

REVIEWED

By Anne Nord at 11:18 am, Oct 31, 2019

10/30/2019

Worklist: 3798

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
C2019-1739	2	UCK	AM 27 Blood THC Quant by LC-QQQ	
C2019-1770	2	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2627	1	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2658	1	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2659	3	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2734	1	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2772	1	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2797	1	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2885	1	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2893	1	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2915	1	UCK	AM 27 Blood THC Quant by LC-QQQ	
P2019-2934	1	UCK	AM 27 Blood THC Quant by LC-QQQ	

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

Extraction Date: 10/11/2019
 Plate lot#: 190716

Analyst: Celena Shrum
 Plate Expiration: 01/16/2020

Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: 445283-3
LCMS-QQQ ID: 069901

Mobile phase B: 0.1% Formic acid in Acetonitrile
Column: UCT Selectra DA 100 x 2.1mm 3um
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: #3**
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 067105*
- 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: 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? (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 ranges: THC: 1-100, THC-OH: 3-100, THC-COOH: 5-250



Idaho State Police Forensic Services

AM #27 Quantitative Analysis of THC and Metabolites in Blood and Urine by LCMS-QQQ

Methanol External Control Solution (Lot: WS041619)

10 ul of 1mg/mL THC, 100 ul of 100 ug/mL THC-OH, C-THC in 9790 ul MeOH
Approximate concentration 1ug/mL.

Component	Source	Source Lot Number	Expiration Date
Methanol (LCMS)	Fisher	184782	
THC	Cerilliant	FE09101501	11/30/2020
C-THC	Cerilliant	FE07171501	09/30/2020
THC-OH	Cerilliant	FE01121503	01/31/2020
Prepared:	04/16/2019		
Prepared By:	Tamara Salazar		
Expires:	01/31/2020		

Urine External Control Solution (Lot: 100719)

100 ul of methanol external control solution was added to 9900 ul of urine.
Approximately 10ng/mL of each compound.

Component	Source	Source Lot Number
Negative Urine	Pocatello Lab	POC031319
Methanol External Control Solution	-	WS041619
Prepared:	10/07/19	
Prepared by:	Celena Shrum	
Expires:	01/31/2020	

AM #27 Cannabinoids Quant. Results

**Batch results**

D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

10/29/2019 10:23:28 AM

Instrument

Falco

Data File

QC Control.d

Type

Sample

Sample

QC Control

Acq. Method

AM 27 THC quant.m

Sample Position

P3-H1

Comment**Injection Volume**

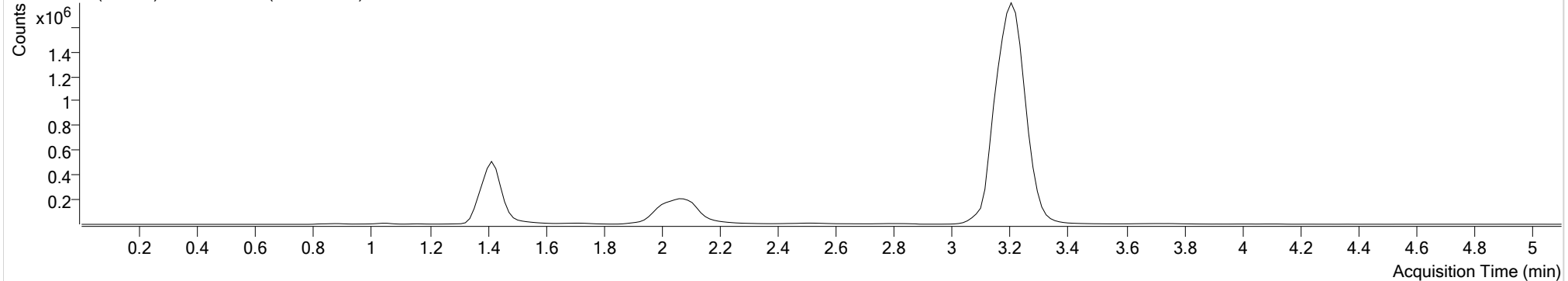
10

Acq. Date-Time

10/11/2019 3:01:29 PM

Sample Info.**Sample Chromatogram**

+ TIC MRM (** -> **) QC Control.d (QC Control)



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.224	470879	1849.04	26.9	∞	12520117	4.4598 ng/ml
THC-COOH	1.444	156260	∞	61.4	∞	421066	14.7831 ng/ml
THC-OH	1.423	132633	423.22	10.9	∞	1756075	4.4634 ng/ml

AM #27 Cannabinoids Quant. Results

**Batch results**

D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

10/29/2019 10:23:28 AM

Instrument

Falco

Data File

Blood Negative.d

Type

Sample

Sample

Blood Negative

Acq. Method

AM 27 THC quant.m

Sample Position

P3-A2

Comment**Injection Volume**

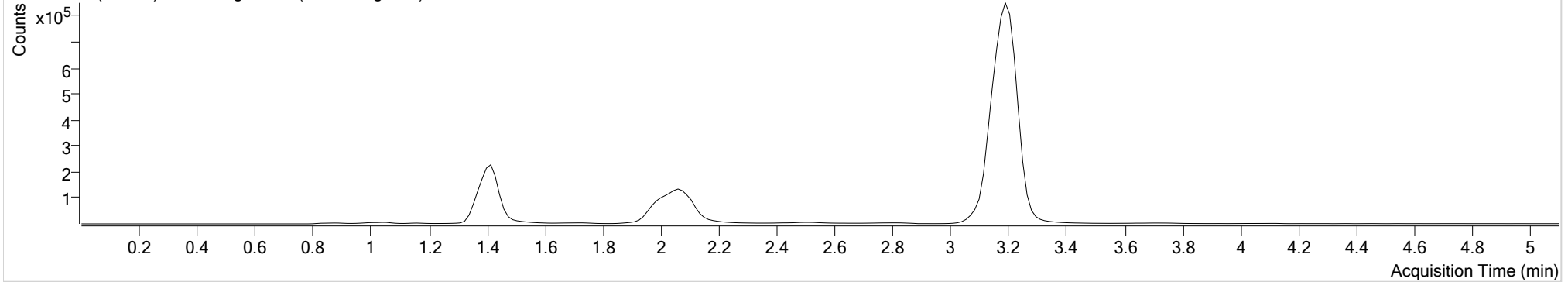
10

Acq. Date-Time

10/11/2019 3:16:40 PM

Sample Info.**Sample Chromatogram**

+ TIC MRM (** -> **) Blood Negative.d (Blood Negative)



AM #27 Cannabinoids Quant. Results



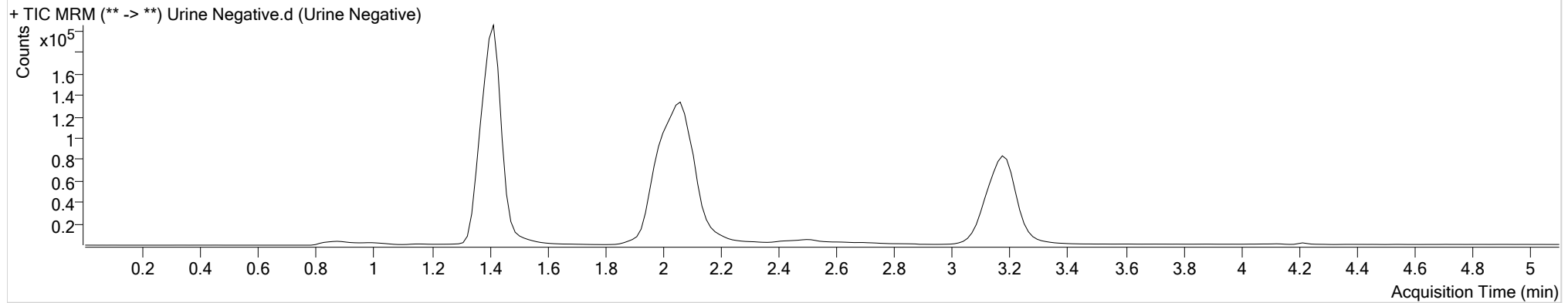
Batch results
Calibration Last Update

D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin
10/29/2019 10:23:28 AM

Instrument Type Falco
Acq. Method AM 27 THC quant.m
Sample Position P3-B2
Injection Volume 10
Acq. Date-Time 10/11/2019 3:31:53 PM
Sample Info.

Data File Urine Negative.d
Sample Urine Negative
Comment

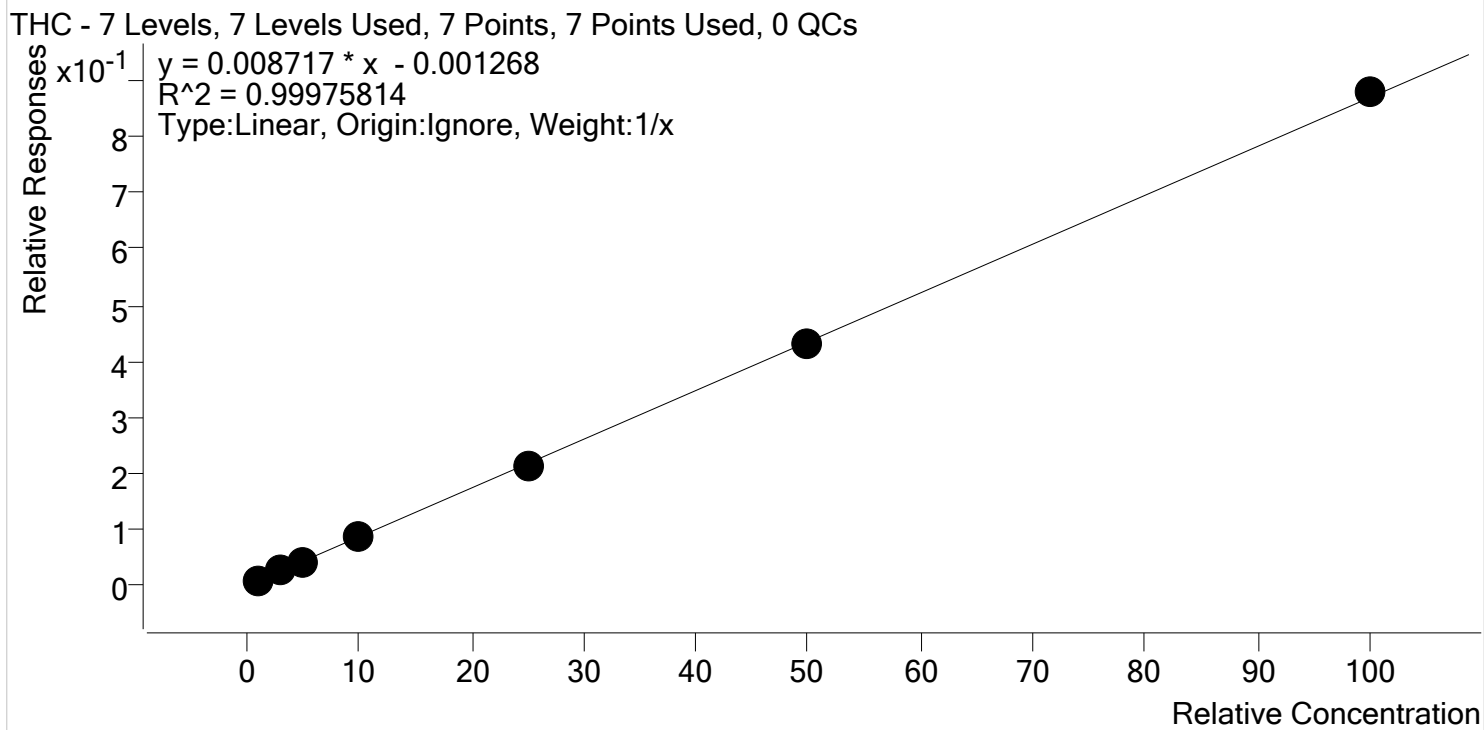
Sample Chromatogram





AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty
 CS\QuantResults\THCQ CS.batch.bin
Last Cal. Update 10/29/2019 10:23 AM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-D3

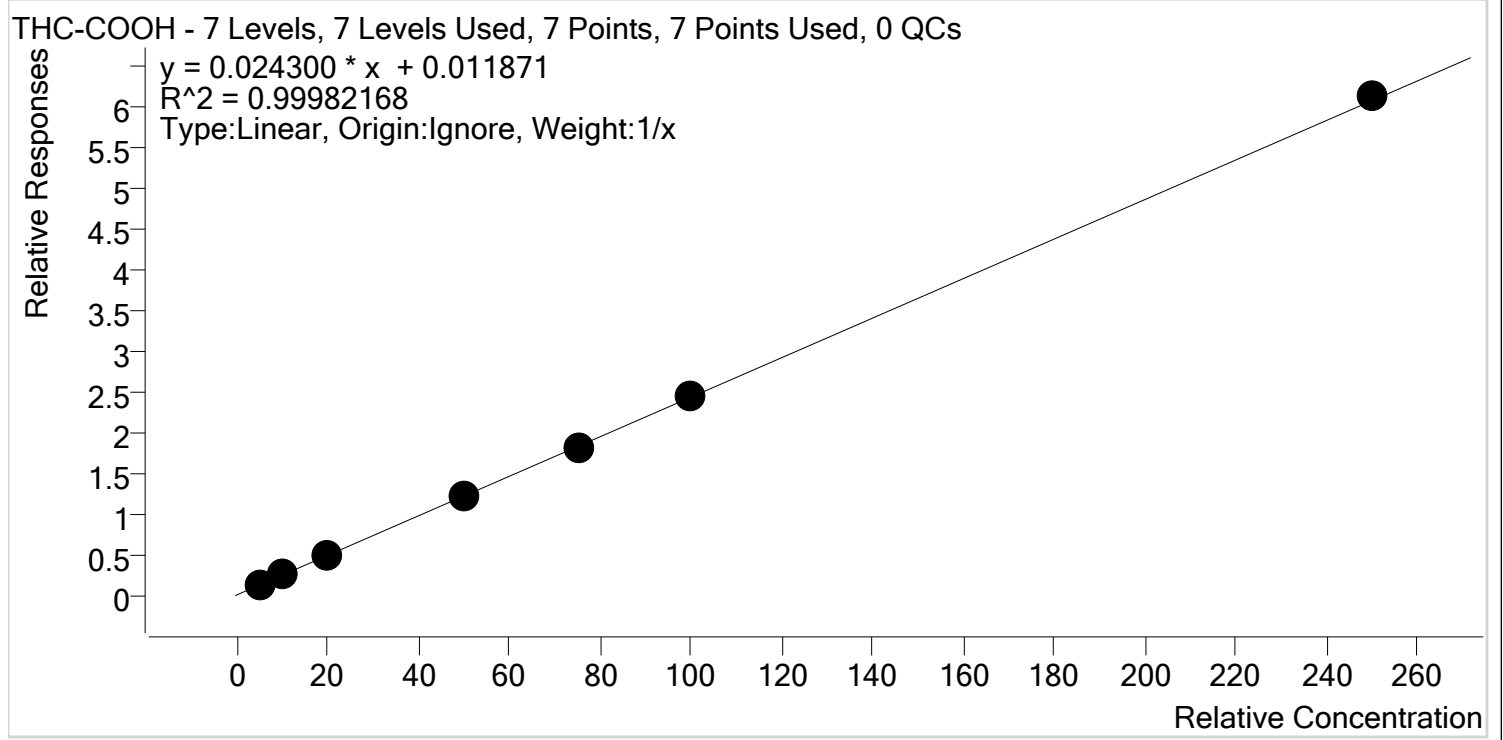


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1	1	✓	1.0	1.1	107.8
Cal 2	2	✓	3.0	2.9	97.9
Cal 3	3	✓	5.0	4.9	97.9
Cal 4	4	✓	10.0	9.8	98.1
Cal 5	5	✓	25.0	24.4	97.6
Cal 6	6	✓	50.0	49.8	99.6
Cal 7	7	✓	100.0	101.1	101.1



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty
 CS\QuantResults\THCQ CS.batch.bin
Last Cal. Update 10/29/2019 10:23 AM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9

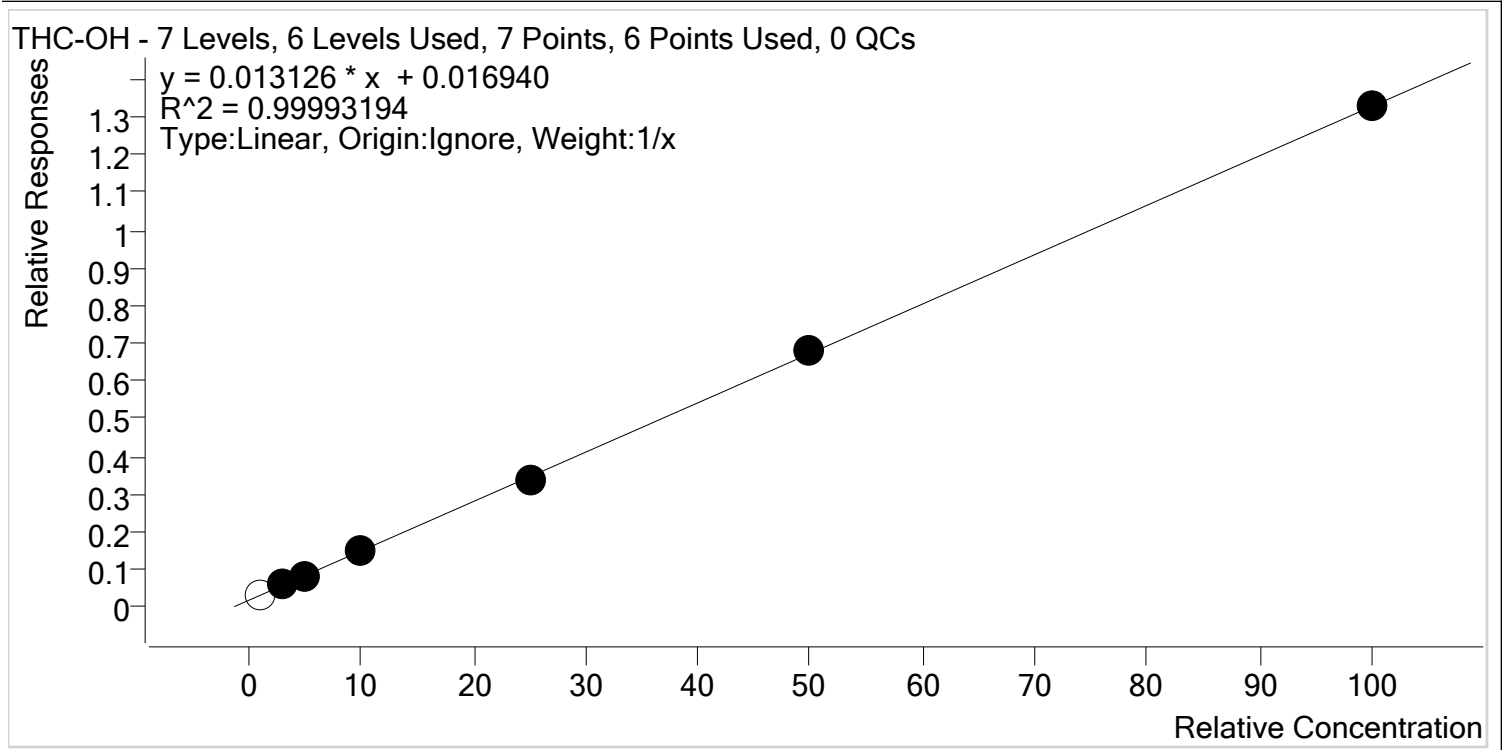


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1	1	✓	5.0	5.2	104.4
Cal 2	2	✓	10.0	10.0	99.8
Cal 3	3	✓	20.0	19.6	98.0
Cal 4	4	✓	50.0	49.4	98.8
Cal 5	5	✓	75.0	73.6	98.2
Cal 6	6	✓	100.0	100.0	100.0
Cal 7	7	✓	250.0	252.2	100.9



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty
 CS\QuantResults\THCQ CS.batch.bin
Last Cal. Update 10/29/2019 10:23 AM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1	1	x	1.0	1.2	120.2
Cal 2	2	✓	3.0	3.0	99.0
Cal 3	3	✓	5.0	5.1	102.0
Cal 4	4	✓	10.0	10.0	99.8
Cal 5	5	✓	25.0	24.6	98.5
Cal 6	6	✓	50.0	50.3	100.6
Cal 7	7	✓	100.0	100.0	100.0

AM #27 Cannabinoids Quant. Results



Batch results

D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

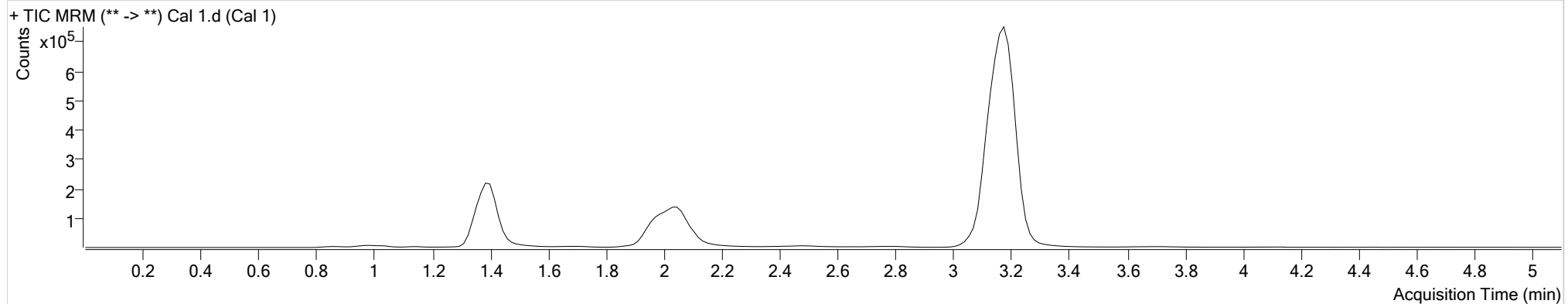
10/29/2019 10:23:28 AM

Instrument Falco
Type Cal
Acq. Method AM 27 THC quant.m
Sample Position P3-A1
Injection Volume 10
Acq. Date-Time 10/11/2019 2:08:14 PM
Sample Info.

Data File Cal 1.d
Sample Cal 1

Comment

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.194	40601	350.22	30.3	74.94	4994660	1.0780 ng/ml
THC-COOH	1.414	35970	∞	50.7	275.80	259317	5.2196 ng/ml
THC-OH	1.408	27349	∞	6.5 Low	16.17	836050	1.2015 ng/ml

AM #27 Cannabinoids Quant. Results

**Batch results**

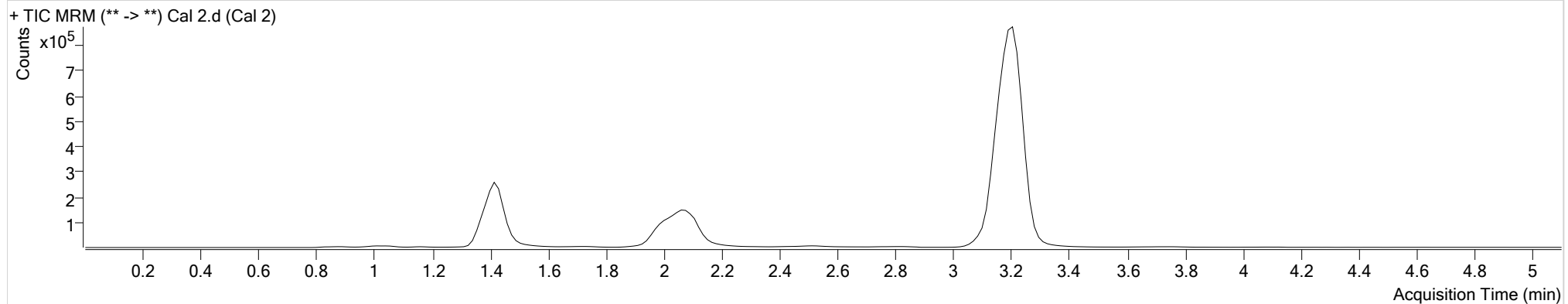
D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

10/29/2019 10:23:28 AM

Instrument Type Falco
Acq. Method AM 27 THC quant.m
Sample Position P3-B1
Injection Volume 10
Acq. Date-Time 10/11/2019 2:15:59 PM
Sample Info.

Data File Cal 2.d
Sample Cal 2

Comment**Sample Chromatogram**

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.209	133332	636.95	27.4	288.83	5480249	2.9364 ng/ml
THC-COOH	1.444	68077	553.97	57.2	1459.91	267718	9.9757 ng/ml
THC-OH	1.423	49477	55.92	9.8	64.14	884877	2.9691 ng/ml

AM #27 Cannabinoids Quant. Results

**Batch results**

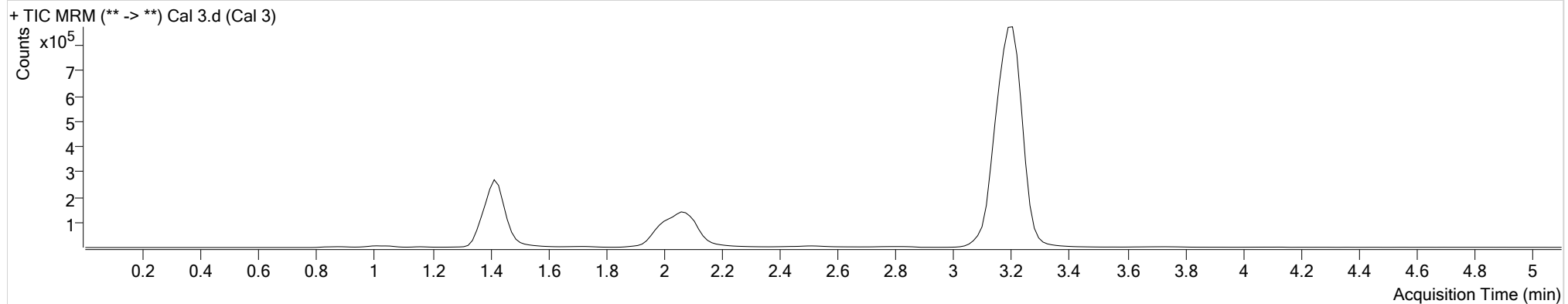
D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

10/29/2019 10:23:28 AM

Instrument Falco
Type Cal
Acq. Method AM 27 THC quant.m
Sample Position P3-C1
Injection Volume 10
Acq. Date-Time 10/11/2019 2:23:33 PM
Sample Info.

Data File Cal 3.d
Sample Cal 3

Comment**Sample Chromatogram**

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.209	224913	7608.72	27.0	457.02	5431259	4.8958 ng/ml
THC-COOH	1.444	126455	1000.33	59.6	1299.03	258998	19.6036 ng/ml
THC-OH	1.423	70791	∞	11.6	289.97	843589	5.1024 ng/ml

AM #27 Cannabinoids Quant. Results

**Batch results**

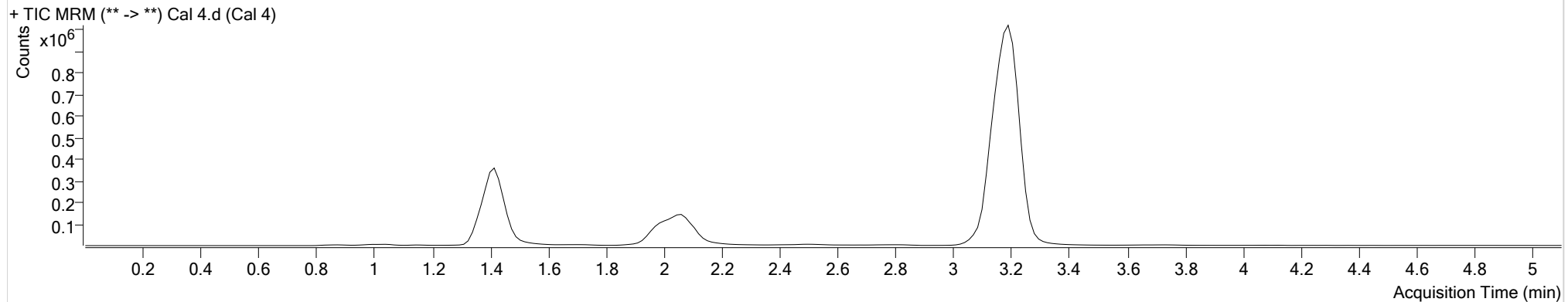
D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

10/29/2019 10:23:28 AM

Instrument Type Falco
Acq. Method AM 27 THC quant.m
Sample Position P3-D1
Injection Volume 10
Acq. Date-Time 10/11/2019 2:31:10 PM
Sample Info.

Data File Cal 4.d
Sample Cal 4

Comment**Sample Chromatogram**

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.209	509616	4749.91	26.5	543.62	6046300	9.8141 ng/ml
THC-COOH	1.429	334082	707.83	62.0	12477.76	275542	49.4060 ng/ml
THC-OH	1.408	138906	∞	12.0	427.84	938755	9.9820 ng/ml

AM #27 Cannabinoids Quant. Results

**Batch results**

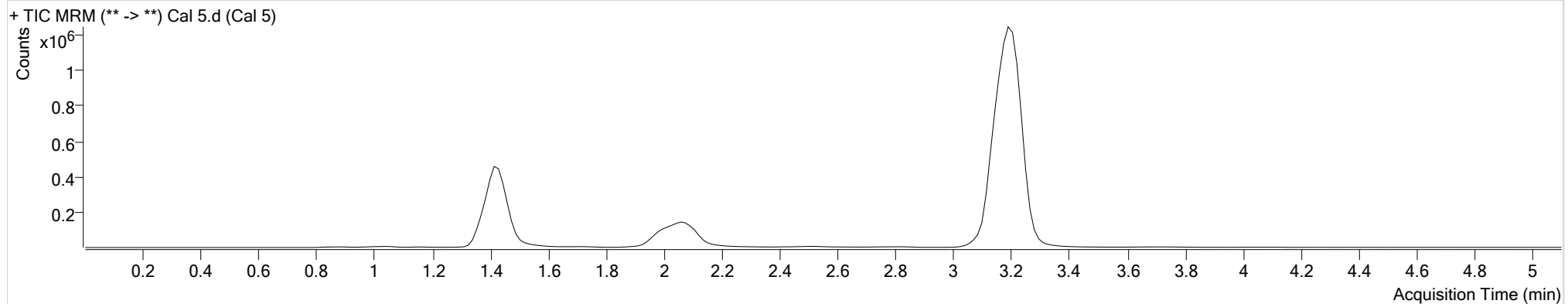
D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

10/29/2019 10:23:28 AM

Instrument Type Falco
Acq. Method AM 27 THC quant.m
Sample Position P3-E1
Injection Volume 10
Acq. Date-Time 10/11/2019 2:38:46 PM
Sample Info.

Data File Cal 5.d
Sample Cal 5

Comment**Sample Chromatogram**

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.209	1388886	7194.99	26.4	∞	6570098	24.3951 ng/ml
THC-COOH	1.444	505180	∞	62.0	8694.40	280485	73.6292 ng/ml
THC-OH	1.423	329296	683.19	13.4	960.32	967608	24.6359 ng/ml

AM #27 Cannabinoids Quant. Results

**Batch results**

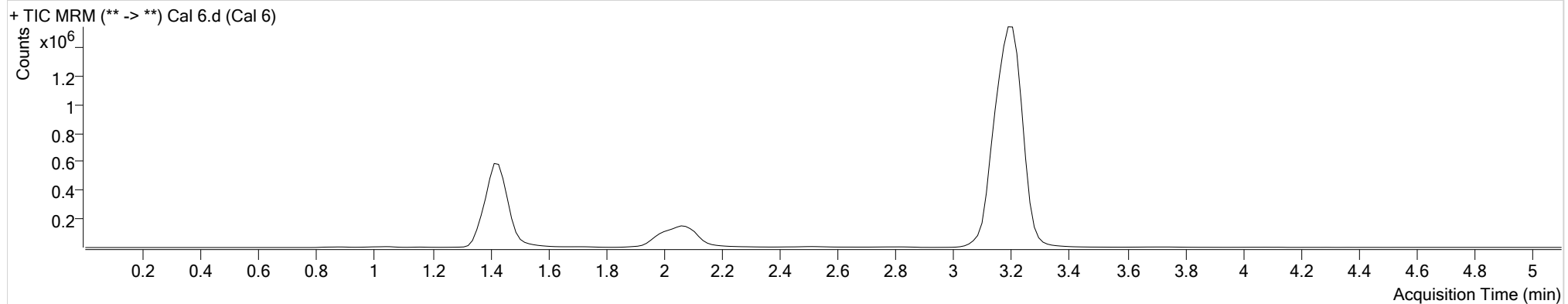
D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

10/29/2019 10:23:28 AM

Instrument Falco
Type Cal
Acq. Method AM 27 THC quant.m
Sample Position P3-F1
Injection Volume 10
Acq. Date-Time 10/11/2019 2:46:20 PM
Sample Info.

Data File Cal 6.d
Sample Cal 6

Comment**Sample Chromatogram**

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.209	2938878	36383.50	26.8	∞	6788539	49.8066 ng/ml
THC-COOH	1.444	686586	5050.03	61.8	3906.99	281251	99.9702 ng/ml
THC-OH	1.423	670032	∞	13.7	2657.42	989261	50.3083 ng/ml

AM #27 Cannabinoids Quant. Results

**Batch results**

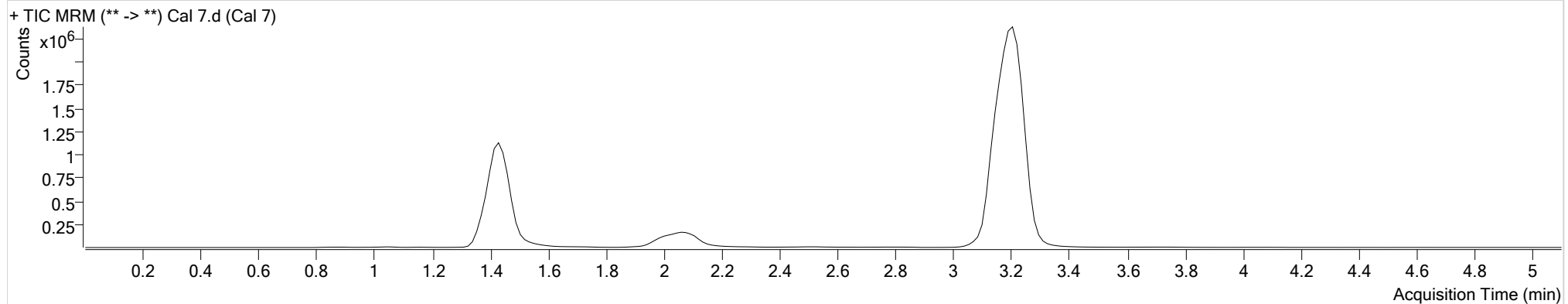
D:\MassHunter\Data\2019\AM 28\101119 MDQ THCQ and MDQ Uncertainty CS\QuantResults\THCQ CS.batch.bin

Calibration Last Update

10/29/2019 10:23:28 AM

Instrument Falco
Type Cal
Acq. Method AM 27 THC quant.m
Sample Position P3-G1
Injection Volume 10
Acq. Date-Time 10/11/2019 2:53:55 PM
Sample Info.

Data File Cal 7.d
Sample Cal 7

Comment**Sample Chromatogram**

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.209	6965539	23940.80	26.9	∞	7916838	101.0741 ng/ml
THC-COOH	1.444	1776390	∞	63.1	12517.01	289299	252.1957 ng/ml
THC-OH	1.423	1468791	∞	13.4	1130.41	1104680	100.0023 ng/ml