REVIEWED
By Sarah Pickle at 8:35 am, Aug 05, 2019

7/24/2019 TS

Worklist: 3564

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
M2019-2774	1	158081	AM 27 Blood THC Quant by LC-QQQ
M2019-3002	1	158082	AM 27 Blood THC Quant by LC-QQQ
P2019-1944	1	158083	AM 27 Blood THC Quant by LC-QQQ
P2019-2137	1	158084	AM 27 Blood THC Quant by LC-QQQ
P2019-2147	1	158085	AM 27 Blood THC Quant by LC-QQQ
P2019-2183	1	158086	AM 27 Blood THC Quant by LC-QQQ
P2019-2188	1	158087	AM 27 Blood THC Quant by LC-QQQ

TS

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

Extraction Date: 07/25/2019 Analyst: Tamara Salazar
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: Hemostat 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.
☒ 3. Create worklist:

Analytic:

☒ 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
☒ 2. Pipette **1000µL blood/urine (calibrated pipette) Pipette ID: 3** in wells of analytical (standards) plate.
☒ 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 067105*
☒ 4. Pipette **500µL 0.1% formic acid in water** in wells of analytical plate for blood samples.
☒ 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: 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.
Worklist path: D:\MassHunter\Data\2019\AM 27\072319 wklst 3556 MDS TS_reinjects
Batch Name THCQ TS
☒ 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: *Curves limited: THC-COOH 10-100*

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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	09/30/2020
THC-OH	Cerilliant	FE01121503	01/31/2020
Prepared:	04/16/2019		
Prepared By:	Tamara Salazar		
Expires:	01/31/2020		

Blood External Control Solution (Lot: 072319)

100 ul of methanol external control solution was added to 9900 ul of blood.

Approximately 10ng/mL of each compound.

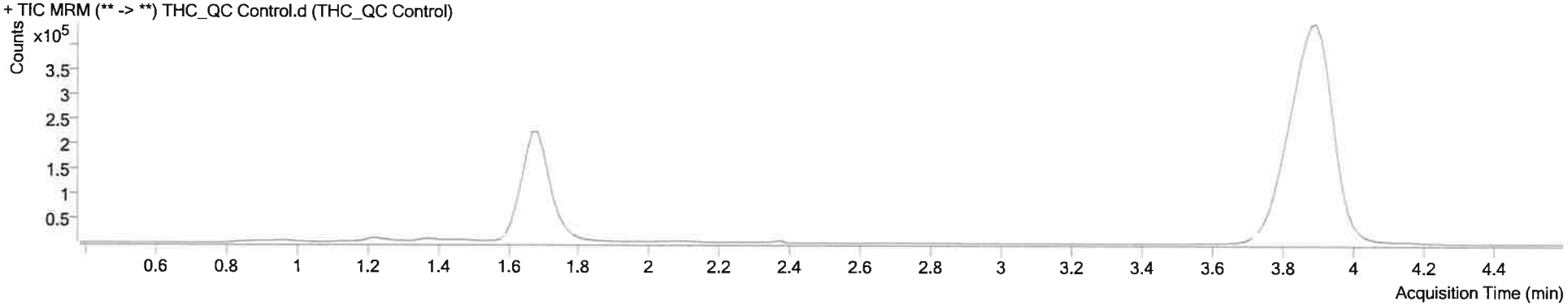
Component	*Source*	*Source Lot Number*
Negative Blood	Hemostat	445283-2
Methanol External Control Solution	-	WS041619
Prepared:	07/23/19	
Prepared by:	Tamara Salazar	
Expires:	01/31/2020	

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_QC Control.d
Type	Sample	**Sample**	THC_QC Control
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 6:18:27 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.912	138003	332.02	29.7	84.56	3684098	4.6844 ng/ml
THC-COOH	1.730	52393	∞	46.9	536.52	244550	9.7077 ng/ml
THC-OH	1.693	73305	307.53	10.9	111.48	894159	4.9648 ng/ml

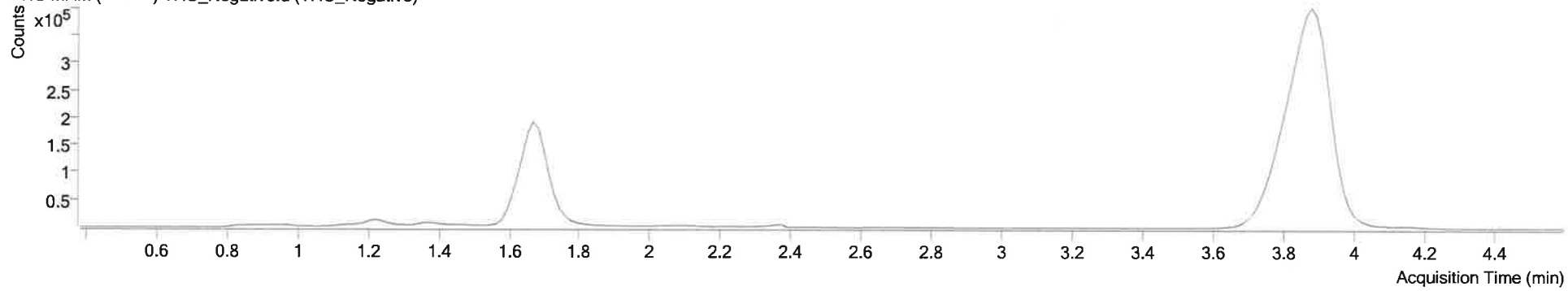
AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_Negative.d
Type	Sample	**Sample**	THC_Negative
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 6:33:37 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) THC_Negative.d (THC_Negative)



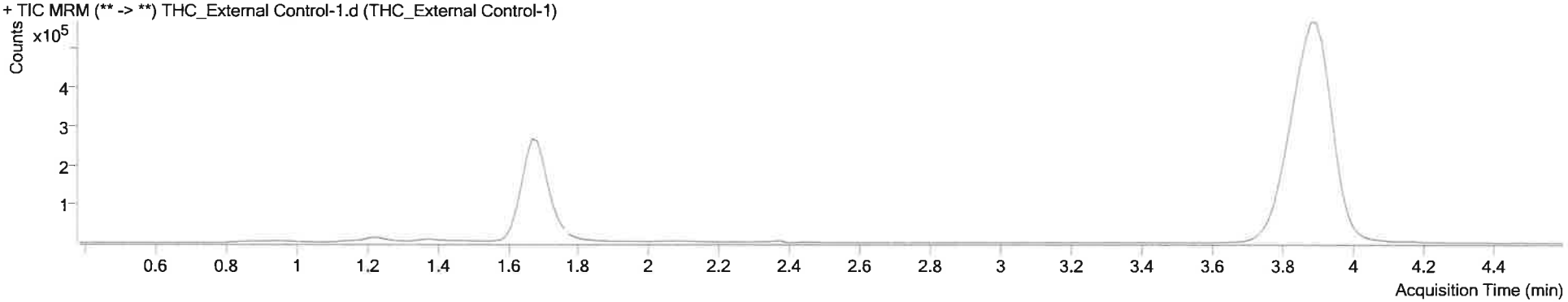
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.768 High	16242	∞	3.3 Low	3.22 Low	845587	0.4328 ng/ml <3

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_External Control-1.d
Type	Sample	**Sample**	THC_External Control-1
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-B2	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 6:48:48 PM		
Sample Info.			

Sample Chromatogram

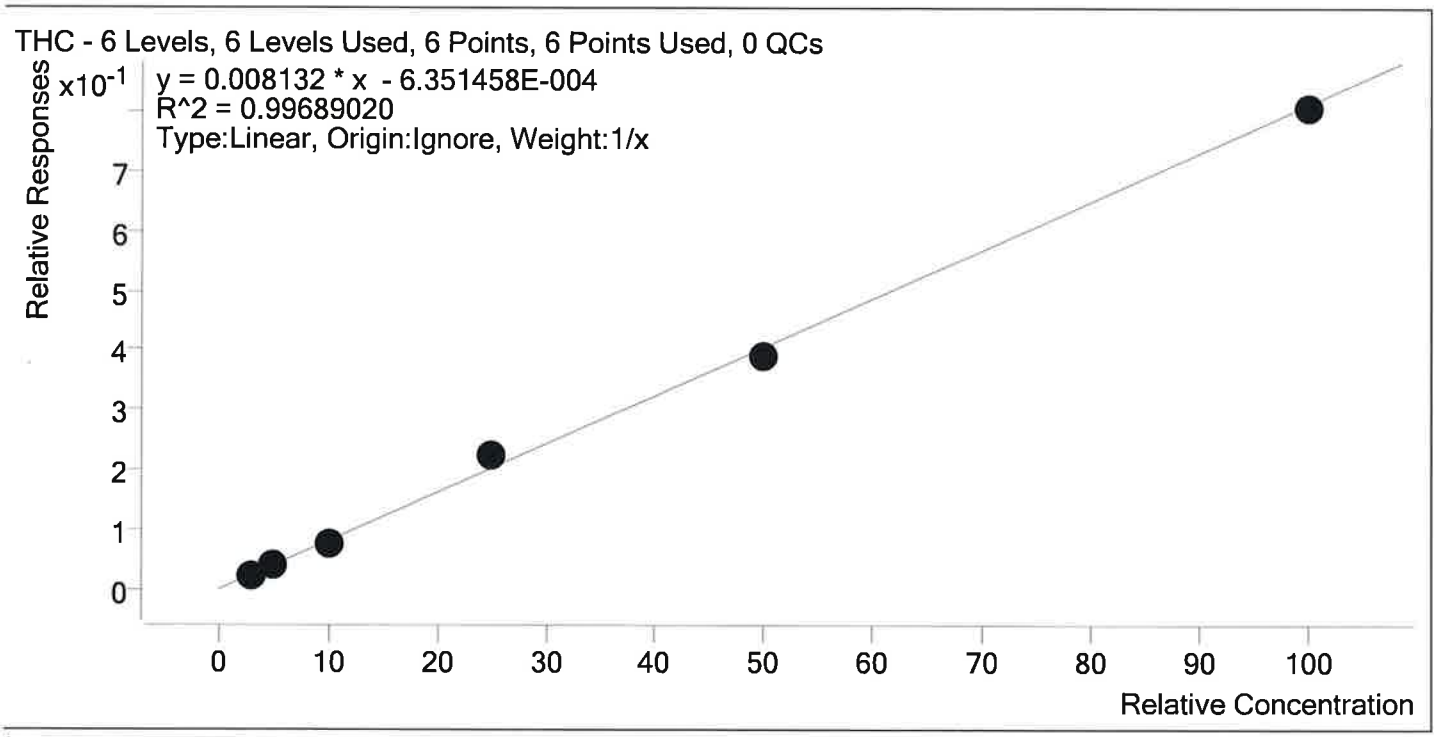


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.897	321628	1733.14	30.4	132.99	4326349	9.2197 ng/ml
THC-COOH	1.730	54616	903.77	45.7	481.45	271688	8.9869 ng/ml
THC-OH	1.678	129604	∞	11.5	362.79	962040	8.7720 ng/ml

TS

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Last Cal. Update	7/26/2019 11:40 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3

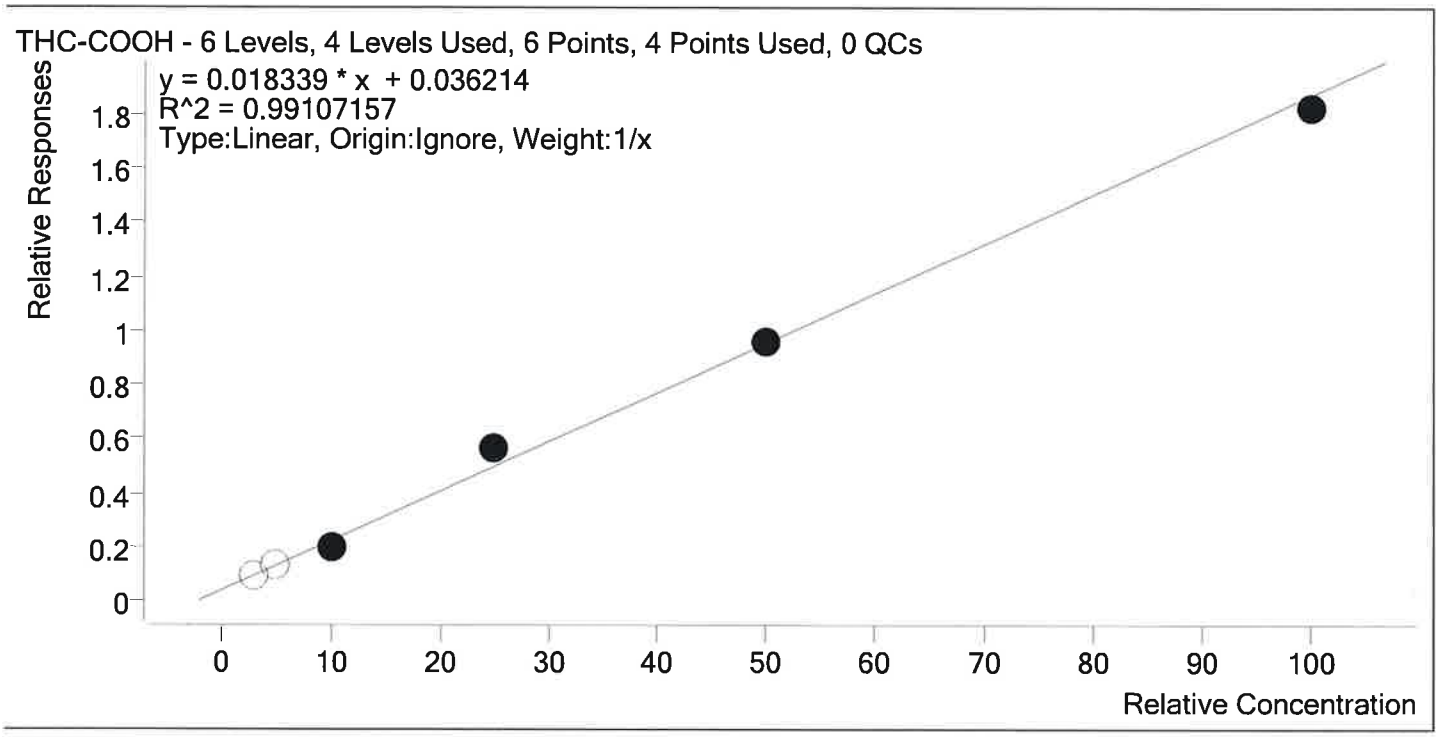


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
THC_Cal 1-3ng	1	✓	3.0	3.0	100.2
THC_Cal 2- 5ng	2	✓	5.0	4.8	96.2
THC_Cal 3 -10ng	3	✓	10.0	9.5	95.4
THC_Cal 4-25ng	4	✓	25.0	28.0	112.0
THC_Cal 5-50ng	5	✓	50.0	48.5	97.0
THC_Cal 6-100ng	6	✓	100.0	99.1	99.1

Generated at 11:41 AM on 7/26/2019

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Last Cal. Update	7/26/2019 11:40 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9

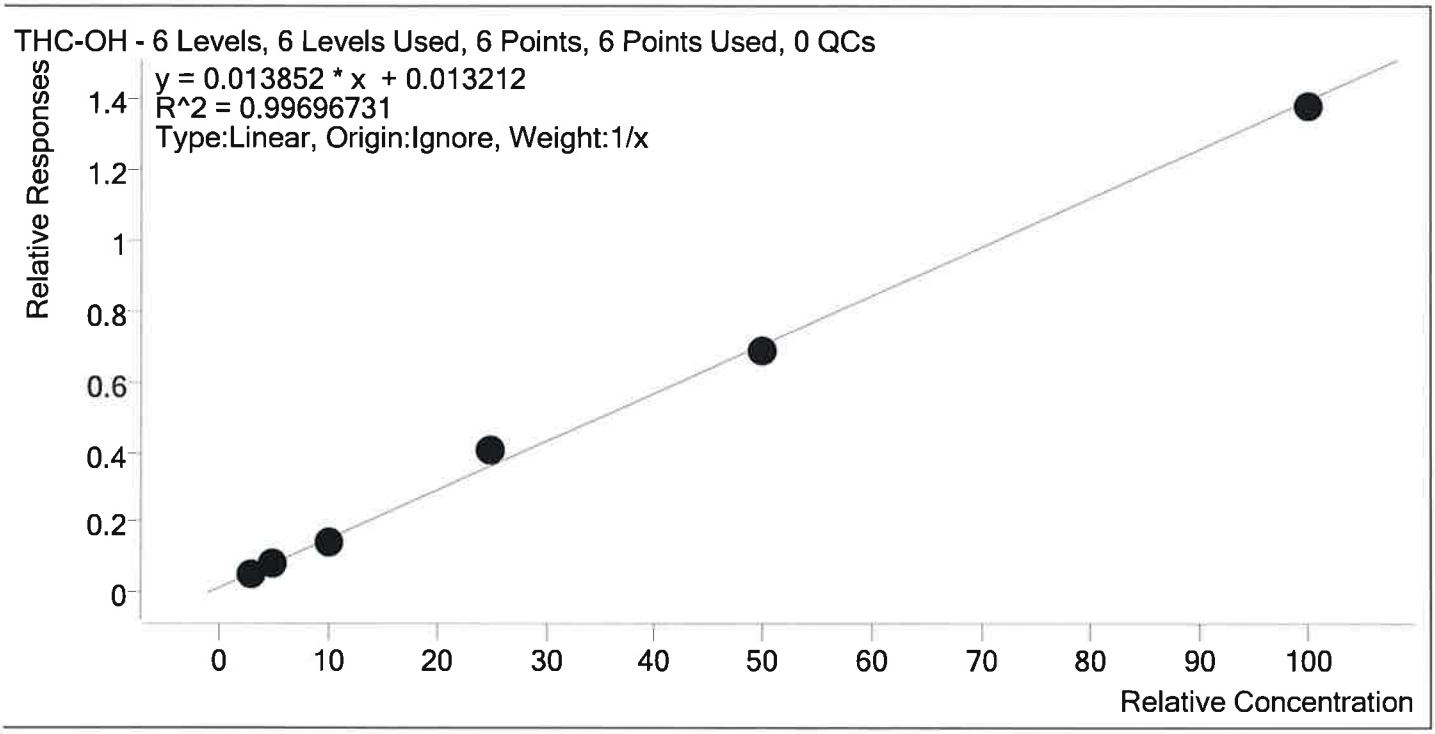


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
THC_Cal 1-3ng	1	×	3.0	3.2	105.4
THC_Cal 2- 5ng	2	×	5.0	5.0	99.3
THC_Cal 3 -10ng	3	✓	10.0	8.7	87.1
THC_Cal 4-25ng	4	✓	25.0	28.8	115.3
THC_Cal 5-50ng	5	✓	50.0	50.1	100.2
THC_Cal 6-100ng	6	✓	100.0	97.3	97.3

Generated at 11:41 AM on 7/26/2019

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Last Cal. Update	7/26/2019 11:40 AM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



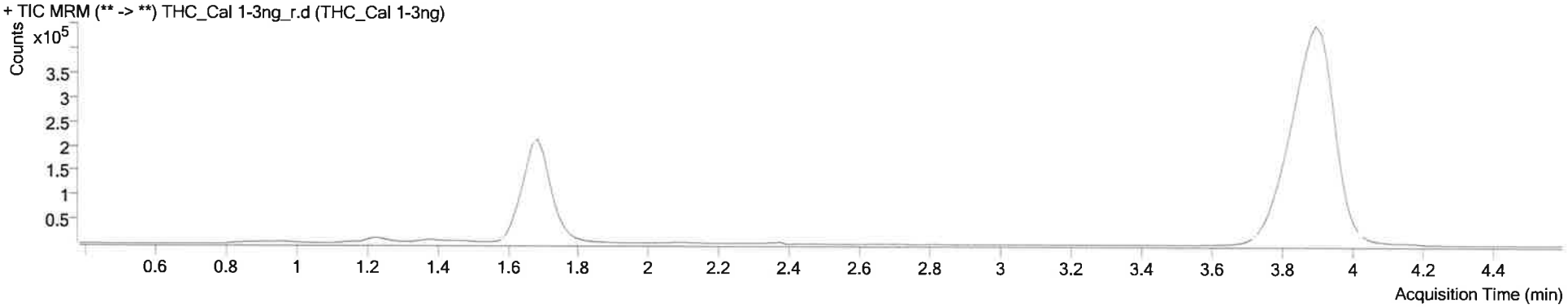
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
THC_Cal 1-3ng	1	✓	3.0	3.0	99.8
THC_Cal 2- 5ng	2	✓	5.0	4.7	94.8
THC_Cal 3 -10ng	3	✓	10.0	9.7	96.9
THC_Cal 4-25ng	4	✓	25.0	28.0	112.1
THC_Cal 5-50ng	5	✓	50.0	48.8	97.7
THC_Cal 6-100ng	6	✓	100.0	98.7	98.7

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_Cal 1-3ng_r.d
Type	Cal	**Sample**	THC_Cal 1-3ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 8:58:19 PM		
Sample Info.			

Sample Chromatogram



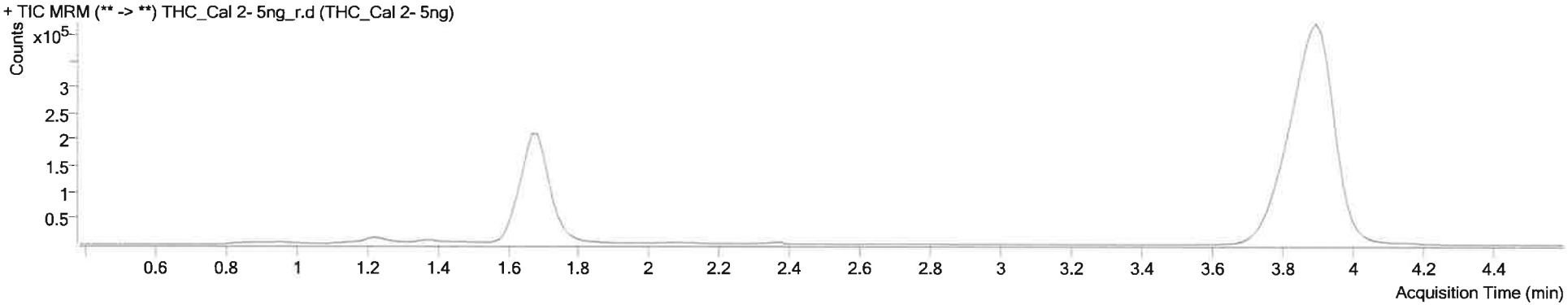
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.912	91078	∞	29.1	15.61	3824081	3.0068 ng/ml
THC-COOH	1.730	24219	∞	34.2 Low	95.28	257100	3.1619 ng/ml
THC-OH	1.693	48944	178.00	10.3	34.90	895309	2.9927 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_Cal 2- 5ng_r.d
Type	Cal	**Sample**	THC_Cal 2- 5ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 9:05:54 PM		
Sample Info.			

Sample Chromatogram



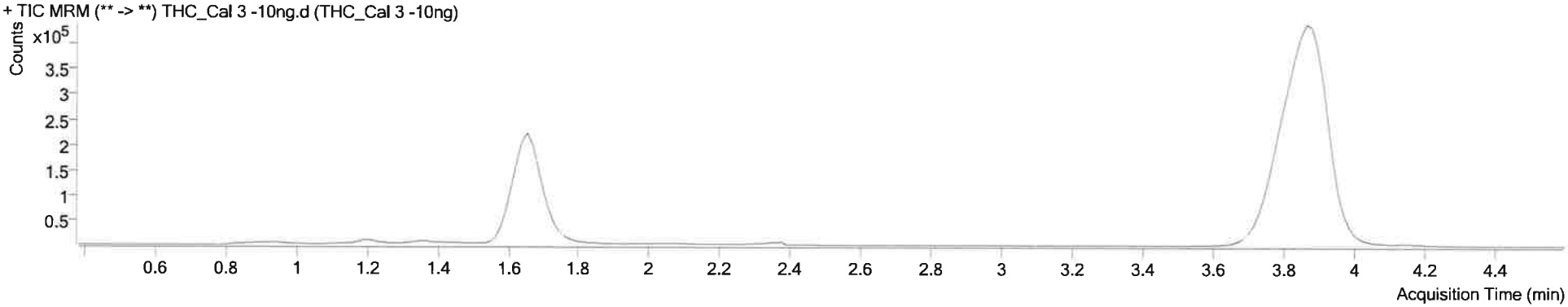
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.912	138823	1163.98	28.2	26.61	3607145	4.8106 ng/ml
THC-COOH	1.730	32018	∞	41.6 Low	155.25	251548	4.9659 ng/ml
THC-OH	1.693	69853	∞	10.8	82.94	885525	4.7410 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_Cal 3 -10ng.d
Type	Cal	**Sample**	THC_Cal 3 -10ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 5:47:59 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.882	288271	850.04	28.7	495.15	3746092	9.5407 ng/ml
THC-COOH	1.715	49400	351.64	52.4	370.41	252010	8.7142 ng/ml
THC-OH	1.663	126790	1298.63	11.8	269.50	859623	9.6944 ng/ml

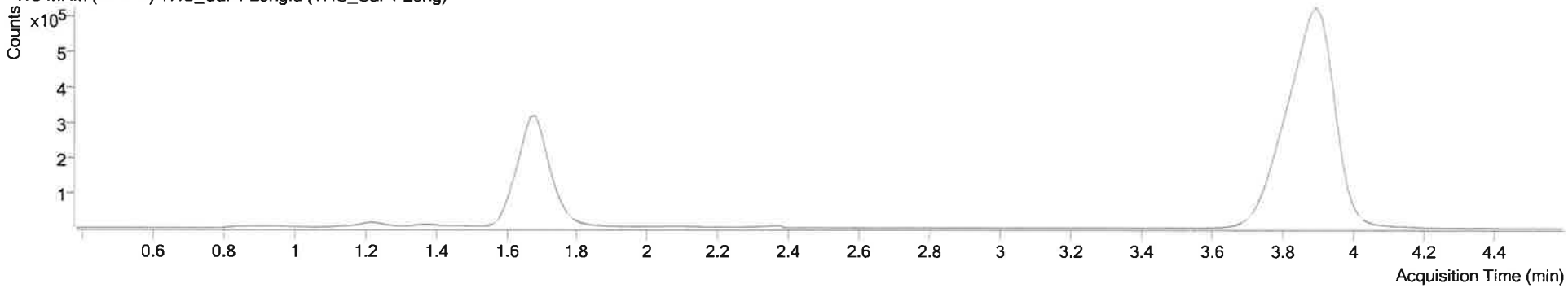
AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_Cal 4-25ng.d
Type	Cal	**Sample**	THC_Cal 4-25ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 5:55:43 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) THC_Cal 4-25ng.d (THC_Cal 4-25ng)



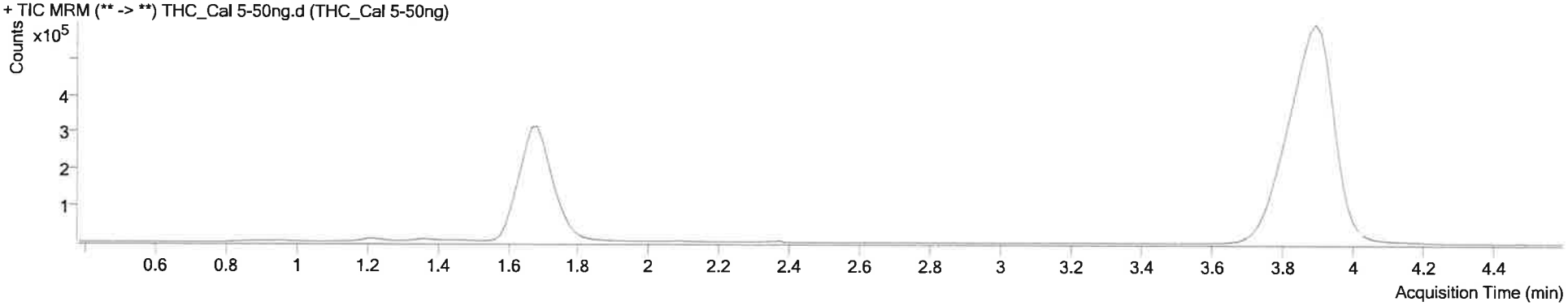
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.912	1056265	8187.51	27.6	2454.94	4650796	28.0058 ng/ml
THC-COOH	1.730	163985	465.93	54.9	695.43	290397	28.8170 ng/ml
THC-OH	1.693	419950	636.96	12.5	330.73	1046343	28.0213 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_Cal 5-50ng.d
Type	Cal	**Sample**	THC_Cal 5-50ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 6:03:18 PM		
Sample Info.			

Sample Chromatogram



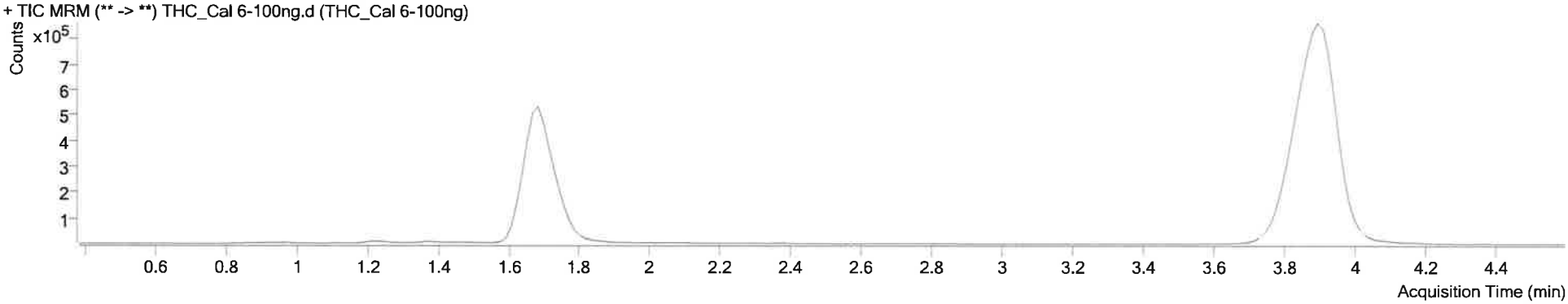
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.912	1433246	4757.77	27.5	2134.48	3640152	48.4944 ng/ml
THC-COOH	1.730	225537	∞	55.1	1926.32	236070	50.1209 ng/ml
THC-OH	1.678	577189	∞	12.6	1481.21	836812	48.8419 ng/ml

AM #27 Cannabinoids Quant. Results

Batch results	D:\MassHunter\Data\2019\AM 25\072319 wklst 3556 MDS TS_reinjects\QuantResults\THCQ TS.batch.bin
Calibration Last Update	7/26/2019 11:40:49 AM

Instrument	Falco	**Data File**	THC_Cal 6-100ng.d
Type	Cal	**Sample**	THC_Cal 6-100ng
Acq. Method	AM 27 THC quant.m		
Sample Position	P3-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	7/24/2019 6:10:52 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.912	2892744	15973.09	27.7	4122.69	3590759	99.1417 ng/ml
THC-COOH	1.730	431748	∞	58.3	1965.57	237031	97.3479 ng/ml
THC-OH	1.678	1221791	2543.04	12.9	4740.85	885048	98.7087 ng/ml