

REVIEWED

By Anne Nord at 2:31 pm, Nov 19, 2018

REVIEWED

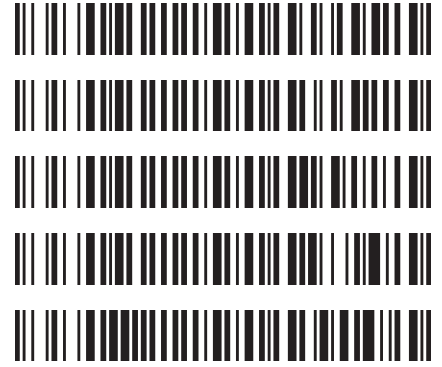
By tsalazar at 12:12 pm, Dec 17, 2018

11/1/2018



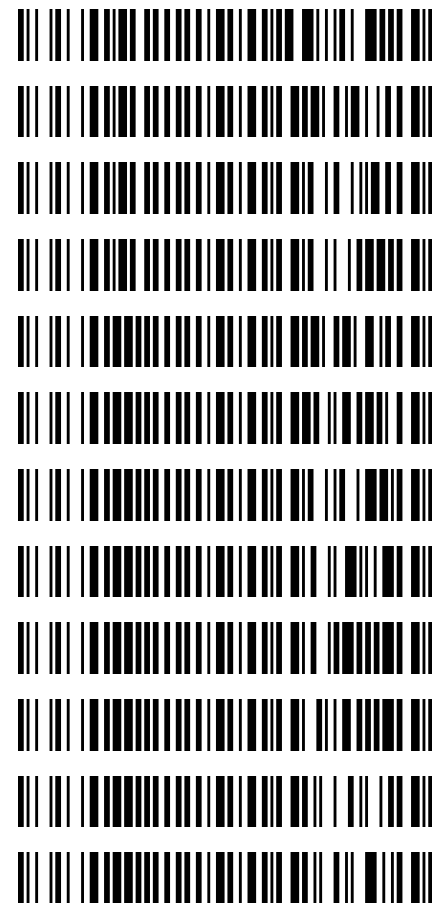
Worklist: 2774

<u>LAB_CASE</u>	<u>ITEM</u>	<u>TASK_ID</u>	<u>DESCRIPTION</u>
M2018-5040	2	130933	AM 27 Blood THC Quant by LC-QQQ
M2018-5085	1	130934	AM 27 Blood THC Quant by LC-QQQ
M2018-5167	4	130935	AM 27 Blood THC Quant by LC-QQQ
M2018-5222	5	130936	AM 27 Blood THC Quant by LC-QQQ
P2018-3016	1	130937	AM 27 Blood THC Quant by LC-QQQ



Worklist: 2791

<u>LAB_CASE</u>	<u>ITEM</u>	<u>TASK_ID</u>	<u>DESCRIPTION</u>
M2018-4768	1	131989	AM 27 Blood THC Quant by LC-QQQ
M2018-5224	1	131990	AM 27 Blood THC Quant by LC-QQQ
M2018-5424	3	131991	AM 27 Blood THC Quant by LC-QQQ
M2018-5473	3	131992	AM 27 Blood THC Quant by LC-QQQ
P2018-3025	1	131993	AM 27 Blood THC Quant by LC-QQQ
P2018-3075	1	132023	AM 27 Blood THC Quant by LC-QQQ
P2018-3121	1	131994	AM 27 Blood THC Quant by LC-QQQ
P2018-3146	1	131996	AM 27 Blood THC Quant by LC-QQQ
P2018-3149	1	131997	AM 27 Blood THC Quant by LC-QQQ
P2018-3150	1	131998	AM 27 Blood THC Quant by LC-QQQ
P2018-3180	1	131999	AM 27 Blood THC Quant by LC-QQQ
P2018-3181	1	132000	AM 27 Blood THC Quant by LC-QQQ



CS S

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

Extraction Date: 11/08/18
Plate lot#: 0539904

Analyst: Sarah Pickle and Celena Shrum
Plate Expiration: 09/10/19

Mobile phase A: 0.1% Formic Acid in LCMS Water
MTBE
Mobile phase B: 0.1% Formic acid in Acetonitrile
LCMS Methanol Hexane
Blank Blood Lot: 445283-1
Column: UCT Selectra DA 100 x 2.1mm 3um
LCMS-QQQ ID: 59740

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: Data Path: _____

Analytic:

- 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
- 2. Pipette **1000µL blood (calibrated pipette) Pipette ID: 3382167** 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.
- 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: 110818 THCQ SP CS Batch Name: 110818 THCQ Final
- 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? Y / N
- 6. Enter QCs into control charting.
- 7. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: *Due to poor peak shape, THC will only be reported out qualitatively for this batch. Steps 3-16 were performed by Sarah Pickle. I witnessed and approved of all steps performed in the method.* CS



Idaho State Police Forensic Services

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

Analyst: Sarah Pickle and Celena Shrum
Extraction Date: 11/08/18
Worklist Number: 2791 and 2774

<i>Reagent</i>	<i>Lot Number</i>	<i>Expiration Date</i>	<i>Date in Service</i>	<i>Date Out of Service</i>	<i>Initials</i>
ToxBox THC/THC Metabolite Plate	0539904	09/10/19			
Negative Blood	445283-1		10/25/18		
Methanol External Control Solution	WS102418	02/08/19	10/24/18		
Blood External Control Solution	102418	02/08/19	10/24/18		
Methyl Tert-Butyl Ether (MTBE) 99.9%	A0375555		6/26/17		
Hexanes (ACS)	101642		10/26/17		
Methanol (LCMS Grade)	177145		4/11/18		
0.1% Formic Acid in Water (Mobile Phase A)	100518		10/05/18		
0.1% Formic Acid in Acetonitrile (Mobile Phase B)	181692		11/08/18		
Needle Rinse--75% LCMS MeOH in LCMS Water	110818		11/08/18		

Methanol External Control Solution (Lot: WS102418)

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

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>	<i>Expiration Date</i>
Methanol (LCMS)	Fisher	177145	
THC	Cerilliant	FE04231406	04/30/2019
C-THC	Cayman	0497429	02/08/2019
THC-OH	Cerilliant	FE01121503	01/31/2020
Prepared:	10/24/18		
Prepared By:	Tamara Salazar		
Expires:	02/08/19		

Blood External Control Solution (Lot: 102418)

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

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Negative Blood	Lampire	18G207D7
Methanol External Control Solution		WS102418
Prepared:	10/24/18	
Prepared by:	Sarah Pickle	
Expires:	02/08/19	

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

0.1% Formic Acid in LCMS Water (Mobile Phase A) (Lot: 100518)

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Formic Acid (LCMS Grade)	Fisher	095180B
Water (LCMS Grade)	Fisher	182702
Prepared:	10/05/18	
Prepared By:	Sarah Pickle	

Needle Rinse (75% LCMS MeOH in LCMS Water) (Lot: 110818)

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
MeOH (LCMS Grade)	Fisher	177145
Water (LCMS Grade)	Fisher	182702
Prepared:	11/08/18	
Prepared By:	Sarah Pickle	

ISP FORENSICS - Pocatello Instrument # 59740

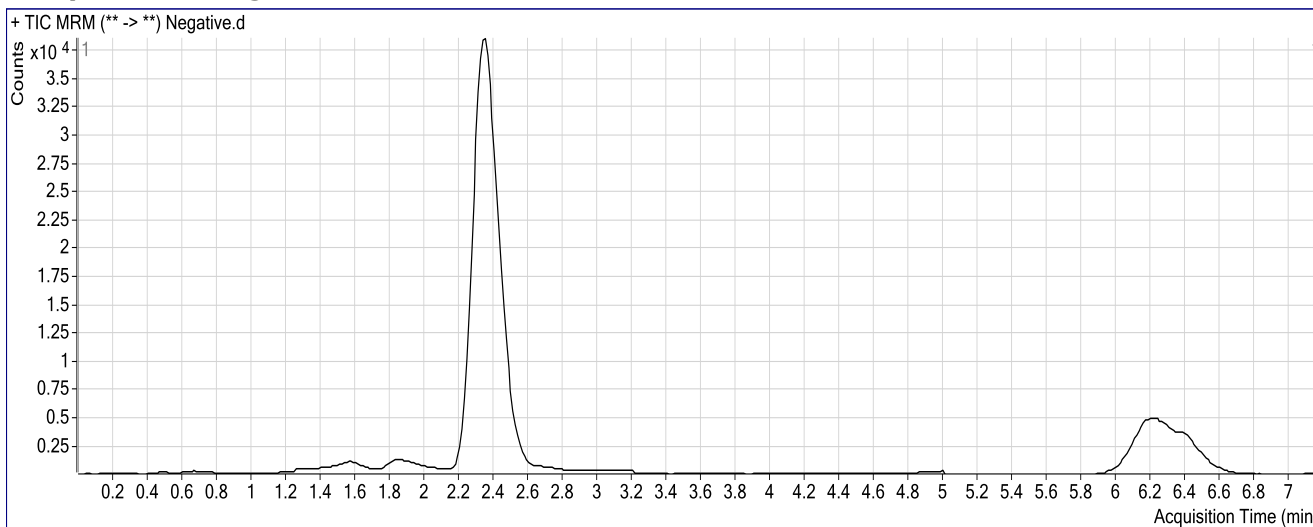
Cannabinoids Analysis Report

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Analysis Time	11/16/2018 10:16 AM	Analyst Name	ISPUser
Report Time	11/16/2018 10:18 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 13:34	Data File	Negative.d
Sample Type	Sample	Sample Name	Negative
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-A6	Sample Info	
Inj Vol	-1	Comment	Hemostat 445283-1

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-COOH	THC-COOH-D9	2.325	3055	107586	0.0284	0.1713

ISP FORENSICS - Pocatello Instrument # 59740

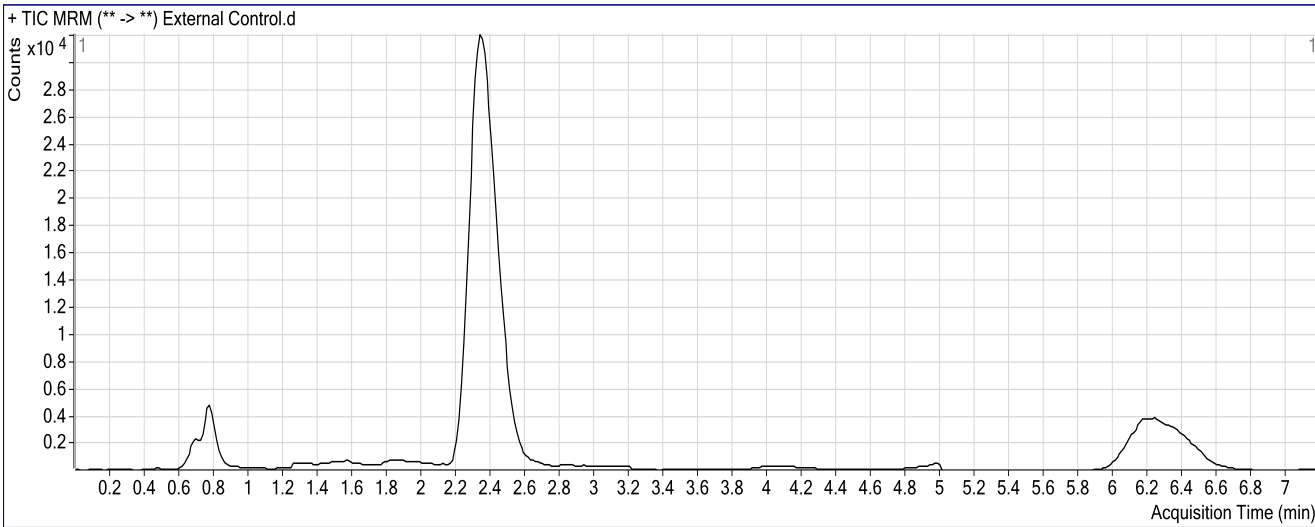
Cannabinoids Analysis Report

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Report Time	11/16/2018 10:18 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 13:58	Data File	External Control.d
Sample Type	Sample	Sample Name	External Control
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-B6	Sample Info	
Inj Vol	-1	Comment	Lampire 18G207D7 + WS 102418

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.345	21696	220272	0.0985	9.5582
THC-COOH	THC-COOH-D9	2.446	15908	88773	0.1792	8.6638
THC	THC-D3	6.252	6857	80867	0.0848	10.2084

ISP FORENSICS - Pocatello Instrument # 59740

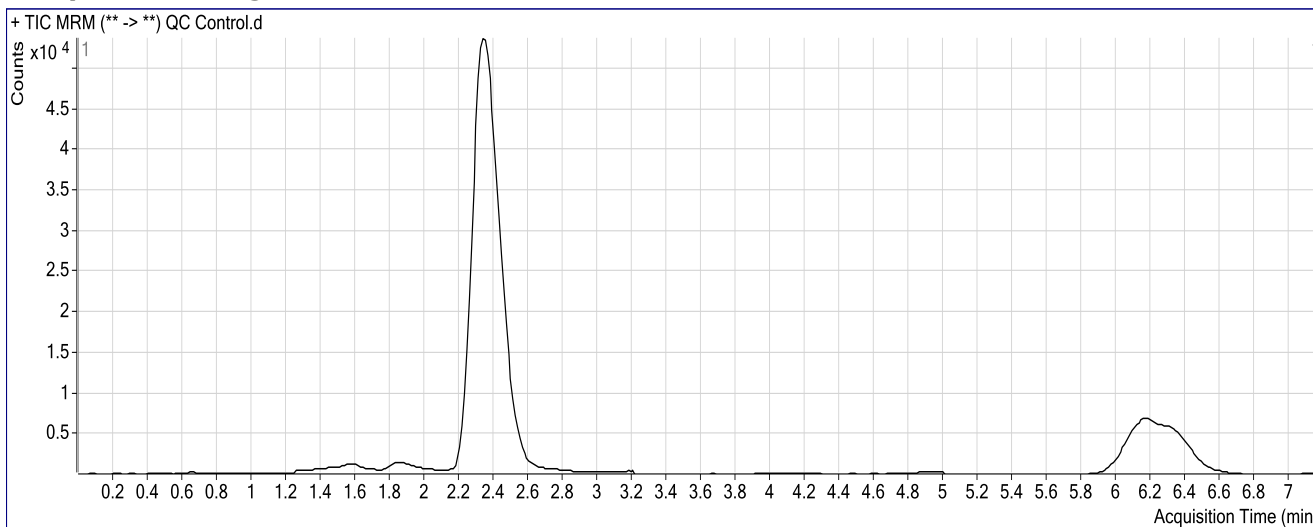
Cannabinoids Analysis Report

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Report Time	11/16/2018 10:17 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 13:11	Data File	QC Control.d
Sample Type	Sample	Sample Name	QC Control
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-H5	Sample Info	
Inj Vol	-1	Comment	

Sample Chromatogram



Results

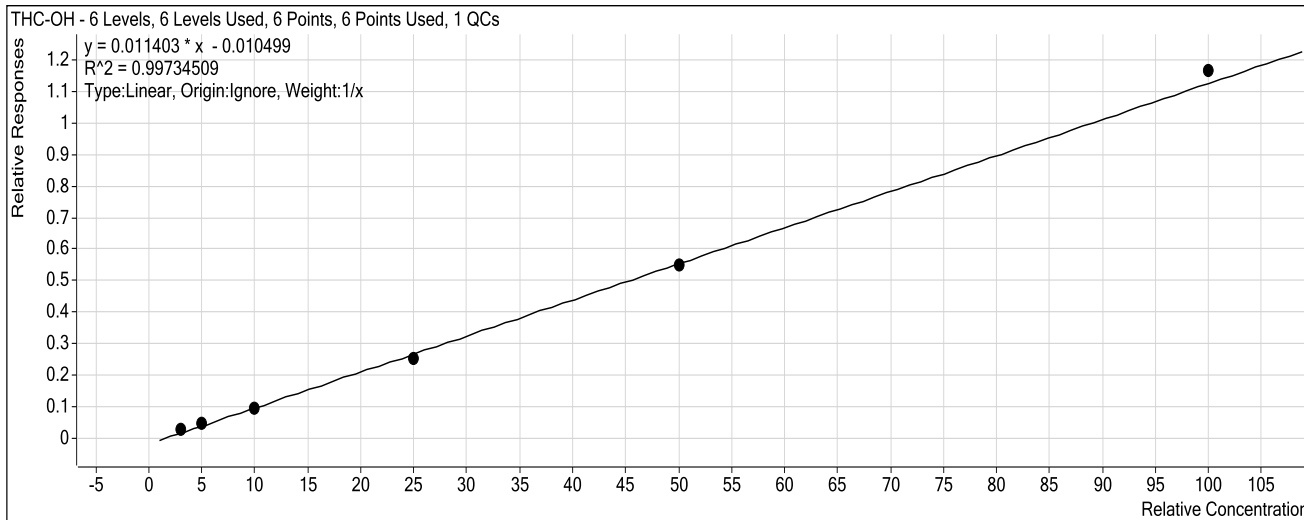
Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.359	20265	396698	0.0511	5.4004
THC-COOH	THC-COOH-D9	2.459	26831	137300	0.1954	9.5773
THC	THC-D3	6.252	6209	150850	0.0412	4.9145

ISP Forensics Calibration Curve Report



Batch Data Path C:\MassHunter\Data\2018\THC Quant\110818 THCQ SP CS\QuantResults\110818 THCQ Final.batch.bin
Last Calib Update 11/16/2018 10:16 AM **Analyst Name** ISP TOX

Target Compound *THC-OH*
Internal Standard *THC-OH-D3*



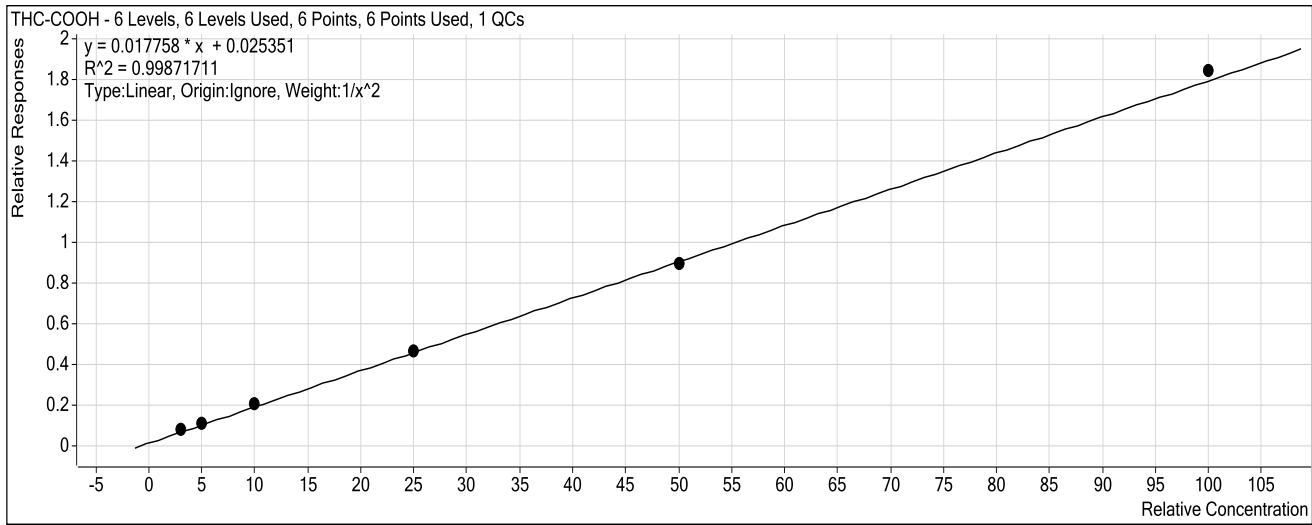
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Cal 1-1ng	1	<input type="checkbox"/>	1	1.8	183.2
Cal 2-3ng	2	<input checked="" type="checkbox"/>	3	3.4	112.4
Cal 3-5ng	3	<input checked="" type="checkbox"/>	5	5.1	101.3
Cal 4-10ng	4	<input checked="" type="checkbox"/>	10	9.3	93.0
Cal 5-25ng	5	<input checked="" type="checkbox"/>	25	23.0	92.1
Cal 6-50ng	6	<input checked="" type="checkbox"/>	50	49.0	98.0
Cal 7-100ng	7	<input checked="" type="checkbox"/>	100	103.3	103.3

ISP Forensics Calibration Curve Report



Batch Data Path C:\MassHunter\Data\2018\THC Quant\110818 THCQ SP CS\QuantResults\110818 THCQ Final.batch.bin
Last Calib Update 11/16/2018 10:16 AM **Analyst Name** ISP TOX

Target Compound *THC-COOH*
Internal Standard *THC-COOH-D9*



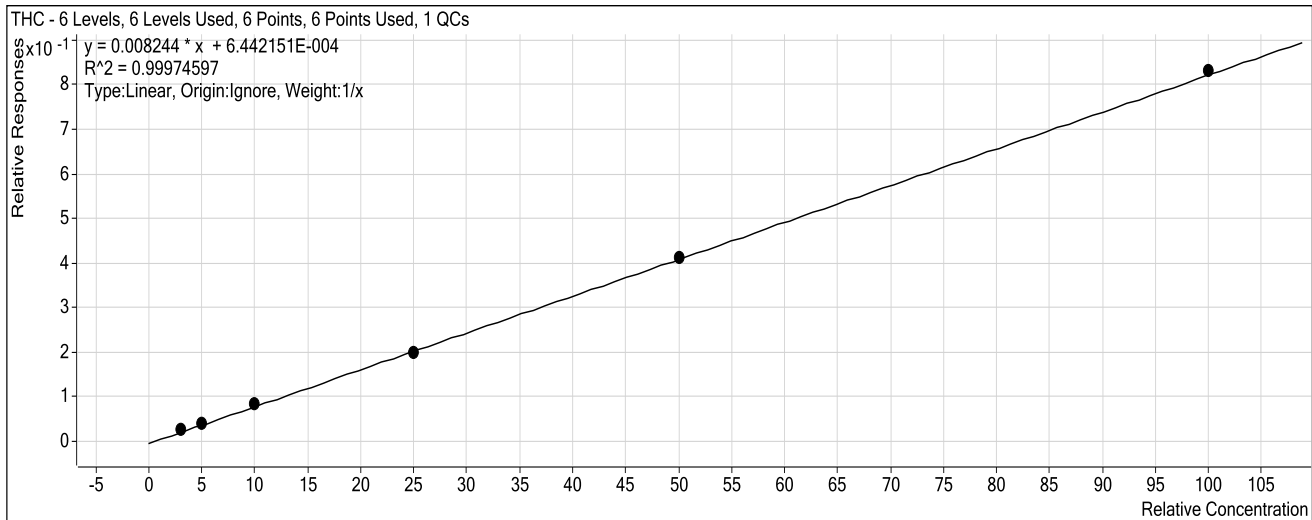
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-1ng	1	<input type="checkbox"/>	1	2.4	243.3
Cal 2-3ng	2	<input checked="" type="checkbox"/>	3	3.1	102.0
Cal 3-5ng	3	<input checked="" type="checkbox"/>	5	4.8	95.7
Cal 4-10ng	4	<input checked="" type="checkbox"/>	10	10.2	102.3
Cal 5-25ng	5	<input checked="" type="checkbox"/>	25	24.9	99.5
Cal 6-50ng	6	<input checked="" type="checkbox"/>	50	49.0	98.0
Cal 7-100ng	7	<input checked="" type="checkbox"/>	100	102.5	102.5

ISP Forensics Calibration Curve Report



Batch Data Path C:\MassHunter\Data\2018\THC Quant\110818 THCQ SP CS\QuantResults\110818 THCQ Final.batch.bin
Last Calib Update 11/16/2018 10:16 AM **Analyst Name** ISP TOX

Target Compound *THC*
Internal Standard *THC-D3*



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-1ng	1	<input type="checkbox"/>	1	0.6	55.1
Cal 2-3ng	2	<input checked="" type="checkbox"/>	3	3.1	104.2
Cal 3-5ng	3	<input checked="" type="checkbox"/>	5	4.9	98.3
Cal 4-10ng	4	<input checked="" type="checkbox"/>	10	10.0	99.9
Cal 5-25ng	5	<input checked="" type="checkbox"/>	25	24.3	97.2
Cal 6-50ng	6	<input checked="" type="checkbox"/>	50	49.8	99.6
Cal 7-100ng	7	<input checked="" type="checkbox"/>	100	100.9	100.9

ISP FORENSICS - Pocatello Instrument # 59740

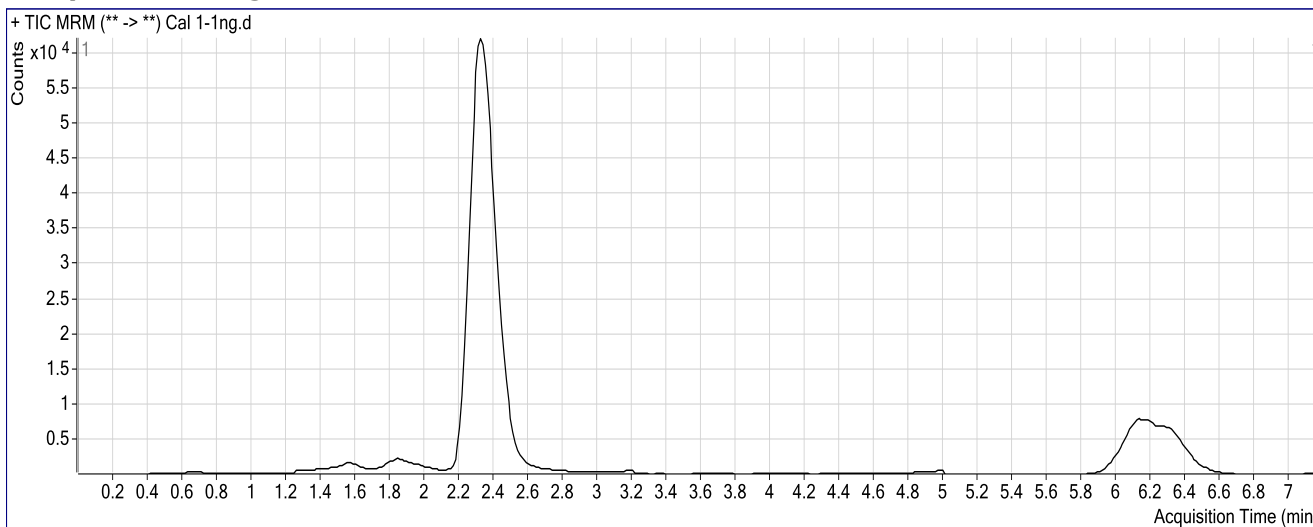
Cannabinoids Analysis Report

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Report Time	11/16/2018 10:17 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 11:36	Data File	Cal 1-1ng.d
Sample Type	QC	Sample Name	Cal 1-1ng
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-A5	Sample Info	
Inj Vol	-1	Comment	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.332	4918	473364	0.0104	1.8319
THC-COOH	THC-COOH-D9	2.432	11320	165128	0.0686	2.4329
THC	THC-D3	6.306	925	178416	0.0052	0.5511

ISP FORENSICS - Pocatello Instrument # 59740

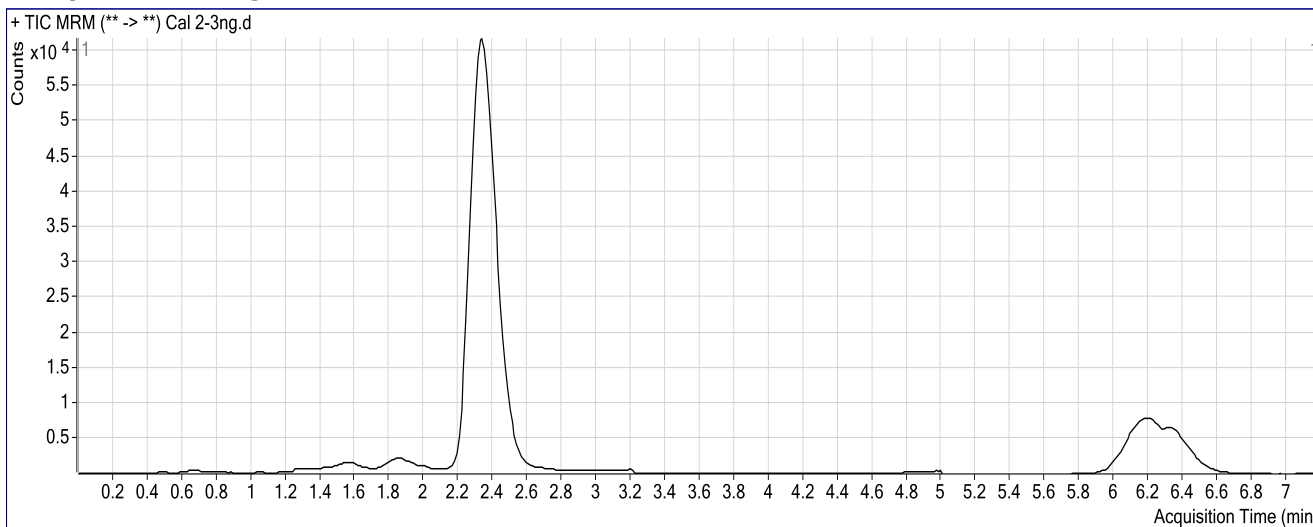
Cannabinoids Analysis Report

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Report Time	11/16/2018 10:17 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 11:48	Data File	Cal 2-3ng.d
Sample Type	Calibration	Sample Name	Cal 2-3ng
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-B5	Sample Info	
Inj Vol	-1	Comment	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.332	12897	461477	0.0279	3.3715
THC-COOH	THC-COOH-D9	2.446	12993	163070	0.0797	3.0595
THC	THC-D3	6.172	4396	166405	0.0264	3.1264

ISP FORENSICS - Pocatello Instrument # 59740

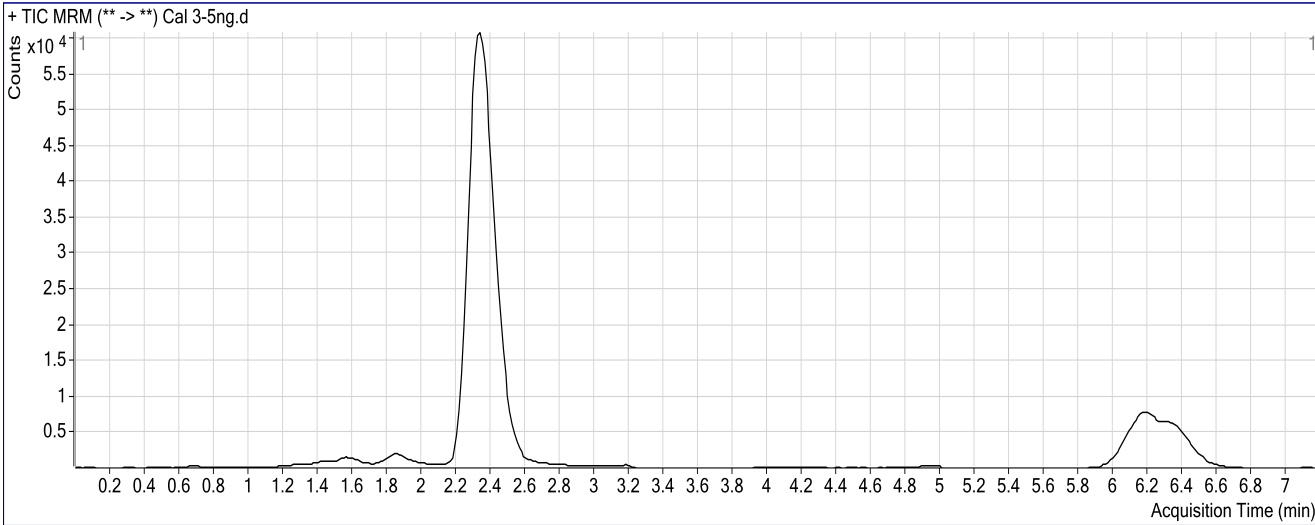
Cannabinoids Analysis Report

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Report Time	11/16/2018 10:17 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 12:00	Data File	Cal 3-5ng.d
Sample Type	Calibration	Sample Name	Cal 3-5ng
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-C5	Sample Info	
Inj Vol	-1	Comment	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.345	21248	449858	0.0472	5.0628
THC-COOH	THC-COOH-D9	2.446	16964	153755	0.1103	4.7855
THC	THC-D3	6.319	6781	164729	0.0412	4.9154

ISP FORENSICS - Pocatello Instrument # 59740

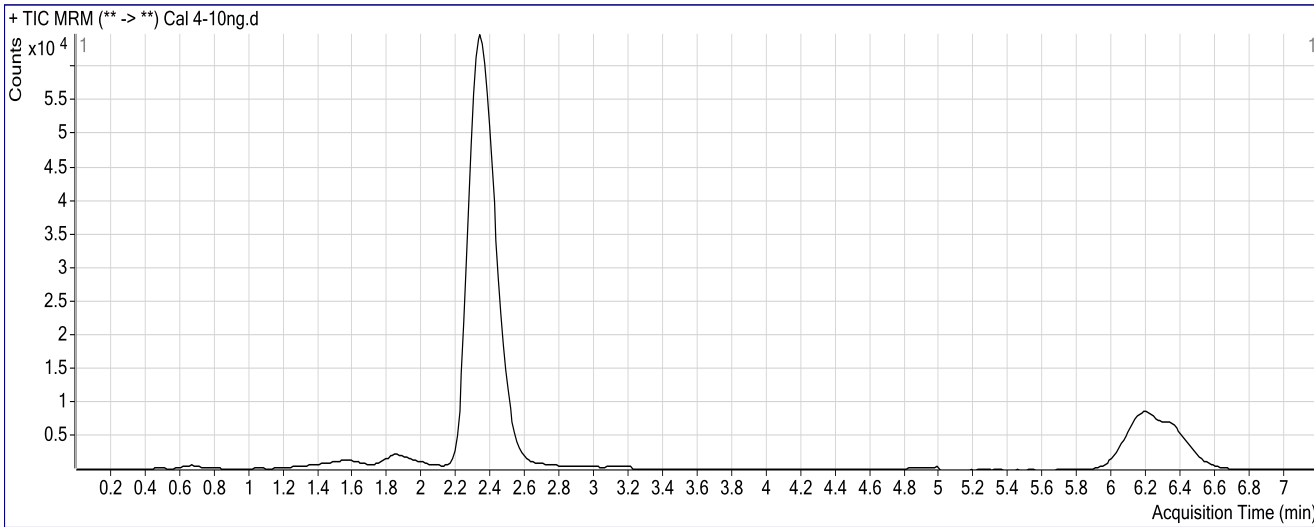
Cannabinoids Analysis Report

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Report Time	11/16/2018 10:17 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 12:11	Data File	Cal 4-10ng.d
Sample Type	Calibration	Sample Name	Cal 4-10ng
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-D5	Sample Info	
Inj Vol	-1	Comment	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.332	43730	457491	0.0956	9.3031
THC-COOH	THC-COOH-D9	2.446	32419	156562	0.2071	10.2330
THC	THC-D3	6.306	14278	172117	0.0830	9.9853

ISP FORENSICS - Pocatello Instrument # 59740

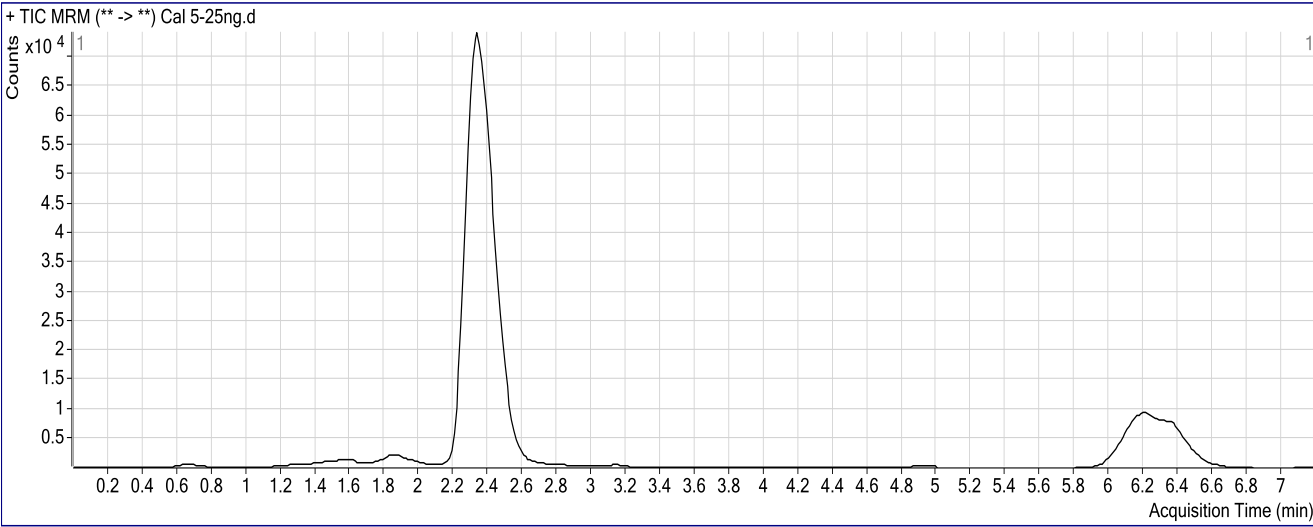
Cannabinoids Analysis Report

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Report Time	11/16/2018 10:17 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 12:23	Data File	Cal 5-25ng.d
Sample Type	Calibration	Sample Name	Cal 5-25ng
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-E5	Sample Info	
Inj Vol	-1	Comment	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.332	114138	452727	0.2521	23.0294
THC-COOH	THC-COOH-D9	2.446	72248	154721	0.4670	24.8685
THC	THC-D3	6.359	33438	166455	0.2009	24.2907

ISP FORENSICS - Pocatello Instrument # 59740

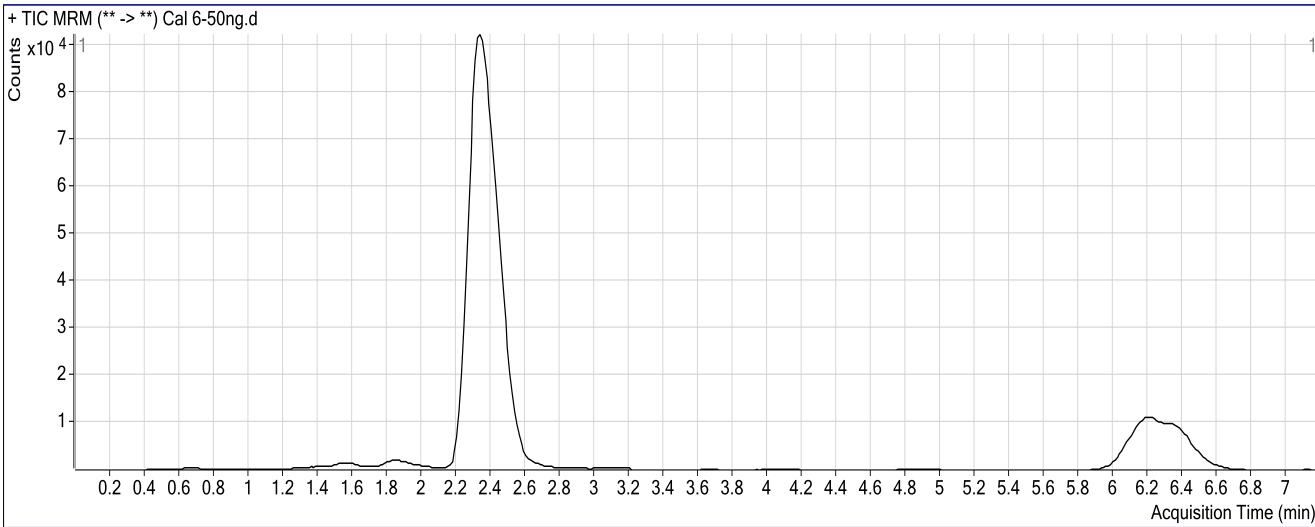
Cannabinoids Analysis Report

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Report Time	11/16/2018 10:17 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 12:35	Data File	Cal 6-50ng.d
Sample Type	Calibration	Sample Name	Cal 6-50ng
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-F5	Sample Info	
Inj Vol	-1	Comment	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.332	247789	452154	0.5480	48.9784
THC-COOH	THC-COOH-D9	2.446	137334	153334	0.8956	49.0096
THC	THC-D3	6.319	65306	158893	0.4110	49.7793

ISP FORENSICS - Pocatello Instrument # 59740

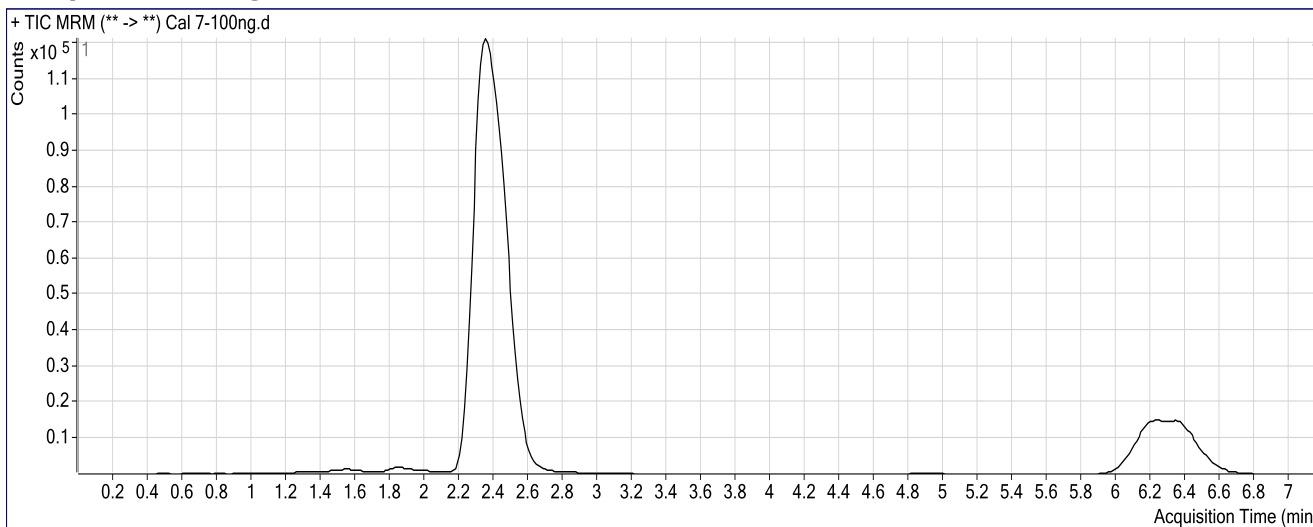
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Report Time	11/16/2018 10:17 AM	Reporter Name	ISPUser
Last Calib Update	11/16/2018 10:16 AM	Batch State	Processed

Analysis Info

Acq Time	2018-11-08 12:47	Data File	Cal 7-100ng.d
Sample Type	Calibration	Sample Name	Cal 7-100ng
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-G5	Sample Info	
Inj Vol	-1	Comment	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.345	498914	427536	1.1670	103.2548
THC-COOH	THC-COOH-D9	2.459	269990	146317	1.8452	102.4841
THC	THC-D3	6.346	133174	159980	0.8324	100.9029