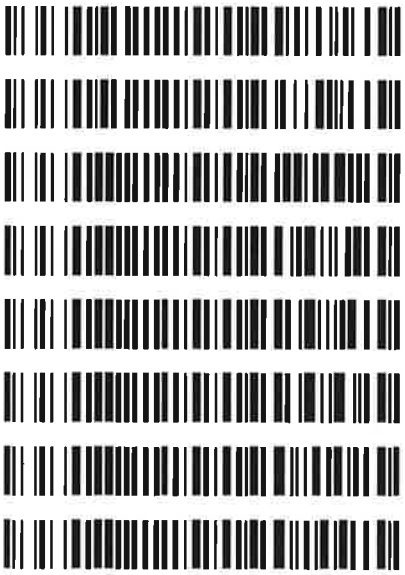
REVIEWED
By Celena Shrum at 1:39 pm, Mar 04, 2019

2/27/2019

Worklist: 2990

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
M2019-0371	1	142963	AM 27 Blood THC Quant by LC-QQQ
M2019-0479	1	142964	AM 27 Blood THC Quant by LC-QQQ
P2019-0474	1	142965	AM 27 Blood THC Quant by LC-QQQ
P2019-0501	1	142966	AM 27 Blood THC Quant by LC-QQQ
P2019-0512	1	142967	AM 27 Blood THC Quant by LC-QQQ
P2019-0513	1	142968	AM 27 Blood THC Quant by LC-QQQ
P2019-0602	1	142969	AM 27 Blood THC Quant by LC-QQQ
P2019-0603	1	142970	AM 27 Blood THC Quant by LC-QQQ



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

Extraction Date: 2/27/19 Analyst: Sarah Pickle
Plate lot#: 0539904 Plate Expiration: 09/10/19

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: 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: #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.

Post-Analytic

☒ 1. Create batch and process data.
Worklist path: 022719 THCQ TS 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: Carboxy-THC 10-100*

Idaho State Police
Forensic Services

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

Methanol External Control Solution (Lot: WS020419)

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	FE04231406	04/30/2019
C-THC	Cerilliant	FE07171501	09/31/2020
THC-OH	Cerilliant	FE01121503	01/31/2020
Prepared:	02/04/19		
Prepared By:	Tamara Salazar		
Expires:	04/30/2019		

Blood External Control Solution (Lot: 020419)

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	-	WS020419
Prepared:	02/04/19	
Prepared by:	Tamara Salazar	
Expires:	04/30/2019	

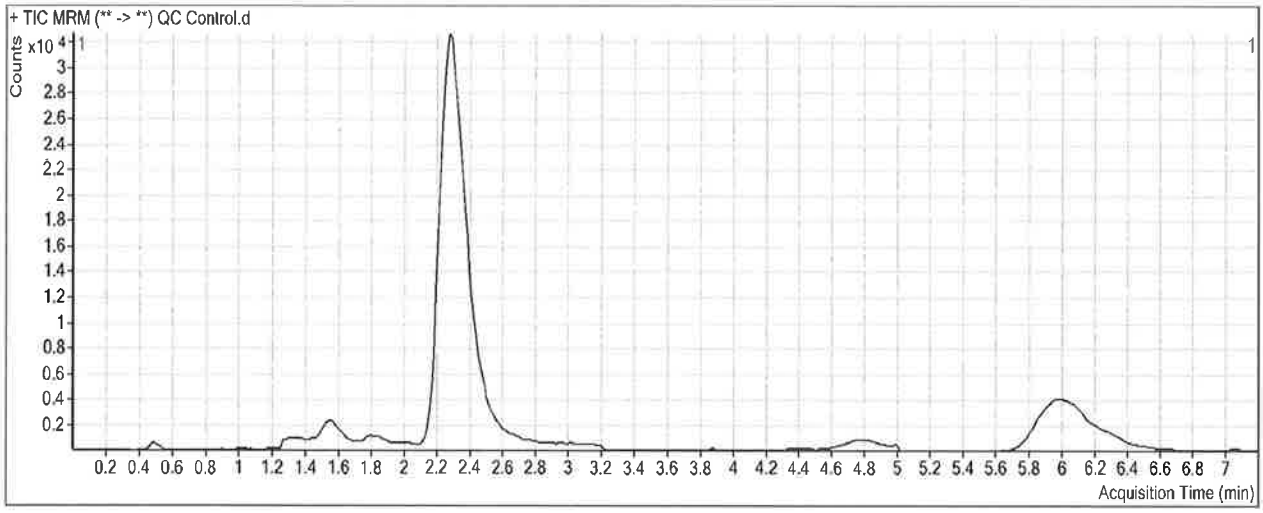
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:15 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 13:11	**Data File**	QC Control.d
Sample Type	QC	**Sample Name**	QC Control
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-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	2.265	12087	252283	0.0479	4.9329
THC-COOH	THC-COOH-D9	2.379	17713	84323	0.2101	9.5760
THC	THC-D3	6.039	3945	87102	0.0453	4.9408

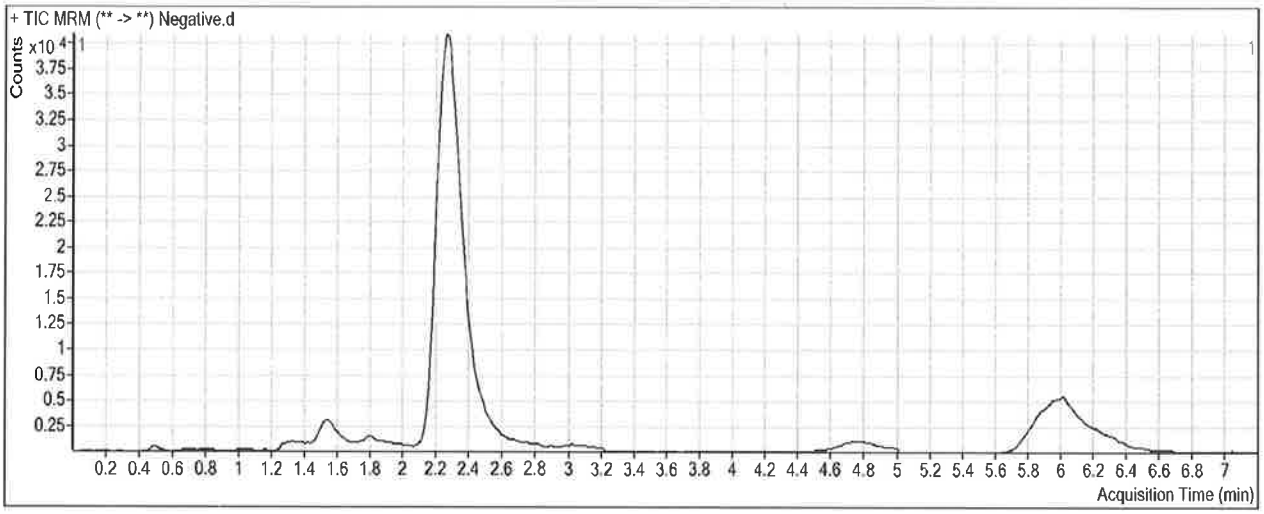
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:15 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 13:34	**Data File**	Negative.d
Sample Type	Sample	**Sample Name**	Negative
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-A2	**Sample Info**	
Inj Vol	-1	**Comment**	Hemostat 445283-1

Sample Chromatogram



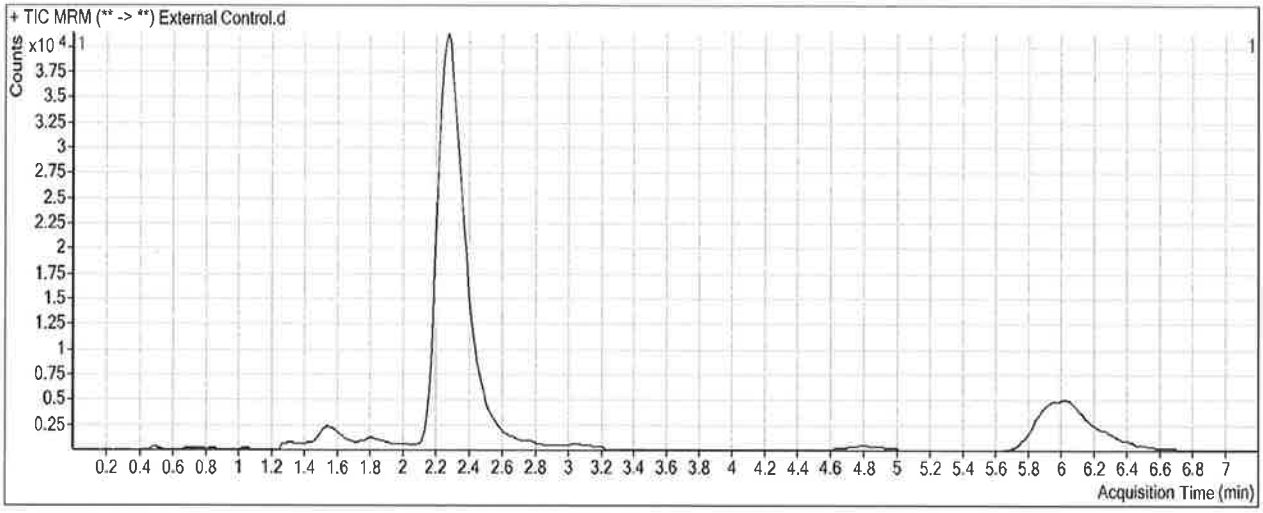
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:15 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 13:58	**Data File**	External Control.d
Sample Type	Sample	**Sample Name**	External Control
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-B2	**Sample Info**	
Inj Vol	-1	**Comment**	Hemostat 445283-1 + WS020419

Sample Chromatogram



Results

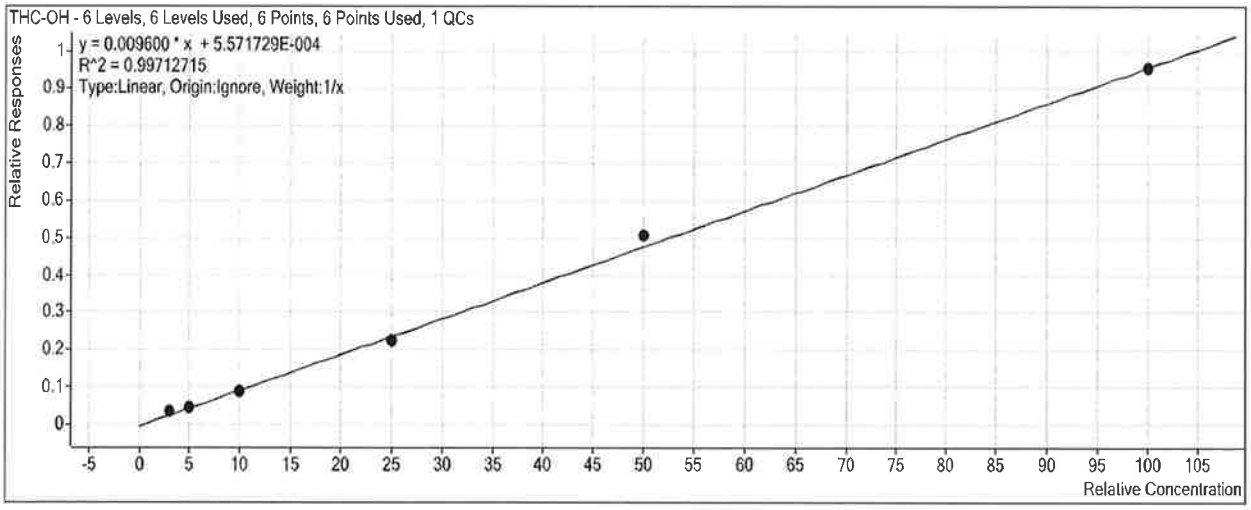
Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.265	23851	315259	0.0757	7.8231
THC-COOH	THC-COOH-D9	2.379	20372	101129	0.2014	9.1015
THC	THC-D3	5.999	7229	105231	0.0687	7.6092

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 2/28/2019 12:45 PM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-3ng	1	☑	3	3.5	115.8
Cal 2-5ng	2	☑	5	4.6	92.6
QC Control	2	☑	5	4.9	98.7
Cal 3-10ng	3	☑	10	9.4	93.6
Cal 4-25ng	4	☑	25	23.3	93.1
Cal 5-50ng	5	☑	50	52.7	105.4
Cal 6-100ng	6	☑	100	99.6	99.6

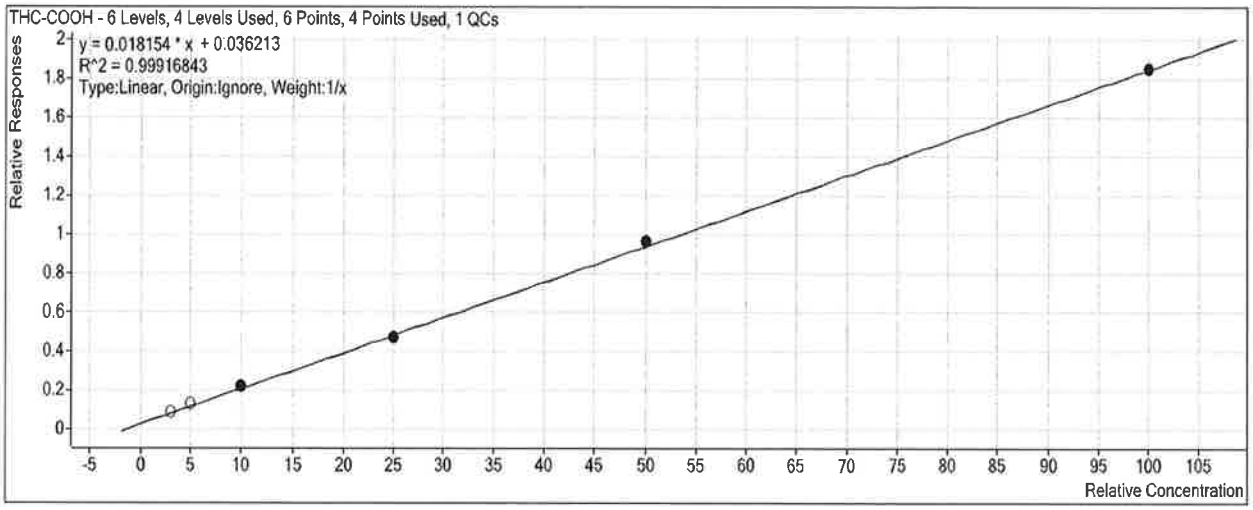
ISP Forensics
Calibration Curve Report

S

Batch Data Path C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 2/28/2019 12:45 PM **Analyst Name** ISP TOX

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



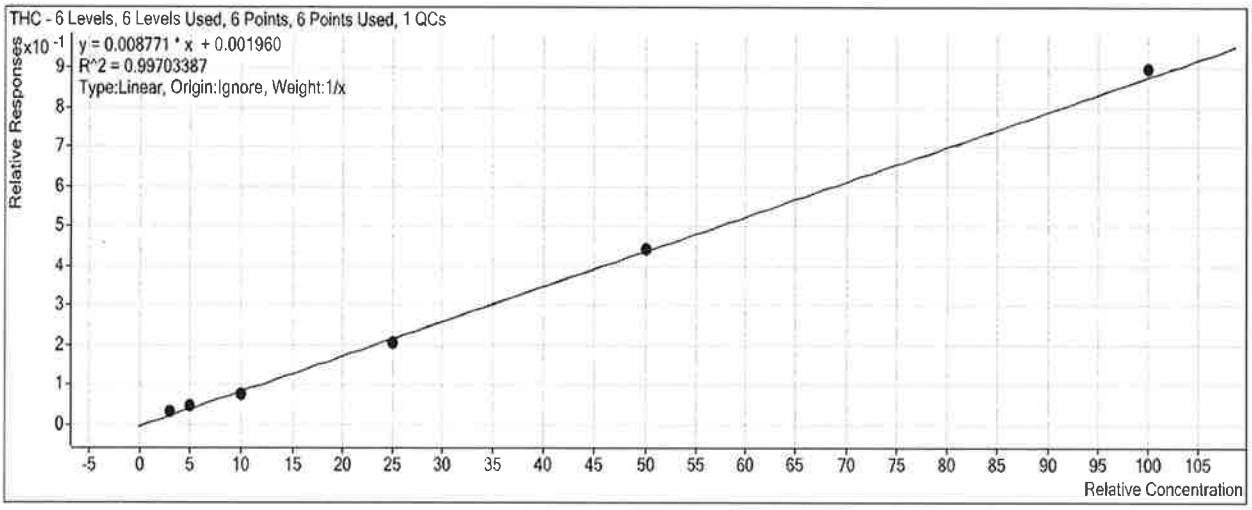
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-3ng	1	☐	3	2.7	89.9
Cal 2-5ng	2	☐	5	5.0	100.7
QC Control	2	☐	5	9.6	191.5
Cal 3-10ng	3	☑	10	10.3	102.7
Cal 4-25ng	4	☑	25	23.9	95.5
Cal 5-50ng	5	☑	50	50.9	101.8
Cal 6-100ng	6	☑	100	99.9	99.9

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 2/28/2019 12:45 PM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-3ng	1	☑	3	3.4	112.4
Cal 2-5ng	2	☑	5	5.3	105.7
QC Control	2	☑	5	4.9	98.8
Cal 3-10ng	3	☑	10	8.6	86.0
Cal 4-25ng	4	☑	25	23.3	93.2
Cal 5-50ng	5	☑	50	50.3	100.6
Cal 6-100ng	6	☑	100	102.1	102.1

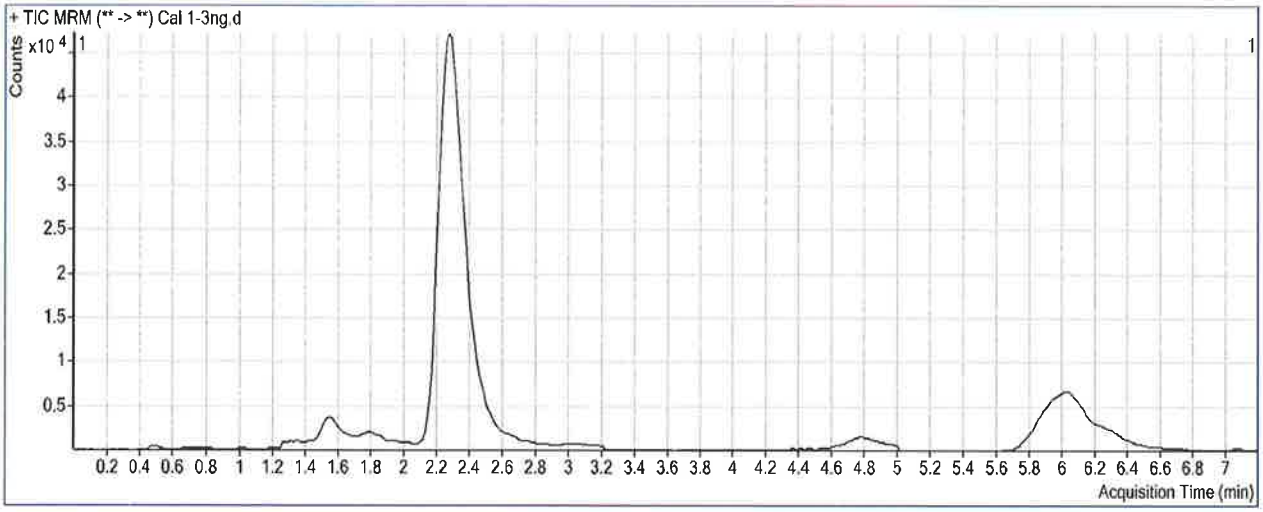
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:14 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 11:48	**Data File**	Cal 1-3ng.d
Sample Type	Calibration	**Sample Name**	Cal 1-3ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-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	2.279	12882	379958	0.0339	3.4737
THC-COOH	THC-COOH-D9	2.379	11233	131844	0.0852	2.6983
THC	THC-D3	6.012	4433	140585	0.0315	3.3714

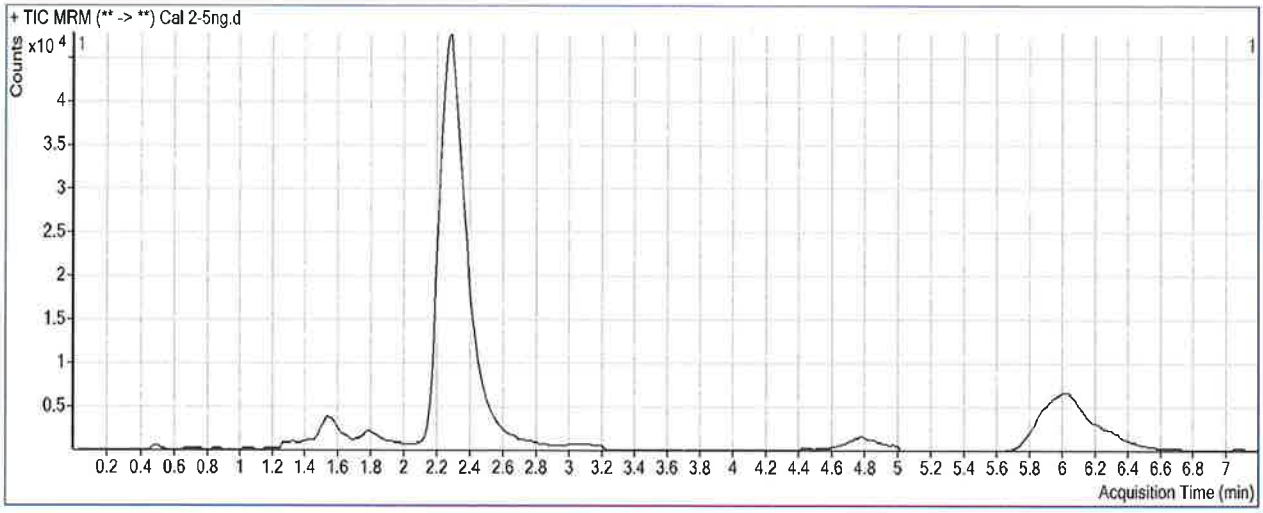
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:14 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 11:59	**Data File**	Cal 2-5ng.d
Sample Type	Calibration	**Sample Name**	Cal 2-5ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-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	2.279	16953	376788	0.0450	4.6288
THC-COOH	THC-COOH-D9	2.379	16281	127552	0.1276	5.0363
THC	THC-D3	5.985	6589	136376	0.0483	5.2849

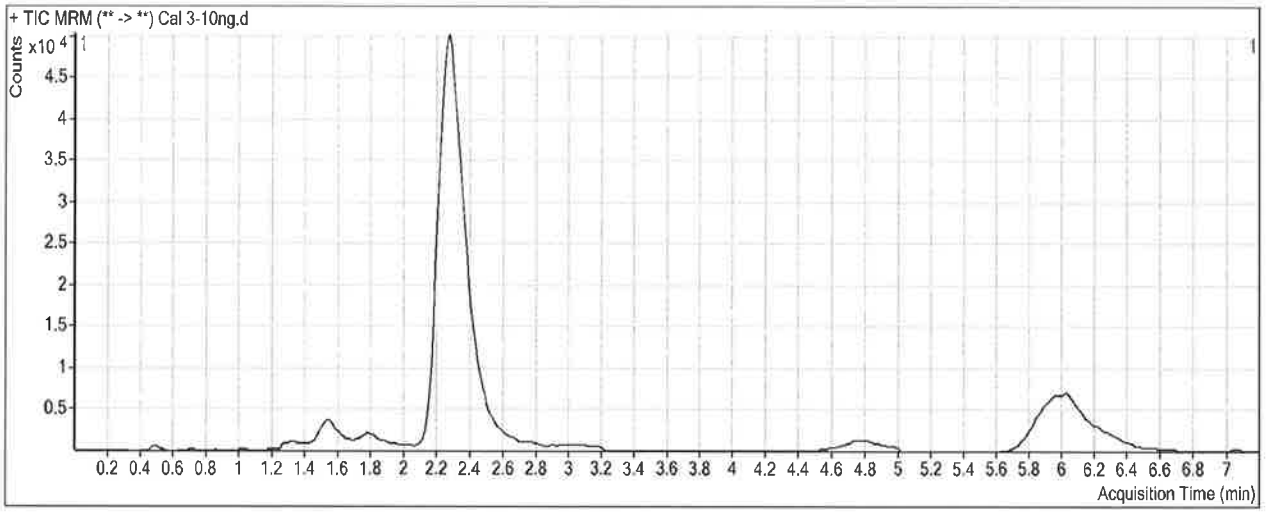
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:14 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 12:11	**Data File**	Cal 3-10ng.d
Sample Type	Calibration	**Sample Name**	Cal 3-10ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-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	2.279	33615	371937	0.0904	9.3566
THC-COOH	THC-COOH-D9	2.365	27935	125419	0.2227	10.2743
THC	THC-D3	6.025	11816	152743	0.0774	8.5963

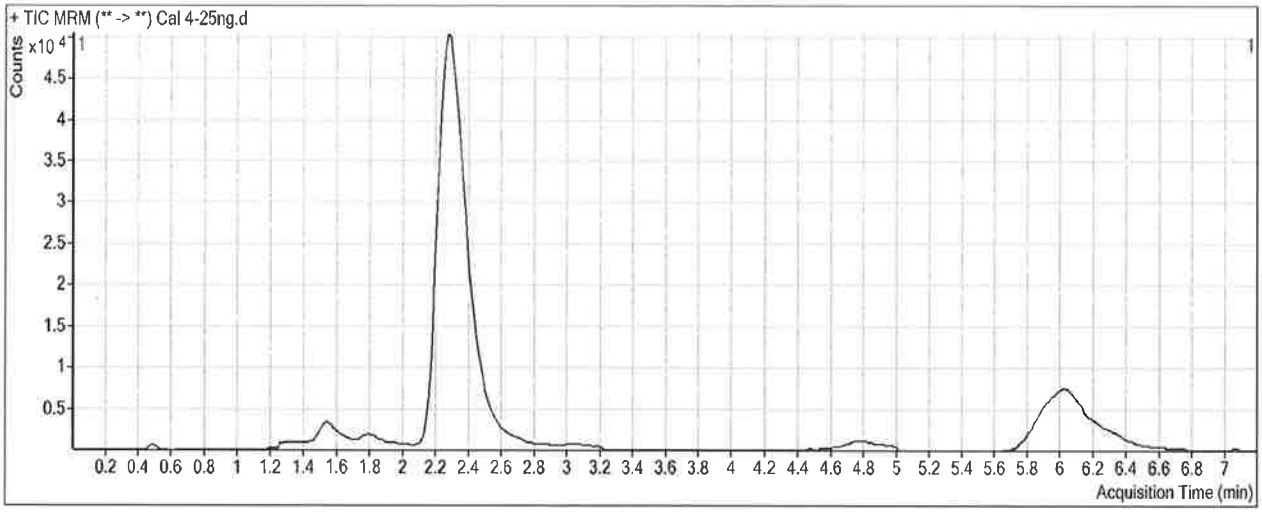
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:14 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 12:23	**Data File**	Cal 4-25ng.d
Sample Type	Calibration	**Sample Name**	Cal 4-25ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-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	2.279	75286	336054	0.2240	23.2794
THC-COOH	THC-COOH-D9	2.365	54597	116274	0.4696	23.8699
THC	THC-D3	6.025	26572	128763	0.2064	23.3052

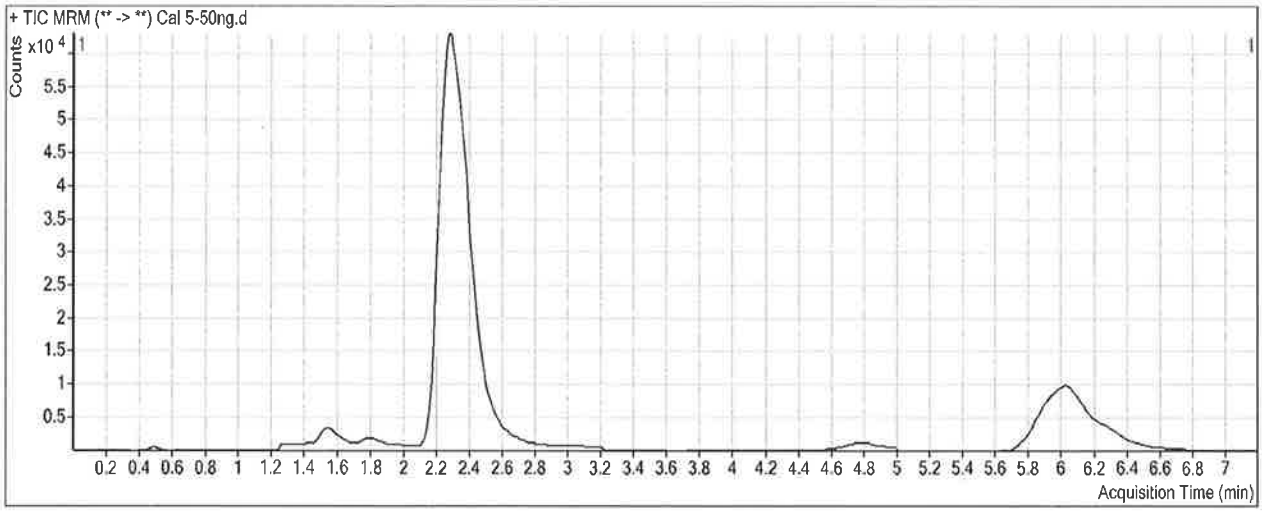
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:14 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 12:35	**Data File**	Cal 5-50ng.d
Sample Type	Calibration	**Sample Name**	Cal 5-50ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-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	2.279	170351	336428	0.5064	52.6888
THC-COOH	THC-COOH-D9	2.379	108077	112504	0.9607	50.9214
THC	THC-D3	6.025	55380	124981	0.4431	50.2975

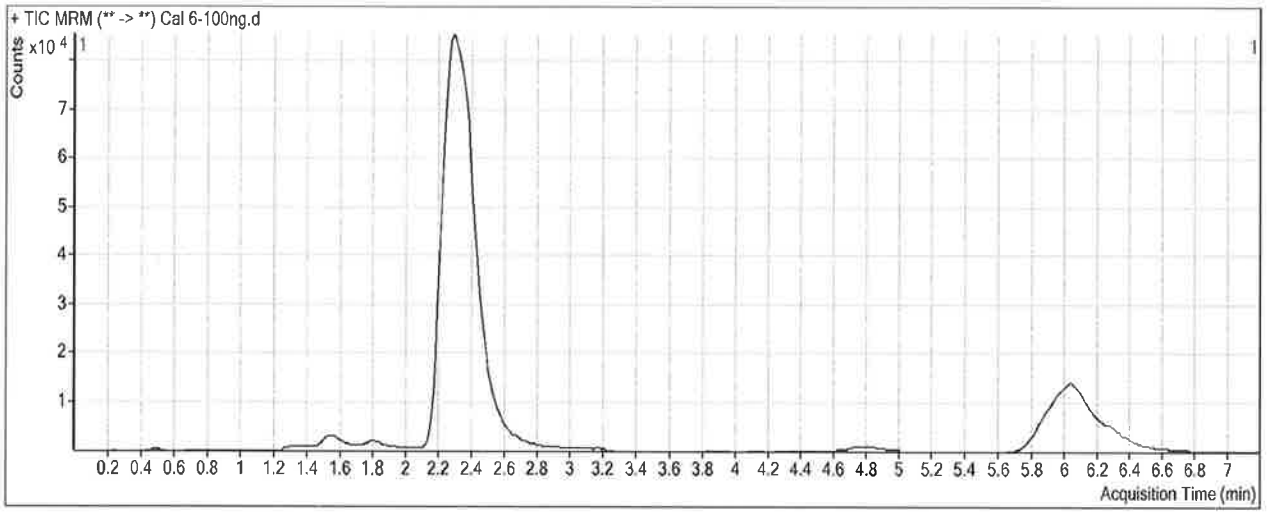
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\022719 THCQ TS SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	2/28/2019 12:45 PM	**Analyst Name**	datastor
Report Time	3/1/2019 2:15 PM	**Reporter Name**	datastor
Last Calib Update	2/28/2019 12:45 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-02-27 12:47	**Data File**	Cal 6-100ng.d
Sample Type	Calibration	**Sample Name**	Cal 6-100ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-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	2.279	327800	342736	0.9564	99.5727
THC-COOH	THC-COOH-D9	2.379	204746	110647	1.8504	99.9344
THC	THC-D3	6.039	109056	121464	0.8978	102.1447