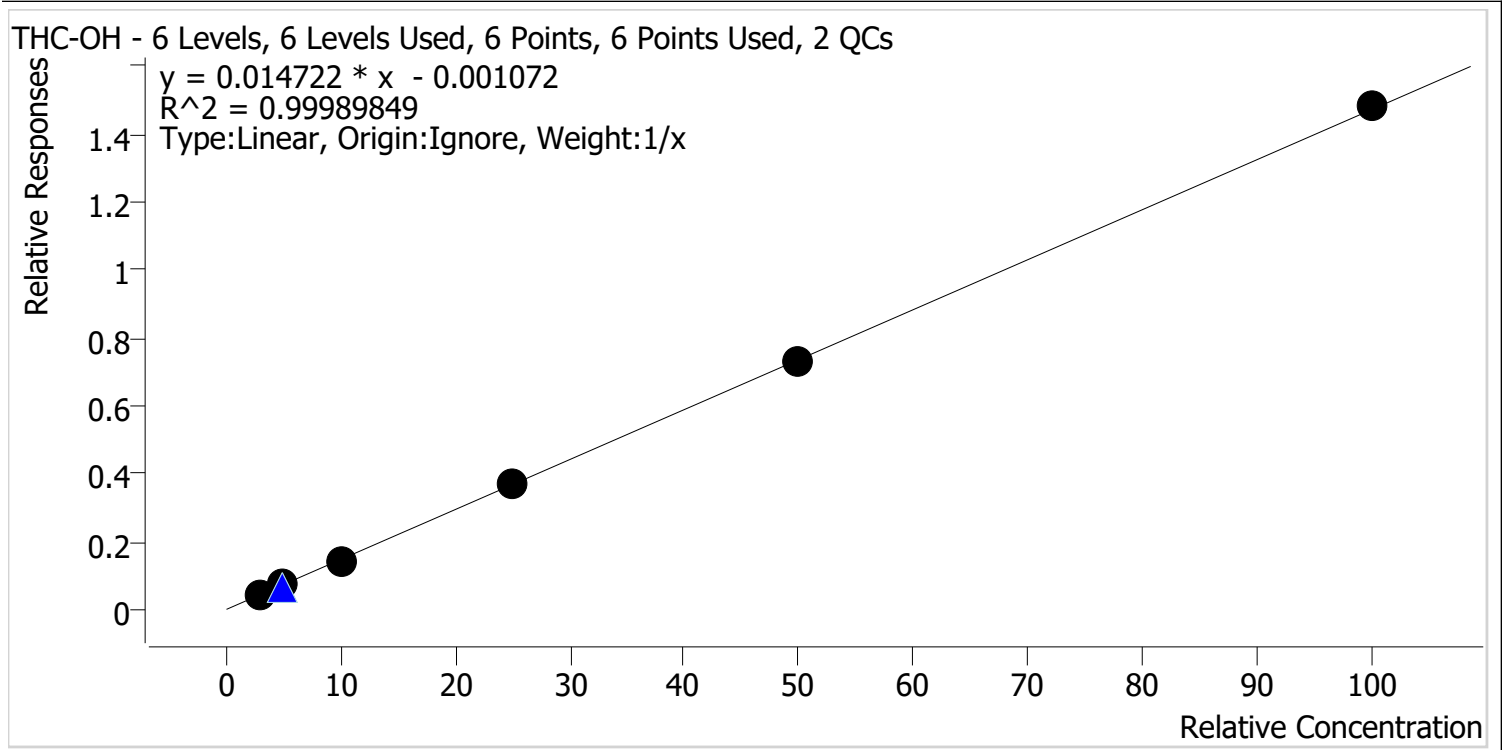
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin

Last Cal. Update 8/2/2022 12:51 PM

Analyst Name ISP\datastor

Analyte THC-OH **Internal Standard** THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 2 MJ	2	✓	3.0	3.1	102.3
Cal 3 MJ	3	✓	5.0	5.0	100.3
Cal 4 MJ	4	✓	10.0	9.8	97.9
Cal 5 MJ	5	✓	25.0	25.0	99.8
Cal 6 MJ	6	✓	50.0	49.5	99.1
Cal 7 MJ	7	✓	100.0	100.6	100.6

Calibrator 1 dropped due to low response.

TS

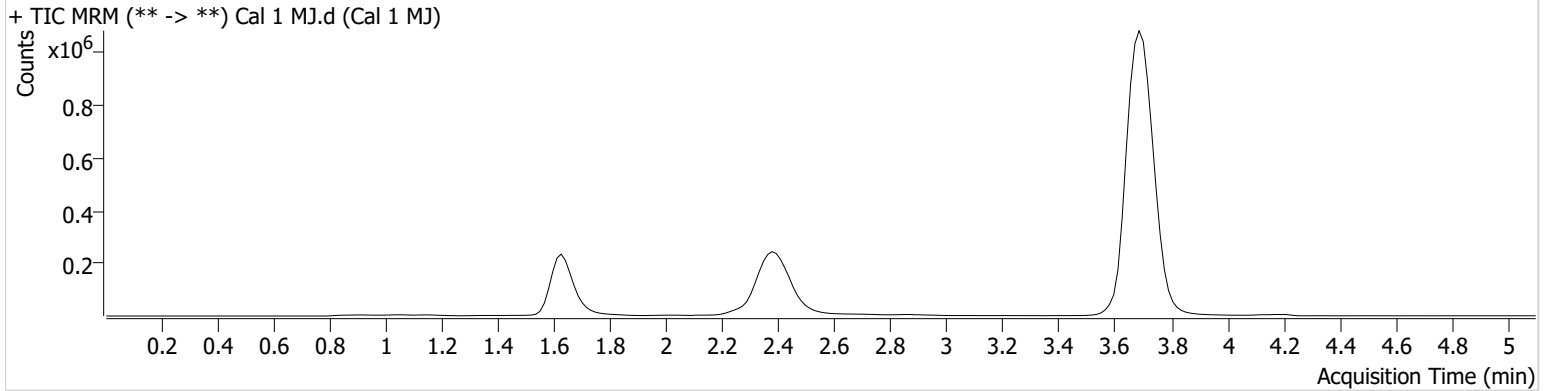


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	Cal 1 MJ.d
Type	Cal	Sample	Cal 1 MJ
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-H6	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 2:58:48 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.670	36307	717.89	47.1	67.17	285053	4.9143 ng/ml Low
THC	3.706	56956	245.61	31.9	160.43	7322639	1.0992 ng/ml

TS

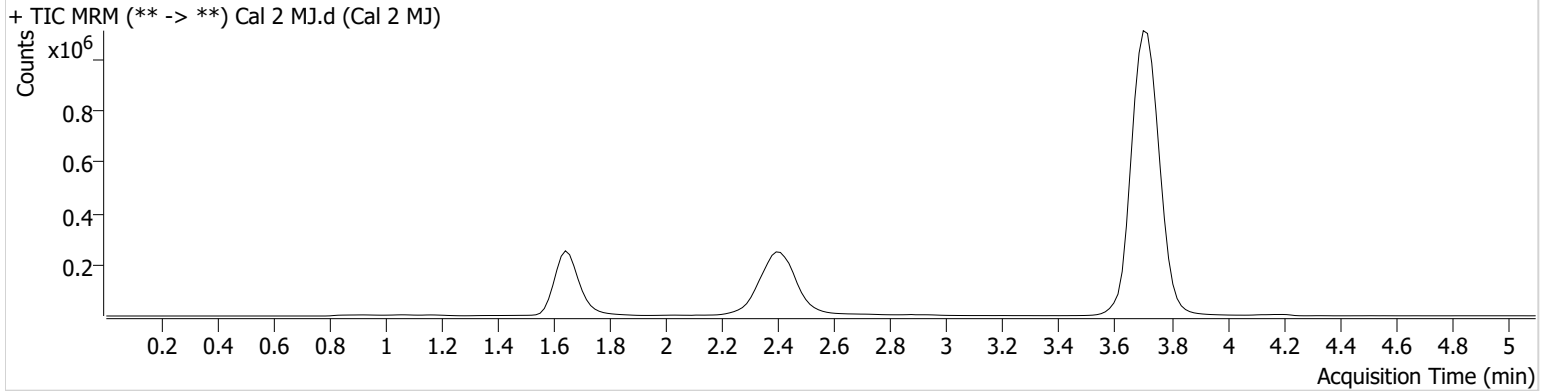


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	Cal 2 MJ.d
Type	Cal	Sample	Cal 2 MJ
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-G6	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 3:06:34 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.648	46890	∞	12.4	56.46	1062739	3.0698 ng/ml
THC-COOH	1.685	76603	195.05	49.2	652.06	303522	10.3367 ng/ml
THC	3.721	181654	966.41	29.4	94.69	7536007	2.9539 ng/ml

TS

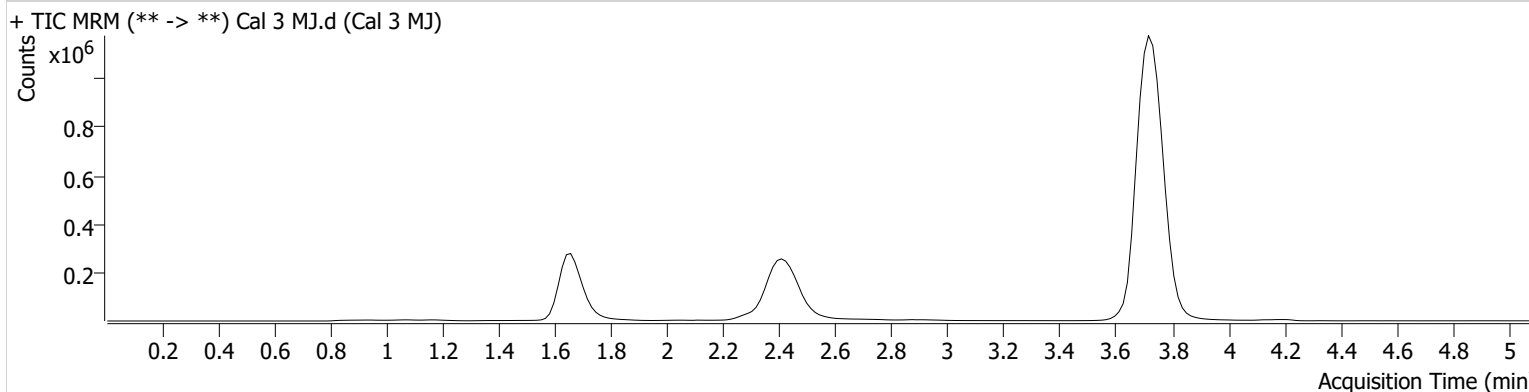


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	Cal 3 MJ.d
Type	Cal	Sample	Cal 3 MJ
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-F6	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 3:14:10 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.663	74649	179.18	13.5	296.61	1026179	5.0139 ng/ml
THC-COOH	1.685	139724	248.72	51.9	615.52	292352	20.1202 ng/ml
THC	3.736	303251	1085.16	28.9	∞	7498531	4.8098 ng/ml

TS

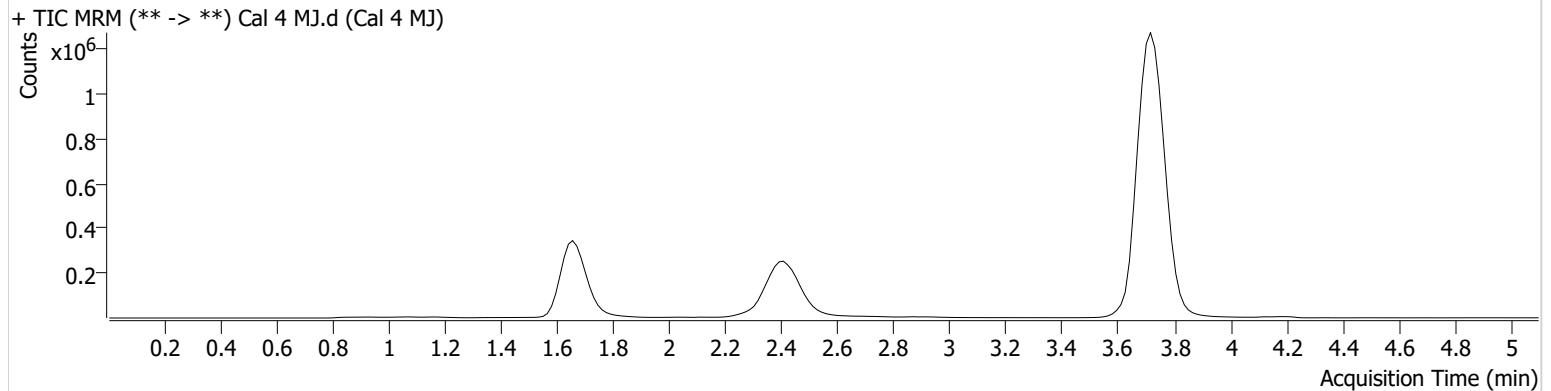


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	Cal 4 MJ.d
Type	Cal	Sample	Cal 4 MJ
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-E6	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 3:21:46 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.648	157269	∞	13.0	225.72	1099827	9.7856 ng/ml
THC-COOH	1.685	356323	∞	56.6	1850.95	307545	49.6452 ng/ml
THC	3.721	655436	1444.73	28.2	∞	7960371	9.5691 ng/ml

TS

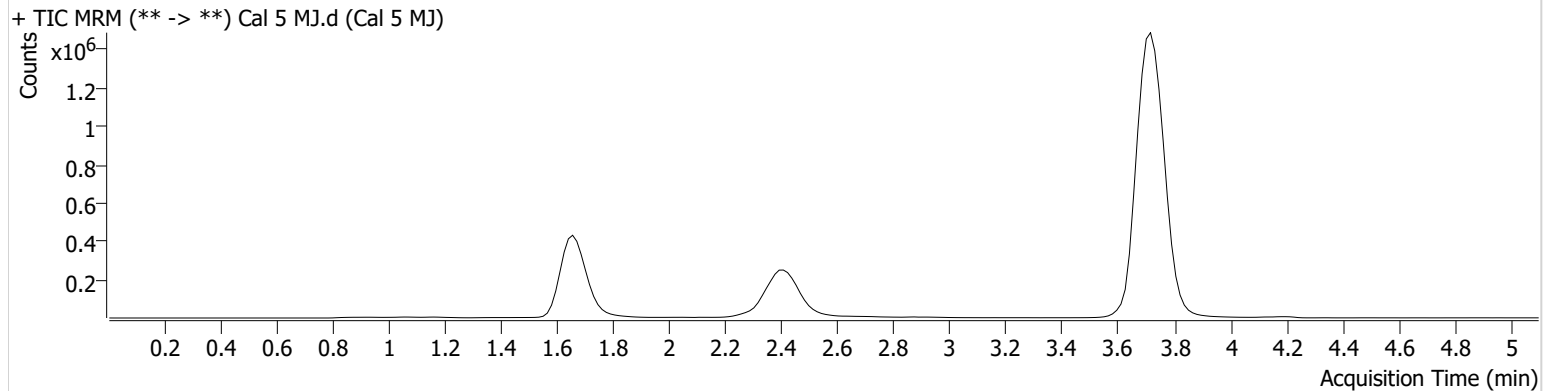


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	Cal 5 MJ.d
Type	Cal	Sample	Cal 5 MJ
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-D6	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 3:29:24 PM		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.648	401902	∞	12.9	1526.44	1097180	24.9538 ng/ml
THC-COOH	1.685	514047	1533.34	57.3	1616.88	302457	73.1103 ng/ml
THC	3.721	1761464	44008.35	27.8	1735.25	8159248	24.7401 ng/ml

TS

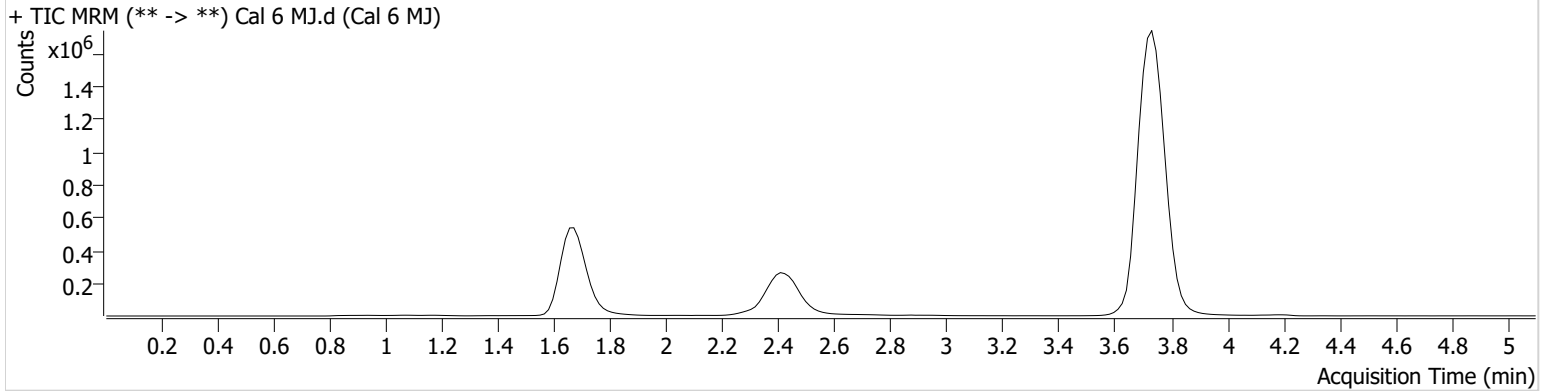


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	Cal 6 MJ.d
Type	Cal	Sample	Cal 6 MJ
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-C6	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 3:37:00 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.663	785738	∞	13.1	∞	1078745	49.5476 ng/ml
THC-COOH	1.685	679111	∞	57.5	5992.38	291693	100.3764 ng/ml
THC	3.736	3328989	4993.35	28.1	∞	7605471	49.9392 ng/ml

TS

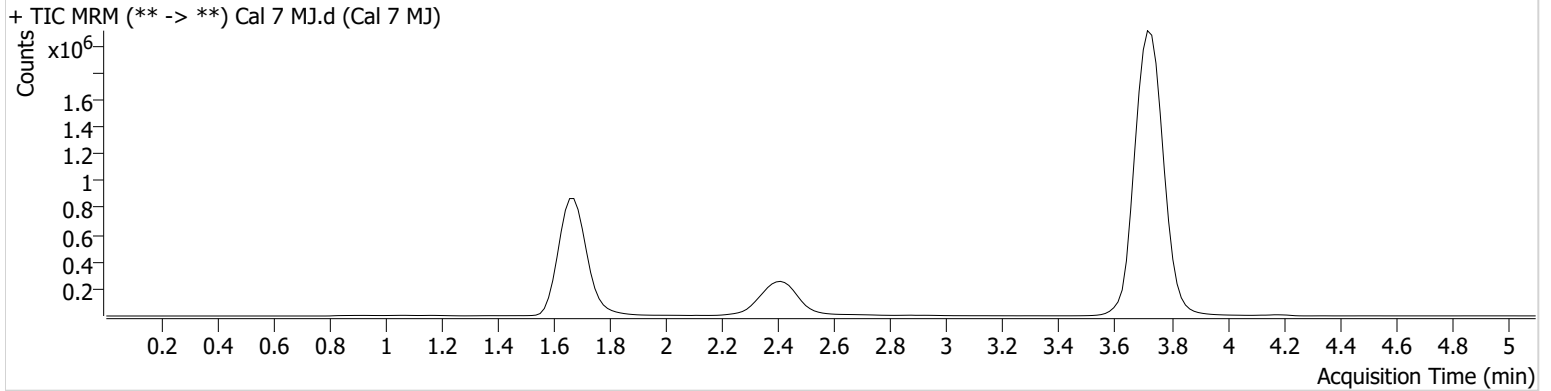


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\072722 AM 27 28 P1 TS_Urines\QuantResults\AM 27.batch.bin
Calibration Last Update 8/2/2022 12:51:11 PM

Instrument	Falco (069901)	Data File	Cal 7 MJ.d
Type	Cal	Sample	Cal 7 MJ
Acq. Method	AM 27 THCQ.m	Operator	Tamara Salazar
Sample Position	P1-B6	Comment	
Injection Volume	10		
Acq. Date-Time	7/27/2022 3:44:37 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.648	1561025	∞	13.3	∞	1054448	100.6293 ng/ml
THC-COOH	1.685	1638579	∞	58.0	∞	281924	251.4968 ng/ml
THC	3.736	6302052	52257.62	28.3	∞	7111223	100.8887 ng/ml