REVIEWED
By Anne Nord at 11:23 am, Jun 11, 2019

6/6/2019

Worklist: 3445

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
M2019-2094	1	153733	AM 27 Blood THC Quant by LC-QQQ
M2019-2231	3	153734	AM 27 Blood THC Quant by LC-QQQ
P2019-1302	1	153735	AM 27 Blood THC Quant by LC-QQQ
P2019-1424	1	153736	AM 27 Blood THC Quant by LC-QQQ
P2019-1425	1	153737	AM 27 Blood THC Quant by LC-QQQ
P2019-1426	2	153738	AM 27 Blood THC Quant by LC-QQQ
P2019-1463	1	153739	AM 27 Blood THC Quant by LC-QQQ
P2019-1467	1	153740	AM 27 Blood THC Quant by LC-QQQ
P2019-1491	1	153741	AM 27 Blood THC Quant by LC-QQQ
P2019-1498	1	153742	AM 27 Blood THC Quant by LC-QQQ
P2019-1511	1	153743	AM 27 Blood THC Quant by LC-QQQ
P2019-1515	1	153744	AM 27 Blood THC Quant by LC-QQQ

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

Extraction Date: 6/3/19 Analyst: Sarah Pickle
Plate lot#: 0539904 Plate Expiration: 9/10/19

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: 445283-2 **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\060319 THCQ SP TS Batch Name: THCQ SP
☒ 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 Range limited: THC-COOH 5-100*

Idaho State Police Forensic Services

AM #27 Quantitative Analysis of THC and Metabolites in Blood 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	09/30/2019
THC-OH	Cerilliant	FE01121503	01/31/2020
Prepared:	04/16/2019		
Prepared By:	Tamara Salazar		
Expires:	01/31/2020		

Blood External Control Solution (Lot: 041619)

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

Component	*Source*	*Source Lot Number*
Negative Blood	Hemostat	445283-1
Methanol External Control Solution	-	WS041619
Prepared:	04/16/19	
Prepared by:	Tamara Salazar	
Expires:	01/31/2020	

AM #27 Cannabinoids Quant. Results

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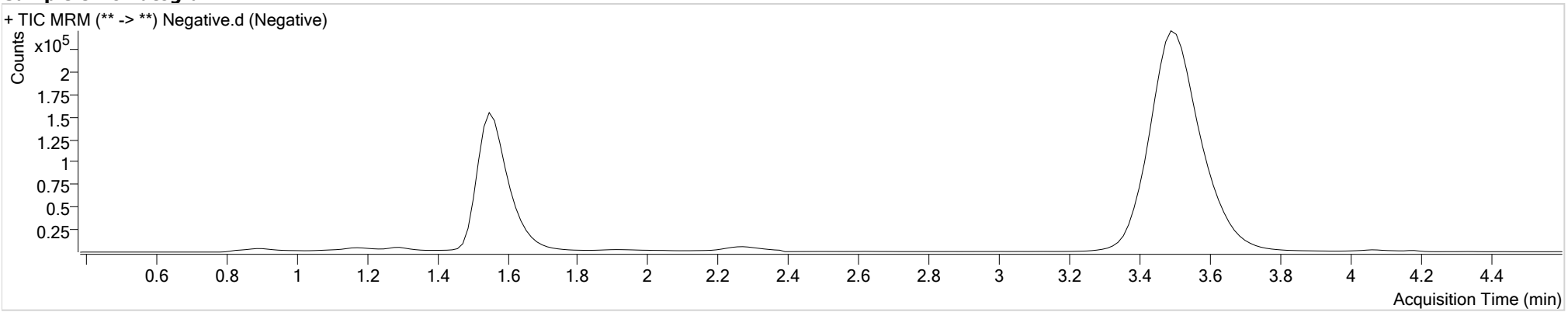
Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/7/2019 10:20:25 AM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	Negative.d
Type	Sample	**Sample**	Negative
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-A5	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 12:09:35 PM		
Sample Info.			

Sample Chromatogram

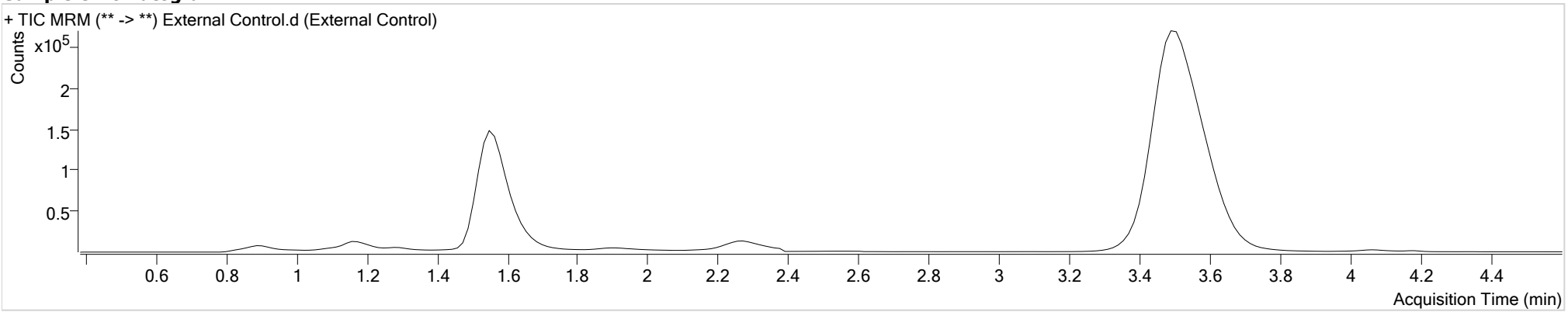


AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/7/2019 10:20:25 AM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	External Control.d
Type	Sample	**Sample**	External Control
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-B5	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 12:24:46 PM		
Sample Info.			

Sample Chromatogram



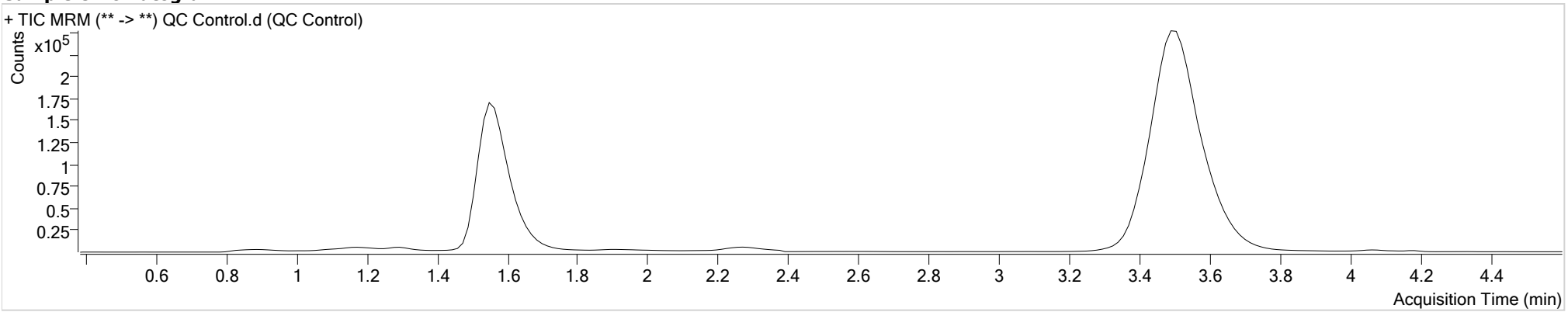
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.506	158066	619.60	28.6	143.57	2569463	7.7360 ng/ml
THC-COOH	1.580	34176	104.49	50.0	87.21	186079	8.4337 ng/ml
THC-OH	1.558	80184	∞	12.4	254.49	602267	8.5866 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/10/2019 1:49:13 PM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	QC Control.d
Type	Sample	**Sample**	QC Control
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-H4	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 11:54:24 AM		
Sample Info.			

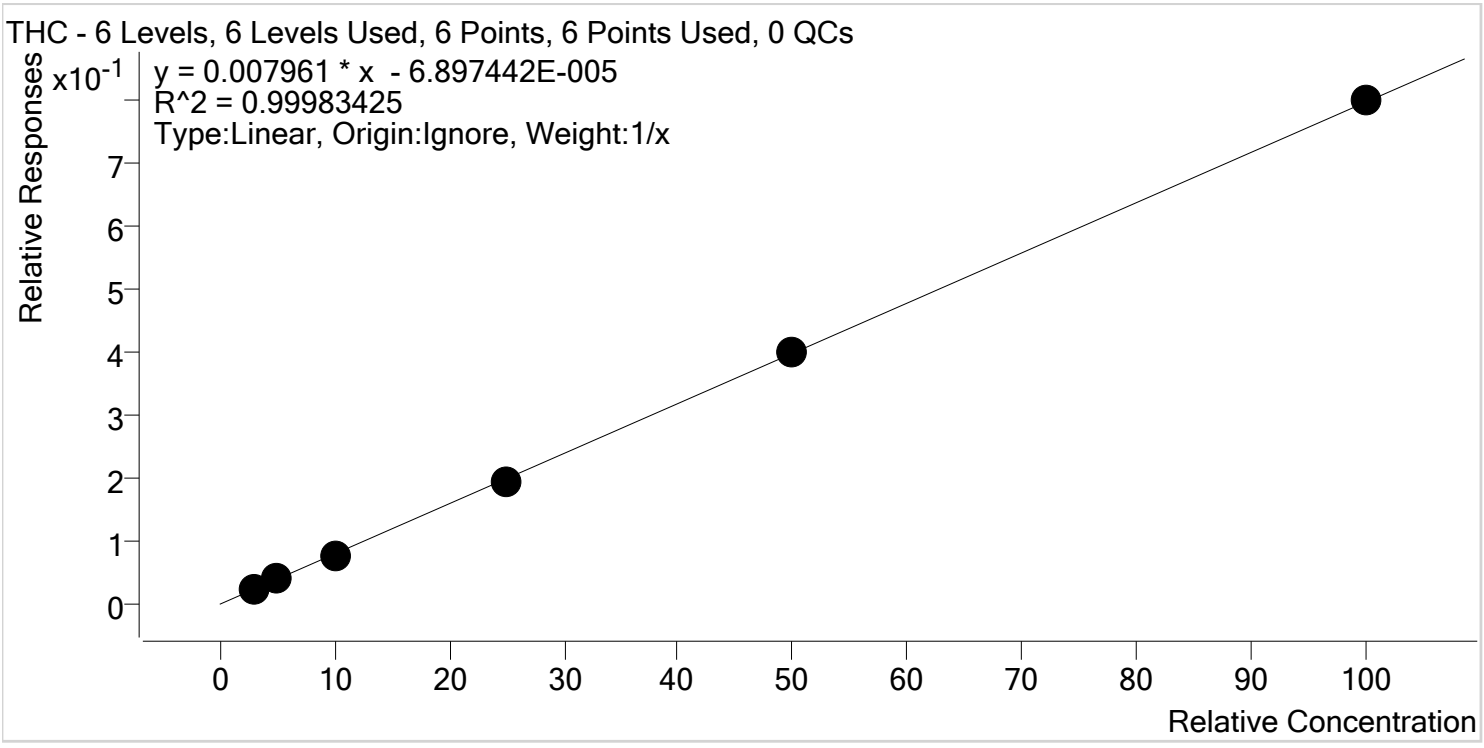
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.506	95272	661.46	28.4	∞	2432603	4.9282 ng/ml
THC-COOH	1.580	42221	175.16	52.8	425.16	209701	9.3542 ng/ml
THC-OH	1.558	59001	152.02	11.8	71.82	732008	4.7568 ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

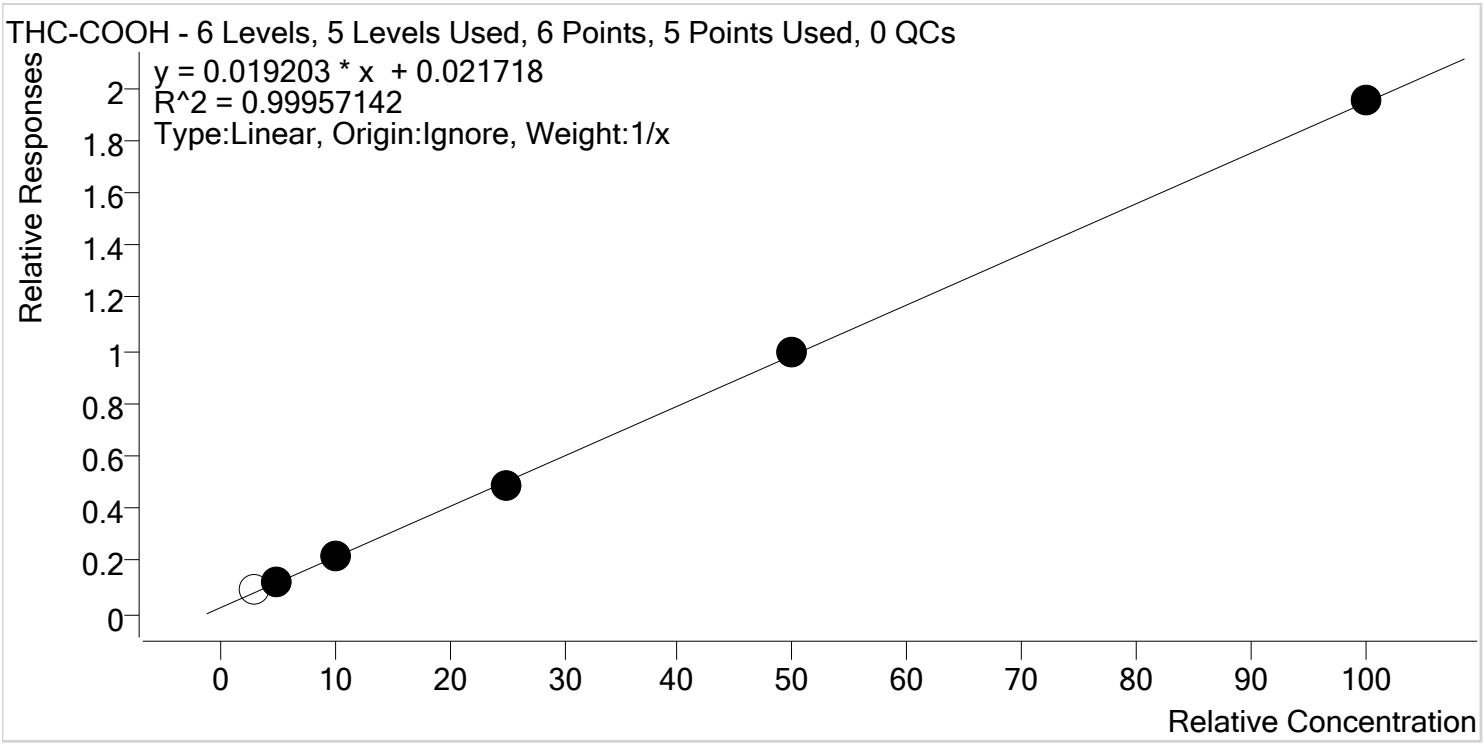
Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Last Cal. Update	6/7/2019 10:20 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1-3ng	1	✔	3.0	3.1	103.6
Cal 2- 5ng	2	✔	5.0	5.0	99.5
Cal 3 -10ng	3	✔	10.0	9.8	98.0
Cal 4-25ng	4	✔	25.0	24.5	97.8
Cal 5-50ng	5	✔	50.0	50.4	100.8
Cal 6-100ng	6	✔	100.0	100.3	100.3

AM #27 Cannabinoids Quant. Calibration Curve Report

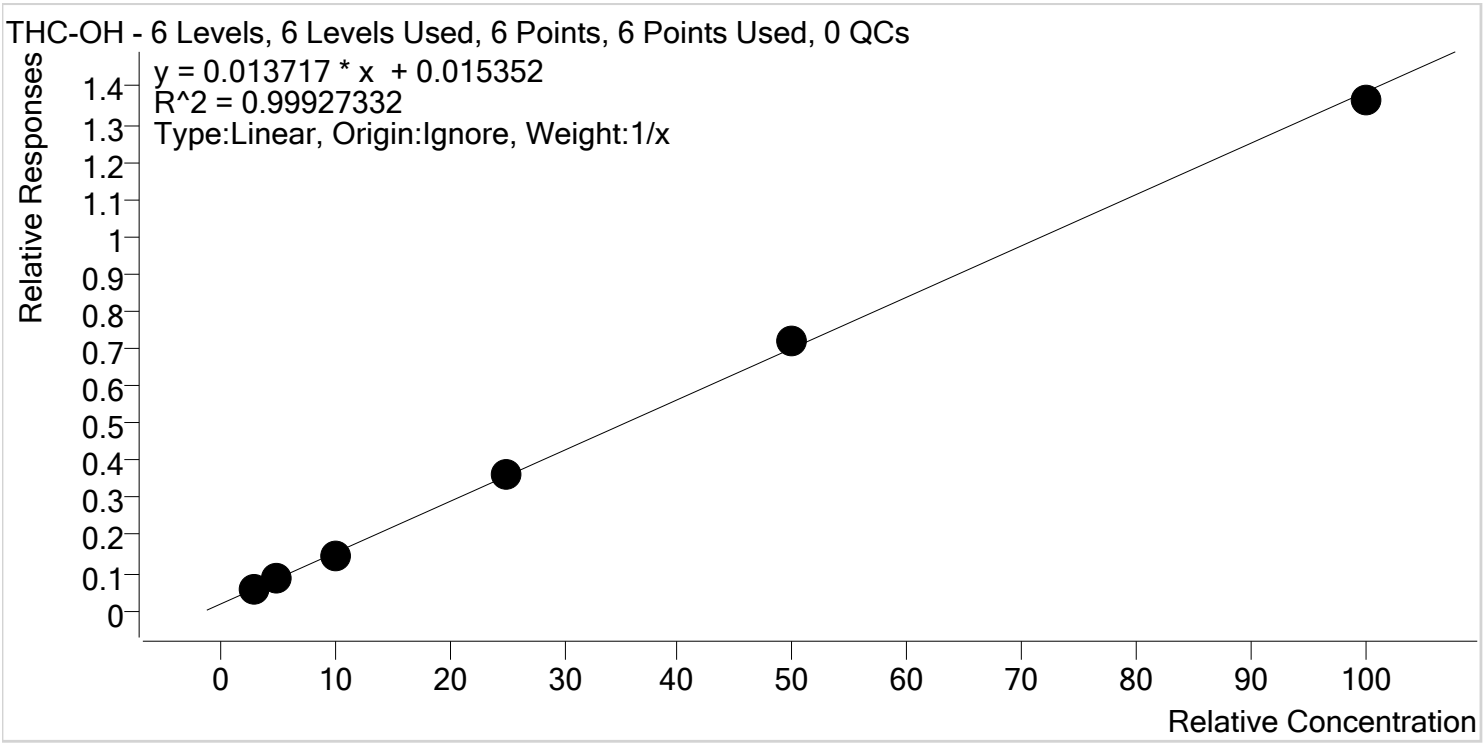
Batch results D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Last Cal. Update 6/7/2019 10:20 AM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1-3ng	1	✗	3.0	3.8	127.8
Cal 2- 5ng	2	✔	5.0	5.0	100.0
Cal 3 -10ng	3	✔	10.0	10.3	102.9
Cal 4-25ng	4	✔	25.0	24.0	96.0
Cal 5-50ng	5	✔	50.0	50.4	100.7
Cal 6-100ng	6	✔	100.0	100.3	100.3

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Last Cal. Update 6/7/2019 10:20 AM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-D3



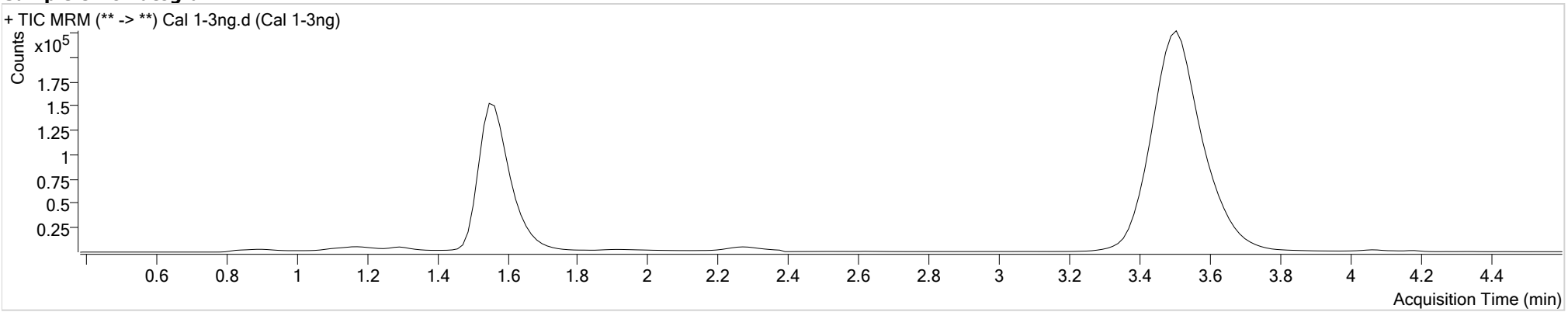
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1-3ng	1	✔	3.0	3.1	102.3
Cal 2- 5ng	2	✔	5.0	4.8	96.8
Cal 3 -10ng	3	✔	10.0	9.8	97.8
Cal 4-25ng	4	✔	25.0	25.4	101.6
Cal 5-50ng	5	✔	50.0	51.7	103.3
Cal 6-100ng	6	✔	100.0	98.3	98.3

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/7/2019 10:20:25 AM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	Cal 1-3ng.d
Type	Cal	**Sample**	Cal 1-3ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-B4	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 11:08:53 AM		
Sample Info.			

Sample Chromatogram



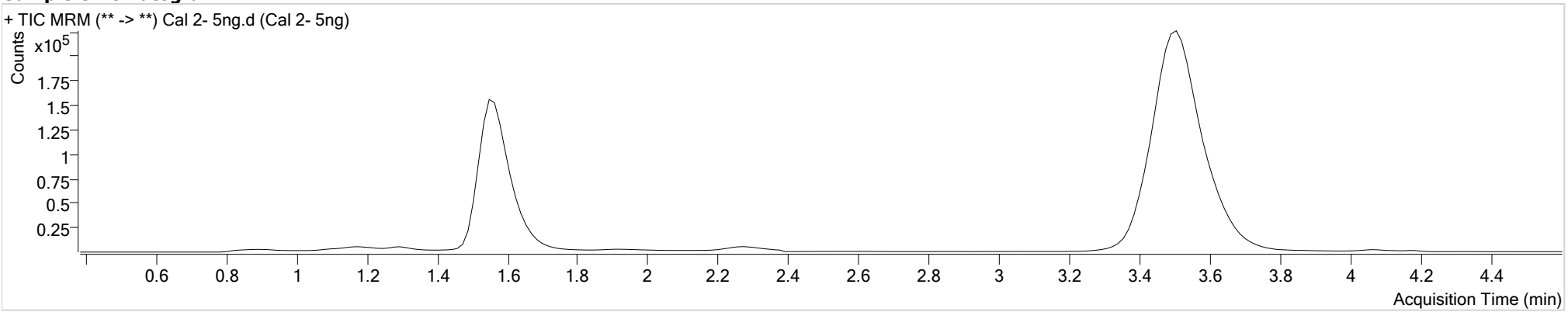
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.506	53914	323.26	29.2	38.88	2184878	3.1083 ng/ml
THC-COOH	1.595	18866	∞	42.7 **Low**	83.38	197909	3.8332 ng/ml
THC-OH	1.573	39232	∞	11.2	20.53	683007	3.0682 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/7/2019 10:20:25 AM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	Cal 2- 5ng.d
Type	Cal	**Sample**	Cal 2- 5ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-C4	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 11:16:28 AM		
Sample Info.			

Sample Chromatogram



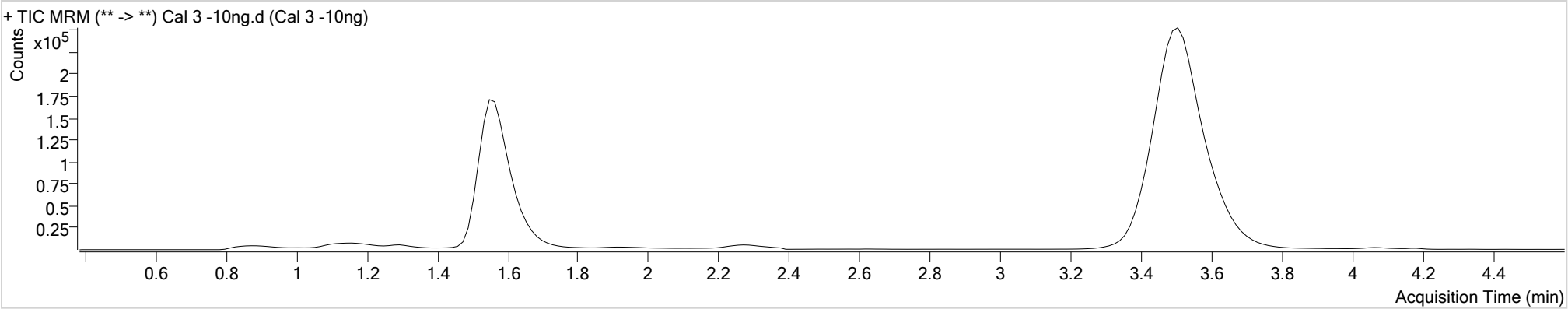
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.506	86688	2671.23	27.5	45.46	2191515	4.9774 ng/ml
THC-COOH	1.595	23215	∞	47.5	120.27	197202	4.9996 ng/ml
THC-OH	1.573	55200	71.38	12.0	50.23	675180	4.8409 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/7/2019 10:20:25 AM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	Cal 3 -10ng.d
Type	Cal	**Sample**	Cal 3 -10ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-D4	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 11:24:03 AM		
Sample Info.			

Sample Chromatogram



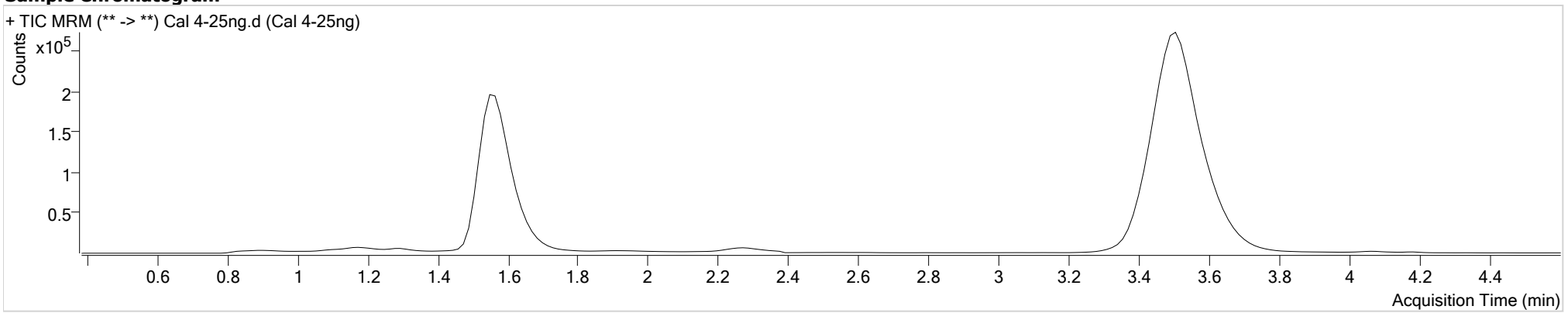
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.506	183089	695.71	28.1	186.48	2349587	9.7969 ng/ml
THC-COOH	1.595	43308	91.02	51.4	239.91	197415	10.2934 ng/ml
THC-OH	1.558	103508	245.67	12.1	181.01	692552	9.7766 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/7/2019 10:20:25 AM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	Cal 4-25ng.d
Type	Cal	**Sample**	Cal 4-25ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-E4	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 11:31:38 AM		
Sample Info.			

Sample Chromatogram



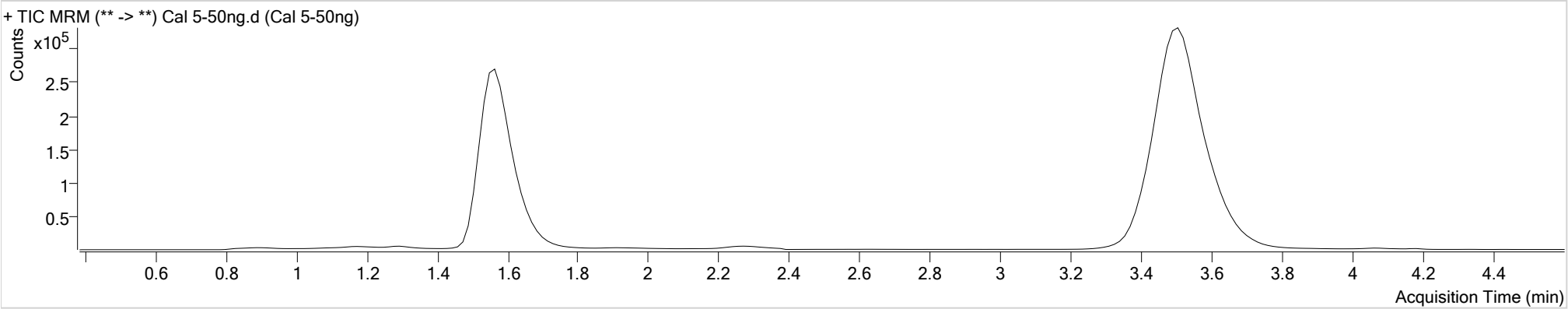
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.506	433507	∞	27.6	∞	2227133	24.4589 ng/ml
THC-COOH	1.595	91994	249.18	56.2	686.75	190633	23.9997 ng/ml
THC-OH	1.558	239479	600.80	13.4	520.79	658631	25.3879 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/7/2019 10:20:25 AM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	Cal 5-50ng.d
Type	Cal	**Sample**	Cal 5-50ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-F4	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 11:39:13 AM		
Sample Info.			

Sample Chromatogram



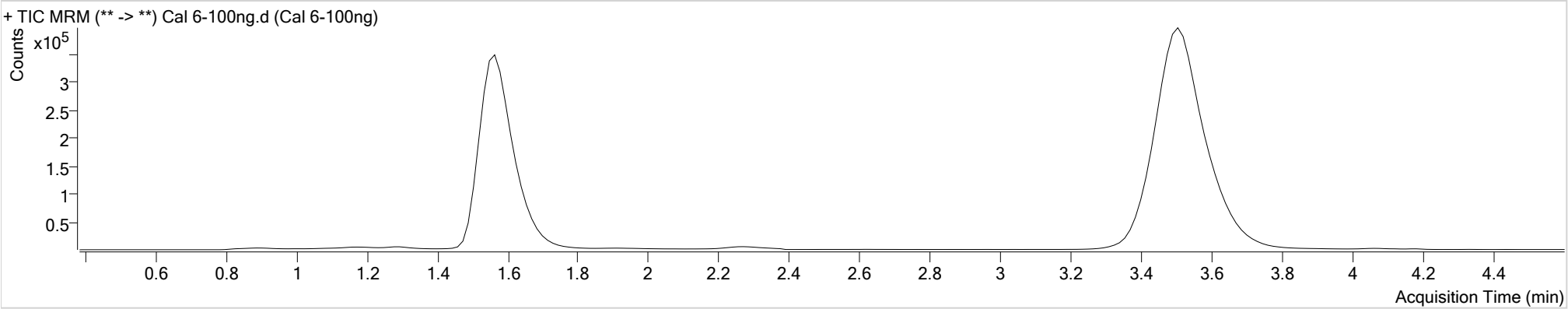
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.506	890690	4015.70	27.7	480.34	2221145	50.3799 ng/ml
THC-COOH	1.595	189681	∞	56.8	986.19	191808	50.3681 ng/ml
THC-OH	1.558	502997	∞	12.8	1846.46	694722	51.6633 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 27\060319 THCQ SP TS\QuantResults\THCQ SP.batch.bin
Calibration Last Update	6/7/2019 10:20:25 AM

Instrument	FALCO-LCMS (Property ID 069901)	**Data File**	Cal 6-100ng.d
Type	Cal	**Sample**	Cal 6-100ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-G4	**Comment**	
Injection Volume	10		
Acq. Date-Time	6/3/2019 11:46:49 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.506	1555625	4936.02	27.4	∞	1948802	100.2786 ng/ml
THC-COOH	1.595	323443	661.18	58.5	2074.23	165997	100.3393 ng/ml
THC-OH	1.558	819913	1778.30	14.4	∞	601444	98.2630 ng/ml