REVIEWED
By Anne Nord at 11:32 am, Oct 24, 2019

10/23/2019

Worklist: 3769

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
M2019-4464	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2019-4621	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2019-4654	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-3186	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-3208	1	BCK	AM 27 Blood THC Quant by LC-QQQ	

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

Extraction Date: 10/23/19 Analyst: Sarah Pickle
Plate lot#: Lot # 190716 Item # IDP-108 Plate Expiration: 1/16/20

Mobile phase A: 0.1% Formic Acid in LCMS Water **Mobile phase B:** 0.1% Formic acid in Acetonitrile
MTBE LCMS Methanol Hexane
Blank Blood Lot: 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: #3**
☒ 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\2019\AM 27\102219 MDQ THCQ SP Batch Name: THCQ
☒ 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

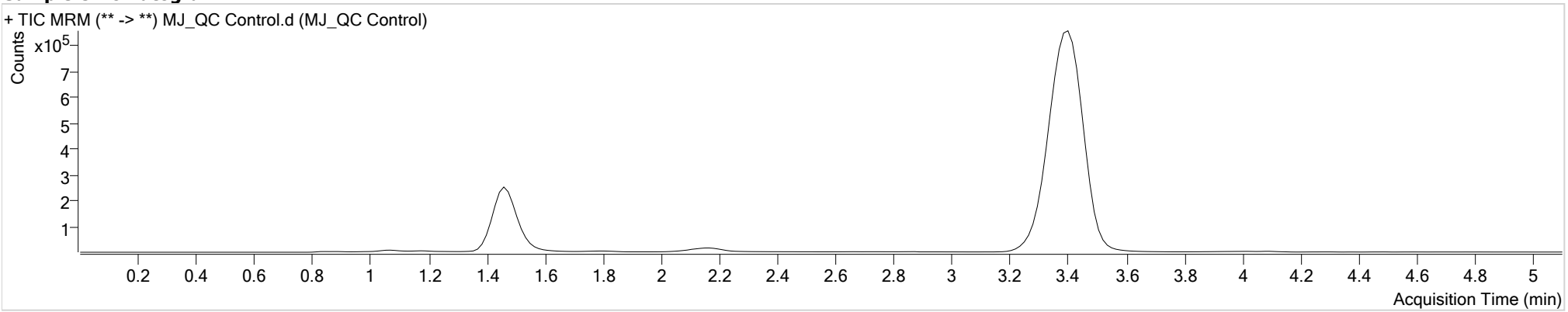
COMMENTS: *Curve Ranges: THC: 1-100, THC-COOH 5-250, THC-OH 3-100*

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update	10/23/2019 2:11:38 PM

Instrument	Falco	**Data File**	MJ_QC Control.d
Type	Sample	**Sample**	MJ_QC Control
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 12:29:41 PM		
Sample Info.			

Sample Chromatogram



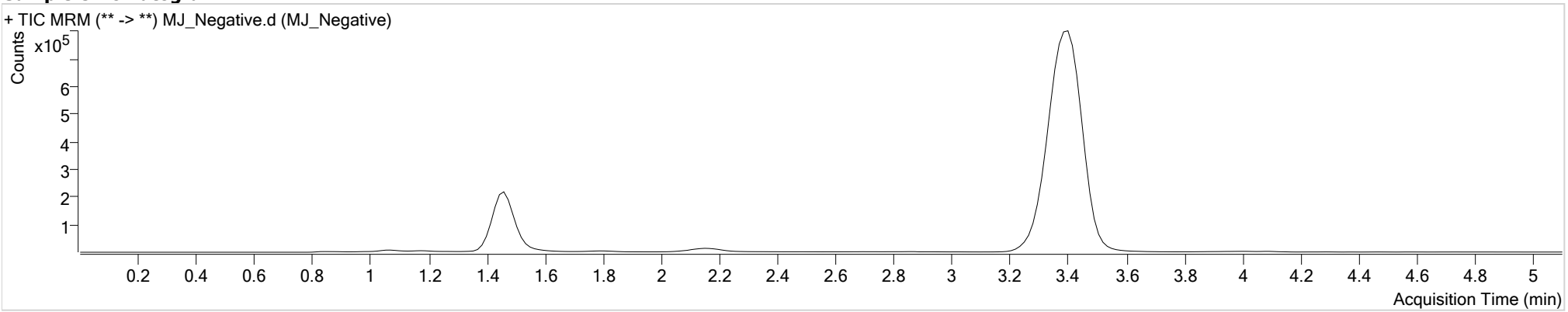
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.405	223830	1469.96	27.0	282.29	6870181	4.2290 ng/ml
THC-COOH	1.504	105990	250.84	56.9	702.40	303163	14.3486 ng/ml
THC-OH	1.468	72493	94.12	10.5	122.79	936480	4.4085 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update	10/23/2019 2:11:38 PM

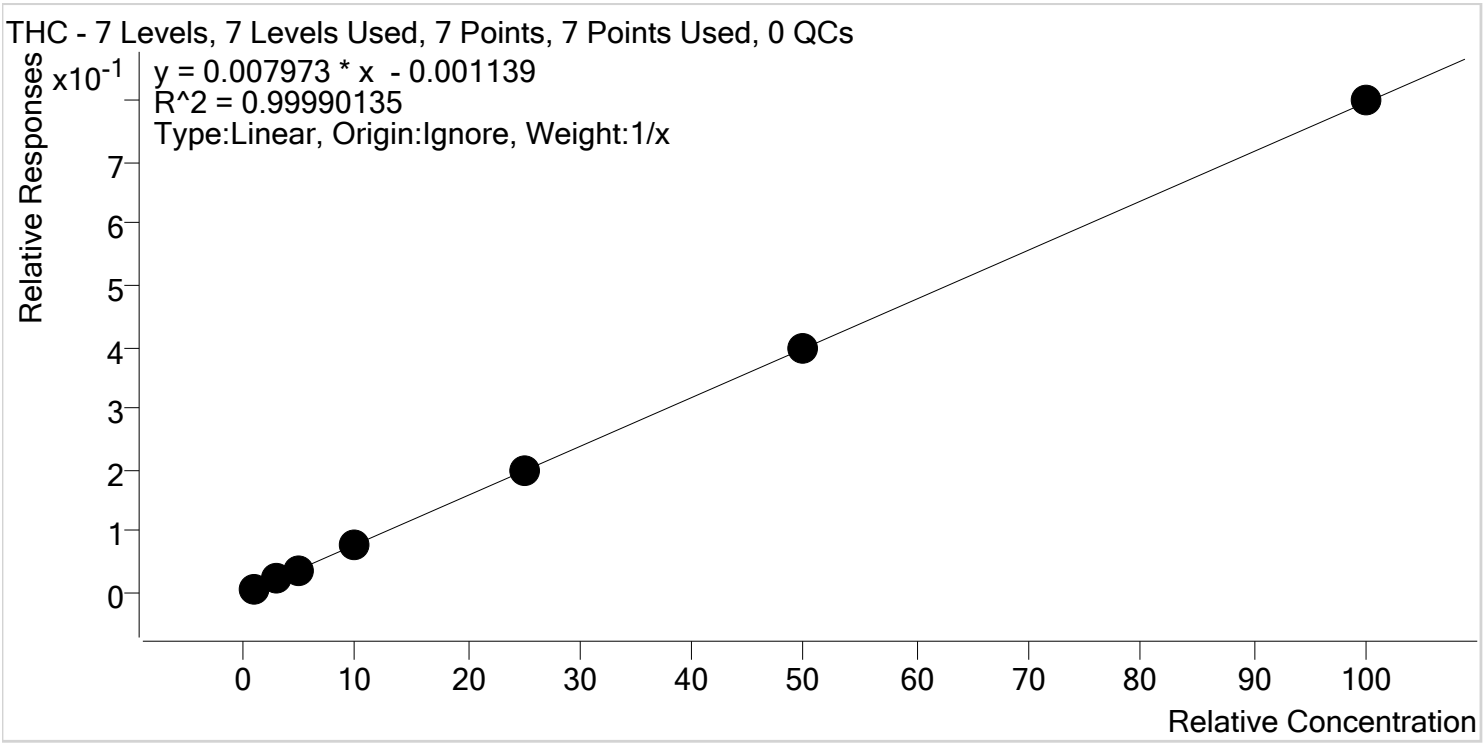
Instrument	Falco	**Data File**	MJ_Negative.d
Type	Sample	**Sample**	MJ_Negative
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 12:44:53 PM		
Sample Info.			

Sample Chromatogram



AM #27 Cannabinoids Quant. Calibration Curve Report

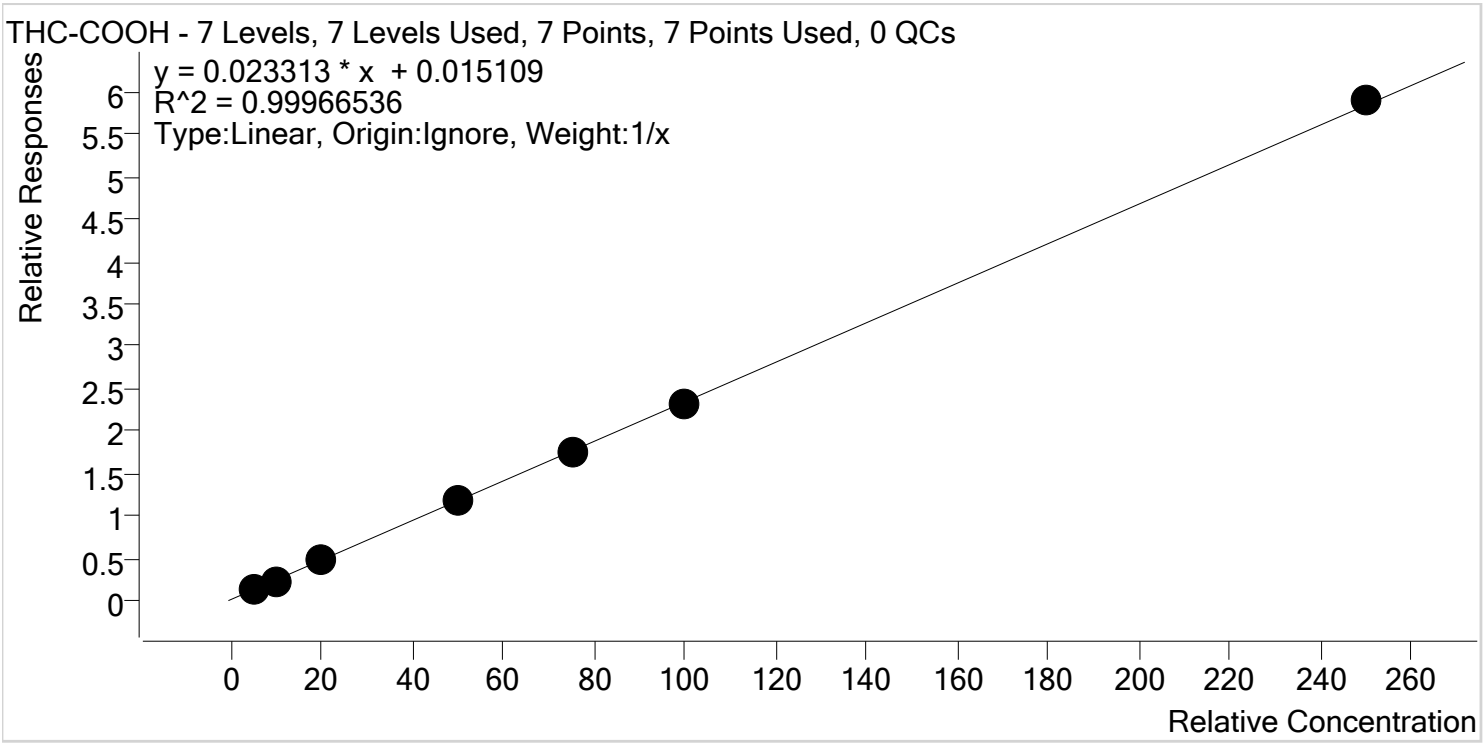
Batch results	D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Last Cal. Update	10/23/2019 2:11 PM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✔	1.0	1.1	106.8
MJ_Cal 2	2	✔	3.0	2.9	97.7
MJ_Cal 3	3	✔	5.0	4.8	96.5
MJ_Cal 4	4	✔	10.0	9.9	99.3
MJ_Cal 5	5	✔	25.0	24.8	99.1
MJ_Cal 6	6	✔	50.0	50.1	100.2
MJ_Cal 7	7	✔	100.0	100.4	100.4

AM #27 Cannabinoids Quant. Calibration Curve Report

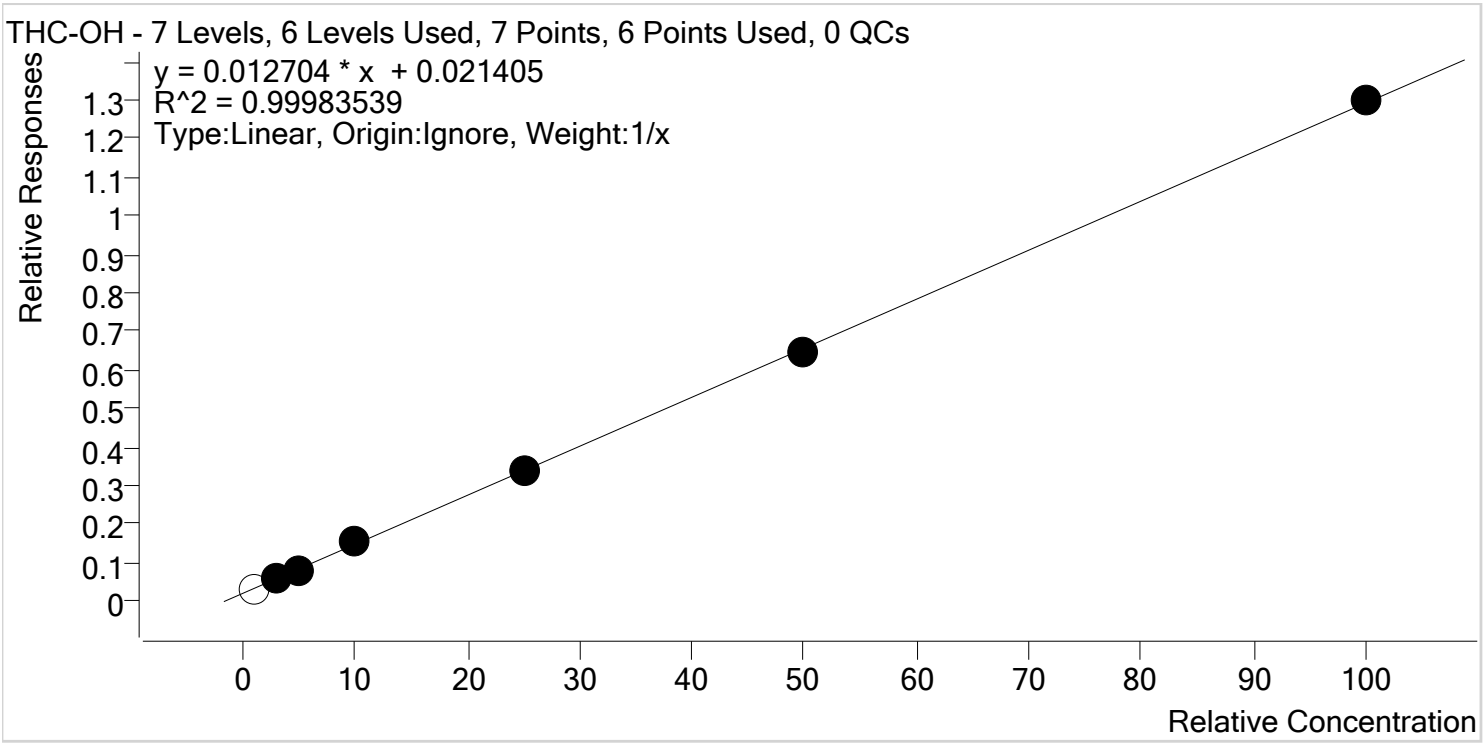
Batch results D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Last Cal. Update 10/23/2019 2:11 PM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✔	5.0	5.4	107.8
MJ_Cal 2	2	✔	10.0	9.5	94.6
MJ_Cal 3	3	✔	20.0	19.7	98.3
MJ_Cal 4	4	✔	50.0	50.6	101.3
MJ_Cal 5	5	✔	75.0	73.8	98.3
MJ_Cal 6	6	✔	100.0	98.8	98.8
MJ_Cal 7	7	✔	250.0	252.3	100.9

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Last Cal. Update	10/23/2019 2:11 PM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



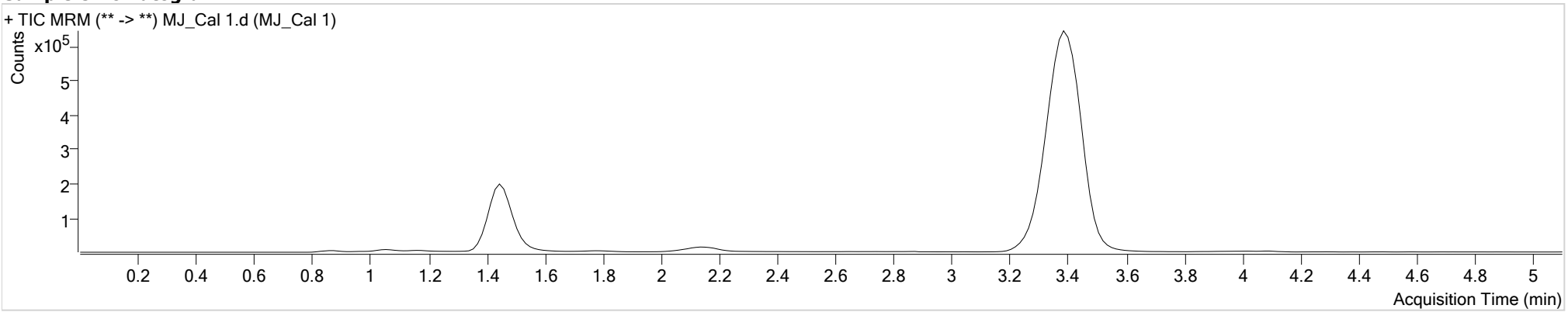
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✗	1.0	0.9	94.8
MJ_Cal 2	2	✔	3.0	3.0	100.4
MJ_Cal 3	3	✔	5.0	4.9	97.4
MJ_Cal 4	4	✔	10.0	10.3	103.3
MJ_Cal 5	5	✔	25.0	24.9	99.5
MJ_Cal 6	6	✔	50.0	49.4	98.9
MJ_Cal 7	7	✔	100.0	100.5	100.5

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update	10/23/2019 2:11:38 PM

Instrument	Falco	**Data File**	MJ_Cal 1.d
Type	Cal	**Sample**	MJ_Cal 1
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 11:28:48 AM		
Sample Info.			

Sample Chromatogram



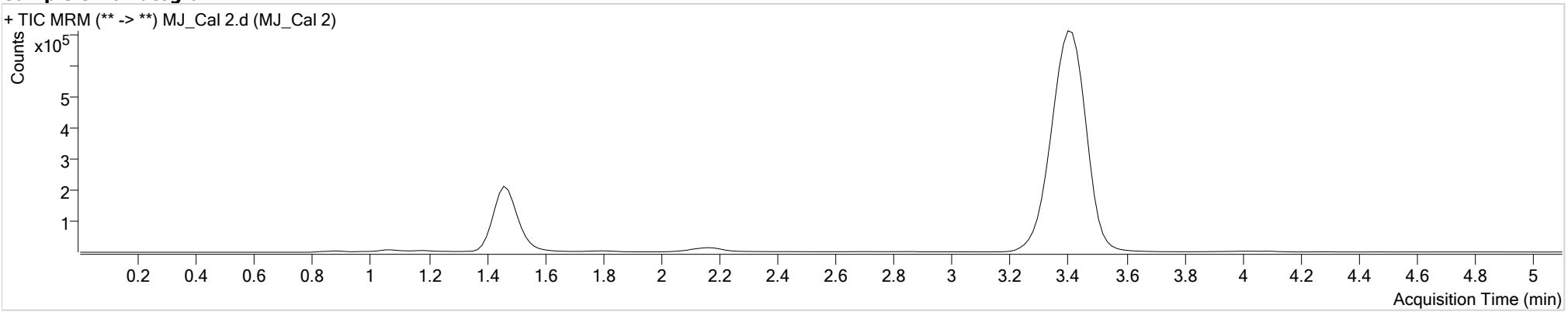
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.405	40203	159.62	29.7	22.90	5451096	1.0678 ng/ml
THC-COOH	1.489	38514	∞	48.9	162.75	273643	5.3891 ng/ml
THC-OH	1.498	27183	∞	7.2 **Low**	18.92	812678	0.9480 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update	10/23/2019 2:11:38 PM

Instrument	Falco	**Data File**	MJ_Cal 2.d
Type	Cal	**Sample**	MJ_Cal 2
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 11:36:34 AM		
Sample Info.			

Sample Chromatogram



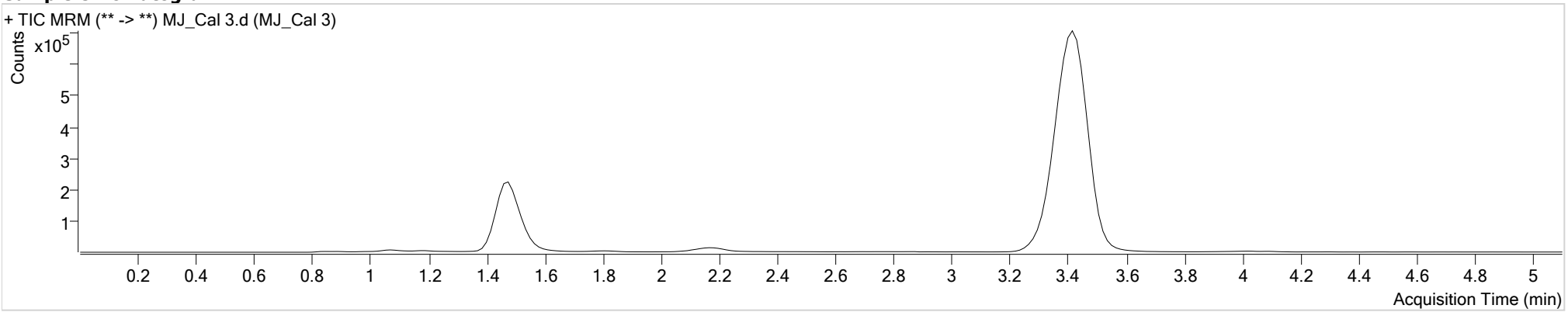
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.420	128767	348.41	27.2	130.25	5790107	2.9321 ng/ml
THC-COOH	1.504	64015	∞	57.4	334.55	271652	9.4601 ng/ml
THC-OH	1.483	48724	∞	10.1	34.64	816681	3.0113 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update	10/23/2019 2:11:38 PM

Instrument	Falco	**Data File**	MJ_Cal 3.d
Type	Cal	**Sample**	MJ_Cal 3
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 11:44:09 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.435	199637	1930.59	27.8	∞	5350136	4.8228 ng/ml
THC-COOH	1.519	123784	∞	58.2	657.53	261531	19.6543 ng/ml
THC-OH	1.483	65560	∞	11.1	131.36	787155	4.8712 ng/ml

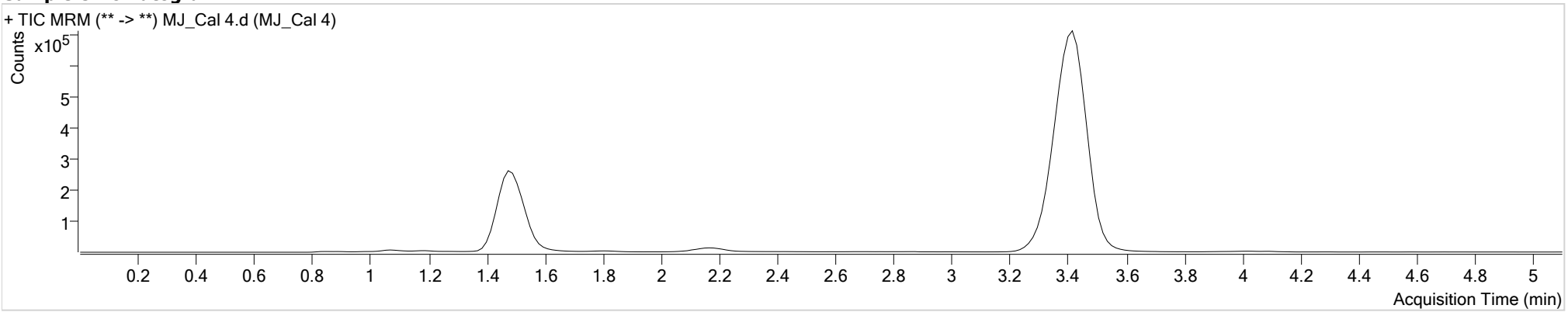
AM #27 Cannabinoids Quant. Results

S

Batch results	D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update	10/23/2019 2:11:38 PM

Instrument	Falco	**Data File**	MJ_Cal 4.d
Type	Cal	**Sample**	MJ_Cal 4
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-E6	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 11:51:44 AM		
Sample Info.			

Sample Chromatogram



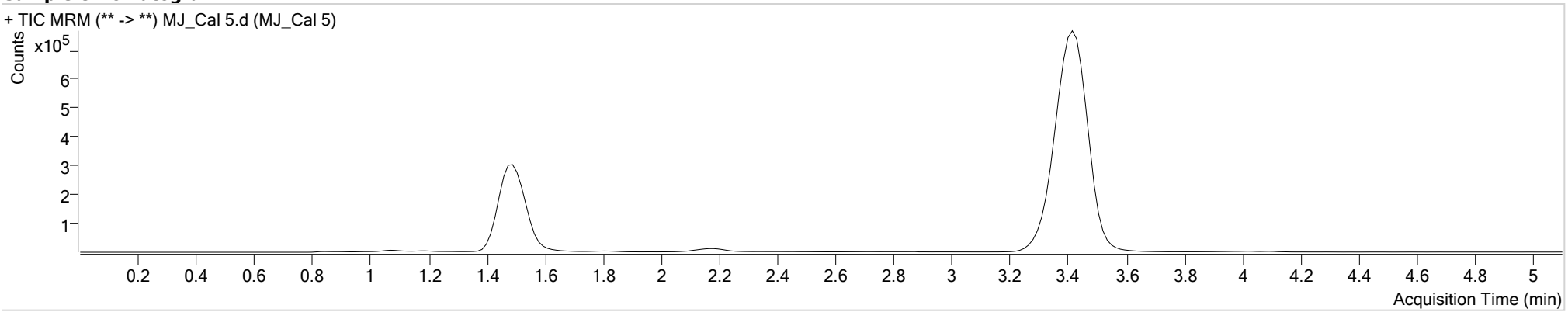
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.420	398305	1322.14	27.6	∞	5101833	9.9346 ng/ml
THC-COOH	1.519	299140	1558.22	60.6	1064.61	250194	50.6384 ng/ml
THC-OH	1.483	116127	∞	12.0	210.23	760678	10.3322 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update 10/23/2019 2:11:38 PM

Instrument	Falco	**Data File**	MJ_Cal 5.d
Type	Cal	**Sample**	MJ_Cal 5
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 11:59:18 AM		
Sample Info.			

Sample Chromatogram



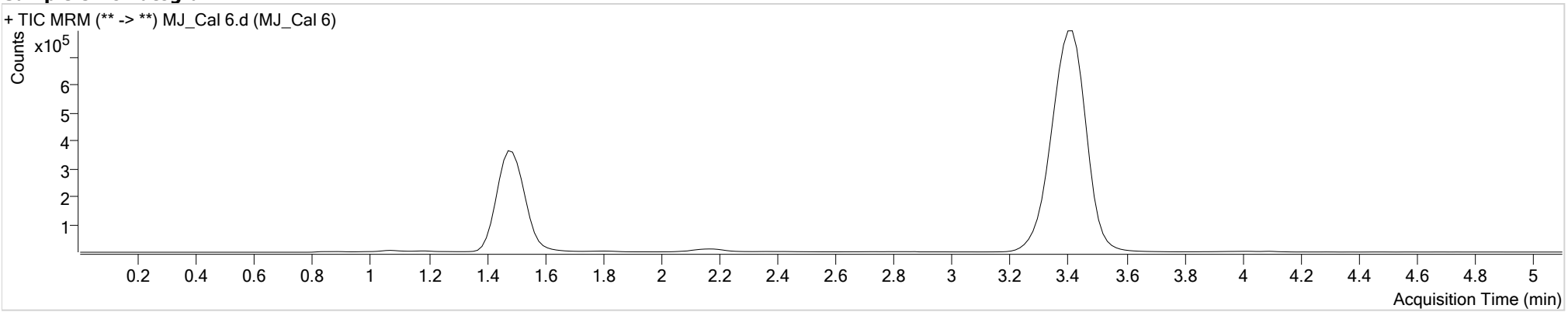
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.435	952737	5330.20	26.6	∞	4849981	24.7807 ng/ml
THC-COOH	1.519	409715	∞	61.2	4934.40	236192	73.7604 ng/ml
THC-OH	1.483	241469	∞	13.1	544.30	715419	24.8837 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update 10/23/2019 2:11:38 PM

Instrument	Falco	**Data File**	MJ_Cal 6.d
Type	Cal	**Sample**	MJ_Cal 6
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 12:06:53 PM		
Sample Info.			

Sample Chromatogram



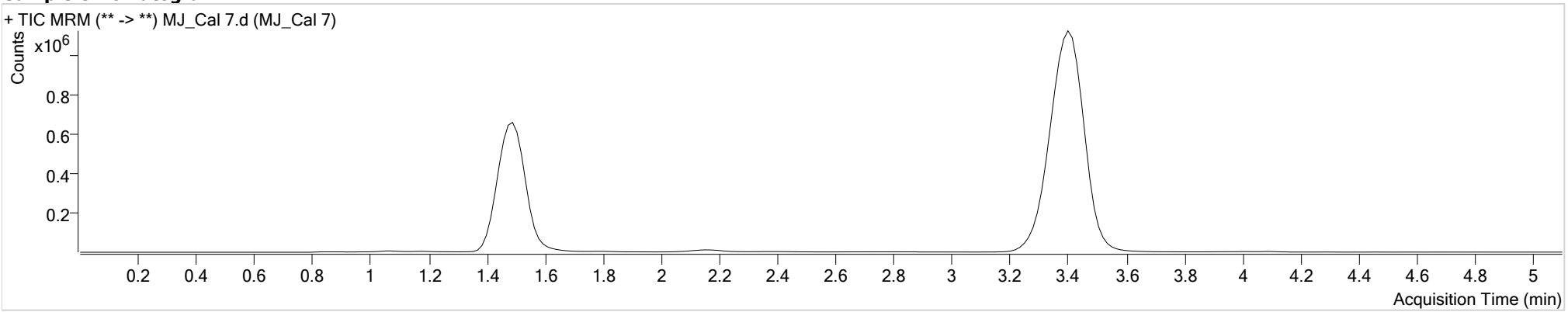
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.420	1747428	12123.15	26.7	∞	4387639	50.0932 ng/ml
THC-COOH	1.504	532904	∞	61.2	4476.51	229850	98.8029 ng/ml
THC-OH	1.468	454076	∞	13.7	872.73	699143	49.4398 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results D:\MassHunter\Data\2019\AM 28\102219 MDQ THCQ SP\QuantResults\THCQ.batch.bin
Calibration Last Update 10/23/2019 2:11:38 PM

Instrument	Falco	**Data File**	MJ_Cal 7.d
Type	Cal	**Sample**	MJ_Cal 7
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	10/23/2019 12:14:29 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.405	3679621	13614.02	26.8	1476.59	4604612	100.3688 ng/ml
THC-COOH	1.504	1297062	∞	61.5	3249.22	219960	252.2948 ng/ml
THC-OH	1.468	917305	∞	14.1	1390.48	706899	100.4618 ng/ml