

TS

reviewed 1/24/18

1/10/2018

BWylee

Worklist: 2120

<u>LAB CASE</u>	<u>ITEM</u>	<u>TASK ID</u>	<u>DESCRIPTION</u>
M2017-4452	1	104698	AM 27 Blood THC Quant by LC
M2017-4509	1	104700	AM 27 Blood THC Quant by LC
P2017-2635	1	104695	AM 27 Blood THC Quant by LC



Quantitation of THC and Metabolites in Blood by LC-MS/MS

TS

Extraction Date: 01/04/18

Analyst: Tamara Salazar

Plate lot#: 0499102

Plate Expiration: 01/29/18

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

Mobile phase B: 0.1% Formic acid in Acetonitrile
Hexane

Blank Blood Lot: 361331-3

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:


Analytic:

- 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
- 2. Pipette **1000µL blood (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.
- 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.

Performed by
Witnessed by
Sarah Pickle.
Tamara Salazar

Post-Analytic C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant

TS

- 1. Create batch and process data.
Worklist path:  Batch Name: 010418 THC Quant TS.batch.bin
- 2. Make any necessary integration changes, r^2 values ≥ 0.98 for each analyte.
- 3. Did all QCs pass for each analyte? Y/N Enter QCs into control charting?
- 4. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: Analysis was performed with Sarah Pickle acting as the primary technician. I pipetted and transferred my own case samples. I witnessed, reviewed, and approved of all work performed during the extraction. -TS

TS

Toxicology AM method 27 panel 1 external prep information

Working Solution 1 ug/ml in MeOH C-THC, THC-OH, THC

Stock Solution 1mg/ml 10 ul THC, 100ug/ml 100ul THC-OH, C-THC in 9790 ul MeOH (Fisher Lot # 172516)

Prepared: 01/04/18

Expires: 06/30/18

By:SP

reviewed 2/7/18

Byyle

TS

Drug	Lot	Expiration
C-THC	FE05311303	6/30/2018
THC-OH	FE04231406	4/30/2019
THC	FE01121503	4/30/2020

THC-OH:	Lot #: FE01121503	Expiration: 01/31/2020
THC:	Lot # FE04231406	Expiration: 04/30/2019

AM 27 control 100 ul working solution lot (WS010418) in 9900ul blood (Hemostat Lot# 361331-3)

Prepared: 01/04/18

Expires: 06/30/18

By:SP

TS

**Idaho State Police
Forensic Services
Toxicology Discipline**

Request for Departure from an Analytical Method

Date of Request
01/10/2017

Forensic Scientist
Tamara Salazar

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

Request

I am formally requesting a deviation to report the results from this run qualitatively due to the external control concentrations of THC and metabolites being slightly above the 10ng/mL limit. A new control with new standards will be made for the next run.

Discipline Leader Review

Departure approved
Comments:

Departure Not Approved
Comments:

Celena Shrum

Date: 01/10/18

Celena Shrum
Toxicology Discipline Lead

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ISP FORENSICS - Pocatello Instrument # 59740

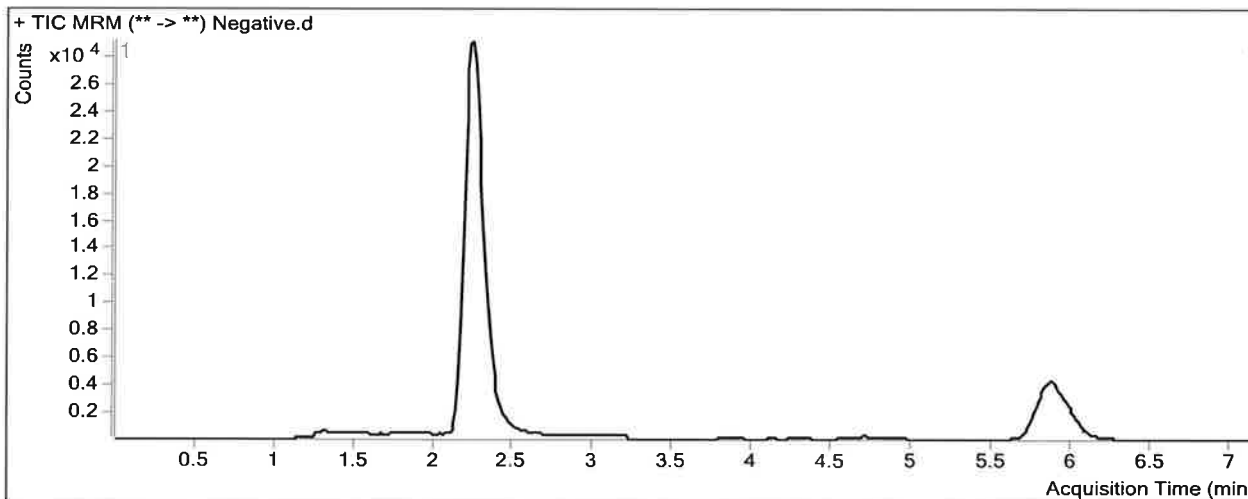
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418 THC Quant TS.bat
Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:40 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 18:08 **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 361331-3

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.078	524	192983	0.0027	0.7868
THC-COOH	THC-COOH-D9	2.312	2758	50254	0.0549	1.7830

ISP FORENSICS - Pocatello Instrument # 59740

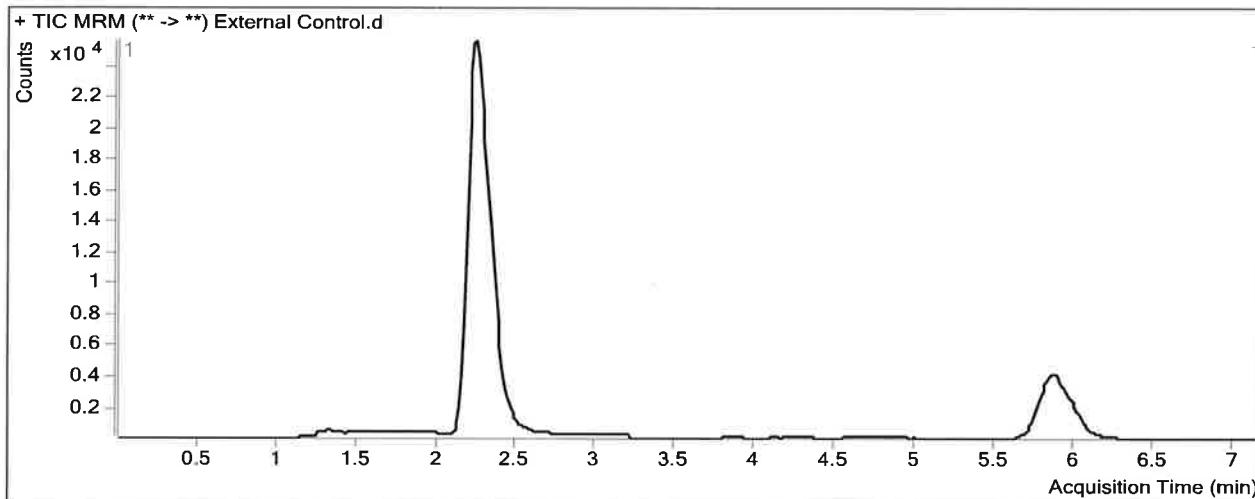
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418 THC Quant TS.bat
Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:40 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 20:54 **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** Hemostat 361331-3 + WS 010418

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.252	13994	155357	0.0901	9.9179
THC-COOH	THC-COOH-D9	2.339	23103	41413	0.5579	27.8318
THC	THC-D3	5.879	7142	47241	0.1512	13.8873

**Concentrations above 10ng/mL limit. Results reported qualitatively.*

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ISP FORENSICS - Pocatello Instrument # 59740

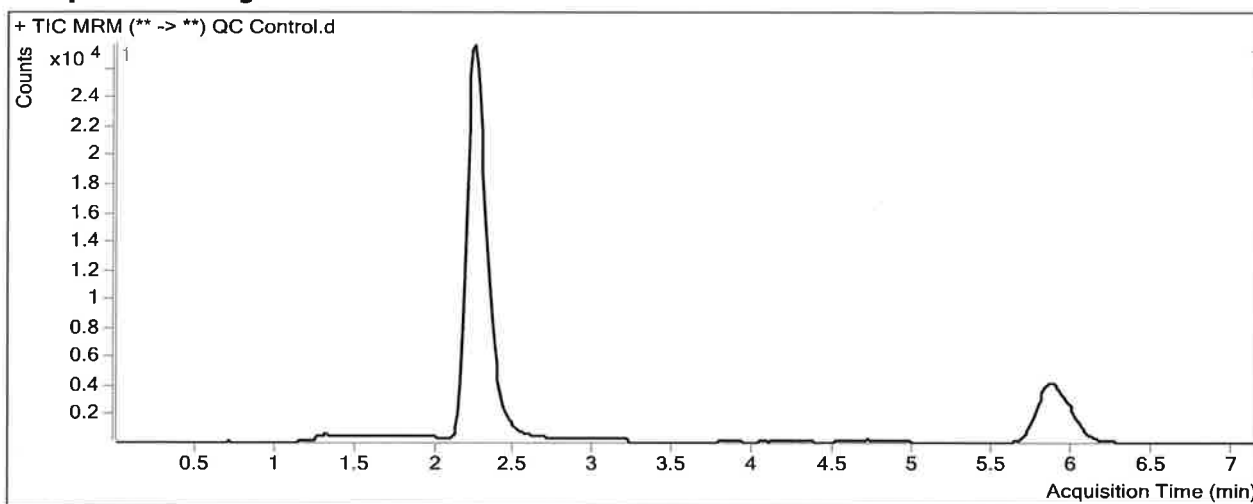
Cannabinoids Analysis Report

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Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:40 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 17:44 **Data File** QC Control.d
Sample Type QC **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

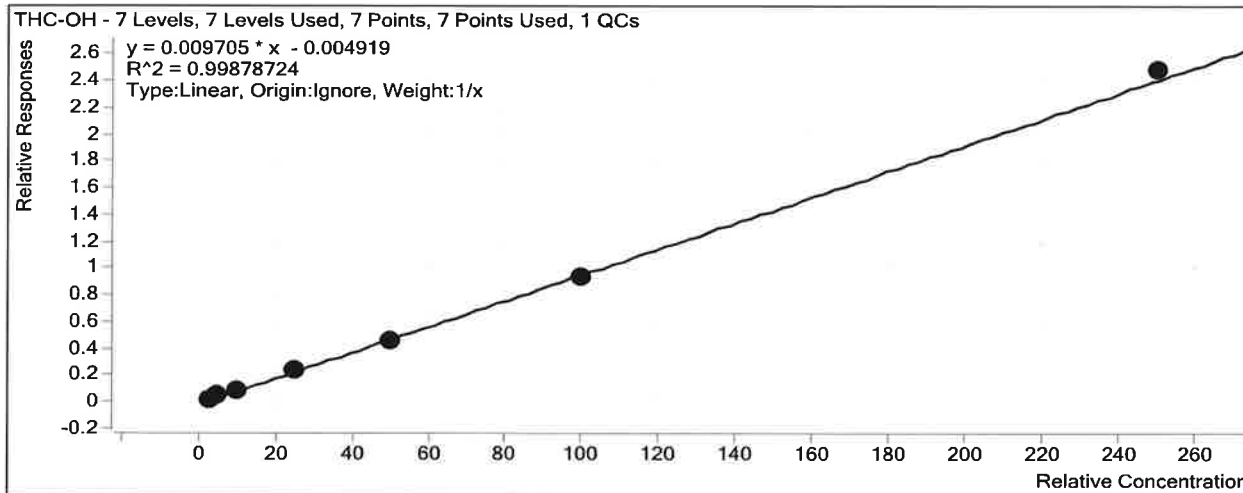
Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.252	16438	171091	0.0961	10.4068
THC-COOH	THC-COOH-D9	2.339	9352	43241	0.2163	10.1421
THC	THC-D3	5.919	5588	50766	0.1101	10.0735

ISP Forensics Calibration Curve Report

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Batch Data Path C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418
THC Quant TS.batch.bin
Last Calib Update 1/24/2018 8:37 AM **Analyst Name** ISP TOX

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



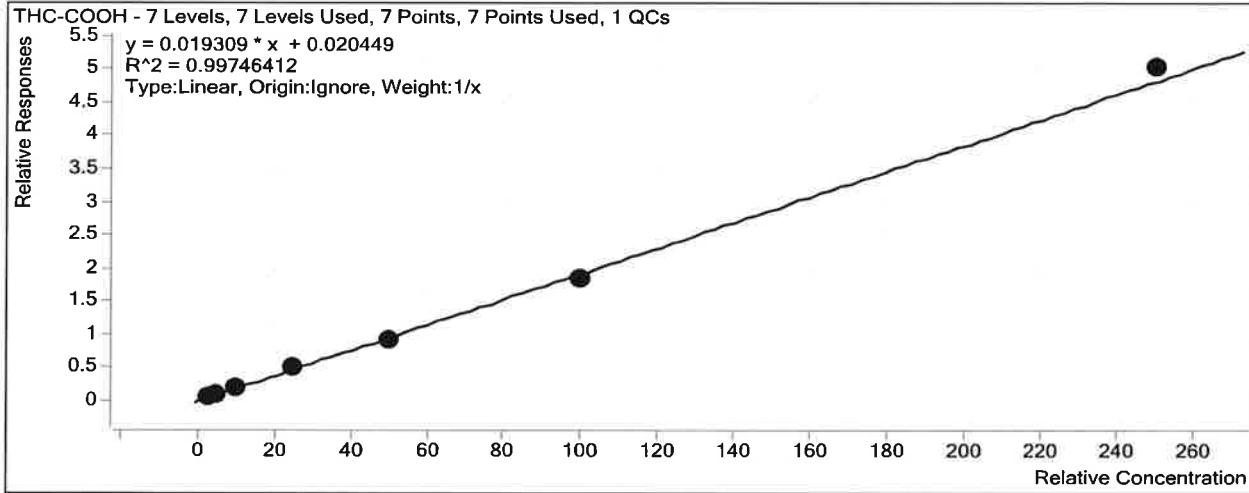
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	<input checked="" type="checkbox"/>	3	3.1	102.8
Cal 2	2	<input checked="" type="checkbox"/>	5	5.5	110.4
Cal 3	3	<input checked="" type="checkbox"/>	10	9.4	93.5
QC Control	3	<input checked="" type="checkbox"/>	10	10.4	104.1
Cal 4	4	<input checked="" type="checkbox"/>	25	24.7	98.6
Cal 5	5	<input checked="" type="checkbox"/>	50	47.9	95.8
Cal 6	6	<input checked="" type="checkbox"/>	100	96.5	96.5
Cal 7	7	<input checked="" type="checkbox"/>	250	256.0	102.4

ISP Forensics Calibration Curve Report

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Batch Data Path C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418
THC Quant TS.batch.bin
Last Calib Update 1/24/2018 8:37 AM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	<input checked="" type="checkbox"/>	3	3.3	108.7
Cal 2	2	<input checked="" type="checkbox"/>	5	5.1	101.9
Cal 3	3	<input checked="" type="checkbox"/>	10	9.9	98.7
QC Control	3	<input checked="" type="checkbox"/>	10	10.1	101.4
Cal 4	4	<input checked="" type="checkbox"/>	25	24.9	99.7
Cal 5	5	<input checked="" type="checkbox"/>	50	46.6	93.3
Cal 6	6	<input checked="" type="checkbox"/>	100	94.0	94.0
Cal 7	7	<input checked="" type="checkbox"/>	250	259.2	103.7

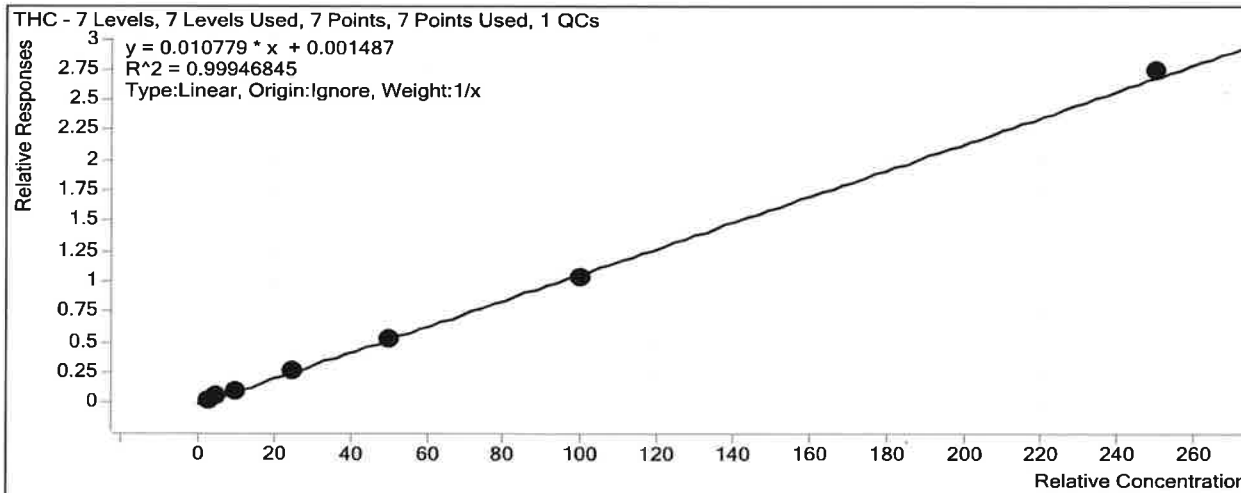
ISP Forensics Calibration Curve Report

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Batch Data Path C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418
THC Quant TS.batch.bin

Last Calib Update 1/24/2018 8:37 AM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	<input checked="" type="checkbox"/>	3	3.1	102.4
Cal 2	2	<input checked="" type="checkbox"/>	5	5.1	102.6
Cal 3	3	<input checked="" type="checkbox"/>	10	9.8	97.9
QC Control	3	<input checked="" type="checkbox"/>	10	10.1	100.7
Cal 4	4	<input checked="" type="checkbox"/>	25	25.0	99.9
Cal 5	5	<input checked="" type="checkbox"/>	50	49.5	99.1
Cal 6	6	<input checked="" type="checkbox"/>	100	96.5	96.5
Cal 7	7	<input checked="" type="checkbox"/>	250	254.0	101.6

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ISP FORENSICS - Pocatello Instrument # 59740

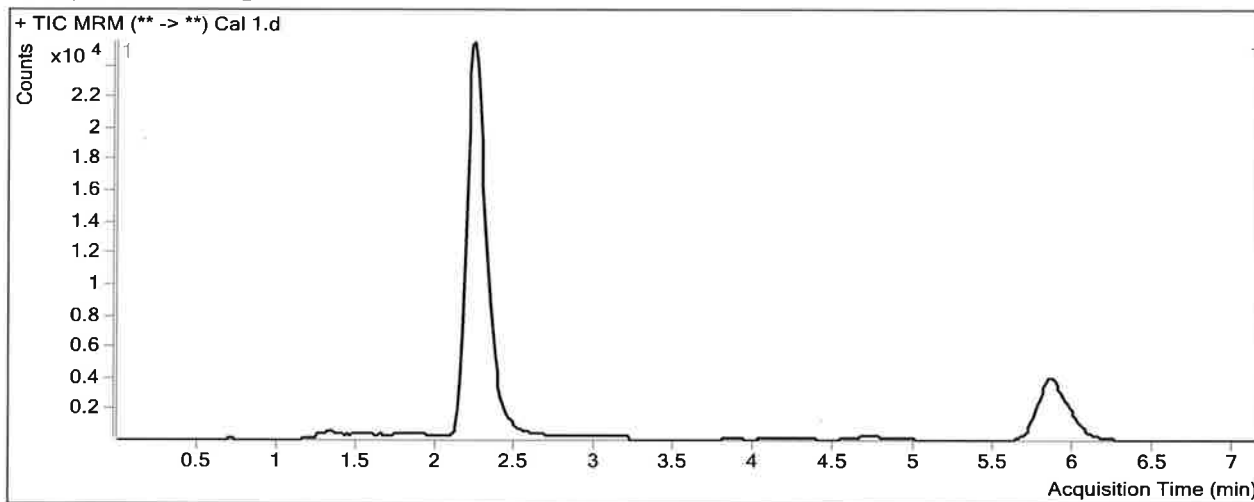
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418 THC Quant TS.bat
Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:39 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 16:10 **Data File** Cal 1.d
Sample Type Calibration **Sample Name** Cal 1
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	2.252	4228	169123	0.0250	3.0825
THC-COOH	THC-COOH-D9	2.339	3374	40435	0.0834	3.2624
THC	THC-D3	5.879	1751	50595	0.0346	3.0725

ISP FORENSICS - Pocatello Instrument # 59740

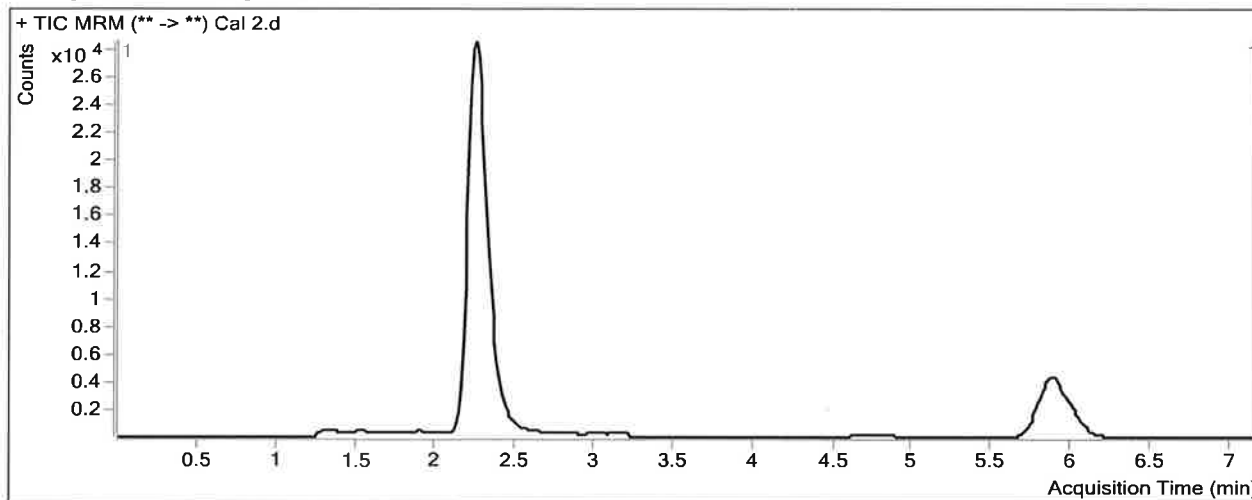
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418 THC Quant TS.bat
Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:39 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 16:21 **Data File** Cal 2.d
Sample Type Calibration **Sample Name** Cal 2
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	2.252	8678	178442	0.0486	5.5181
THC-COOH	THC-COOH-D9	2.352	5463	45975	0.1188	5.0947
THC	THC-D3	5.905	3145	55355	0.0568	5.1323

ISP FORENSICS - Pocatello Instrument # 59740

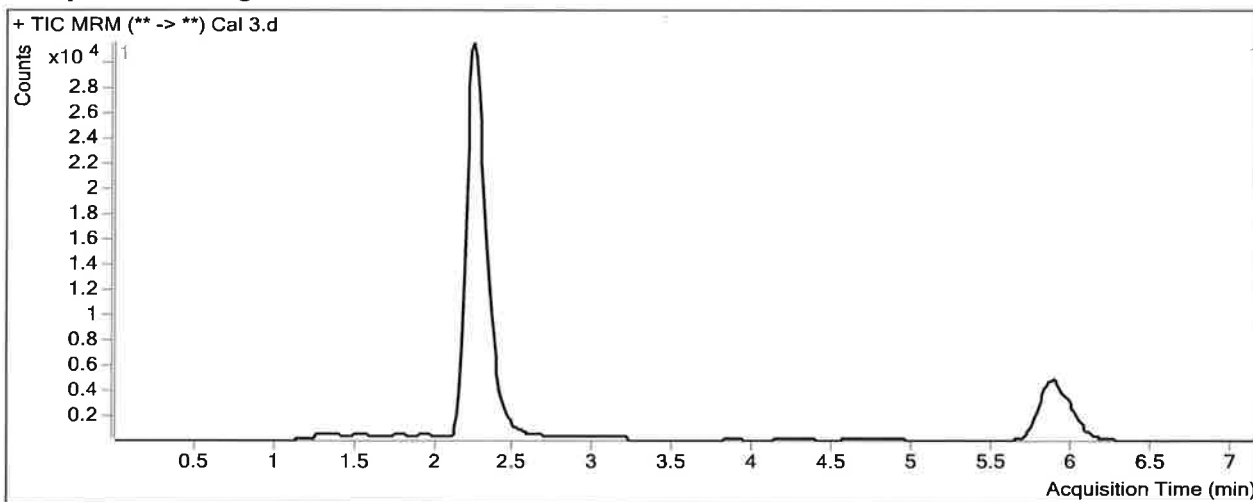
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418 THC Quant TS.bat
Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:39 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 16:33 **Data File** Cal 3.d
Sample Type Calibration **Sample Name** Cal 3
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	2.252	16926	197158	0.0858	9.3528
THC-COOH	THC-COOH-D9	2.352	10181	48225	0.2111	9.8745
THC	THC-D3	5.905	6166	57625	0.1070	9.7890

TS

ISP FORENSICS - Pocatello Instrument # 59740

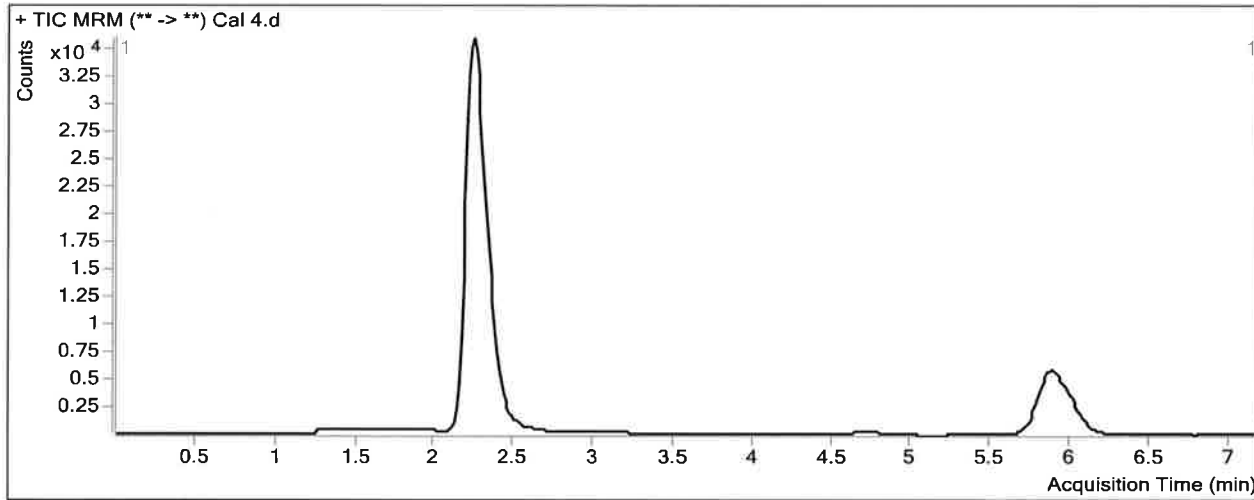
Cannabinoids Analysis Report

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Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:40 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 16:45 **Data File** Cal 4.d
Sample Type Calibration **Sample Name** Cal 4
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	2.252	45441	193841	0.2344	24.6624
THC-COOH	THC-COOH-D9	2.352	23832	47520	0.5015	24.9140
THC	THC-D3	5.892	15908	58792	0.2706	24.9638

ISP FORENSICS - Pocatello Instrument # 59740

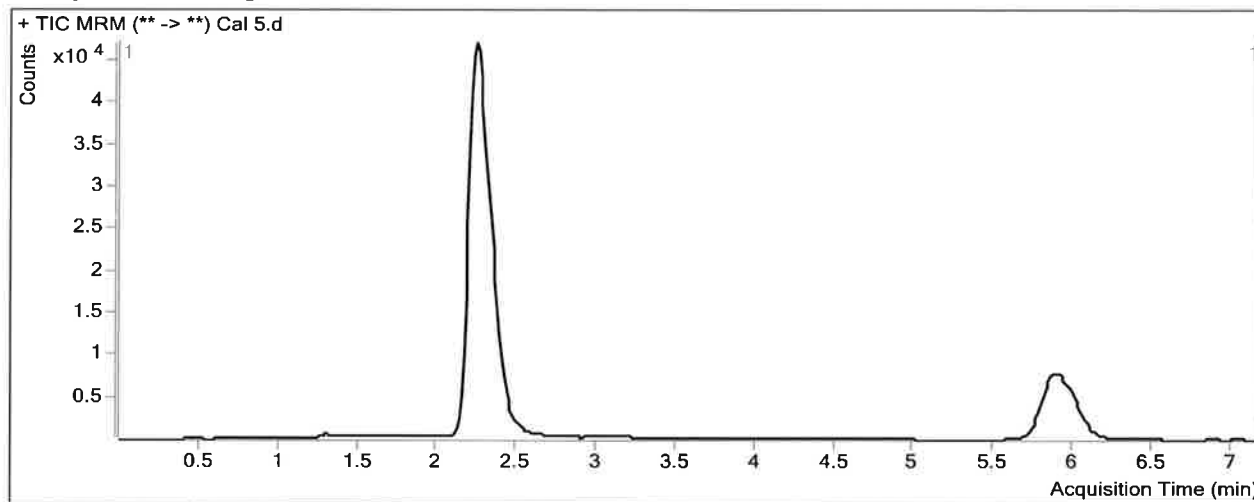
Cannabinoids Analysis Report

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Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:40 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 16:57 **Data File** Cal 5.d
Sample Type Calibration **Sample Name** Cal 5
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	2.252	98109	213291	0.4600	47.9037
THC-COOH	THC-COOH-D9	2.352	47638	51735	0.9208	46.6278
THC	THC-D3	5.905	33661	62860	0.5355	49.5404

ISP FORENSICS - Pocatello Instrument # 59740

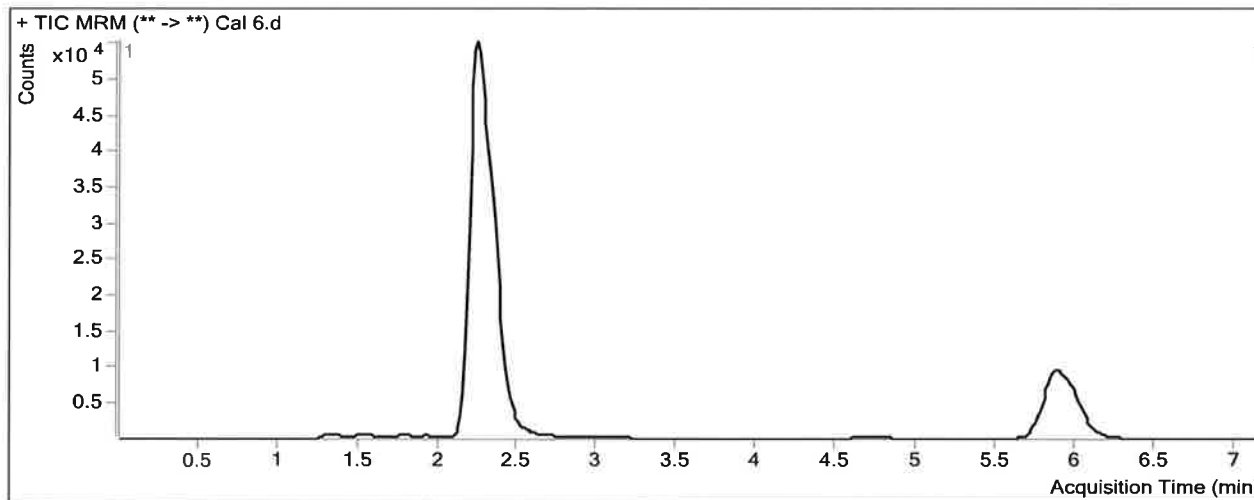
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\010418 TS and SP THC Quant\QuantResults\010418 THC Quant TS.bat		
Analysis Time	1/24/2018 8:37 AM	Analyst Name	ISPUser
Report Time	1/24/2018 8:40 AM	Reporter Name	ISPUser
Last Calib Update	1/24/2018 8:37 AM	Batch State	Processed

Analysis Info

Acq Time	2018-01-04 17:09	Data File	Cal 6.d
Sample Type	Calibration	Sample Name	Cal 6
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	2.252	173388	186086	0.9318	96.5175
THC-COOH	THC-COOH-D9	2.339	81964	44643	1.8360	94.0229
THC	THC-D3	5.905	55608	53374	1.0419	96.5175

ISP FORENSICS - Pocatello Instrument # 59740

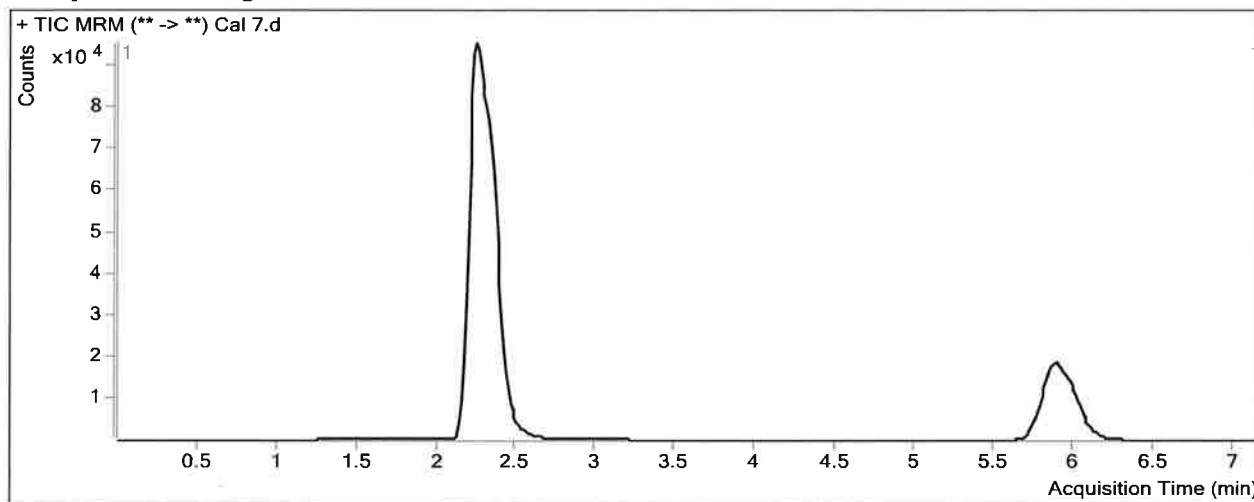
Cannabinoids Analysis Report

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Analysis Time 1/24/2018 8:37 AM **Analyst Name** ISPUser
Report Time 1/24/2018 8:40 AM **Reporter Name** ISPUser
Last Calib Update 1/24/2018 8:37 AM **Batch State** Processed

Analysis Info

Acq Time 2018-01-04 17:21 **Data File** Cal 7.d
Sample Type Calibration **Sample Name** Cal 7
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	2.252	433006	174659	2.4791	255.9630
THC-COOH	THC-COOH-D9	2.339	203305	40455	5.0255	259.2037
THC	THC-D3	5.892	139701	51001	2.7392	253.9844