REVIEWED
By Anne Nord at 7:25 am, Oct 17, 2018

10/10/2018

Worklist: 2728

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
M2018-3955	1	128859	AM 27 Blood THC Quant by LC-QQQ
M2018-4517	1	128860	AM 27 Blood THC Quant by LC-QQQ
M2018-4522	1	128861	AM 27 Blood THC Quant by LC-QQQ
M2018-4572	1	128862	AM 27 Blood THC Quant by LC-QQQ
M2018-4630	1	128863	AM 27 Blood THC Quant by LC-QQQ
M2018-4670	1	128864	AM 27 Blood THC Quant by LC-QQQ
M2018-4887	4	128865	AM 27 Blood THC Quant by LC-QQQ
P2018-2728	1	128866	AM 27 Blood THC Quant by LC-QQQ
P2018-2734	1	128867	AM 27 Blood THC Quant by LC-QQQ
P2018-2754	1	128868	AM 27 Blood THC Quant by LC-QQQ
P2018-2800	1	128869	AM 27 Blood THC Quant by LC-QQQ
P2018-2818	1	128870	AM 27 Blood THC Quant by LC-QQQ

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

Extraction Date: 10/05/18 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: 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: 100518 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*~~
Did not evaluate THC-COOH.

Idaho State Police Forensic Services

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

Analyst: Sarah Pickle
Extraction Date: 10/05/18
Worklist Number: 2728

Reagent	*Lot Number*	*Expiration Date*	*Date in Service*	*Date Out of Service*	*Initials*
ToxBox THC/THC Metabolite Plate	0539904	09/10/19			
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)	100518		10/05/18		
0.1% Formic Acid in Acetonitrile (Mobile Phase B)	176190		2/6/18		
Needle Rinse--75% LCMS MeOH in LCMS Water	092418		09/24/18		

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	

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

Component	*Source*	*Source Lot Number*
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: 092418)

Component	*Source*	*Source Lot Number*
MeOH (LCMS Grade)	Fisher	177145
Water (LCMS Grade)	Fisher	182702
Prepared:	09/24/18	
Prepared By:	Tamara Salazar	

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Idaho State Police
Forensic Services
Toxicology Discipline

Request for Departure from an Analytical Method

Date of Request
10/16/2018

Forensic Scientist
Sarah Pickle

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

Request

I am formally requesting a deviation to not evaluate carboxy-THC for my current batch (worklist 2728) due to a possible interferant in the carboxy-THC confirmation data. Samples that contain THC and/or OH-THC will be evaluated and reported. Any samples that possibly contain carboxy-THC and do not contain either THC or OH-THC will be re-extracted and ran at a later date.

Discipline Leader Review

☒ Departure approved
Comments:

☐ Departure Not Approved
Comments:

Date: 10/16/2018

Celena Shrum

Celena Shrum
Toxicology Discipline Lead

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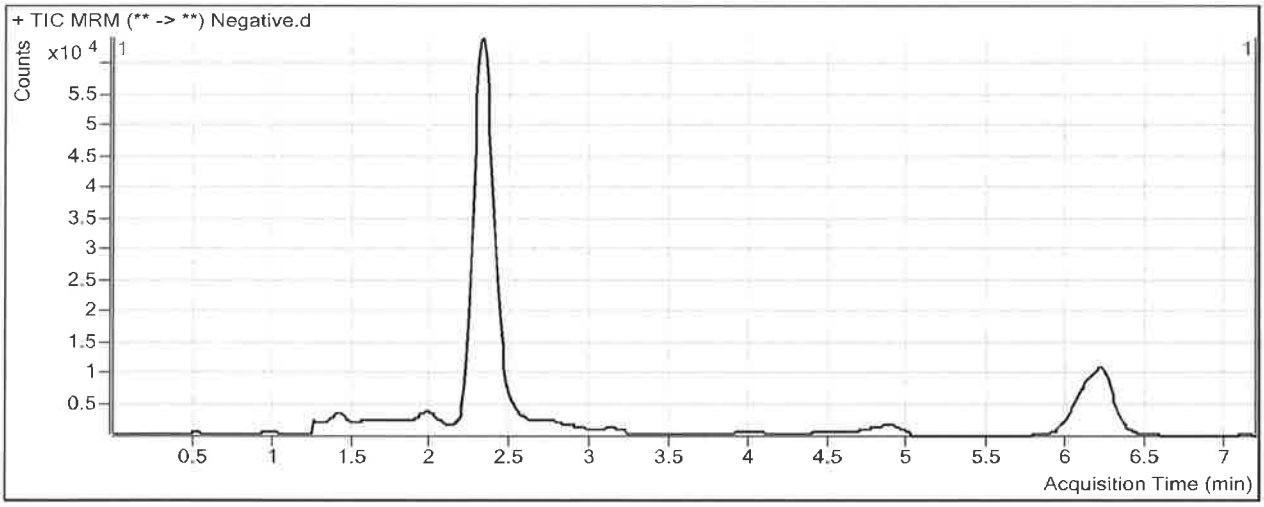
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:58 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 17:39	**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	2.145	2630	416226	0.0063	1.1525
THC-COOH	THC-COOH-D9	2.406	19834	138448	0.1433	0.0000

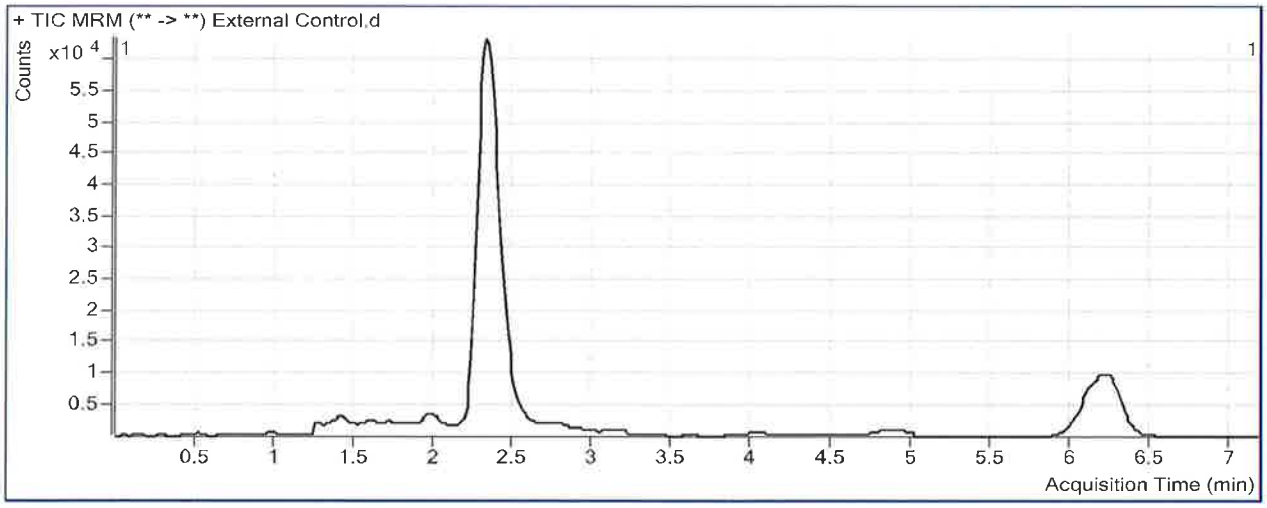
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:58 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 18:02	**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	2.332	33043	380336	0.0869	8.3901
~~THC-COOH~~	~~THC-COOH-D9~~	~~2.432~~	~~49781~~	~~128560~~	~~0.3872~~	~~9.1362~~ DNC
THC	THC-D3	6.239	10177	149047	0.0683	7.9555

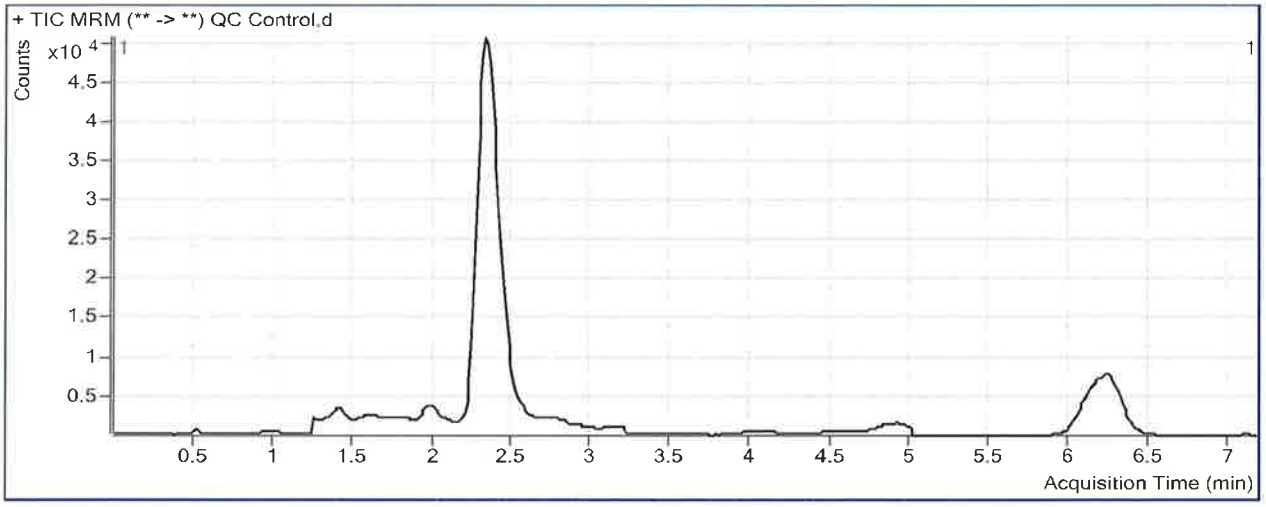
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:58 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 17:15	**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	2.332	18123	310939	0.0583	5.8212
~~THC-COOH~~	~~THC-COOH-D9~~	~~2.432~~	~~43058~~	~~103050~~	~~0.4178~~	~~10.7385~~ DNE
THC	THC-D3	6.252	5054	118657	0.0426	5.1534

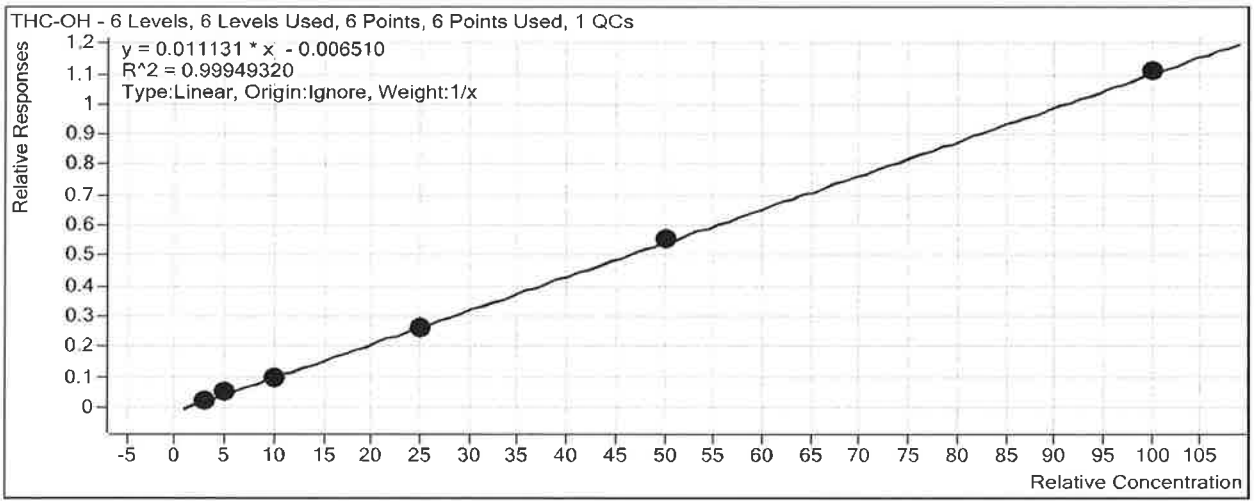
ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 10/16/2018 3:12 PM **Analyst Name** ISP TOX

Target Compound *THC-OH*

Internal Standard *THC-OH-D3*



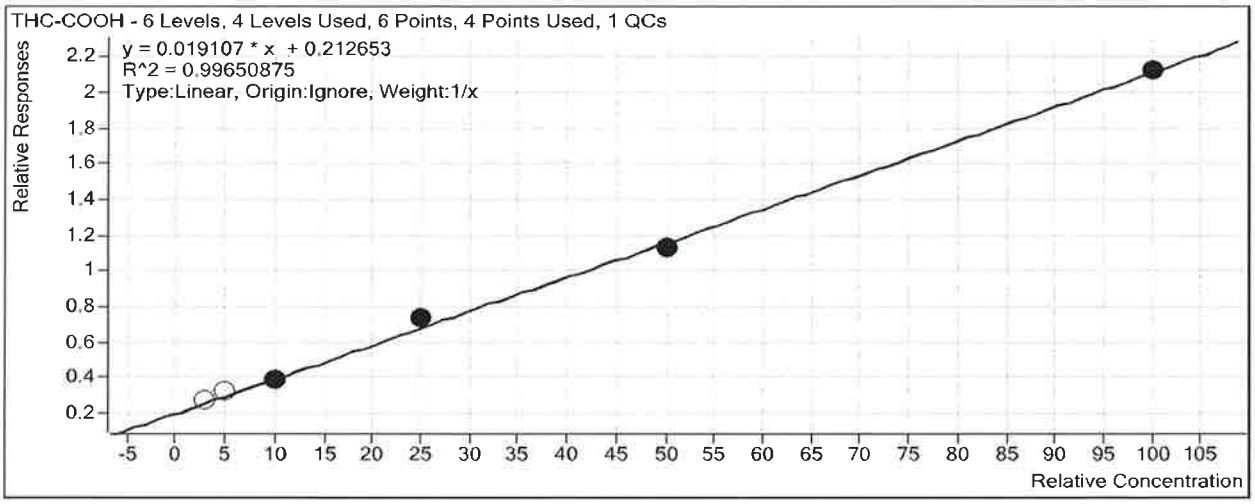
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-1ng	1	☐	1	1.3	127.0
Cal 2-3ng	2	☑	3	2.9	97.1
Cal 3-5ng	3	☑	5	5.4	108.1
Cal 4-10ng	4	☑	10	9.6	96.0
Cal 5-25ng	5	☑	25	24.4	97.5
Cal 6-50ng	6	☑	50	50.5	101.1
Cal 7-100ng	7	☑	100	100.2	100.2

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 10/16/2018 3:12 PM **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	☐	1	1.3	129.8
Cal 2-3ng	2	☐	3	3.6	119.4
Cal 3-5ng	3	☐	5	5.7	114.4
Cal 4-10ng	4	☑	10	9.4	93.9
Cal 5-25ng	5	☑	25	27.4	109.5
Cal 6-50ng	6	☑	50	48.3	96.7
Cal 7-100ng	7	☑	100	99.9	99.9

Did not evaluate THC-COOH due to interferant. SP

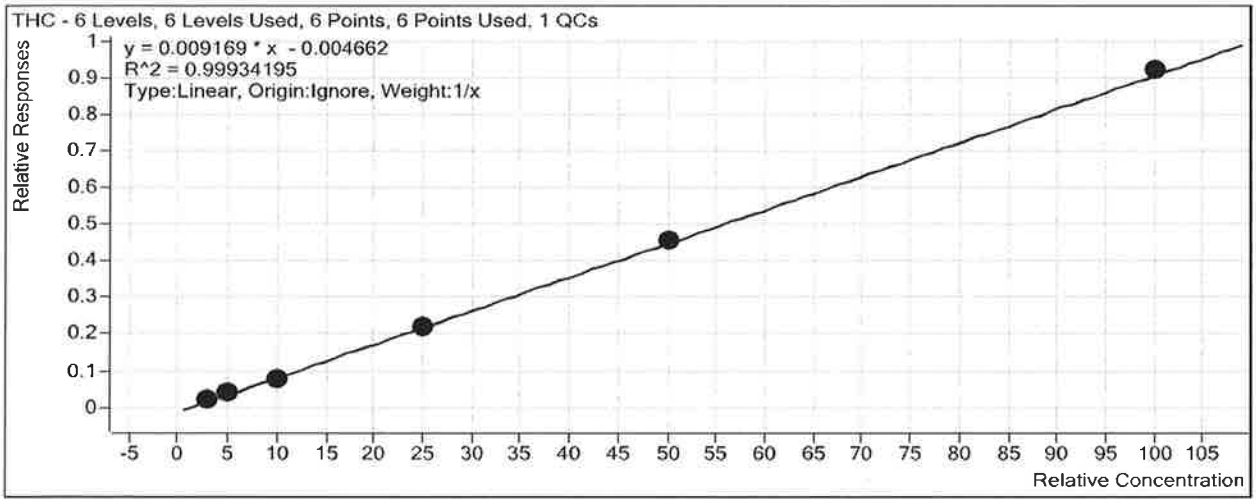
ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin

Last Calib Update 10/16/2018 3:12 PM **Analyst Name** ISP TOX

Target Compound *THC*

Internal Standard *THC-D3*



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-1ng	1	☐	1	1.6	155.6
Cal 2-3ng	2	☑	3	3.3	108.5
Cal 3-5ng	3	☑	5	5.0	99.7
Cal 4-10ng	4	☑	10	9.3	93.0
Cal 5-25ng	5	☑	25	24.4	97.6
Cal 6-50ng	6	☑	50	50.1	100.2
Cal 7-100ng	7	☑	100	100.9	100.9

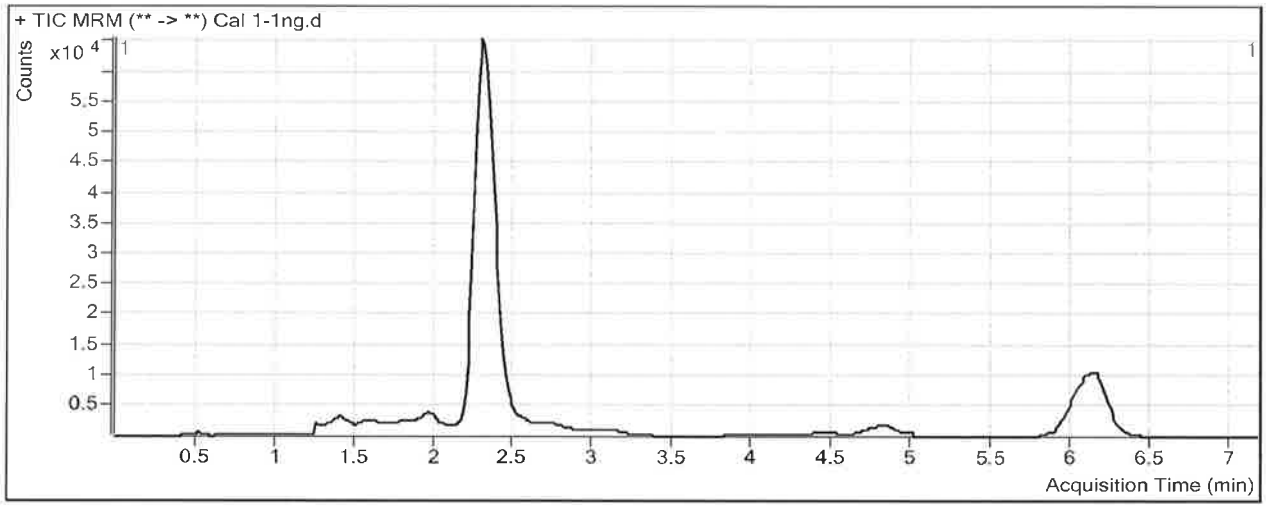
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:57 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 15:40	**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-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	3155	413947	0.0076	1.2696
THC-COOH	THC-COOH-D9	2.392	32404	136470	0.2374	1.2975
THC	THC-D3	6.146	1517	157904	0.0096	1.5562

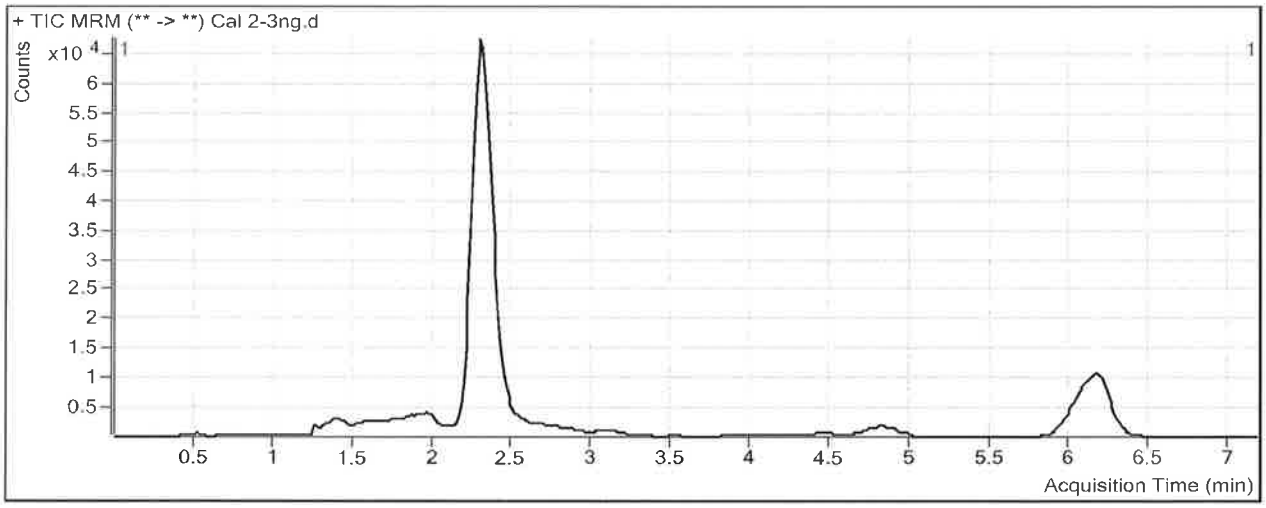
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:57 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 15:52	**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-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	2.305	10753	414719	0.0259	2.9143
THC-COOH	THC-COOH-D9	2.392	37986	135140	0.2811	3.5814
THC	THC-D3	6.186	3945	156620	0.0252	3.2554

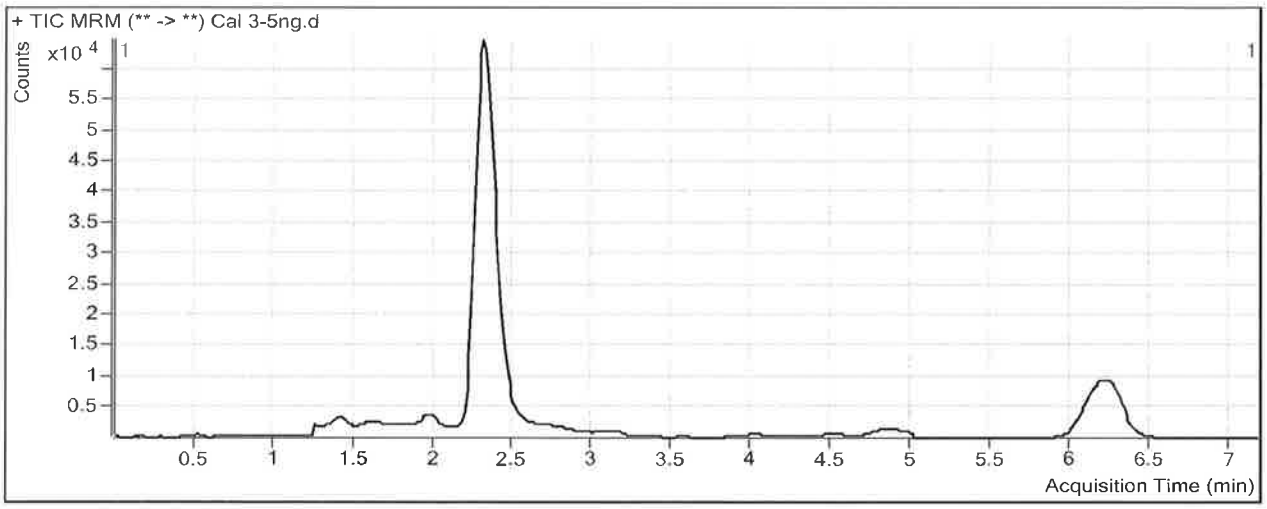
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:58 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 16:04	**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-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	2.319	20851	388739	0.0536	5.4038
THC-COOH	THC-COOH-D9	2.406	41234	128092	0.3219	5.7180
THC	THC-D3	6.266	5983	145833	0.0410	4.9827

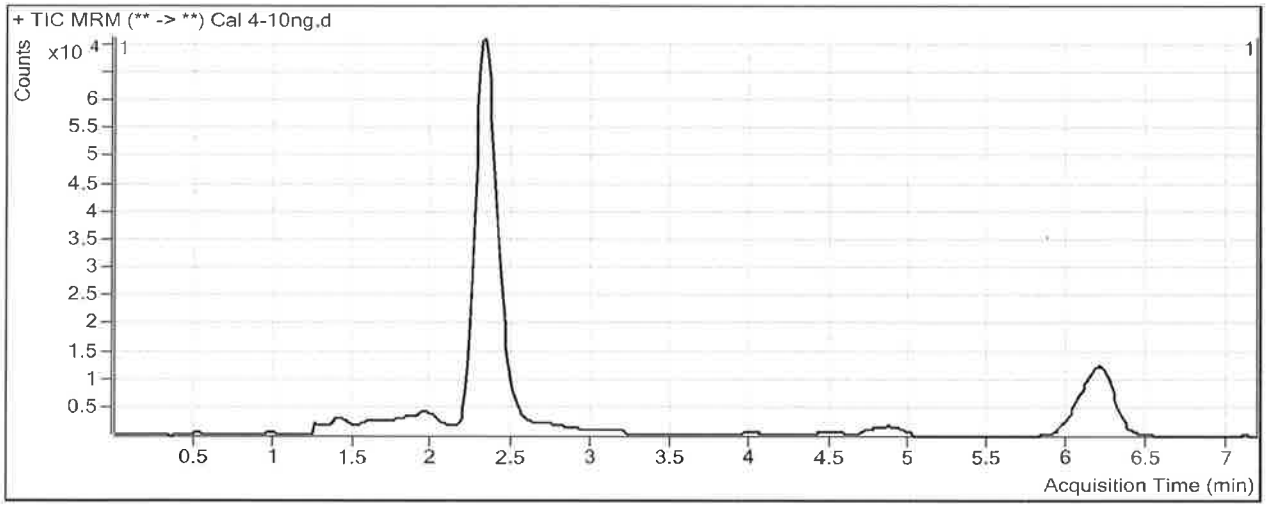
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:58 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 16:16	**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-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	2.319	42728	425664	0.1004	9.6032
THC-COOH	THC-COOH-D9	2.432	54003	137709	0.3922	9.3944
THC	THC-D3	6.226	13948	172962	0.0806	9.3033

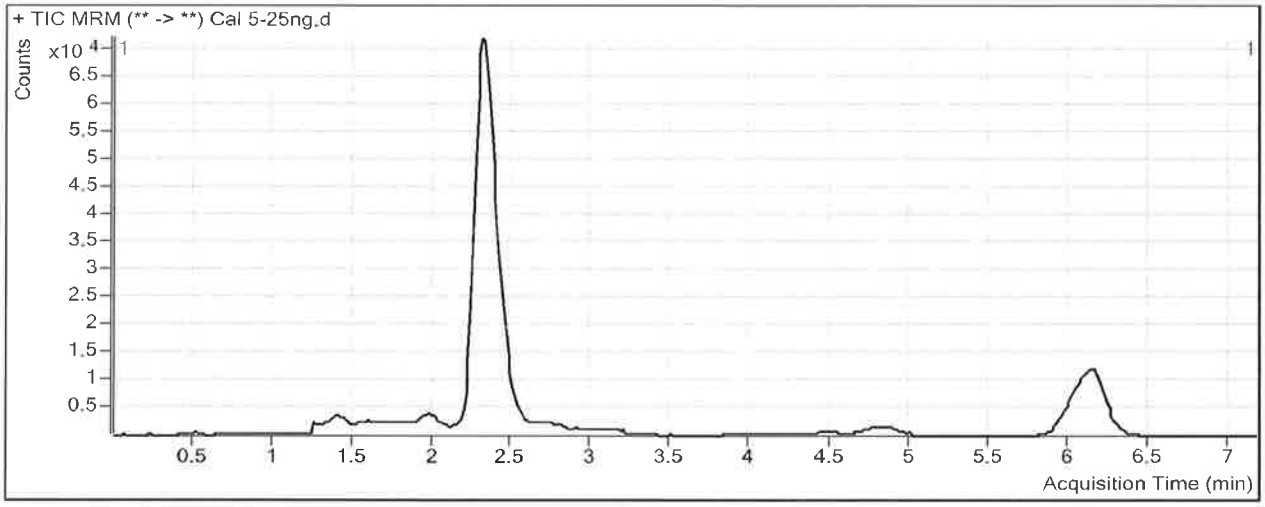
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:58 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 16:28	**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-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	2.319	96391	363907	0.2649	24.3820
THC-COOH	THC-COOH-D9	2.419	84657	115069	0.7357	27.3748
THC	THC-D3	6.159	29729	135674	0.2191	24.4061

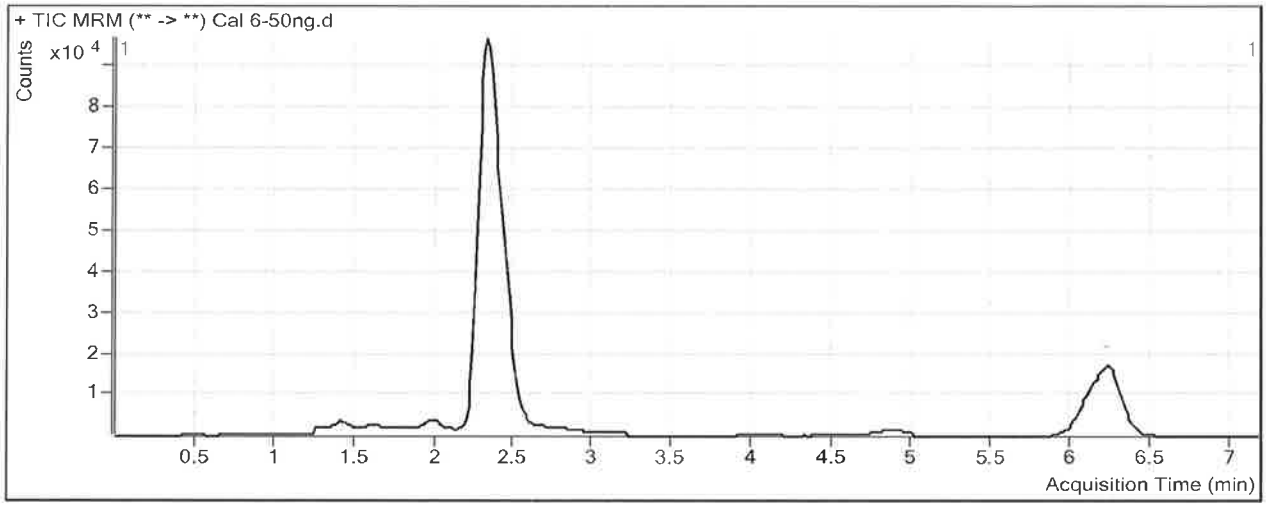
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:58 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 16:39	**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-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	2.332	224592	404030	0.5559	50.5263
THC-COOH	THC-COOH-D9	2.432	148358	130594	1.1360	48.3259
THC	THC-D3	6.239	67976	149424	0.4549	50.1235

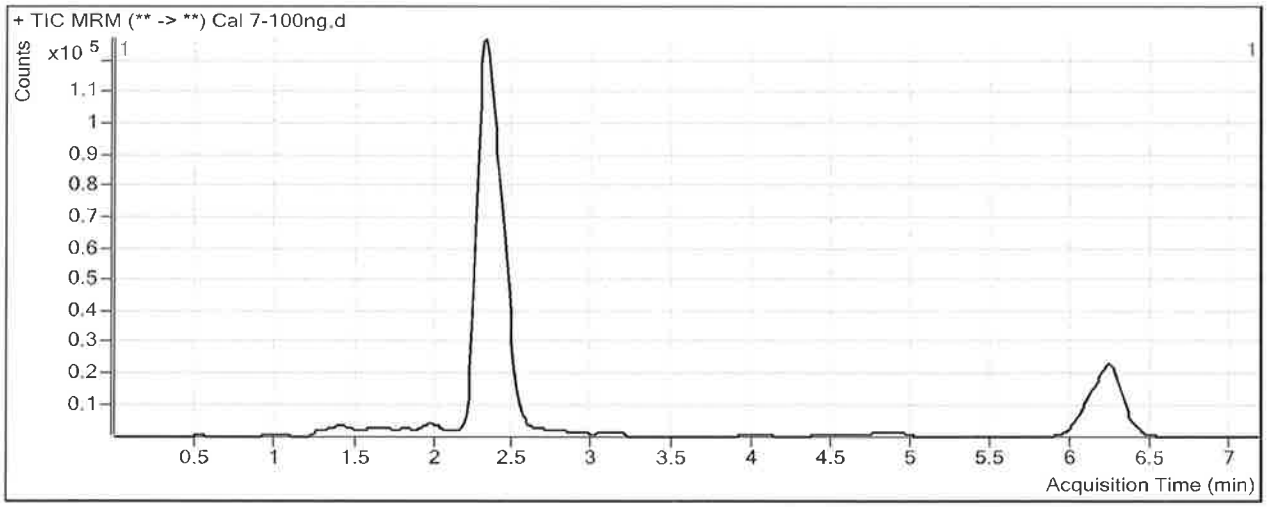
ISP FORENSICS - Pocatello Instrument # 59740
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\100518 THCQ SP\QuantResults\THCQ SP.batch.bin		
Analysis Time	10/10/2018 9:16 AM	**Analyst Name**	ISPUser
Report Time	10/10/2018 1:58 PM	**Reporter Name**	ISPUser
Last Calib Update	10/10/2018 9:16 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-10-05 16:51	**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-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	2.319	422598	381251	1.1084	100.1705
THC-COOH	THC-COOH-D9	2.419	259342	122242	2.1215	99.9049
THC	THC-D3	6.239	130533	141767	0.9208	100.9291