REVIEWED
By Anne Nord at 6:28 pm, Sep 19, 2018

9/14/2018

Worklist: 2688

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
M2018-4176	3	127009	AM 27 Blood THC Quant by LC-QQQ
P2018-2406	1	127010	AM 27 Blood THC Quant by LC-QQQ
P2018-2423	3	127011	AM 27 Blood THC Quant by LC-QQQ
P2018-2430	1	127012	AM 27 Blood THC Quant by LC-QQQ
P2018-2499	1	127013	AM 27 Blood THC Quant by LC-QQQ
P2018-2505	1	127014	AM 27 Blood THC Quant by LC-QQQ
P2018-2508	1	127015	AM 27 Blood THC Quant by LC-QQQ
P2018-2589	1	127016	AM 27 Blood THC Quant by LC-QQQ

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

Extraction Date: 09/12/18 Analyst: Sarah Pickle
Plate lot#: 0515037 Plate Expiration: 09/28/18

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: 361331-1 **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: 091218 THCQ SP Batch Name: THCQ SP
☒ 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: *Curve Range Limited: THC-COOH 10-250*

Idaho State Police Forensic Services

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

Analyst: Sarah Pickle
Extraction Date: 9/12/18
Worklist Number: 2688

Reagent	*Lot Number*	*Expiration Date*	*Date in Service*	*Date Out of Service*	*Initials*
ToxBox THC/THC Metabolite Plate	0515037	09/28/18			
Negative Blood	361331-1		12/27/17		
Methanol External Control Solution	WS020718	02/07/19	02/07/18		
Blood External Control Solution	090418	02/07/19	09/04/18		
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)	180079		08/24/18		
0.1% Formic Acid in Acetonitrile (Mobile Phase B)	176190		2/6/18		
Needle Rinse--75% LCMS MeOH in LCMS Water	090418		09/04/18		

Methanol External Control Solution (Lot: WS020718)
10 ul of 1mg/mL THC, 100 ul of 100 ug/mL THC-OH, C-THC in 9790 ul MeOH

Component	*Source*	*Source Lot Number*	*Expiration Date*
Methanol (LCMS)	Fisher	172516	
THC	Cerilliant	FE04231406	04/30/2019
C-THC	Cayman	0497429	02/08/2019
THC-OH	Cerilliant	FE01121503	01/31/2020
Prepared:	02/07/18		
Prepared By:	Tamara Salazar		
Expires:	02/07/19		

Blood External Control Solution (Lot: 090418)
100 ul of methanol external control solution was added to 9900 ul of blood.

Component	*Source*	*Source Lot Number*
Negative Blood	Hemostat	361331-1
Methanol External Control Solution		WS020718
Prepared:	09/04/18	
Prepared by:	Sarah Pickle	
Expires:	02/07/19	

Needle Rinse (75% LCMS MeOH in LCMS Water) (Lot: 090418)

Component	*Source*	*Source Lot Number*
MeOH (LCMS Grade)	Fisher	177145
Water (LCMS Grade)	Fisher	182702
Prepared:	09/04/18	
Prepared By:	Sarah Pickle	

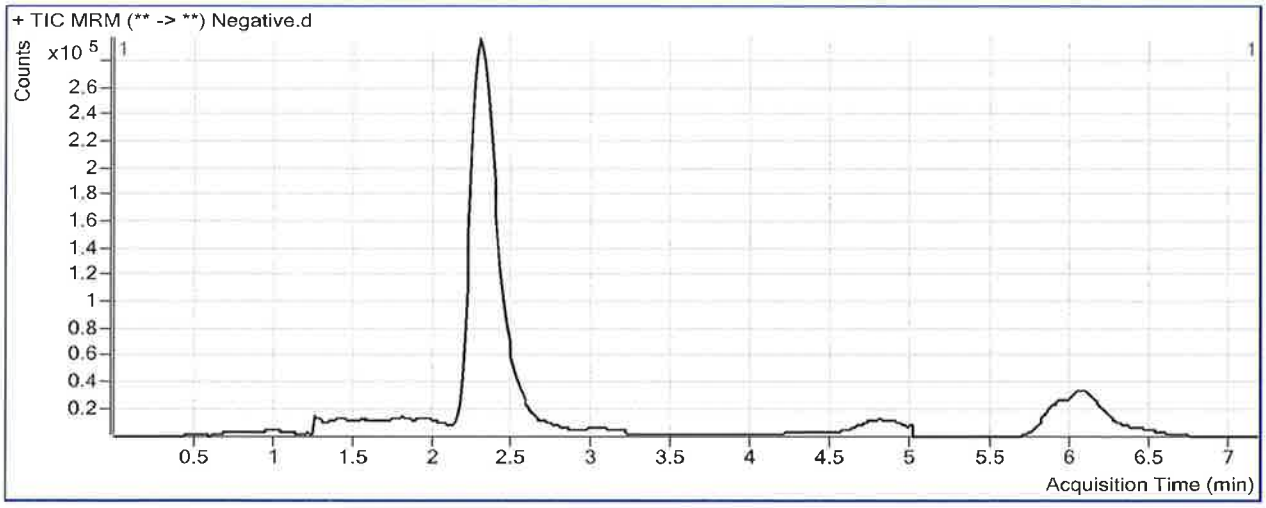
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:05 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 18:13	**Data File**	Negative.d
Sample Type	Sample	**Sample Name**	Negative
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-H10	**Sample Info**	
Inj Vol	-1	**Comment**	Hemostat 361331-1

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.145	14134	2713670	0.0052	1.5526 < 3
THC-COOH	THC-COOH-D9	2.419	44443	715404	0.0621	2.4556 < 10

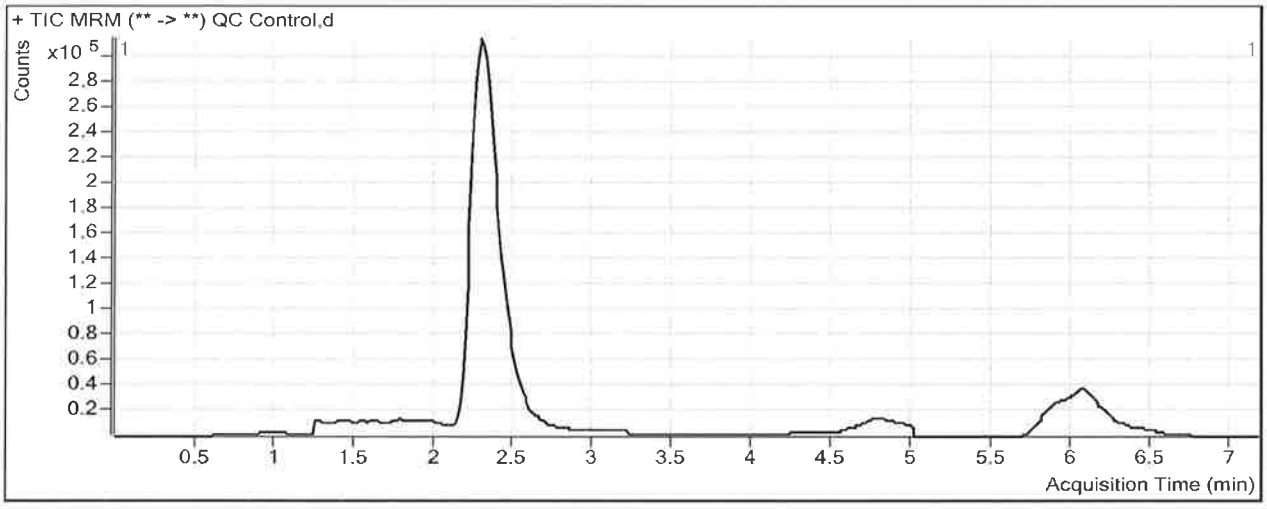
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:04 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 17:49	**Data File**	QC Control.d
Sample Type	Sample	**Sample Name**	QC Control
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-A11	**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	2.292	237813	2595829	0.0916	9.2330
THC-COOH	THC-COOH-D9	2.406	164214	716112	0.2293	9.8136
THC	THC-D3	6.052	74326	808171	0.0920	9.5568

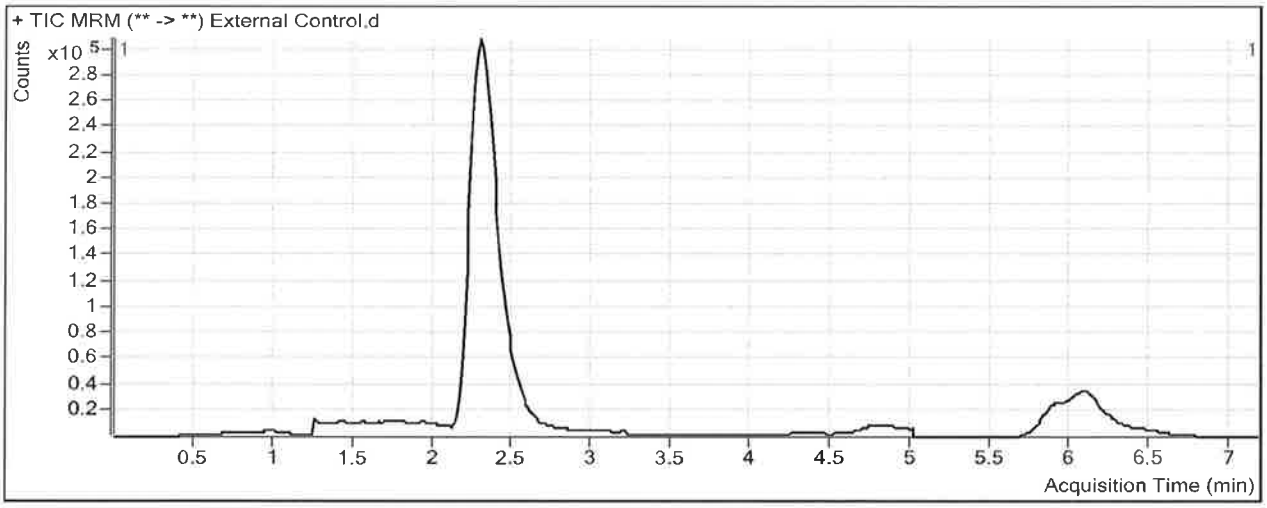
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:05 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 18:37	**Data File**	External Control.d
Sample Type	Sample	**Sample Name**	External Control
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-G10	**Sample Info**	
Inj Vol	-1	**Comment**	Hemostat 361331-1 + WS 020718

Sample Chromatogram



Results

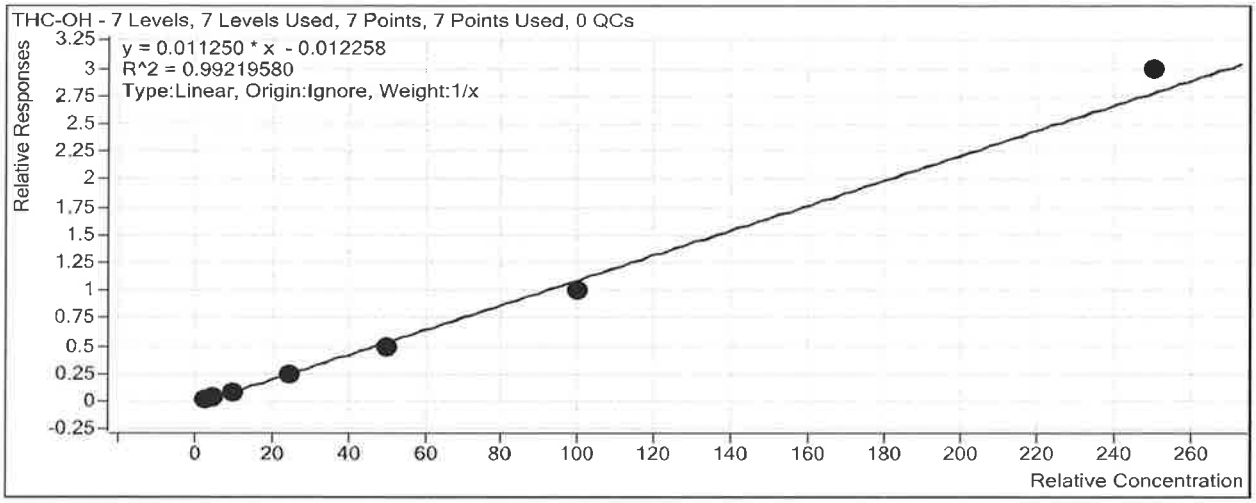
Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.305	232234	2565195	0.0905	9.1369
THC-COOH	THC-COOH-D9	2.379	183651	718349	0.2557	10.9729
THC	THC-D3	6.079	68155	794525	0.0858	8.9914

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 9/13/2018 2:00 PM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	☑	3	3.6	121.4
Cal 2	2	☑	5	5.5	110.8
Cal 3	3	☑	10	8.9	89.2
Cal 4	4	☑	25	22.8	91.2
Cal 5	5	☑	50	45.1	90.2
Cal 6	6	☑	100	90.6	90.6
Cal 7	7	☑	250	266.4	106.5

ISP Forensics
Calibration Curve Report

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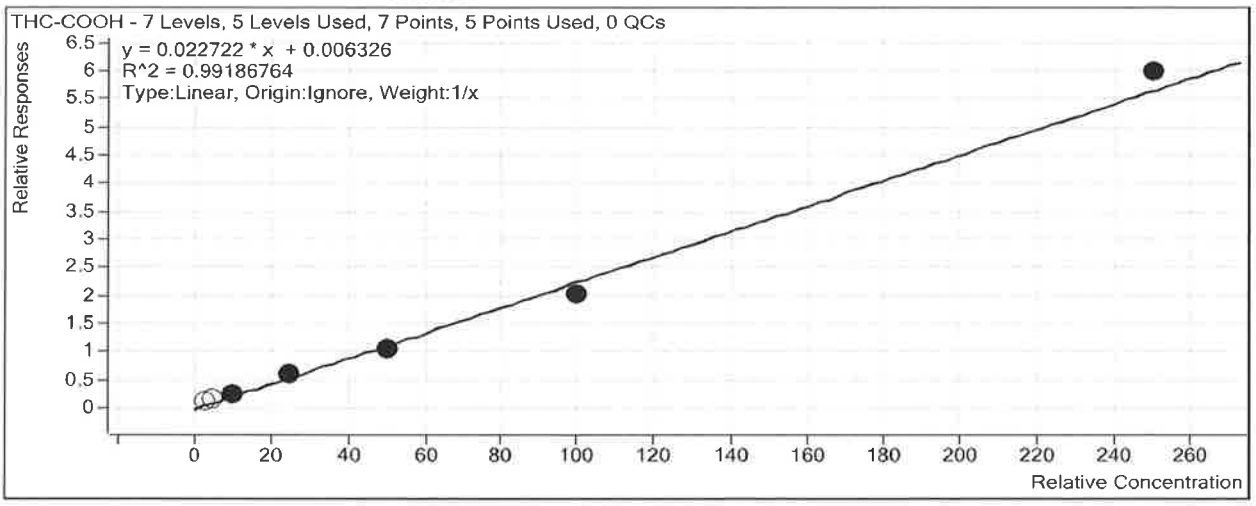
Batch Data Path C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 9/13/2018 2:00 PM **Analyst Name** ISP TOX

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



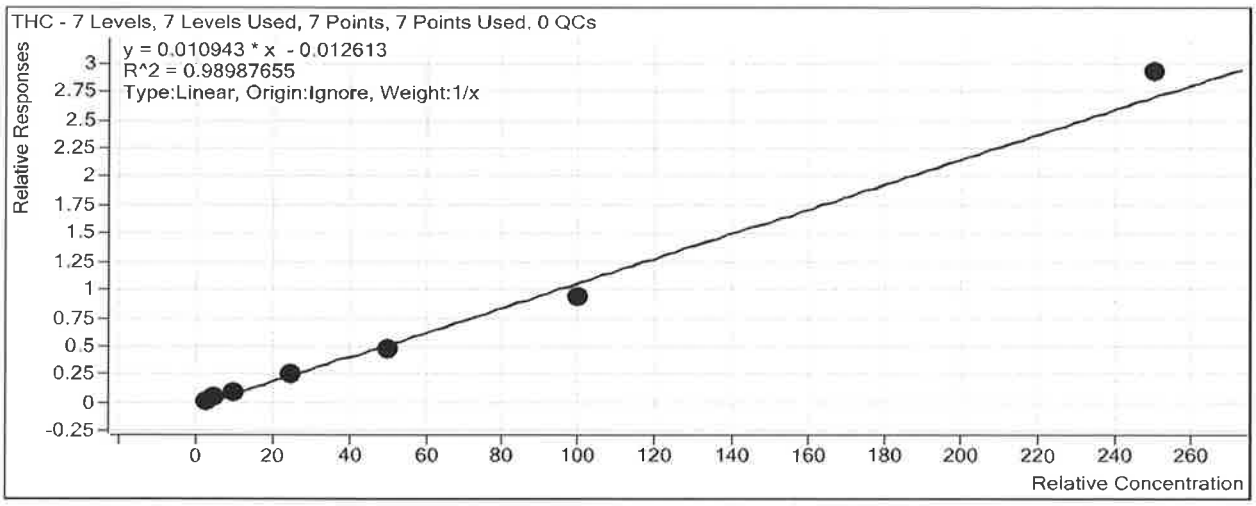
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	☐	3	5.6	187.8
Cal 2	2	☐	5	6.9	139.0
Cal 3	3	☑	10	11.1	111.0
Cal 4	4	☑	25	25.8	103.1
Cal 5	5	☑	50	45.7	91.4
Cal 6	6	☑	100	89.2	89.2
Cal 7	7	☑	250	263.2	105.3

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 9/13/2018 2:00 PM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	☑	3	3.4	114.4
Cal 2	2	☑	5	5.5	109.1
Cal 3	3	☑	10	9.5	94.9
Cal 4	4	☑	25	24.5	98.1
Cal 5	5	☑	50	44.5	89.1
Cal 6	6	☑	100	86.9	86.9
Cal 7	7	☑	250	268.6	107.4

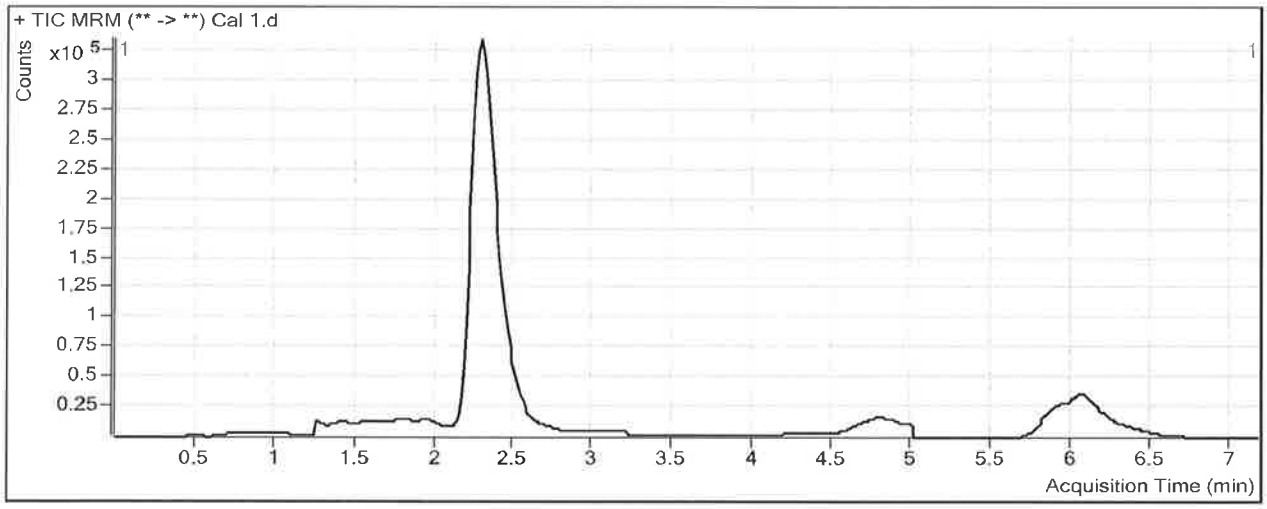
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:04 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 16:14	**Data File**	Cal 1.d
Sample Type	Calibration	**Sample Name**	Cal 1
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-H11	**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	2.279	79960	2783429	0.0287	3.6431
THC-COOH	THC-COOH-D9	2.406	102160	760625	0.1343	5.6325
THC	THC-D3	6.052	21680	869268	0.0249	3.4317

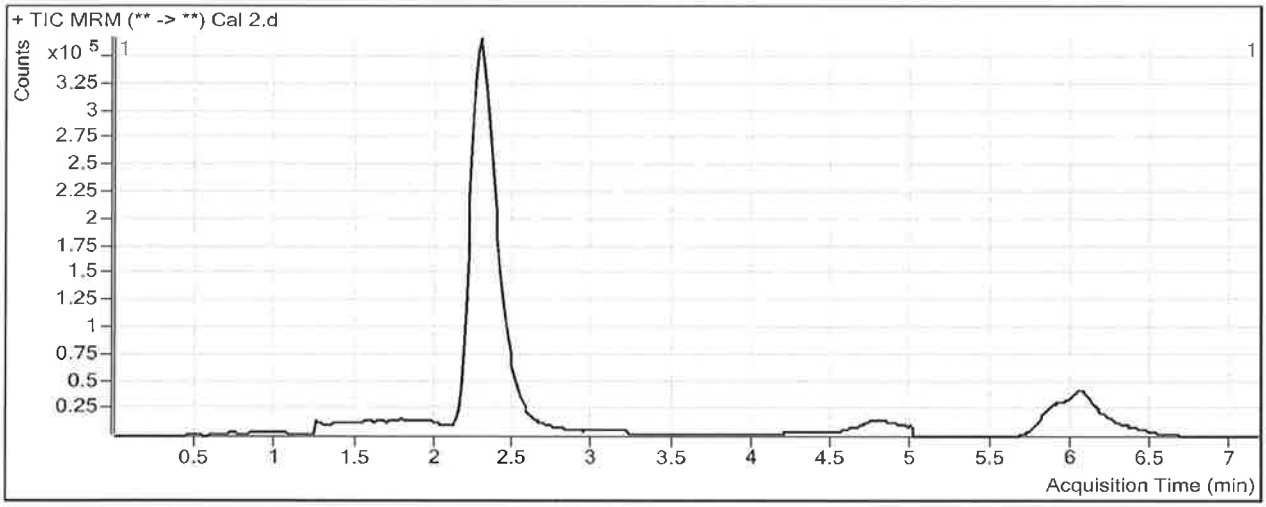
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:04 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 16:26	**Data File**	Cal 2.d
Sample Type	Calibration	**Sample Name**	Cal 2
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-G11	**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	2.292	149206	2978626	0.0501	5.5422
THC-COOH	THC-COOH-D9	2.379	132067	804159	0.1642	6.9492
THC	THC-D3	6.025	43928	932550	0.0471	5.4571

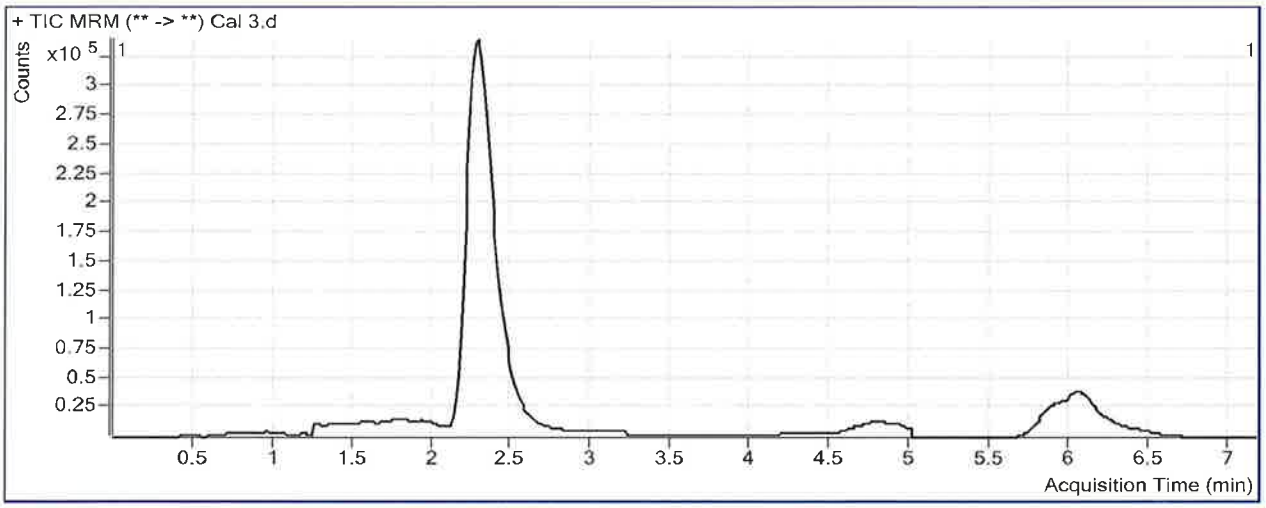
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:04 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 16:38	**Data File**	Cal 3.d
Sample Type	Calibration	**Sample Name**	Cal 3
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-F11	**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	2.292	249632	2835149	0.0880	8.9161
THC-COOH	THC-COOH-D9	2.379	194326	751407	0.2586	11.1031
THC	THC-D3	6.065	75894	832303	0.0912	9.4853

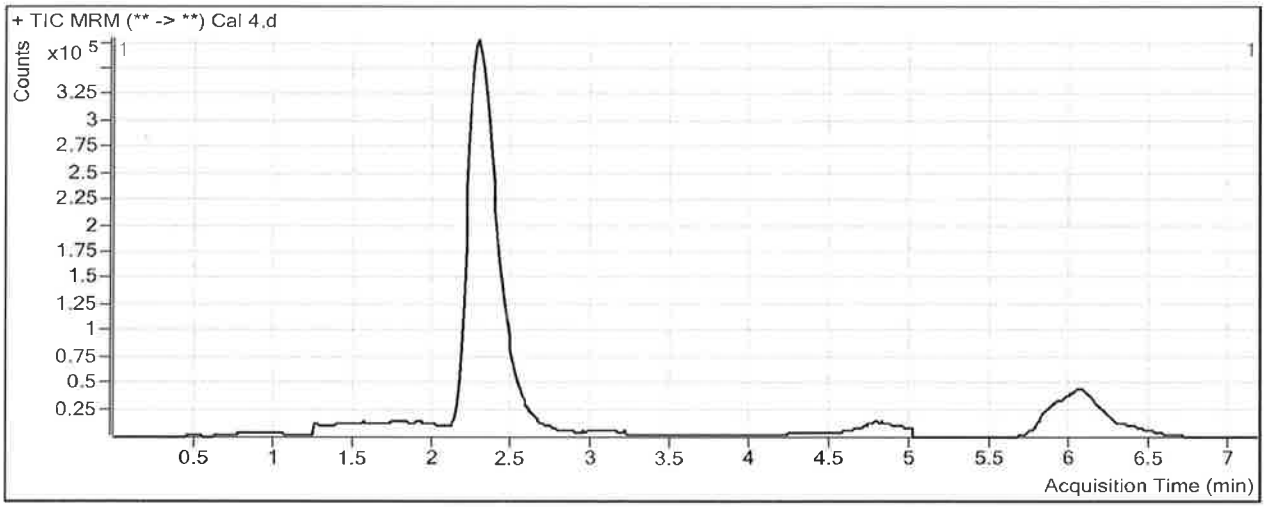
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:04 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 16:50	**Data File**	Cal 4.d
Sample Type	Calibration	**Sample Name**	Cal 4
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-E11	**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	2.279	656773	2689407	0.2442	22.7967
THC-COOH	THC-COOH-D9	2.379	413870	698955	0.5921	25.7807
THC	THC-D3	6.052	205232	802054	0.2559	24.5357

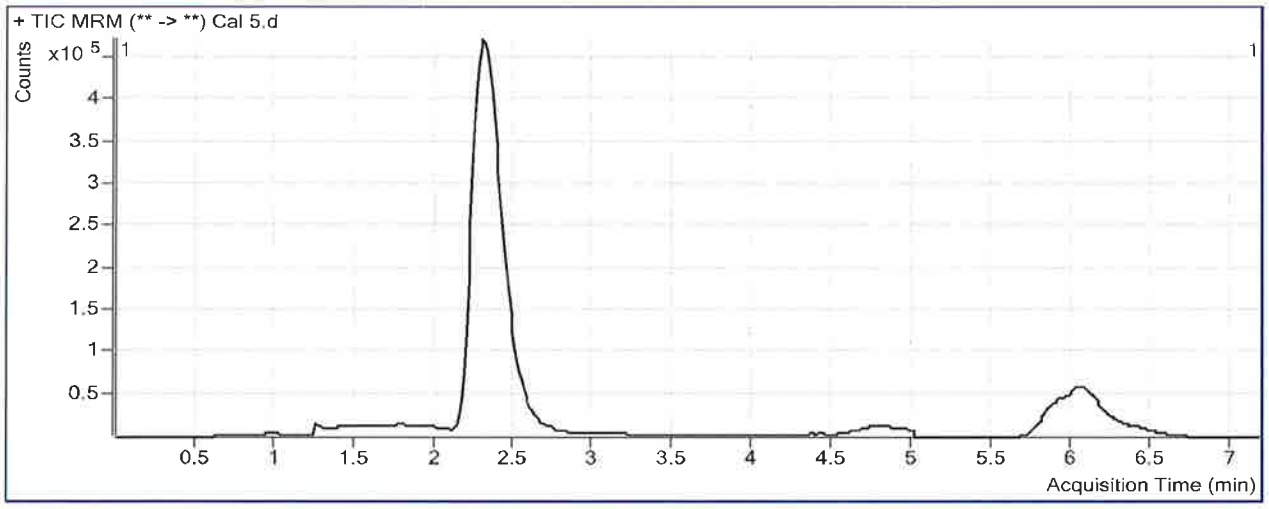
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:04 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 17:02	**Data File**	Cal 5.d
Sample Type	Calibration	**Sample Name**	Cal 5
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-D11	**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	2.292	1355598	2738371	0.4950	45.0926
THC-COOH	THC-COOH-D9	2.392	764094	731765	1.0442	45.6753
THC	THC-D3	6.065	404363	851642	0.4748	44.5411

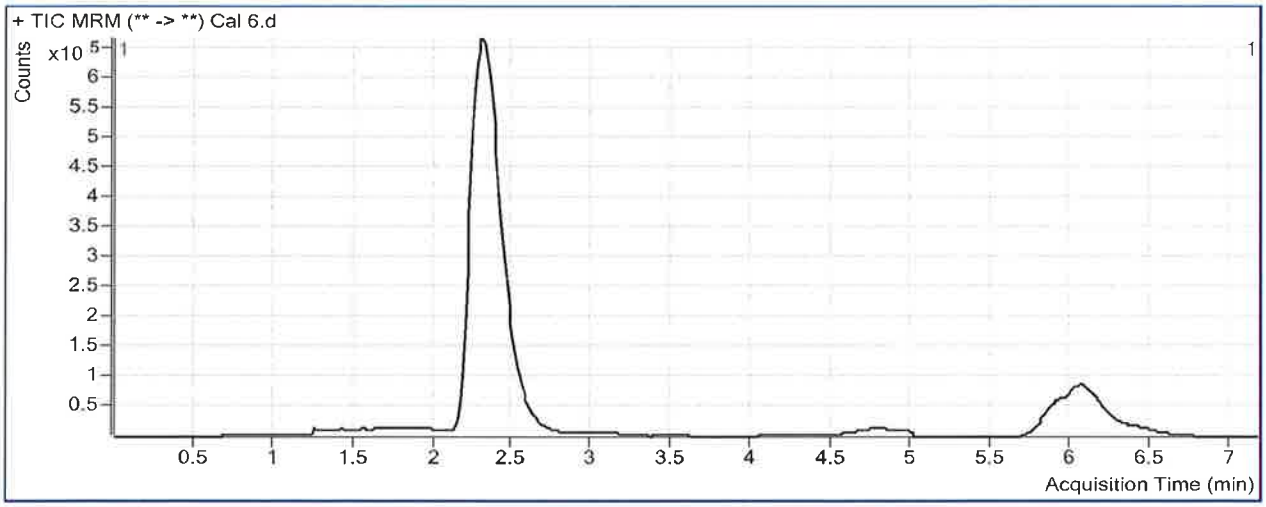
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:04 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 17:14	**Data File**	Cal 6.d
Sample Type	Calibration	**Sample Name**	Cal 6
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-C11	**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	2.292	2892969	2871739	1.0074	90.6347
THC-COOH	THC-COOH-D9	2.379	1512690	744028	2.0331	89.1976
THC	THC-D3	6.065	812524	865418	0.9389	86.9492

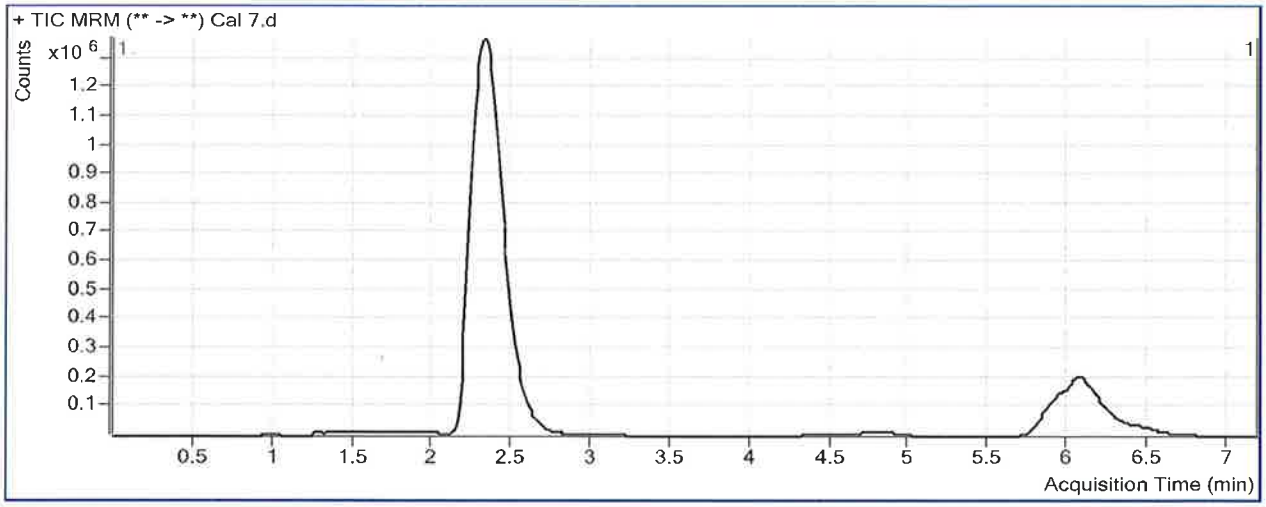
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\091218 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	9/13/2018 2:00 PM	**Analyst Name**	ISPUser
Report Time	9/13/2018 2:04 PM	**Reporter Name**	ISPUser
Last Calib Update	9/13/2018 2:00 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-09-12 17:26	**Data File**	Cal 7.d
Sample Type	Calibration	**Sample Name**	Cal 7
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-B11	**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	2.292	8318992	2787413	2.9845	266.3745
THC-COOH	THC-COOH-D9	2.392	4267297	712661	5.9878	263.2431
THC	THC-D3	6.065	2507246	856680	2.9267	268.5999