reviewed 8/13/18 amn

8/8/2018

Worklist: 2622

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
M2018-3191	1	123596	AM 27 Blood THC Quant by LC-QQQ
M2018-3304	1	123597	AM 27 Blood THC Quant by LC-QQQ
M2018-3304	2	123598	AM 27 Blood THC Quant by LC-QQQ
M2018-3331	1	123599	AM 27 Blood THC Quant by LC-QQQ
M2018-3354	1	123600	AM 27 Blood THC Quant by LC-QQQ
P2018-1940	1	123601	AM 27 Blood THC Quant by LC-QQQ
P2018-1949	1	123602	AM 27 Blood THC Quant by LC-QQQ
P2018-1951	1	123603	AM 27 Blood THC Quant by LC-QQQ
P2018-1952	1	123604	AM 27 Blood THC Quant by LC-QQQ
P2018-1953	1	123605	AM 27 Blood THC Quant by LC-QQQ
P2018-1958	1	123606	AM 27 Blood THC Quant by LC-QQQ
P2018-1963	1	123607	AM 27 Blood THC Quant by LC-QQQ
P2018-1979	1	123608	AM 27 Blood THC Quant by LC-QQQ
P2018-1980	1	123609	AM 27 Blood THC Quant by LC-QQQ
P2018-1981	1	123610	AM 27 Blood THC Quant by LC-QQQ

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

Extraction Date: 08/03/18 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: 080318 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 10-100, THC 3-100, THC-OH 3-100*

Idaho State Police Forensic Services

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

Analyst: Sarah Pickle
Extraction Date: 8/3/18
Worklist Number: 2622

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	061718	~~06/07/19~~ 02/07/19	06/07/18		
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)	166541		6/26/17		
0.1% Formic Acid in Acetonitrile (Mobile Phase B)	176190		2/6/18		
Needle Rinse--75% LCMS MeOH in LCMS Water	080318		08/03/18		

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: 061718)
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:	06/17/18	
Prepared by:	Tamara Salazar	
Expires:	~~06/17/19~~ 02/07/19	

Needle Rinse (75% LCMS MeOH in LCMS Water) (Lot: 080318)

Component	*Source*	*Source Lot Number*
MeOH (LCMS Grade)	Fisher	177145
Water (LCMS Grade)	Fisher	181370
Prepared:	08/03/18	
Prepared By:	Sarah Pickle	

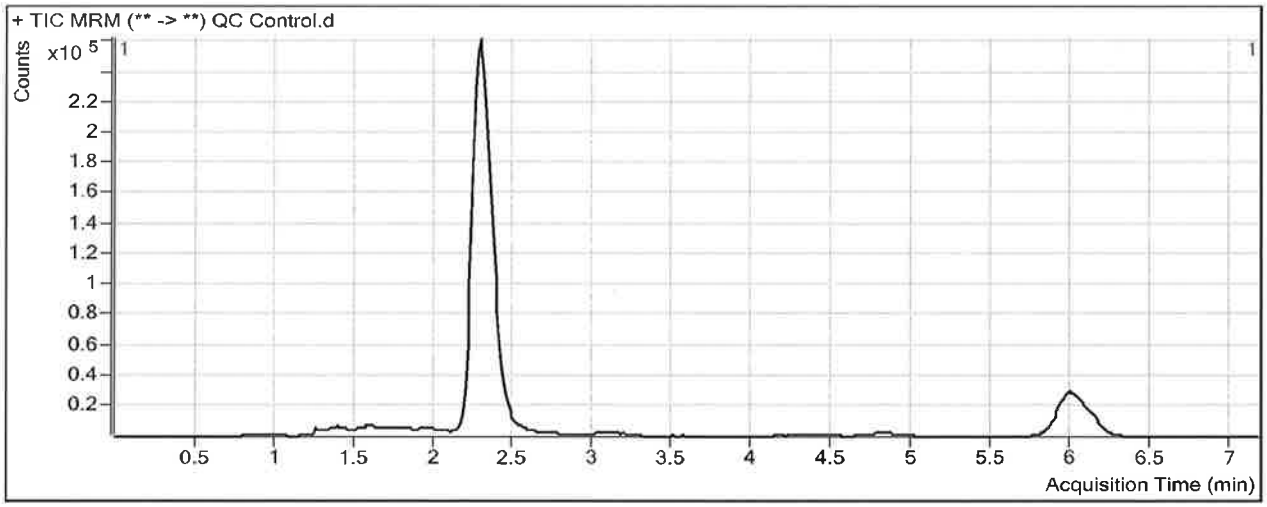
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:48 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 16:19	**Data File**	QC Control.d
Sample Type	Sample	**Sample Name**	QC Control
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-H1	**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	103657	1616563	0.0641	7.7907
THC-COOH	THC-COOH-D9	2.392	85106	401186	0.2121	9.6833
THC	THC-D3	6.052	31037	382709	0.0811	9.5137

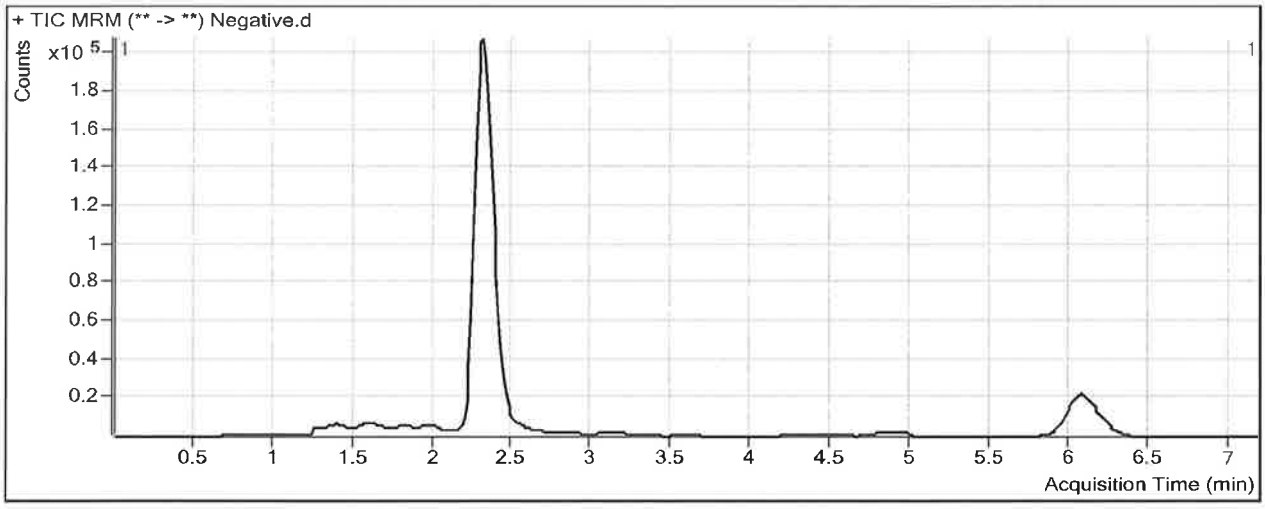
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:48 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 16:43	**Data File**	Negative.d
Sample Type	Sample	**Sample Name**	Negative
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-A2	**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	2.546	13463	1370260	0.0098	1.7581
THC-COOH	THC-COOH-D9	2.379	28365	343281	0.0826	2.4053

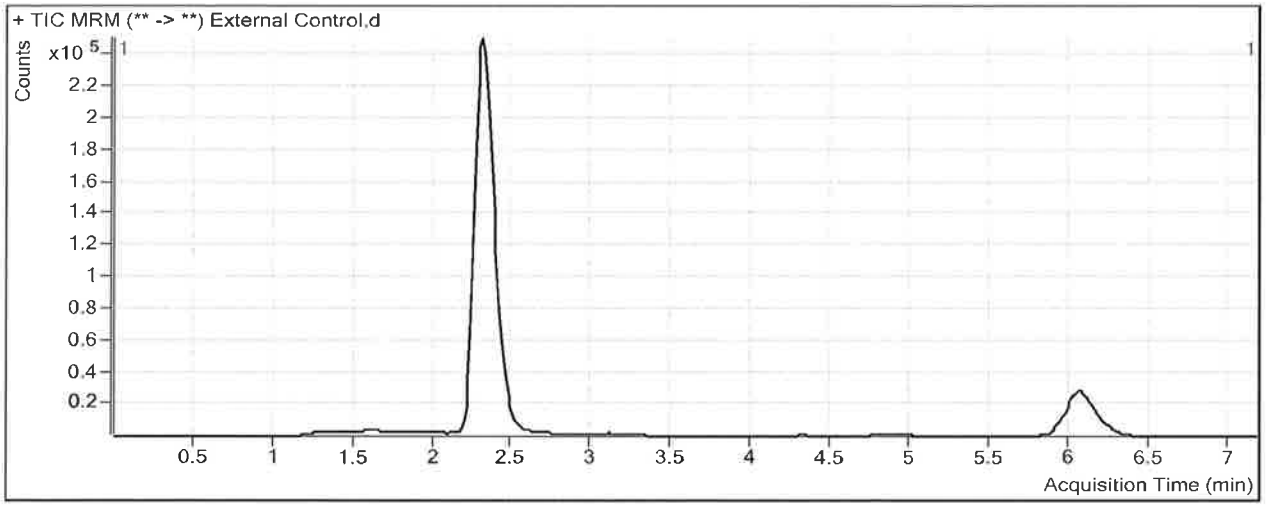
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:48 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 17:07	**Data File**	External Control.d
Sample Type	Sample	**Sample Name**	External Control
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-B2	**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	2.319	125494	1550963	0.0809	9.6564
THC-COOH	THC-COOH-D9	2.419	80449	377160	0.2133	9.7488
THC	THC-D3	6.079	29187	344758	0.0847	9.9379

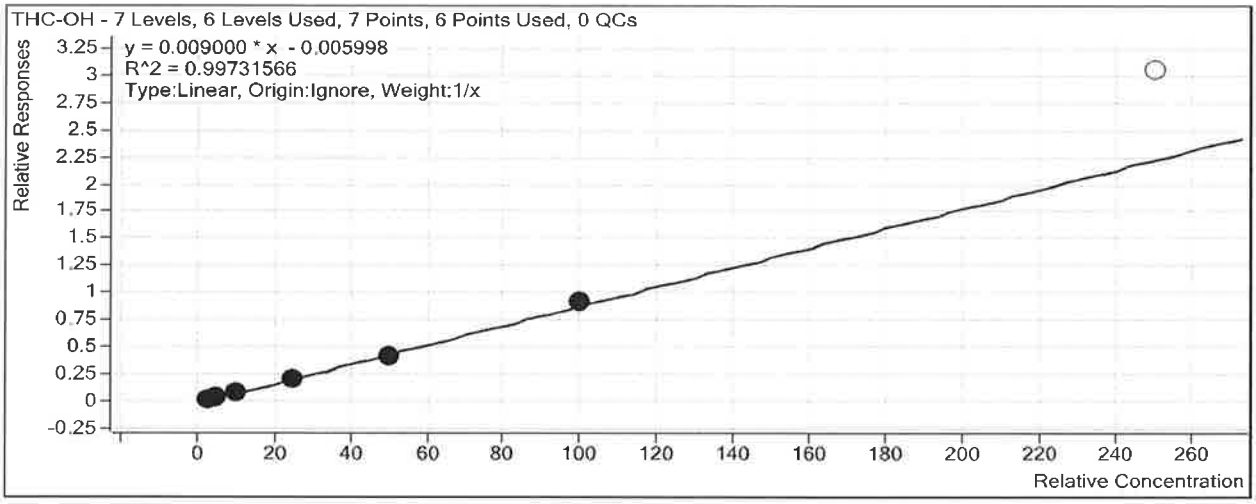
ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 8/8/2018 1:46 PM **Analyst Name** ISP TOX

Target Compound *THC-OH*

Internal Standard *THC-OH-D3*



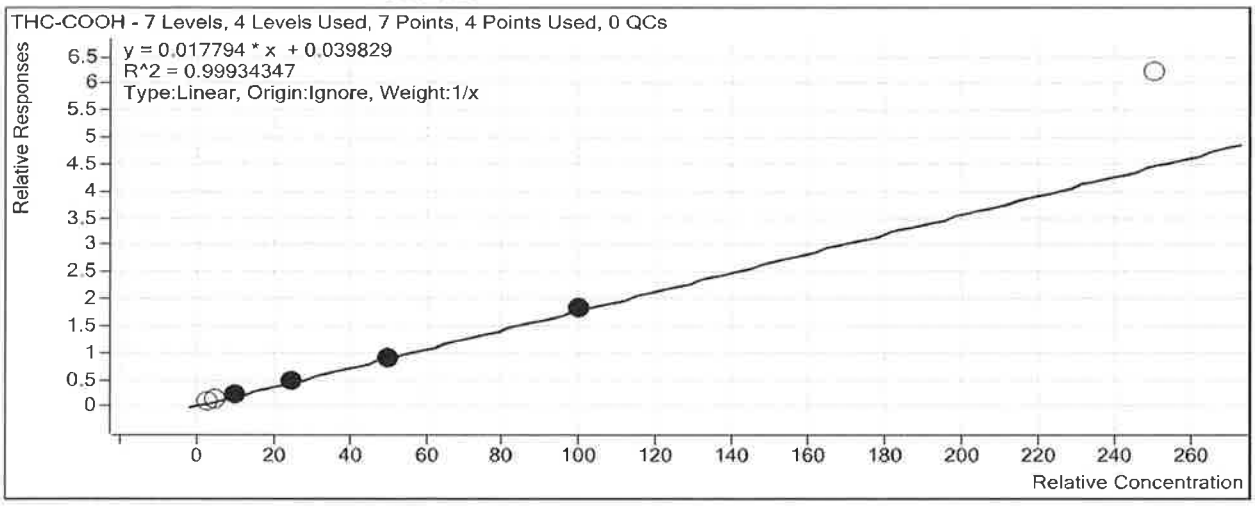
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	☑	3	3.4	111.7
Cal 2	2	☑	5	5.0	99.2
Cal 3	3	☑	10	9.3	93.4
Cal 4	4	☑	25	24.4	97.4
Cal 5	5	☑	50	47.3	94.6
Cal 6	6	☑	100	103.7	103.7
Cal 7	7	☐	250	339.9	135.9

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 8/8/2018 1:46 PM **Analyst Name** ISP TOX

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



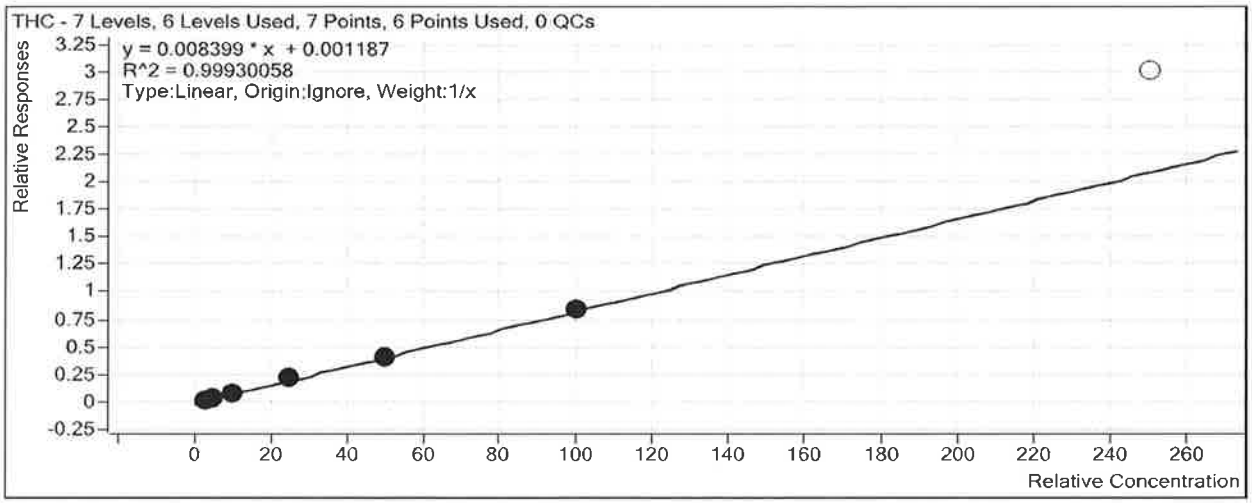
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	☐	3	4.4	145.6
Cal 2	2	☐	5	6.1	122.6
Cal 3	3	☑	10	10.2	102.5
Cal 4	4	☑	25	24.7	98.7
Cal 5	5	☑	50	48.7	97.5
Cal 6	6	☑	100	101.3	101.3
Cal 7	7	☐	250	346.8	138.7

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 8/8/2018 1:46 PM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	☑	3	2.9	96.1
Cal 2	2	☑	5	5.1	101.3
Cal 3	3	☑	10	9.9	98.6
Cal 4	4	☑	25	26.4	105.7
Cal 5	5	☑	50	49.5	99.0
Cal 6	6	☑	100	99.3	99.3
Cal 7	7	☐	250	357.6	143.0

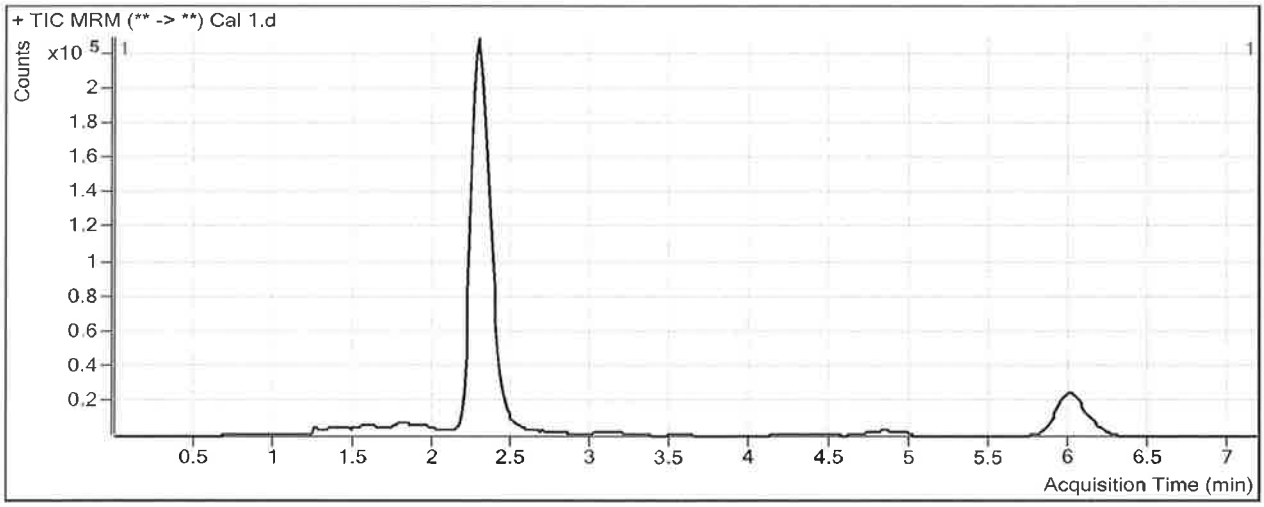
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:47 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 14:45	**Data File**	Cal 1.d
Sample Type	Calibration	**Sample Name**	Cal 1
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-A1	**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	35524	1470500	0.0242	3.3505
THC-COOH	THC-COOH-D9	2.379	42765	363718	0.1176	4.3694
THC	THC-D3	6.039	8595	338207	0.0254	2.8845

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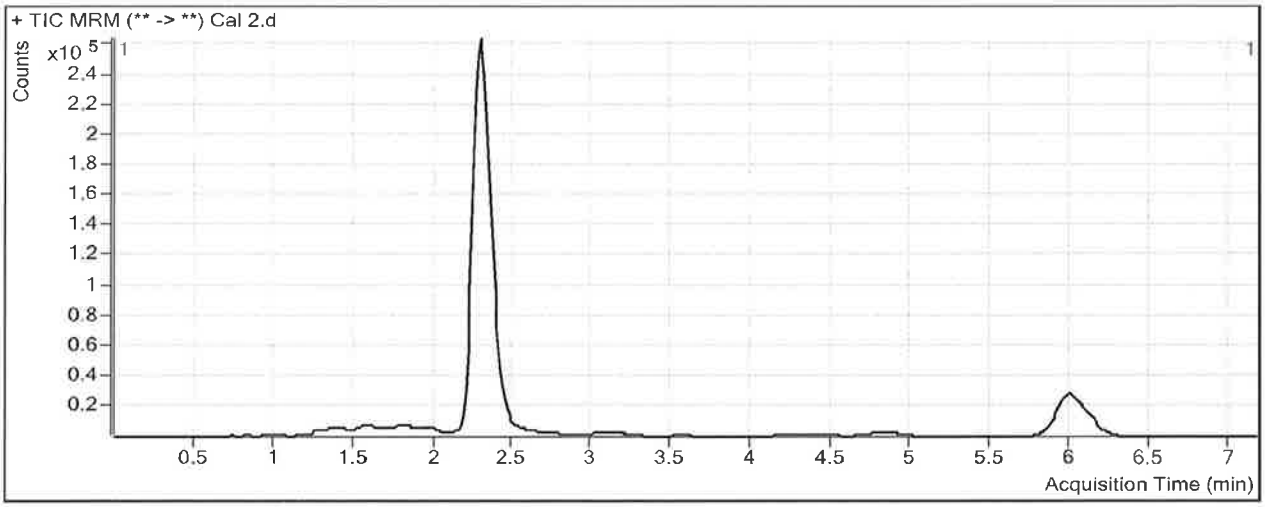
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:47 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 14:57	**Data File**	Cal 2.d
Sample Type	Calibration	**Sample Name**	Cal 2
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-B1	**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	63898	1654041	0.0386	4.9586
THC-COOH	THC-COOH-D9	2.406	59059	396728	0.1489	6.1276
THC	THC-D3	6.052	16237	371388	0.0437	5.0637

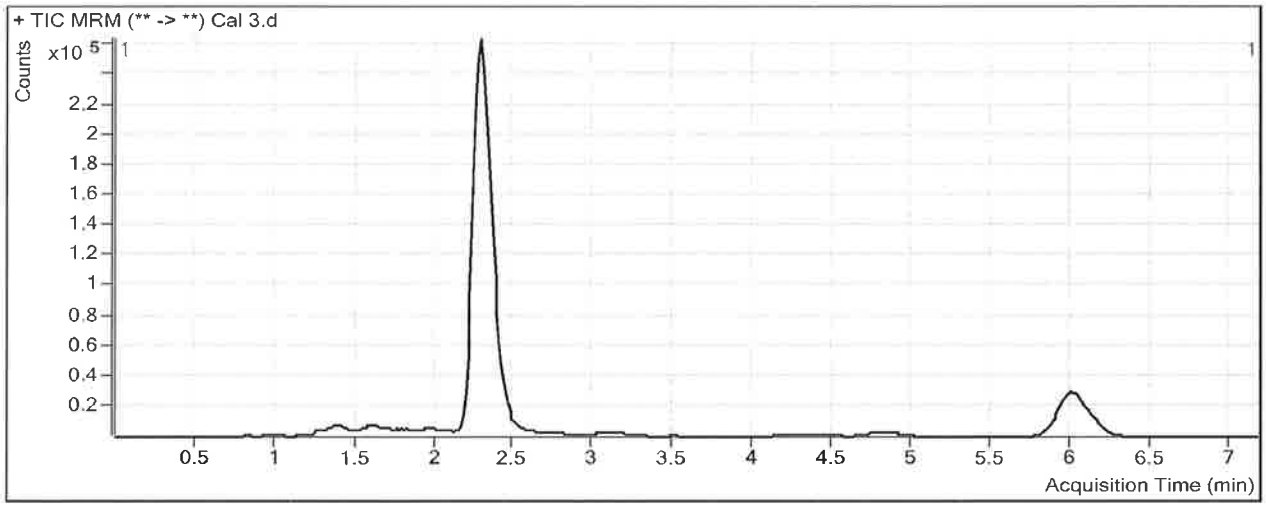
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:47 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 15:08	**Data File**	Cal 3.d
Sample Type	Calibration	**Sample Name**	Cal 3
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-C1	**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	2.305	124200	1590966	0.0781	9.3400
THC-COOH	THC-COOH-D9	2.392	88595	398694	0.2222	10.2496
THC	THC-D3	6.025	30489	362862	0.0840	9.8620

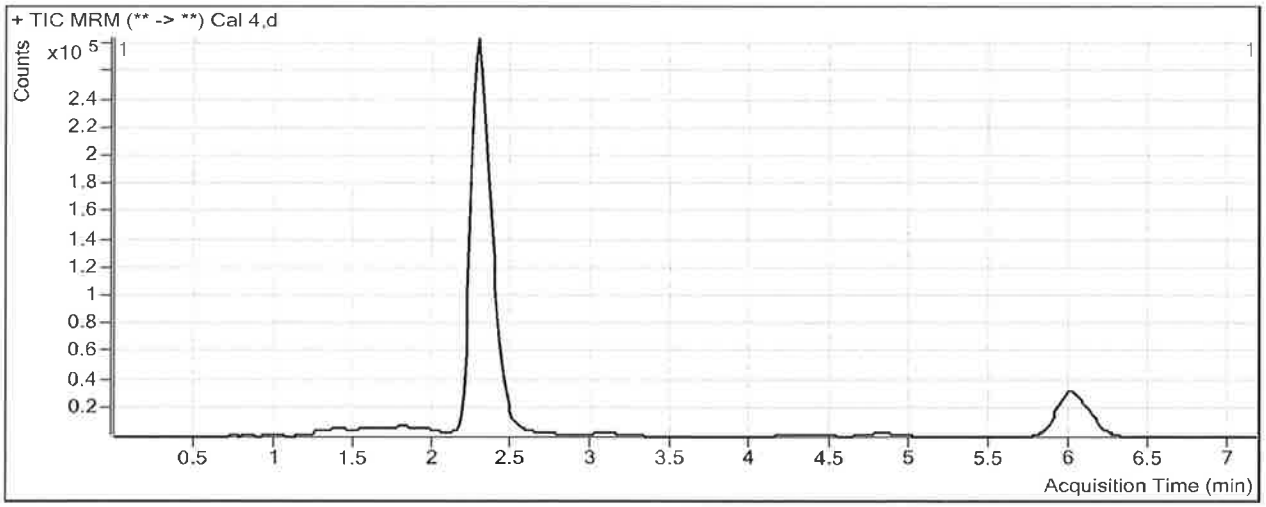
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:47 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 15:20	**Data File**	Cal 4.d
Sample Type	Calibration	**Sample Name**	Cal 4
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-D1	**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	2.305	330206	1548261	0.2133	24.3624
THC-COOH	THC-COOH-D9	2.392	180297	376574	0.4788	24.6684
THC	THC-D3	6.025	76908	344621	0.2232	26.4279

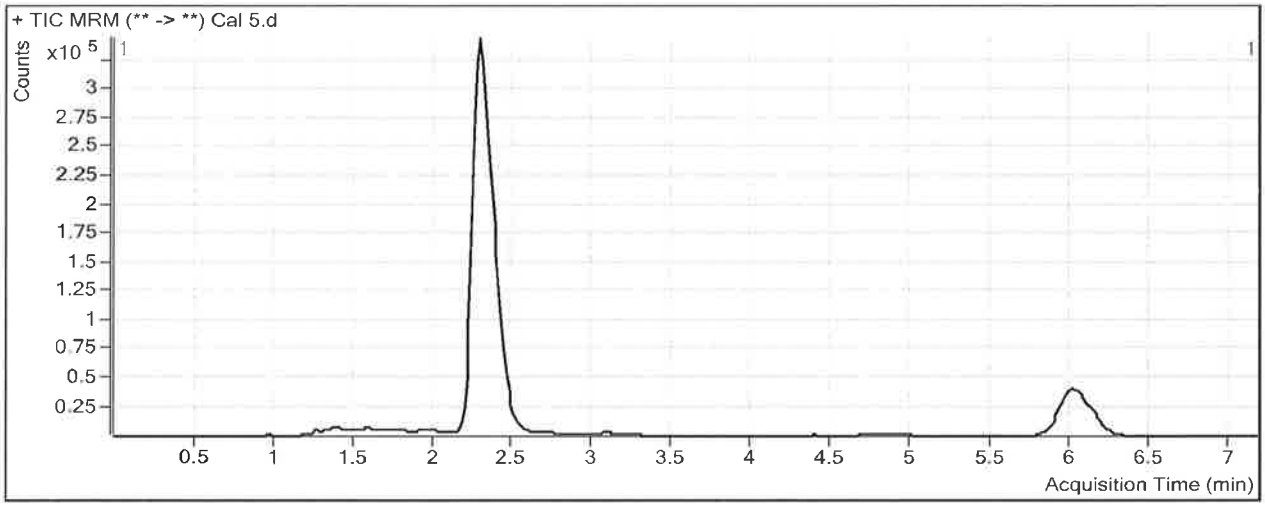
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:47 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 15:32	**Data File**	Cal 5.d
Sample Type	Calibration	**Sample Name**	Cal 5
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-E1	**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	651504	1551987	0.4198	47.3070
THC-COOH	THC-COOH-D9	2.392	348901	384558	0.9073	48.7489
THC	THC-D3	6.025	146717	351982	0.4168	49.4845

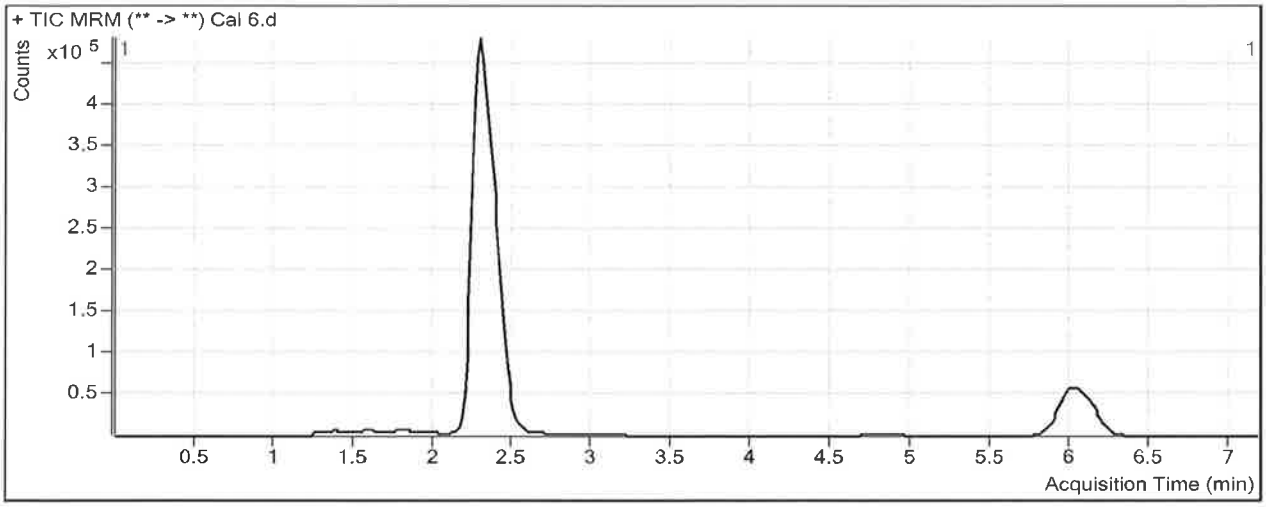
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:48 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 15:44	**Data File**	Cal 6.d
Sample Type	Calibration	**Sample Name**	Cal 6
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-F1	**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	1510770	1629415	0.9272	103.6816
THC-COOH	THC-COOH-D9	2.392	725719	393776	1.8430	101.3332
THC	THC-D3	6.025	326551	391048	0.8351	99.2775

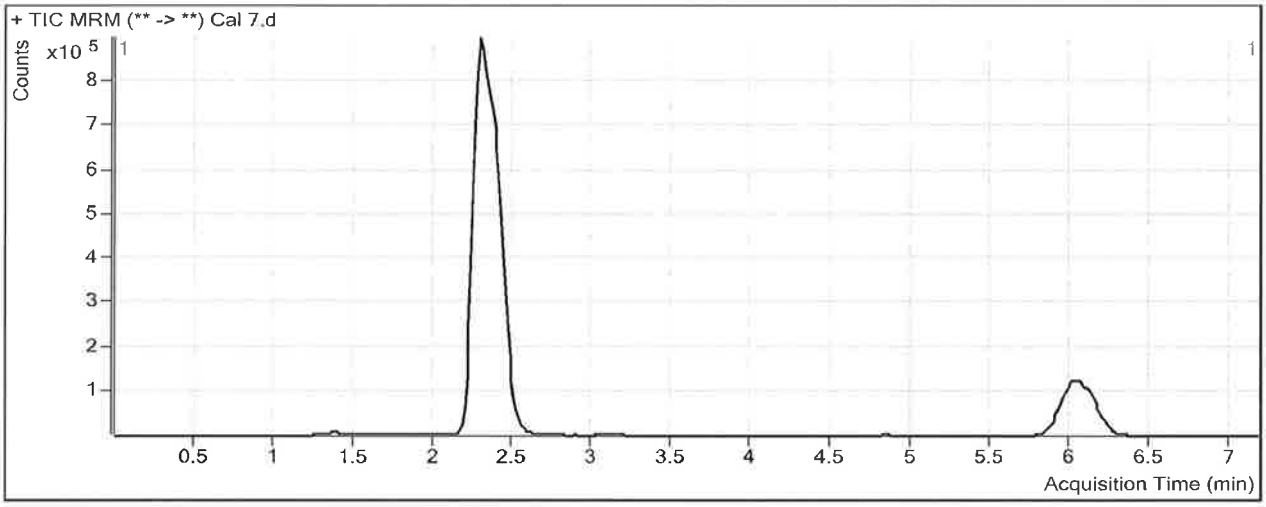
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\080318 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	8/8/2018 1:46 PM	**Analyst Name**	ISPUser
Report Time	8/8/2018 1:48 PM	**Reporter Name**	ISPUser
Last Calib Update	8/8/2018 1:46 PM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-03 15:56	**Data File**	Cal 7.d
Sample Type	Calibration	**Sample Name**	Cal 7
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P1-G1	**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	4400016	1441237	3.0529	339.8642
THC-COOH	THC-COOH-D9	2.392	2067080	332840	6.2104	346.7753
THC	THC-D3	6.039	987415	328625	3.0047	357.5815