

**REVIEWED**

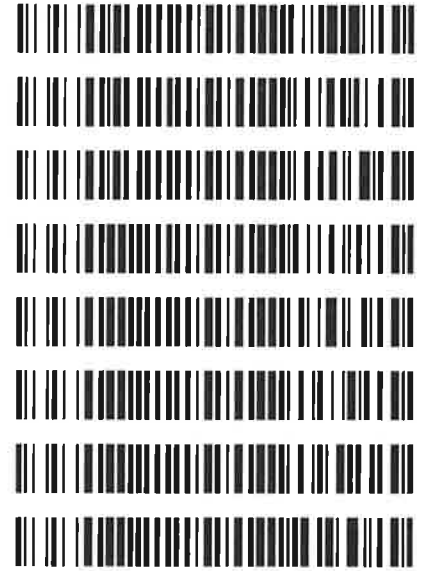
By Celena Shrum at 11:14 am, Jun 27, 2019

6/20/2019

TS

**Worklist: 3501**

<u>LAB CASE</u>	<u>ITEM</u>	<u>TASK ID</u>	<u>DESCRIPTION</u>
M2019-2510	1	155407	AM 27 Blood THC Quant by LC-QQQ
M2019-2511	1	155408	AM 27 Blood THC Quant by LC-QQQ
M2019-2512	1	155409	AM 27 Blood THC Quant by LC-QQQ
P2019-1686	1	155410	AM 27 Blood THC Quant by LC-QQQ
P2019-1710	1	155411	AM 27 Blood THC Quant by LC-QQQ
P2019-1711	1	155412	AM 27 Blood THC Quant by LC-QQQ
P2019-1714	1	155413	AM 27 Blood THC Quant by LC-QQQ
P2019-1746	2	155414	AM 27 Blood THC Quant by LC-QQQ



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

Extraction Date: 06/24/19  
Plate lot#: 0539904

Analyst: Tamara Salazar  
Plate Expiration: 09/10/19

**Mobile phase A:** 0.1% Formic Acid in LCMS Water  
MTBE LCMS Methanol

**Mobile phase B:** 0.1% Formic acid in Acetonitrile  
Hexane

**Blank Blood Lot:** Hemostat 445283-1  
**LCMS-QQQ ID:** 069901

**Column:** UCT Selectra DA 100 x 2.1mm 3um

## 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: 27** 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\062419 MDQ P1 P2 and THCQ TS  
Batch Name THCQ wklst 3501 TS
- 2. Make any necessary integration changes, Curve weighting of Linear 1/x with r<sup>2</sup> 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 5-100*



# Idaho State Police Forensic Services

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## 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 <sup>TS</sup> 20
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	

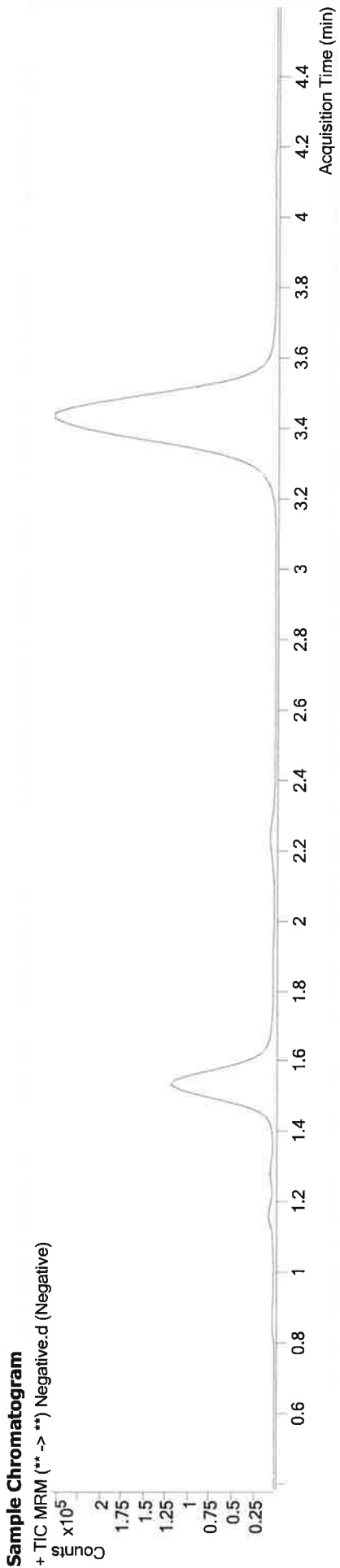
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# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCO TS\QuantResults\THCO wklst 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

<b>Instrument Type</b>	Falco Sample	<b>Data File Sample</b>	Negative.d Negative
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Comment</b>	
<b>Sample Position</b>	P3-A2		
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	6/24/2019 7:04:31 PM		



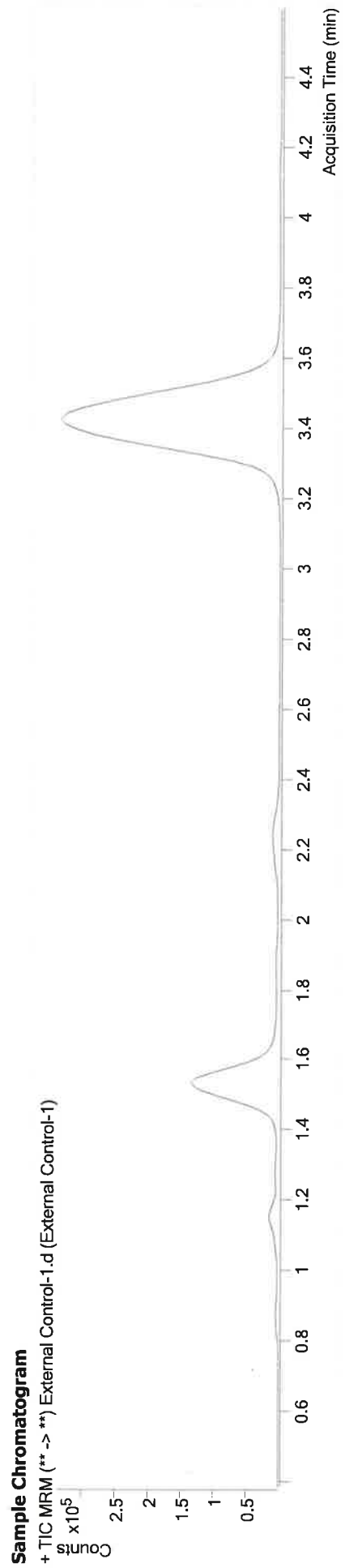
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# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wkfst 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

<b>Instrument Type</b>	Falco	<b>Data File</b>	External Control-1.d
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Sample</b>	External Control-1
<b>Sample Position</b>	P3-B2	<b>Comment</b>	
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	6/24/2019 7:19:43 PM		



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.446	192938	2747.21	27.8	162.69	3307129	7.1359 ng/ml
THC-COOH	1.565	37285	189.46	45.0	260.67	238472	8.5561 ng/ml
THC-OH	1.543	65790	∞	12.4	184.92	499932	8.1145 ng/ml

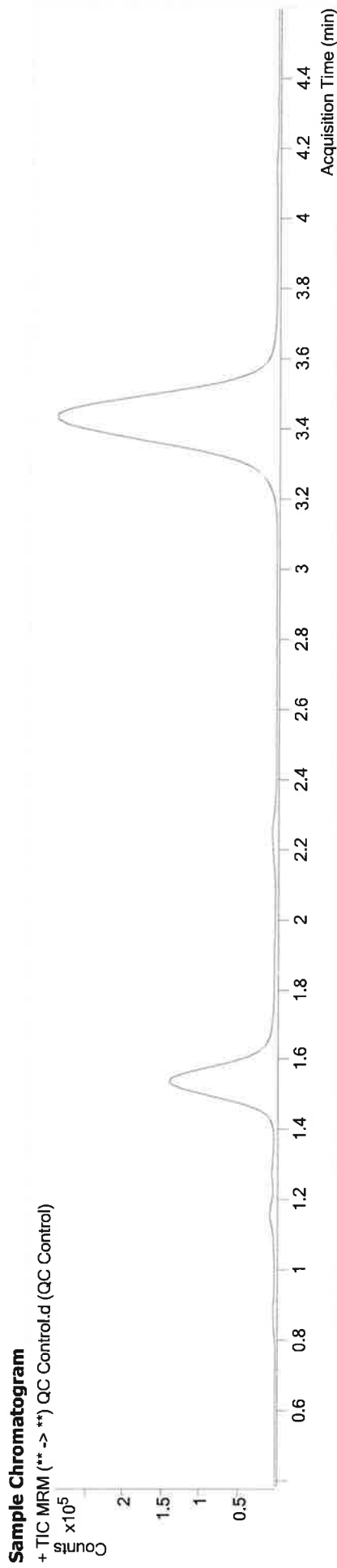
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# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wklet 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

Instrument Type Falco  
Sample QC Control.d  
Acq. Method AM 27 THC quant.m  
Sample Position P3-H1  
Injection Volume 10  
Acq. Date-Time 6/24/2019 6:49:21 PM  
Sample Info. QC Control



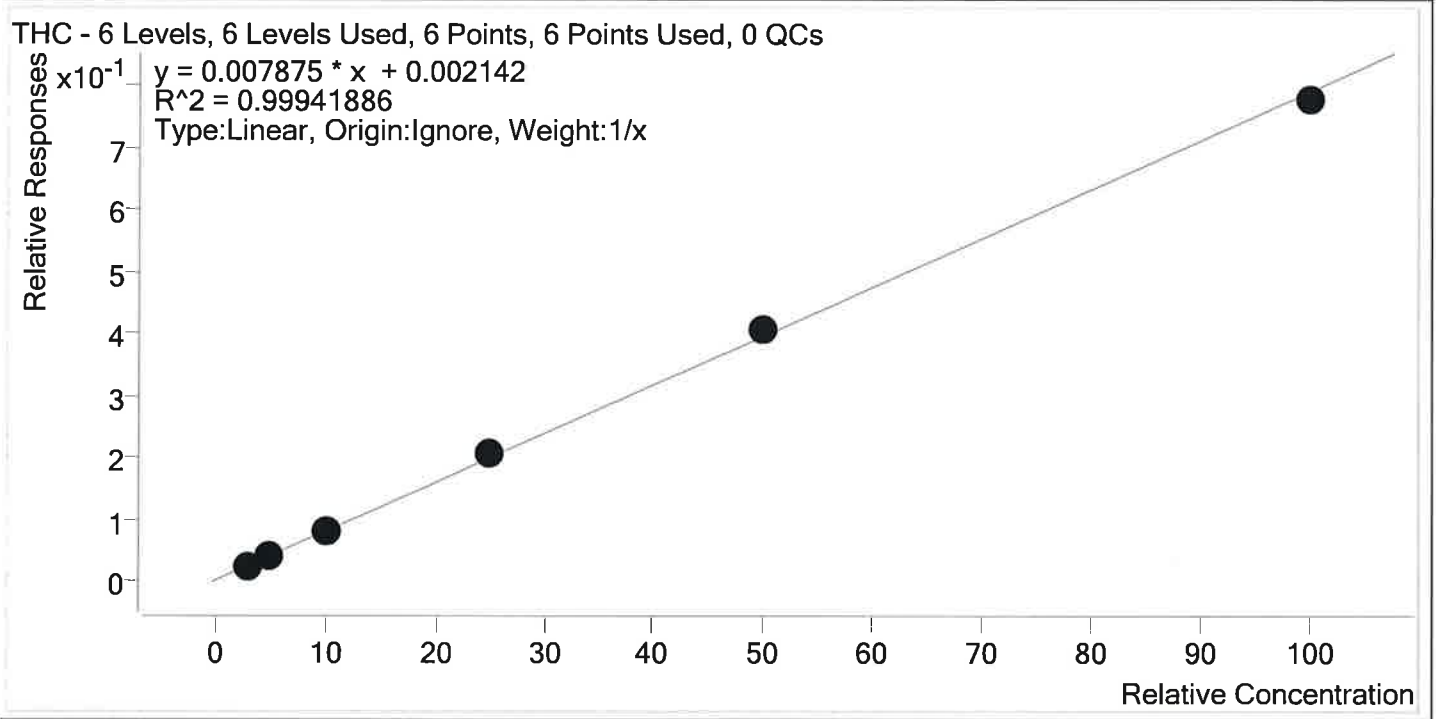
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.446	106227	283.92	28.2	96.50	2670734	4.7785 ng/ml
THC-COOH	1.580	39686	178.23	53.7	326.31	229388	9.6856 ng/ml
THC-OH	1.558	49597	∞	11.5	81.90	548675	5.1979 ng/ml

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# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wk1st  
3501 TS.batch.bin  
**Last Cal. Update** 6/25/2019 9:00 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	3.0	99.5
Cal 2- 5ng	2	✓	5.0	4.9	98.5
Cal 3 -10ng	3	✓	10.0	9.8	98.4
Cal 4-25ng	4	✓	25.0	25.7	102.9
Cal 5-50ng	5	✓	50.0	51.2	102.4
Cal 6-100ng	6	✓	100.0	98.3	98.3

TS



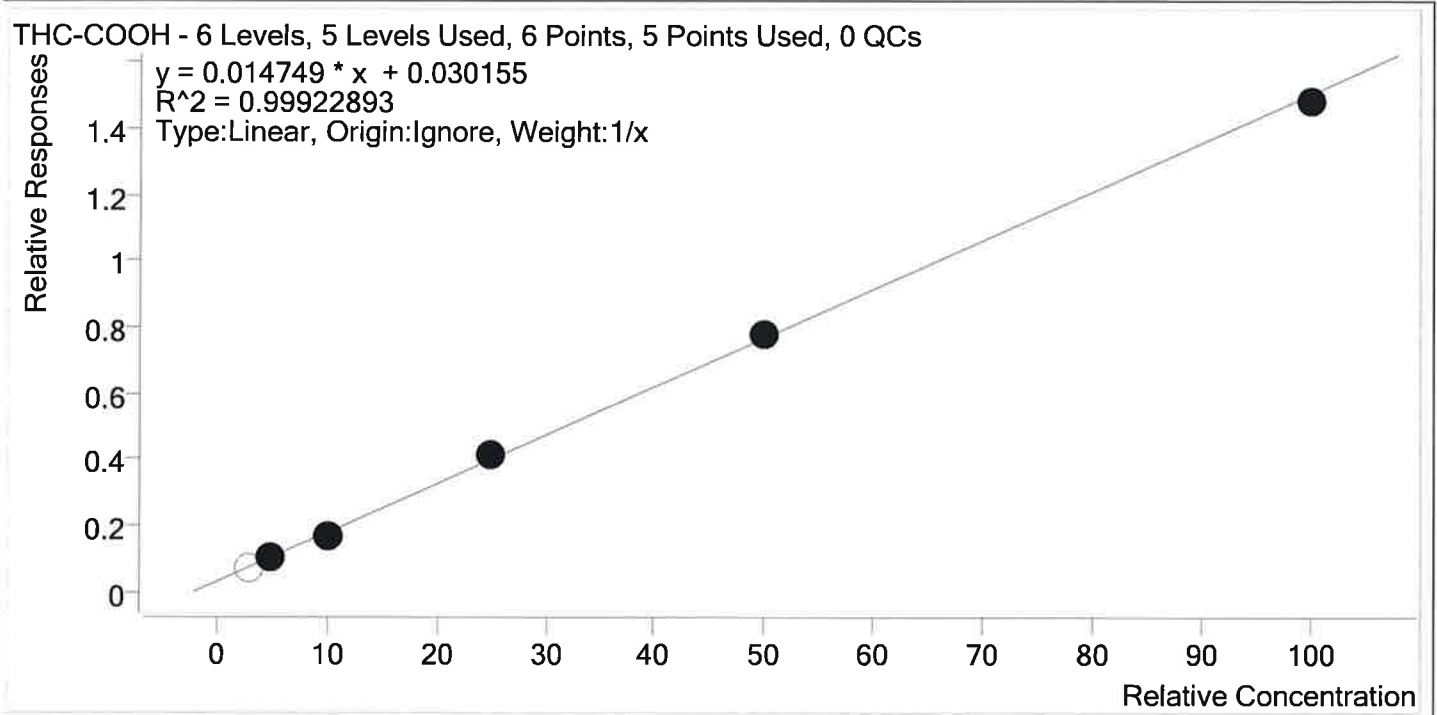
# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wk1st  
3501 TS.batch.bin

**Last Cal. Update** 6/25/2019 9:00 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	x	3.0	3.0	99.7
Cal 2- 5ng	2	✓	5.0	5.0	100.1
Cal 3 -10ng	3	✓	10.0	9.6	95.8
Cal 4-25ng	4	✓	25.0	25.9	103.5
Cal 5-50ng	5	✓	50.0	51.1	102.2
Cal 6-100ng	6	✓	100.0	98.5	98.5

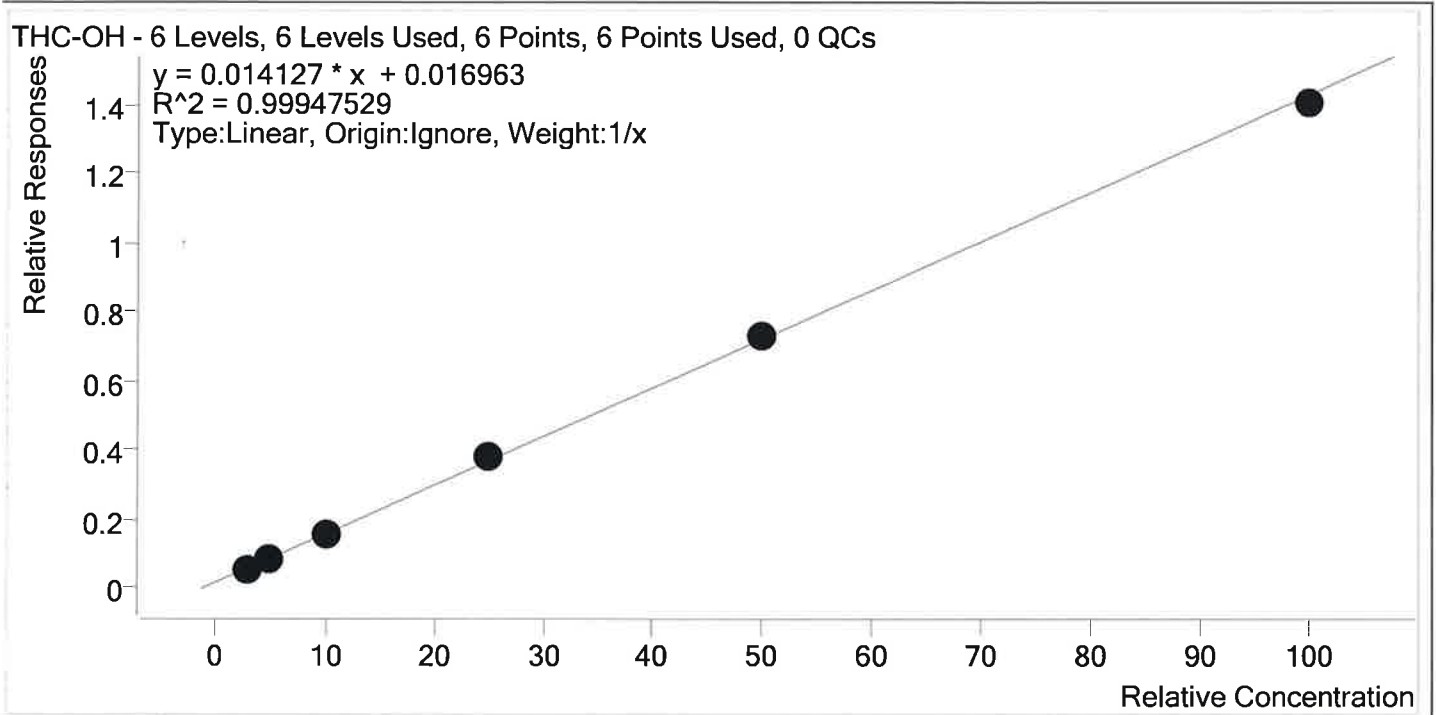


B



# AM #27 Cannabinoids Quant. Calibration Curve Report

**Batch results** D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wk1st  
 3501 TS.batch.bin  
**Last Cal. Update** 6/25/2019 9:00 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	2.9	96.4
Cal 2- 5ng	2	✓	5.0	5.0	99.9
Cal 3 -10ng	3	✓	10.0	10.0	100.2
Cal 4-25ng	4	✓	25.0	25.8	103.2
Cal 5-50ng	5	✓	50.0	50.9	101.9
Cal 6-100ng	6	✓	100.0	98.4	98.4

TS

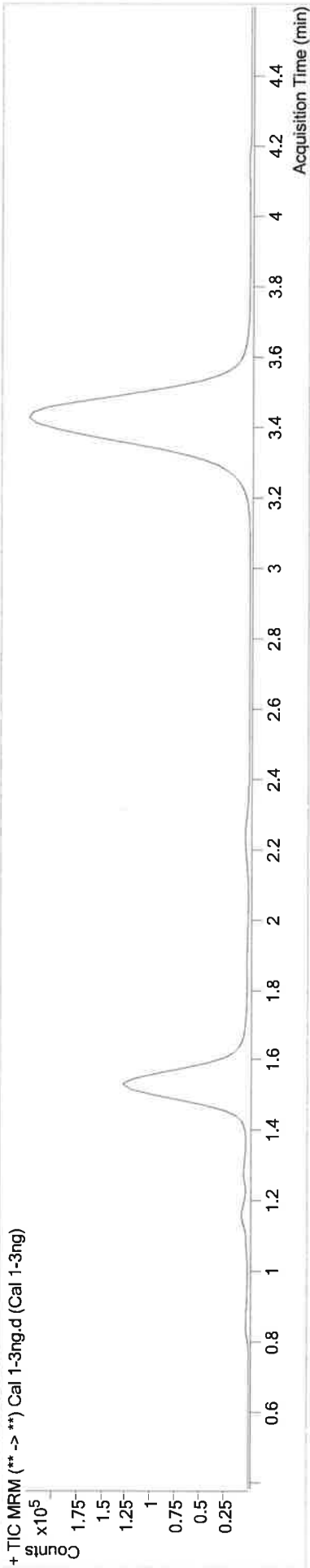


# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wk1st 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

Instrument Type	Data File Sample	Comment
Falco Cal	Cal 1-3ng.d	
Acq. Method	AM 27 THC quant.m	
Sample Position	P3-B1	
Injection Volume	10	
Acq. Date-Time	6/24/2019 6:03:55 PM	

## Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.446	54856	295.37	28.0	105.51	2139456	2.9838 ng/ml
THC-COOH	1.565	16370	∞	38.2 <b>Low</b>	114.32	220361	2.9923 ng/ml
THC-OH	1.558	30286	∞	11.6	32.10	523757	2.8924 ng/ml

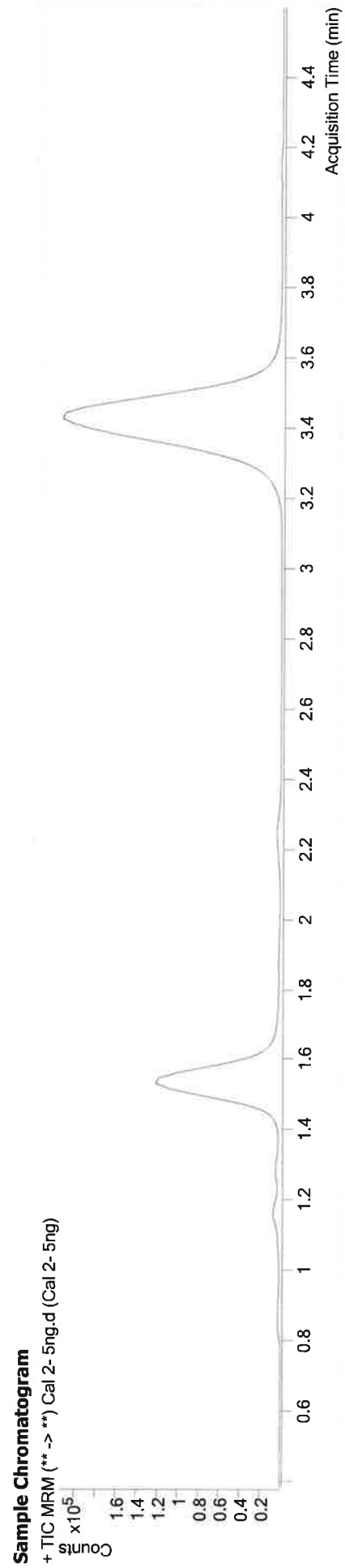
TS



# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wk1st 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

<b>Instrument Type</b>	Falco Cal	<b>Data File Sample</b>	Cal 2- 5ng.d Cal 2- 5ng
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Comment</b>	
<b>Sample Position</b>	P3-C1		
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	6/24/2019 6:11:30 PM		



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.446	80192	386.80	27.9	∞	1958869	4.9262 ng/ml
THC-COOH	1.565	21460	∞	44.2	207.33	206351	5.0067 ng/ml
THC-OH	1.558	42171	∞	12.3	44.98	481707	4.9963 ng/ml

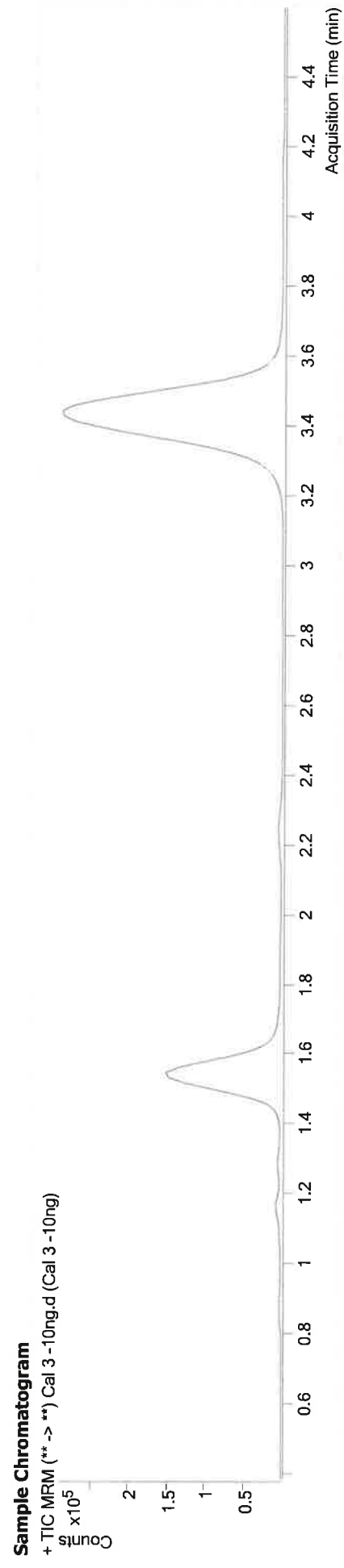
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# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wkfst 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

Instrument Type Falco Cal  
Acq. Method AM 27 THC quant.m  
Sample Position P3-D1  
Injection Volume 10  
Acq. Date-Time 6/24/2019 6:19:04 PM  
Sample Info. Cal 3 -10ng.d  
Cal 3 -10ng



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.446	202477	826.65	27.6	367.94	2543004	9.8382 ng/ml
THC-COOH	1.580	40321	141.58	50.9	331.08	235223	9.5777 ng/ml
THC-OH	1.558	88130	154.06	12.7	96.89	555858	10.0222 ng/ml

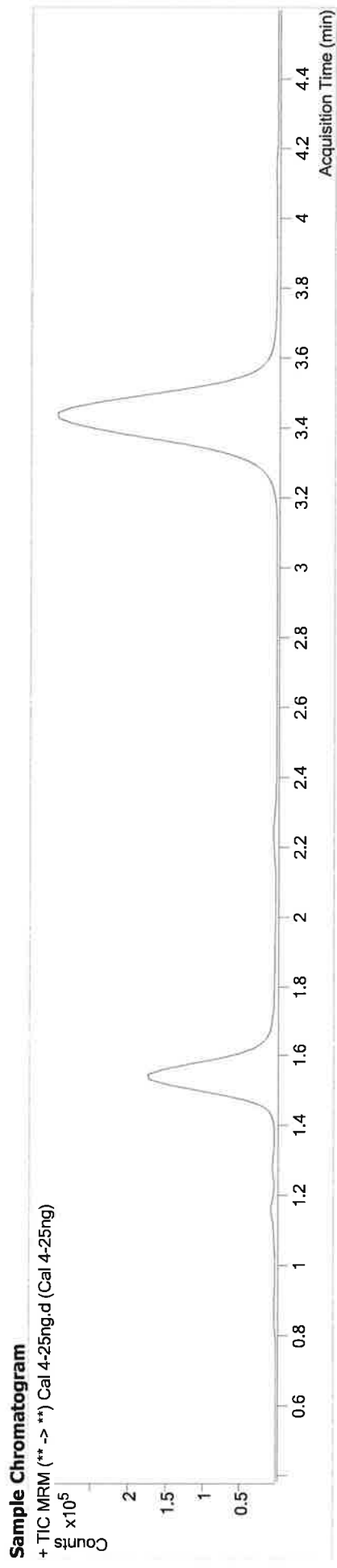
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# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wkst 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

<b>Instrument Type</b>	Falco Cal	<b>Data File Sample</b>	Cal 4-25ng.d Cal 4-25ng
<b>Acq. Method</b>	AM 27 THC quant.m	<b>Comment</b>	
<b>Sample Position</b>	P3-E1		
<b>Injection Volume</b>	10		
<b>Acq. Date-Time</b>	6/24/2019 6:26:38 PM		
<b>Sample Info.</b>			



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.446	451065	1026.55	27.4	996.39	2203289	25.7234 ng/ml
THC-COOH	1.580	89167	389.00	55.5	268.23	216628	25.8636 ng/ml
THC-OH	1.543	188614	∞	13.9	222.57	494357	25.8063 ng/ml

AS

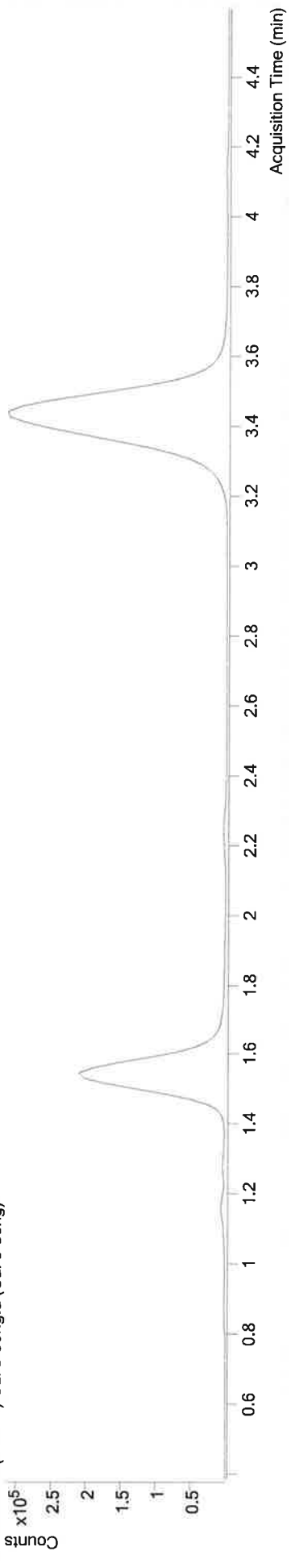


# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wk1st 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

Instrument Type	Falco Cal	Data File Sample	Comment
Acq. Method	AM 27 THC quant.m	Cal 5-50ng.d	
Sample Position	P3-F1	Cal 5-50ng	
Injection Volume	10		
Acq. Date-Time	6/24/2019 6:34:12 PM		

Sample Chromatogram + TIC MRM (\*\* -> \*\*) Cal 5-50ng.d (Cal 5-50ng)



Name	RT	Resp.	S/N	Ratio	ISTD Resp.	Final Conc.
THC	3.446	812204	5629.00	27.6	2003185	51.2121 ng/ml
THC-COOH	1.580	159537	74.37	56.2	203607	51.0818 ng/ml
THC-OH	1.543	346445	∞	14.1	470405	50.9315 ng/ml

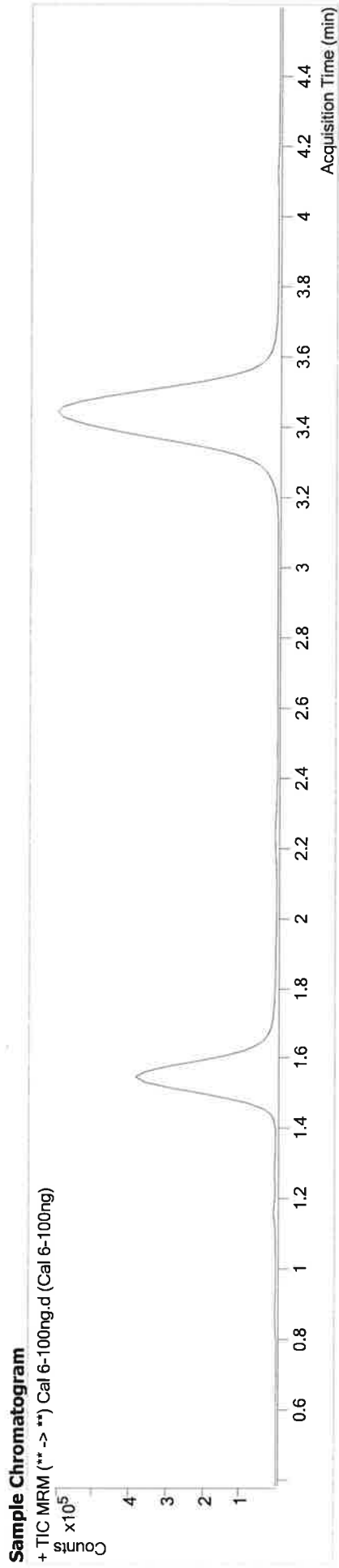
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# AM #27 Cannabinoids Quant. Results

Batch results Calibration Last Update D:\MassHunter\Data\2019\AM 28\062419 MDQ P1 P2 and THCQ TS\QuantResults\THCQ wk1st 3501 TS.batch.bin  
6/25/2019 9:00:03 AM

Instrument Type Falco Cal  
Acq. Method AM 27 THC quant.m  
Sample Position P3-G1  
Injection Volume 10  
Acq. Date-Time 6/24/2019 6:41:46 PM  
Sample Info. Cal 6-100ng.d  
Cal 6-100ng



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC	3.446	2287261	15560.21	27.3	3617.42	2945913	98.3163 ng/ml
THC-COOH	1.580	365879	123.34	57.3	405.62	246803	98.4702 ng/ml
THC-OH	1.558	839962	1364.17	13.9	534.97	597246	98.3513 ng/ml