REVIEWED
By Sarah Pickle at 11:51 am, Jul 22, 2019

7/16/2019

Worklist: 3549

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
C2019-1085	1	157485	AM 27 Blood THC Quant by LC-QQQ
C2019-1097	1	157486	AM 27 Blood THC Quant by LC-QQQ
C2019-1105	1	157487	AM 27 Blood THC Quant by LC-QQQ
C2019-1136	1	157488	AM 27 Blood THC Quant by LC-QQQ
C2019-1217	1	157489	AM 27 Blood THC Quant by LC-QQQ
C2019-1231	1	157490	AM 27 Blood THC Quant by LC-QQQ
C2019-1241	1	157491	AM 27 Blood THC Quant by LC-QQQ
C2019-1268	1	157492	AM 27 Blood THC Quant by LC-QQQ
C2019-1269	2	157493	AM 27 Blood THC Quant by LC-QQQ
C2019-1273	1	157494	AM 27 Blood THC Quant by LC-QQQ
C2019-1284	1	157495	AM 27 Blood THC Quant by LC-QQQ
C2019-1287	1	157496	AM 27 Blood THC Quant by LC-QQQ
C2019-1299	1	157497	AM 27 Blood THC Quant by LC-QQQ

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

Extraction Date: 07/16/19 Analyst: Anne Nord
Plate lot#: 0539904 Plate Expiration: 09/10/19

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: 19A207P3 **Column**: UCT Selectra DA 100 x 2.1mm 3um
LCMS-QQQ ID: 69679

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.
☒ 3. Create worklist:

Analytic:

☒ 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
☒ 2. Pipette **1000µL blood (calibrated pipette) Pipette ID: k52558g** in wells of analytical (standards) plate.
☒ 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 66759*
☒ 4. Pipette **500µL 0.1% formic acid in water** in wells 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)*** Manifold ID: 66792
☒ 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: 66819*
☒ 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. am28-27\0716019 7/22/19
Batch ~~Worklist~~ path: Data\2019\~~AM 27\061119~~ 7/22/19 Batch Name: cann quant
☒ 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: *see deviation for 50ng calibrator.*

Idaho State Police
Forensic Services
Toxicology Discipline

Request for Departure from an Analytical Method

Date of Request
07/18/19

Forensic Scientist
Anne Nord

Analytical Method
Toxicology AM #27: Quantitative Analysis of THC and Metabolites in Blood by LCMS-QQQ

4.3.2.3 The analyte retention time must be within +/- 3% or +/- 0.100 min whichever is greater of the average retention time of the calibrators for that analyte.

Request

For worklist 3549 the 50ng calibrator had a shift that puts it just outside acceptable range for THC. All compounds and internal standards shifted proportionally in this sample. I am requesting to allow the acceptable retention time for the 50 ng cal for THC to be .102 minutes greater than the average of the rest of the calibrators. I am also requesting to drop the 50 ng calibrator calculation to obtain the average retention time.

Discipline Leader Review

☒ Departure approved
Comments: This is a minor deviation.

☐ Departure Not Approved
Comments:

Celena Shrum

Date: 07/19/19

Celena Shrum
Toxicology Discipline Lead

Toxicology AM method 27 external prep information
working solution 1 ug/ml in meoh C-THC, THC-OH, THC
Stock solution 1mg/ml 7.5 ul each THC, 100 ug/ml 150 ul C-THC, 75 ulTHC-OH in 9767.5 ul meOH
Ppd 3/13/19 Exp: 9/13/19 lot 91319 by AMN

Drug	lot	expiration
C-THC	FE01061702 cerillient	3/1/2022
THC-OH	318.1b18.1L1a	12/1/2021
THC	135.1b71.0L6	11/1/2021

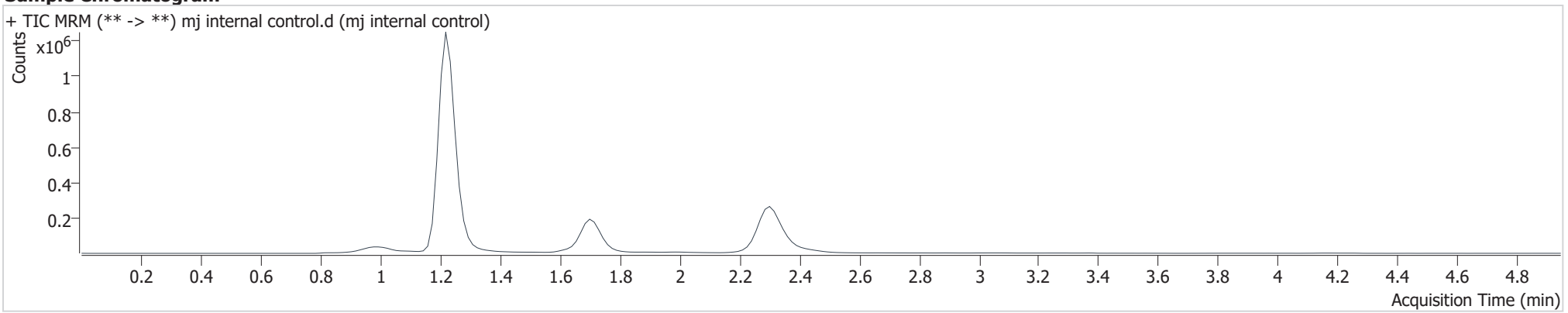
AM 27 control 100 ul working solution lot (91319) in 9900 ul blood lot (19A207p3)
ppd 3/13/19 Exp 9/13/19 lot 31319 Concentration 7.5 ng/ml THC, THC-OH and 15 ng/ml C-TH by AMN

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj internal control.d
Type	QC	**Sample**	mj internal control
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 12:02:47 PM		
Sample Info.			

Sample Chromatogram



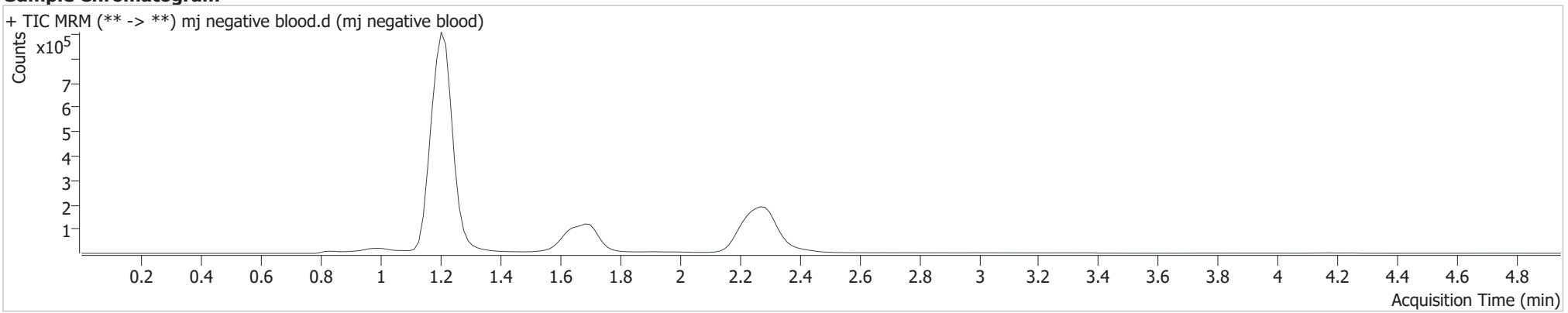
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.235	128947	448.2	35.5	539.2	1320739	9.467 ng/ml Low
THC-OH	1.224	237760	∞	9.4	∞	3031125	5.539 ng/ml
THC	2.311	47212	692.0	25.1	110.9	1526383	4.722 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj negative blood.d
Type	Sample	**Sample**	mj negative blood
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 12:10:29 PM		
Sample Info.			

Sample Chromatogram

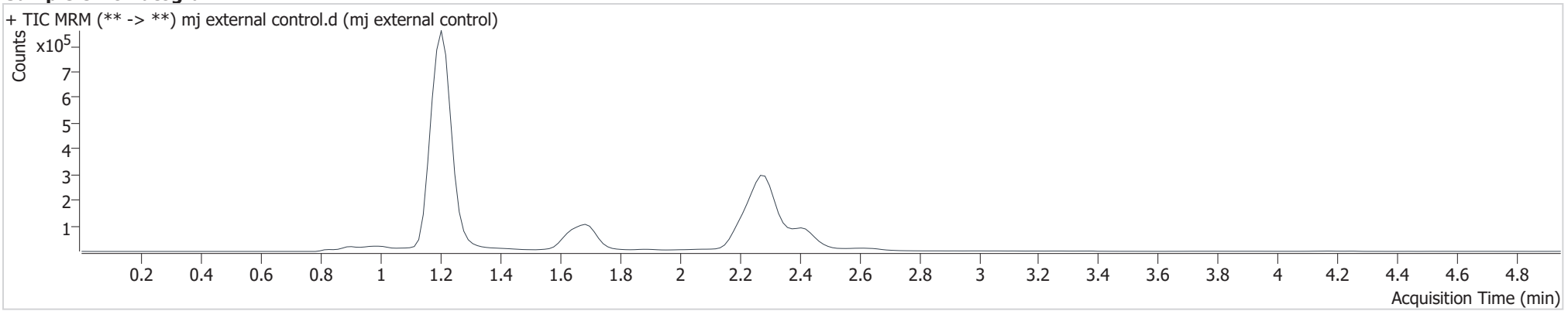


AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj external control.d
Type	QC	**Sample**	mj external control
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-B2	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 12:18:13 PM		
Sample Info.			

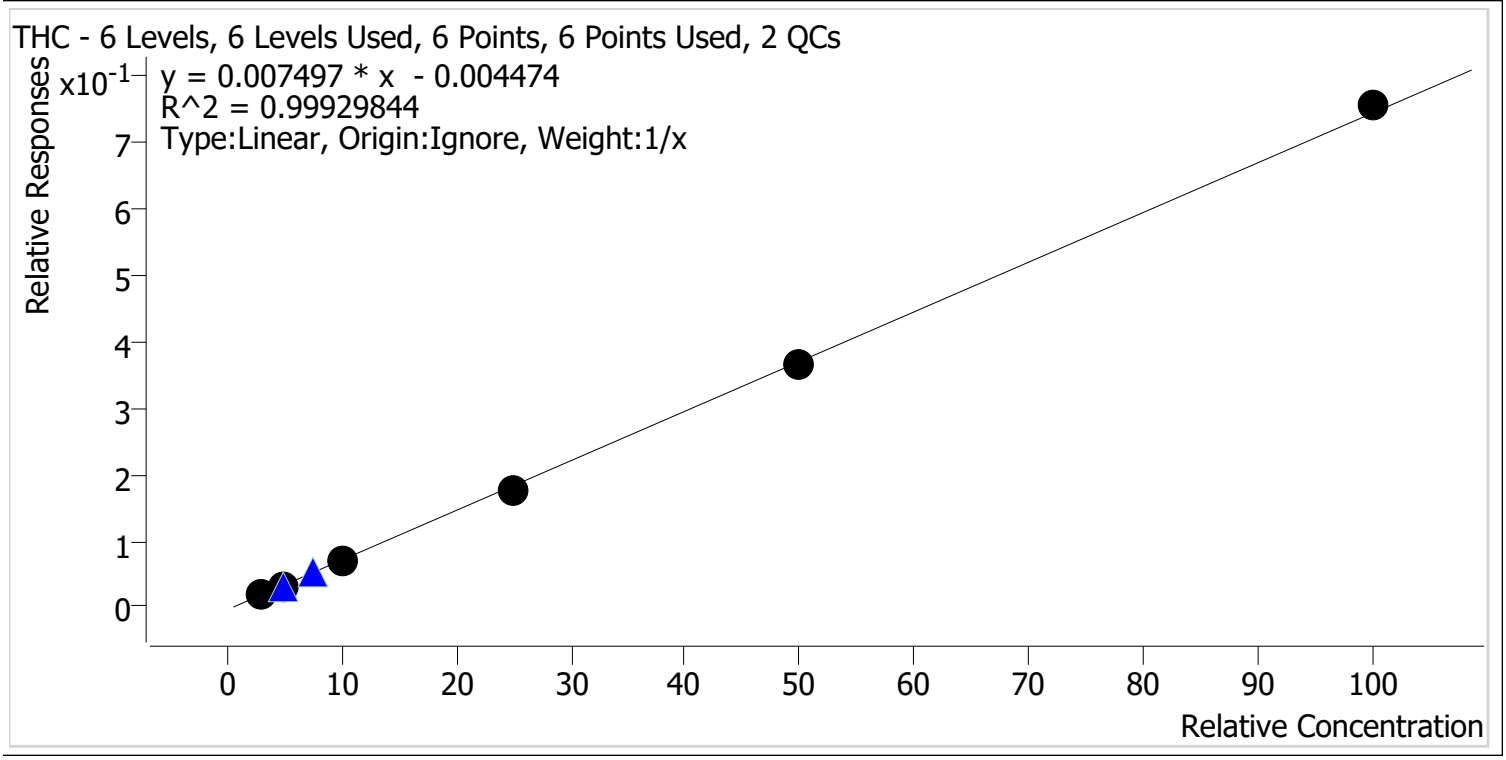
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.220	155939	279.2	32.2	3036.8	1069376	13.999 ng/ml
THC-OH	1.209	240589	∞	8.7	∞	2384334	7.134 ng/ml
THC	2.296	79345	∞	25.0	114.7	1465886	7.816 ng/ml

Compound Calibration Report

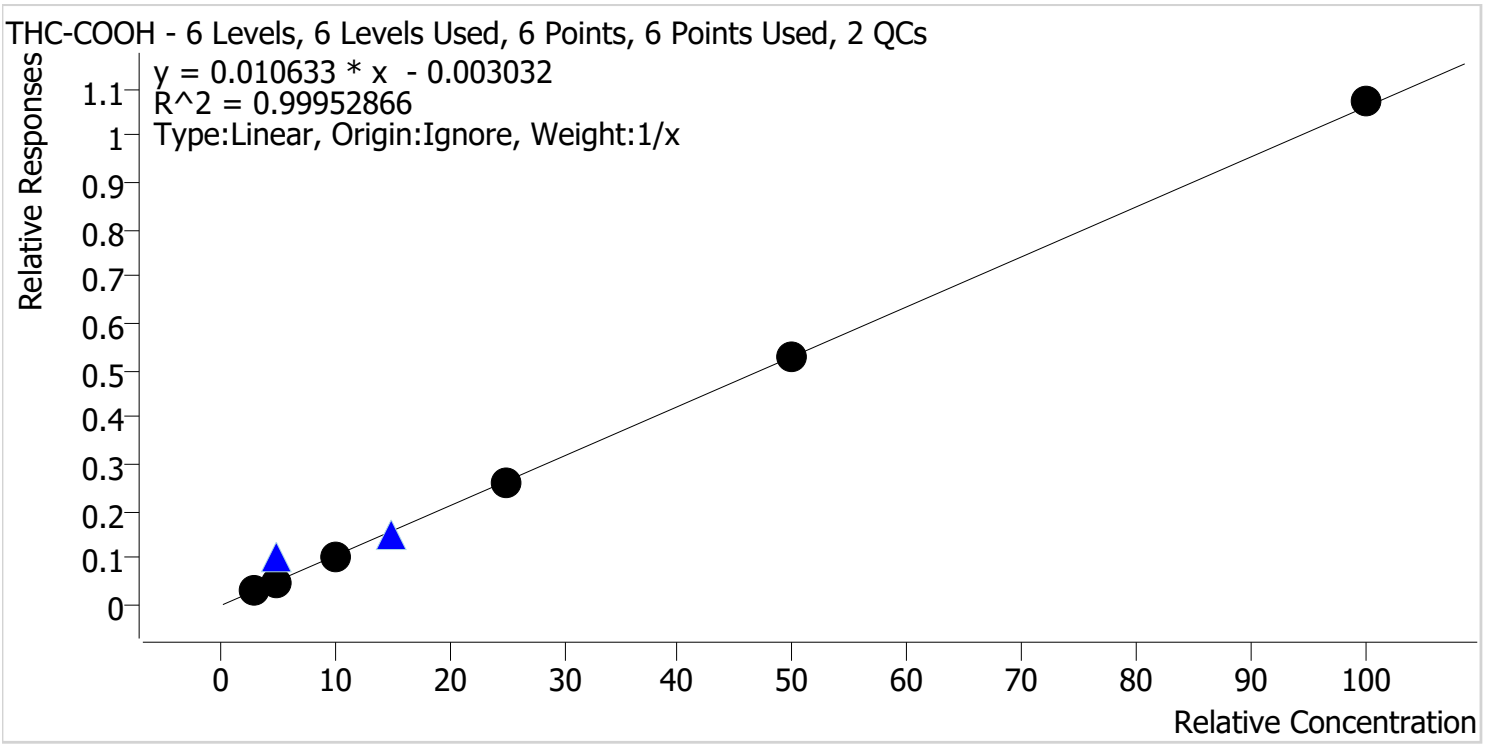
Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Last Cal. Update	7/19/2019 1:07 PM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-d3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal2	2	✔	3.0	3.3	109.2
mj cal 3	3	✔	5.0	4.9	97.8
mj cal 4	4	✔	10.0	9.6	95.7
mj cal 5	5	✔	25.0	24.1	96.5
mj cal 6	6	✔	50.0	49.7	99.4
mj cal 7	7	✔	100.0	101.4	101.4

Compound Calibration Report

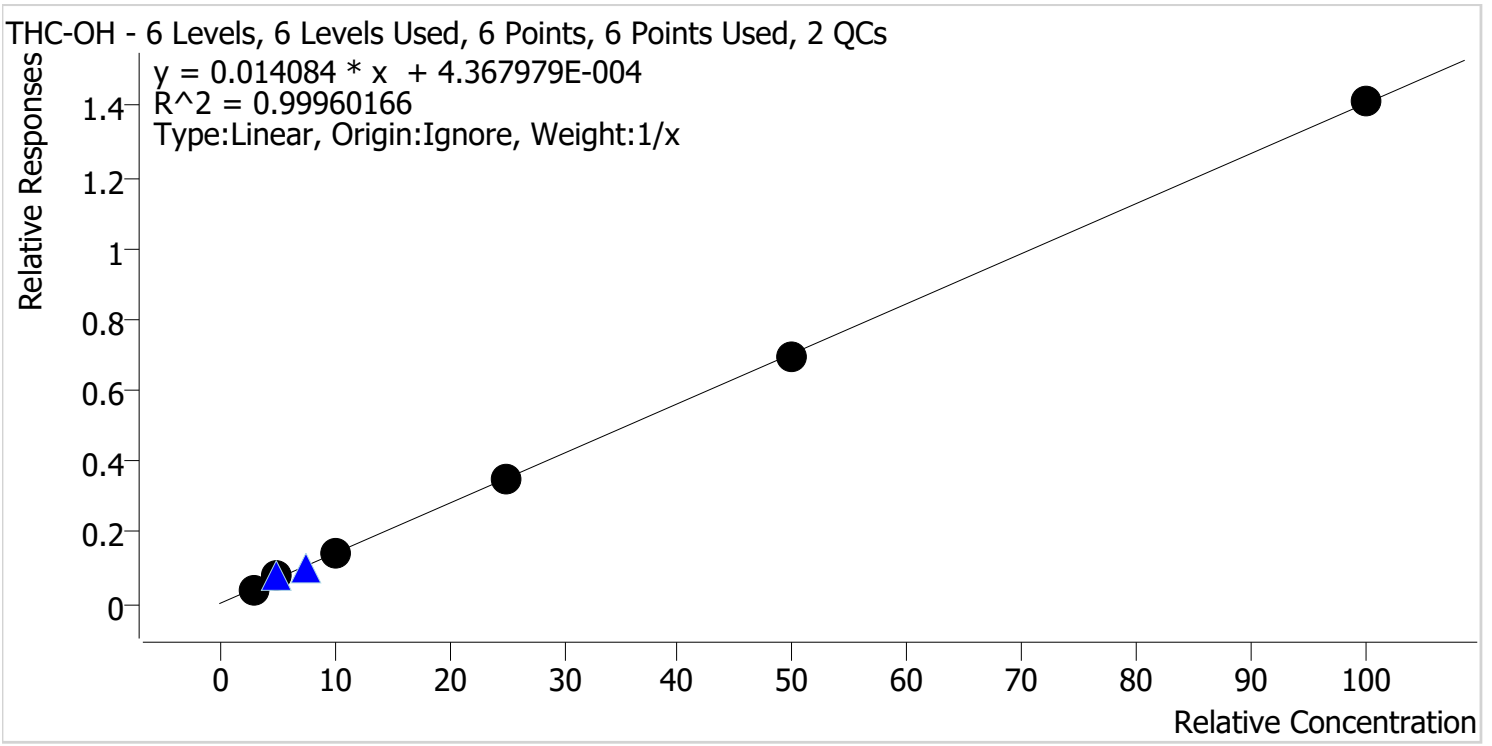
Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Last Cal. Update	7/19/2019 1:07 PM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-d9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal2	2	✔	3.0	3.3	109.0
mj cal 3	3	✔	5.0	4.7	94.7
mj cal 4	4	✔	10.0	9.8	98.0
mj cal 5	5	✔	25.0	24.4	97.8
mj cal 6	6	✔	50.0	49.7	99.4
mj cal 7	7	✔	100.0	101.0	101.0

Compound Calibration Report

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Last Cal. Update	7/19/2019 1:07 PM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-d3



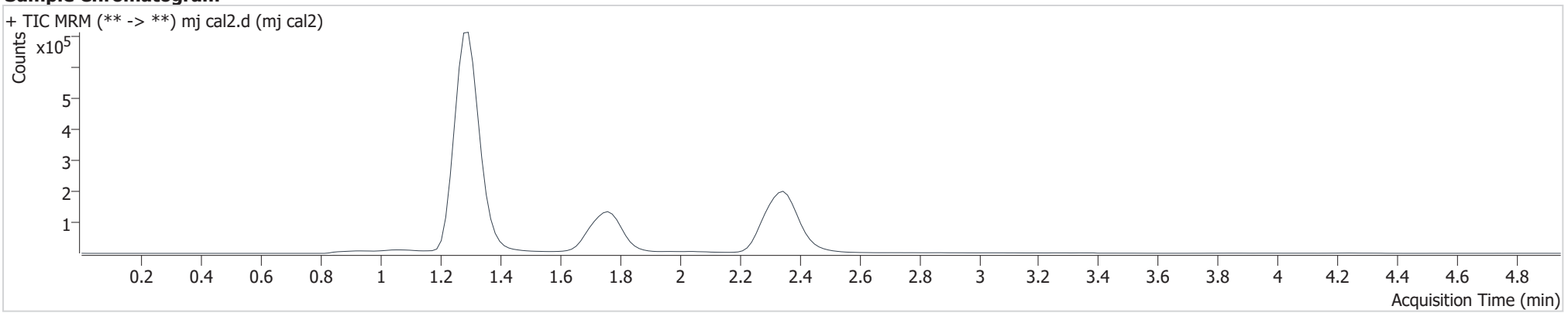
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal2	2	✔	3.0	2.8	92.1
mj cal 3	3	✔	5.0	5.3	106.7
mj cal 4	4	✔	10.0	10.2	101.9
mj cal 5	5	✔	25.0	25.1	100.3
mj cal 6	6	✔	50.0	49.3	98.6
mj cal 7	7	✔	100.0	100.3	100.3

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj cal2.d
Type	Cal	**Sample**	mj cal2
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 9:53:40 AM		
Sample Info.			

Sample Chromatogram



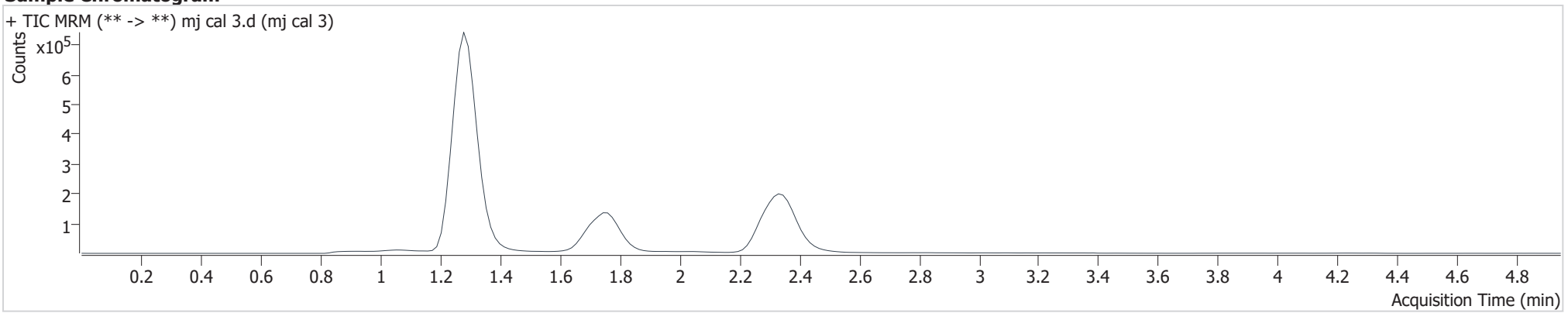
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.295	41029	52.6	32.1	57.6	1292597	3.270 ng/ml Low
THC-OH	1.300	106678	∞	10.2	∞	2711684	2.762 ng/ml Low
THC	2.356	31413	762.3	25.6	66.4	1563712	3.276 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj cal 3.d
Type	Cal	**Sample**	mj cal 3
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 10:01:25 AM		
Sample Info.			

Sample Chromatogram



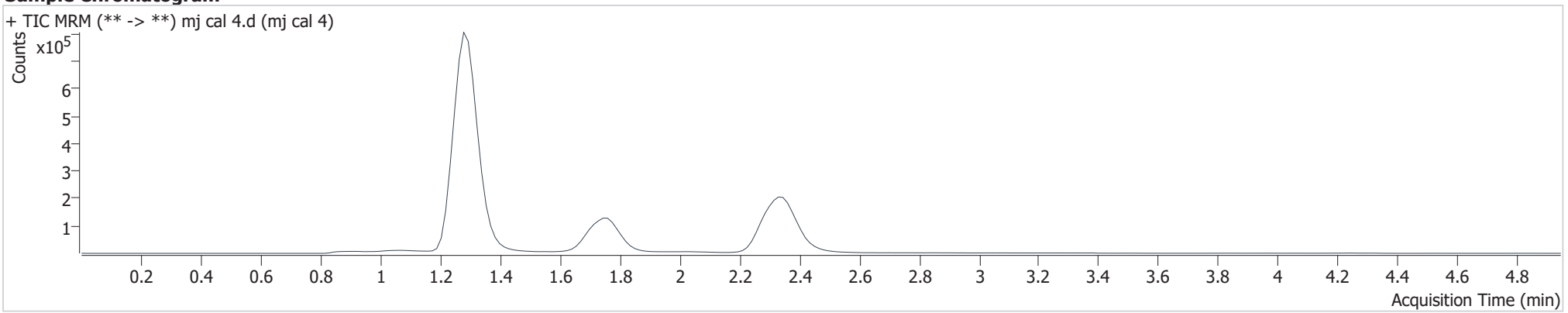
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.295	59419	234.3	36.9	102.1	1255728	4.735 ng/ml Low
THC-OH	1.285	202867	∞	10.6	∞	2683554	5.337 ng/ml
THC	2.356	49945	4706.0	26.0	141.8	1552046	4.889 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj cal 4.d
Type	Cal	**Sample**	mj cal 4
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 10:09:08 AM		
Sample Info.			

Sample Chromatogram



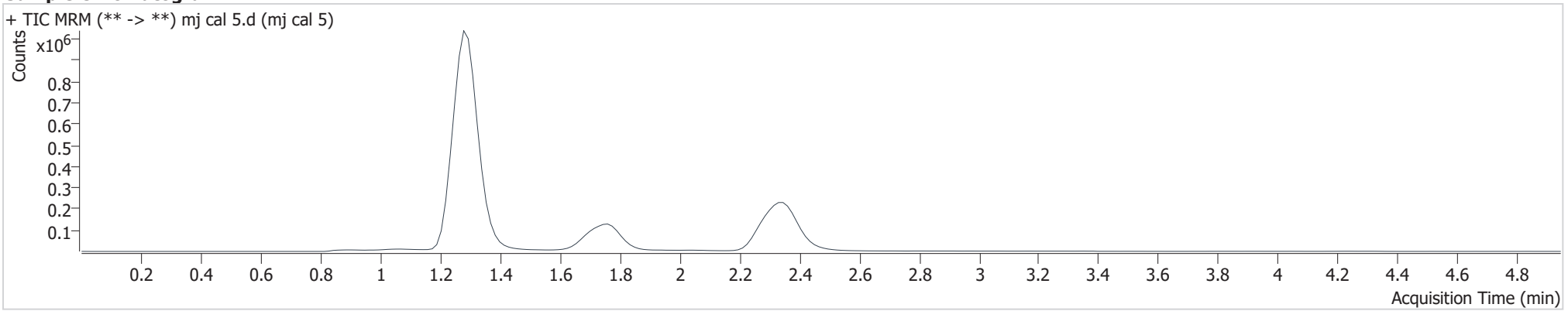
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.295	125472	631.6	33.8	512.9	1239687	9.804 ng/ml Low
THC-OH	1.285	380196	∞	10.1	∞	2640063	10.194 ng/ml
THC	2.356	101579	3935.5	25.1	184.8	1510636	9.566 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj cal 5.d
Type	Cal	**Sample**	mj cal 5
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 10:16:50 AM		
Sample Info.			

Sample Chromatogram



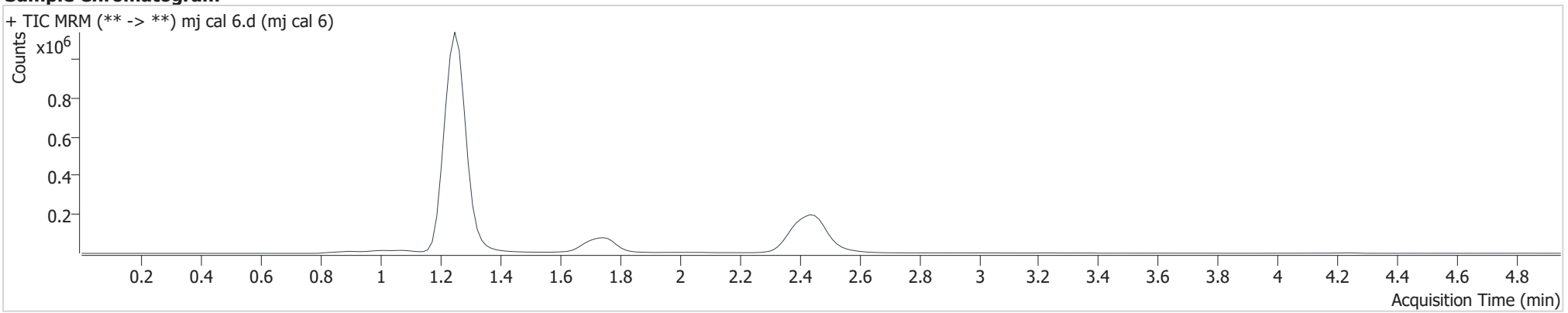
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.295	313283	776.8	35.9	301.8	1219455	24.447 ng/ml
THC-OH	1.285	1013163	∞	10.8	∞	2864766	25.081 ng/ml
THC	2.356	282345	2683.1	23.8	∞	1600094	24.132 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj cal 6.d
Type	Cal	**Sample**	mj cal 6
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 11:47:23 AM		
Sample Info.			

Sample Chromatogram



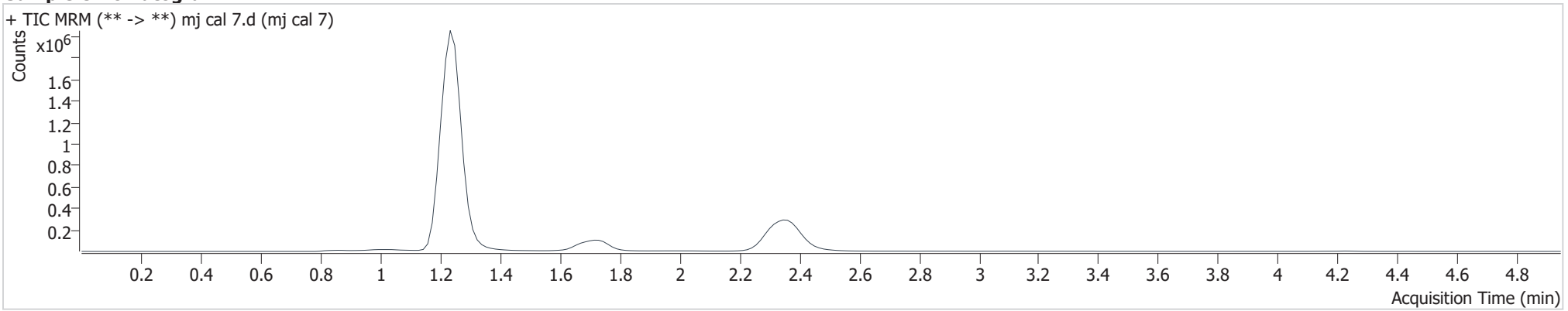
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.265	410701	780.6	37.1	771.6	781524	49.709 ng/ml
THC-OH	1.255	1458053	∞	11.2	∞	2098783	49.297 ng/ml
THC	2.461 High	413307	∞	24.5	529.4	1122804	49.694 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2019\am28-27 0716019\QuantResults\am 27.batch.bin
Calibration Last Update	7/19/2019 1:07:17 PM

Instrument	69679	**Data File**	mj cal 7.d
Type	Cal	**Sample**	mj cal 7
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/17/2019 11:55:05 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.250	1032461	1703.2	37.5	884.2	963789	101.035 ng/ml
THC-OH	1.240	3291860	∞	11.5	∞	2328979	100.329 ng/ml
THC	2.371	918139	4182.6	24.8	1915.0	1214342	101.443 ng/ml