

Worklist: 6086

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>	
C2022-1837	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
M2022-3014	2	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
M2022-3132	3	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
M2022-3348	3	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
M2022-3493	2	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
M2022-3517	3	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
P2022-2327	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
P2022-2328	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
P2022-2408	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
P2022-2589	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
P2022-2597	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
P2022-2646	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
P2022-2668	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	
P2022-2786	1	UCK	AM 25/AM 26 Urine MultiDrug/THC Screen by LC-QQQ	

SC

**Idaho State Police
Forensic Services**

Request for Departure from an Analytical Method or Quality Standard

Deviation Number (assigned by QM): TOX-22-01

Date of Request: **2/3/2022**

Requestor/Discipline: Celena Shrum/Toxicology

Analytical Method/Quality Standard, Revision #: AM #25, AM #28, AM #29, Revision 13

Temporary or Permanent Deviation: Permanent

Scope of Deviation (record specific information, e.g. affected programs, evidence types, expected end date; etc): Deviation will remain in place until the change is made in the next method revision.

Deviation Request (Describe detailed instructions of the changes being made; include reference to specific section number(s) in the method manual): 4.1.4 (Place plate on shaking incubator at approximately 900 rpm for approximately 15 minutes) of AM #25, AM # 28, and AM #29 is being removed. The removal of this step was tested in the validation "Addition of Compounds/Modifications for the MDS" (approved on 2/2/2022) and it was determined that that step is not necessary and can be removed.

Technical Justification for Analytical Method Deviations: Refer to validation "Addition of Compounds/Modifications for the MDS" (approved on 2/2/2022)

Technical Review

Departure approved
Comments:

Departure Not Approved
Comments:

Approver: Rachel Cutler
Title: Laboratory Manager



Date: 2/10/2022

Quality Review

Quality Approver: Jason Crowe
Title: Quality Manager
Date: 2/10/2022



SC

**Idaho State Police
Forensic Services**

Request for Departure from an Analytical Method or Quality Standard

Deviation Number (assigned by QM): TOX-22-02

Date of Request:
03/02/2022

Requestor/Discipline:
Celena Shrum/Toxicology

Analytical Method/Quality Standard, Revision #:
Toxicology AM #25, AM #26, and AM #27, Revision 13

Temporary or Permanent Deviation:
Permanent

Scope of Deviation (record specific information, e.g. affected programs, evidence types, expected end date; etc):

Deviation will remain in place until the change is made in the next method revision.

Deviation Request (Describe detailed instructions of the changes being made; include reference to specific section number(s) in the method manual):

Toxicology AM #25 3.3.1.1 Internal standards are prepared by the ToxBox plate manufacturer and contained on the 96 well plate. If the run contains urine samples, a positive external urine control must also be run.

Toxicology AM #26 3.3.2 A negative control will be run with each extraction. If the run contains urine samples, a negative urine control and external positive urine control must also be included.

Toxicology AM #27 3.3.2 A negative control will be run with each extraction. If the run contains urine samples, a negative urine control and positive external urine control will also be included in the run.

The deviation is to include the option of using an internal urine control in lieu of an external urine control.

SC

Technical Justification for Analytical Method Deviations:

Internal controls serve the same purpose as external controls but also helps to avoid the possible issues that can occur with using external controls (incorrect spiking, incorrect preparation, evaporation of compounds, etc.). If these errors occur, runs need to be repeated and this wastes time, sample, and supplies.

Technical Review

Departure approved

Comments:

Departure Not Approved

Comments:



Approver: Rachel Cutler

Title: Lab Manager

Date: 3/2/22

Quality Review

Quality Approver: Jason Crowe

Title: Quality Manager

Date: 3/2/2022



SC

AM# 25: Multi-Drug Screen in Blood and Urine by LC-MS/MS

Extraction Date: 09/08/2022
Plate lot#: IDP-120-2-220315

Analyst: Sarah Collins
Retest Date: 09/15/22

Mobile phase A: 10mM Amm Form
Instant Buffer I

Mobile phase B: 0.1% Formic Acid in MeOH
Ethyl Acetate LC Methanol

Blank Blood Lot: Lampire 22B52015-1

Blank Urine Lot: POC021022

Column: Phenomenex Phenyl Hexyl (4.6x50mm, 2.6um)

LCMS-QQQ ID: 069901

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.

Analytic:

- 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
- 2. Urine Hydrolysis: In blank well, add 250µL urine, 40µL BG Turbo, and 100µL Instant Buffer I. Place on plate shaker for 5 minutes.
- 3. Using a calibrated pipette, pipette **250µL blood and urine** (if applicable) into wells of analytical (standards) plate.
Pipette ID: #16
- 4. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. (SKIPPED PER DEVIATION)
- 5. Pipette **250µL 0.5 M ammonium hydroxide** in wells of analytical plate.
- 6. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 7. Transfer **200-450µL of blood+base and urine+base (if applicable)** mixture to corresponding wells of SLE+ plate.
Amount transferred: 300 uL
- 8. 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).
- 9. Wait 5 minutes.
- 10. Add **900uL ethyl acetate.**
- 11. Wait 5 minutes.
- 12. Apply positive pressure for approx. 15 seconds. **(10-15 PSI- Selector to the left).**
- 13. Add **900uL ethyl acetate.**
- 14. Wait 5 minutes.
- 15. Apply positive pressure for approx. 15 seconds. **(10-15 PSI- Selector to the left).**
- 16. Remove plate containing eluate. Place on SPE Dry and evaporate to dryness at approx. 35°C. If run contains urine, add 50µL 1% HCl in MeOH to wells and place plate cover on plate before drying.
- 17. Reconstitute in **100µL 20% LC MeOH** and heat seal plate with foil. Place in autosampler and run worklist.

Post-Analytic

- 1. Open quantitation software and create a new quantitation batch.
- 2. Make necessary changes to integration limits
- 3. Evaluate samples, S/N of primary transition >5 and S/N of secondary transition >3 or evaluation of peak symmetry and resolution. Within +/- 2% or 0.1 min RT of administrative control. Calculated concentration of 5 or greater or 2-5 for discretionary range.
- 4. Did all QCs pass for each analyte? If no, describe issue in comments (below).
- 5. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: Samples were originally extracted 9/7/22 on plate IDP-120-2110125 (re-test date 04/15/22); however, an external urine control was not ran. The samples were re-extracted 9/8/22 and ran on 9/9/22. The re-extracted data was used for analysis.

Due to low internal standard response P2022-2328 and P2022-2786 were not evaluated for topiramate and P2022-2668 and P2022-2786 were not evaluated for 6MAM.

SC

	1	2	3	4	5	6	7	8	9	10	11	12
A	IS + Cal. 1										p2022-2589-1	m2022-3014-2
B	IS + Cal. 1										p2022-2408-1	C2022-1837-1
C											p2022-2328-1	external control urine
D											p2022-2327-1	negative urine
E										p2022-2786-1	m2022-3517-3	external control blood
F										p2022-2668-1	m2022-3493-2	negative blood
G										p2022-2646-1	m2022-3348-3	IS + Cal. 1
H										p2022-2597-1	m2022-3132-3	IS + Cal. 1

SC

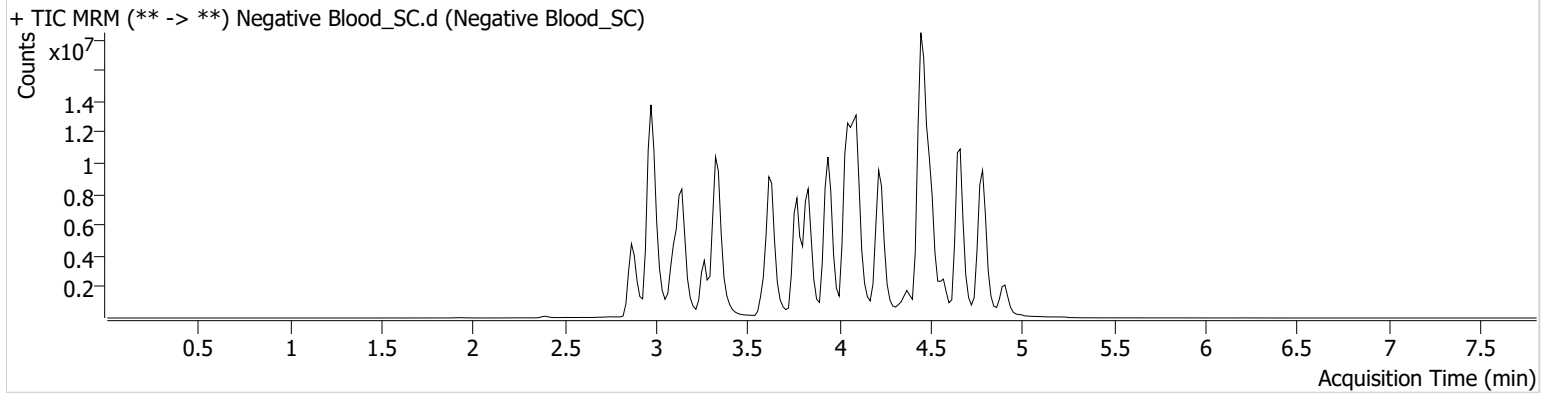


AM #25 Multi-Drug Screen Results

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090922 AM 25 SC reextract\QuantResults\AM 25.batch.bin
Calibration Last Update 9/9/2022 1:26:48 PM

Instrument	Falco (069901)	Data File	Negative Blood_SC.d
Type	Sample	Sample	Negative Blood_SC
Acq. Method	AM 25 MDS.m	Operator	Sarah Collins
Sample Position	P6-F12	Comment	
Injection Volume	5		
Acq. Date-Time	9/9/2022 9:28:59 AM		
Sample Info.			

Sample Chromatogram



SC

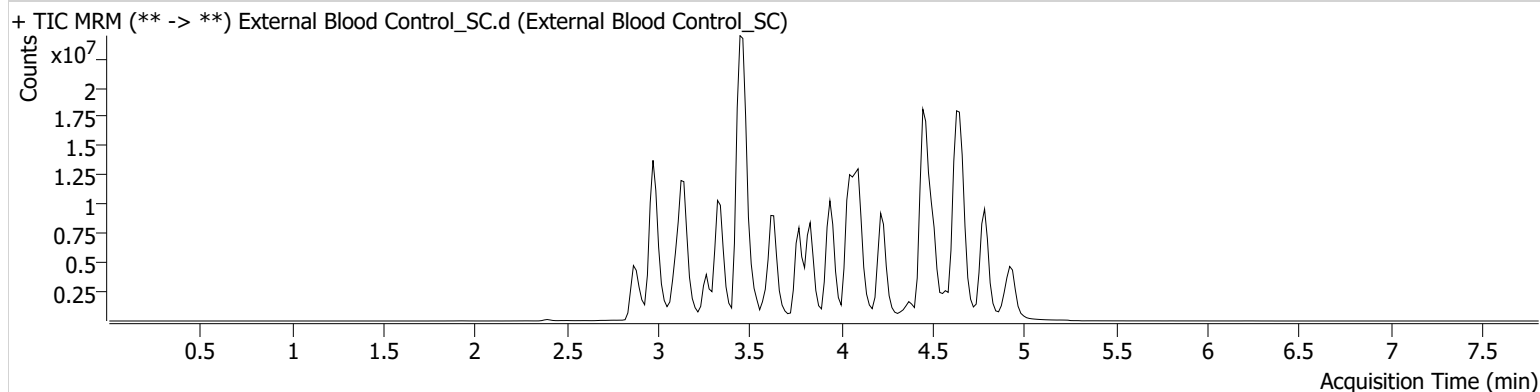
AM #25 Multi-Drug Screen Results



Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090922 AM 25 SC reextract\QuantResults\AM 25.batch.bin
Calibration Last Update 9/9/2022 1:26:48 PM

Instrument	Falco (069901)	Data File	External Blood Control_SC.d
Type	Sample	Sample	External Blood Control_SC
Acq. Method	AM 25 MDS.m	Operator	Sarah Collins
Sample Position	P6-E12	Comment	
Injection Volume	5		
Acq. Date-Time	9/9/2022 9:37:25 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
Alprazolam	4.636	22870904	760.21	4093.77	23902744	67.6336
Buprenorphine	4.933	9824420	154693.86	807709.17	5814438	66.0368
Hydrocodone	3.128	10640517	3324.58	135223.83	13068983	50.3803
Tramadol	3.453	96389328	∞	514.30	47328649	36.4397

SC



Idaho State Police Forensic Services

AM #25 Blood Multi-Drug Screen by LCMS-QQQ And AM #28 Blood Multi-Drug Confirmatory Analysis by LCMS-QQQ---Panel 1

Methanol External Control Solution (Lot: 042222)

100 μ L of 1mg/mL stock was added to each drug to 9700 μ L of LC MeOH.

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>	<i>Expiration Date</i>
Methanol (LCMS)	Fisher	215245	N/A
Tramadol	Cerilliant	FE10051901	12/31/2024
Hydrocodone	Cerilliant	FE04241902	09/30/2024
Alprazolam	Cerilliant	FE06102008	06/30/2025
Buprenorphine	Cerilliant	FE03191903	06/31/2024
Prepared:	04/22/2022		
Expires:	04/22/2023		
Prepared By:	Celena Shrum		

Blood External Control Solution (Lot: WS042222)

200 μ L of methanol external control solution was added to 9800 μ L of blood.

Approximately 200 ng/mL of each compound.

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Negative Blood	Lampire	22B52016-2
Methanol External Control Solution		042222
Prepared:	04/22/2022	
Expires:	04/22/2023	
Prepared by:	Celena Shrum	

SC

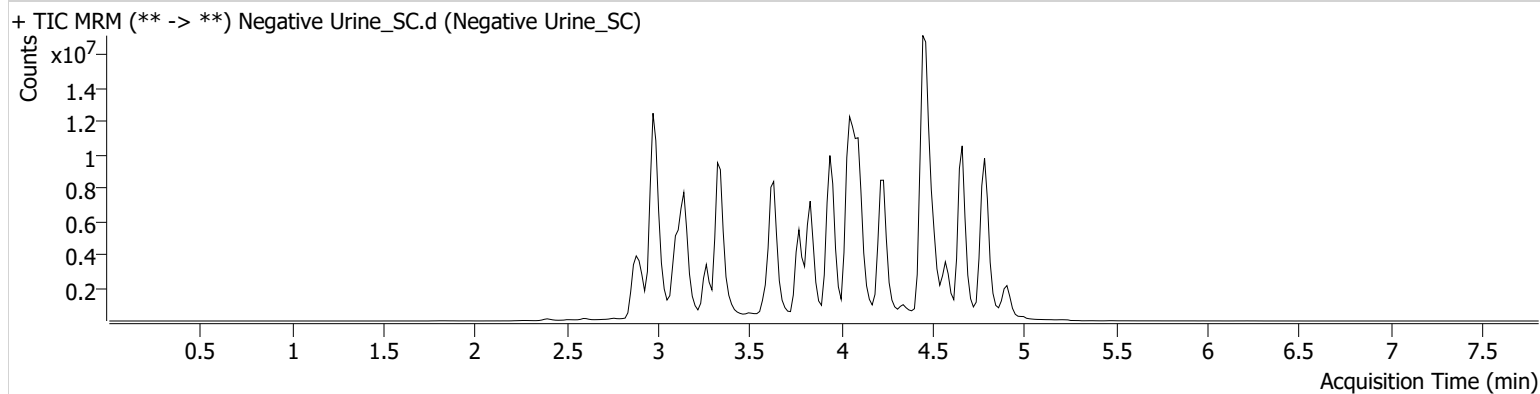
AM #25 Multi-Drug Screen Results



Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090922 AM 25 SC reextract\QuantResults\AM 25.batch.bin
Calibration Last Update 9/9/2022 1:26:48 PM

Instrument	Falco (069901)	Data File	Negative Urine_SC.d
Type	Sample	Sample	Negative Urine_SC
Acq. Method	AM 25 MDS.m	Operator	Sarah Collins
Sample Position	P6-D12	Comment	
Injection Volume	5		
Acq. Date-Time	9/9/2022 9:45:52 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
Amphetamine	2.908	1779432	2964.88	168.67	10305626	4.0431 <32

SC

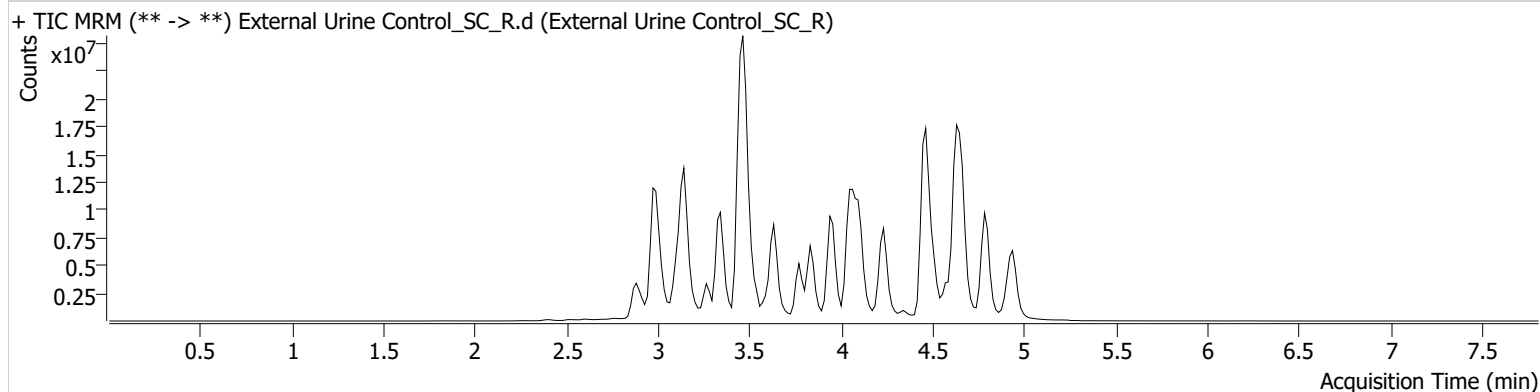
AM #25 Multi-Drug Screen Results



Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090922 AM 25 SC reextract\QuantResults\AM 25.batch.bin
Calibration Last Update 9/9/2022 1:26:48 PM

Instrument	Falco (069901)	Data File	External Urine Control_SC_R.d
Type	Sample	Sample	External Urine Control_SC_R
Acq. Method	AM 25 MDS.m	Operator	Sarah Collins
Sample Position	P6-C12	Comment	
Injection Volume	5		
Acq. Date-Time	9/9/2022 10:53:19 AM		Not a reinject; wrong sample position in initial injection
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
Alprazolam	4.636	24053438	1184.16	9205.96	16096089	105.6291
Buprenorphine	4.933	15441886	11037.75	979105.57	4898240	123.2104
Hydrocodone	3.143	16012393	61599.41	431.85	9495165	104.3503
Tramadol	3.469	99262187	∞	343.05	44631836	39.7932

SC



Idaho State Police Forensic Services

AM #25 Urine Multi-Drug Screen by LCMS-QQQ And AM #28 Urine Multi-Drug Confirmatory Analysis by LCMS-QQQ---Panel 1

Methanol External Control Solution (Lot: 042222)

100 μ L of 1mg/mL stock was added to each drug to 9700 μ L of LC MeOH.

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>	<i>Expiration Date</i>
Methanol (LCMS)	Fisher	215245	N/A
Tramadol	Cerilliant	FE10051901	N/A- Qualitative
Hydrocodone	Cerilliant	FE04241902	N/A- Qualitative
Alprazolam	Cerilliant	FE06102008	N/A- Qualitative
Buprenorphine	Cerilliant	FE03191903	N/A- Qualitative
Prepared:	04/22/2022		
Prepared By:	Celena Shrum		

Urine External Control Solution (Lot: WS042222)

*200 μ L of methanol external control solution was added to 9800 μ L of blood.
Approximately 200 ng/mL of each compound.*

<i>Component</i>	<i>Source</i>	<i>Source Lot Number</i>
Negative Urine	Pocatello Lab	POC021022
Methanol External Control Solution		042222
Prepared:	04/22/2022	
Prepared by:	Celena Shrum	

SC

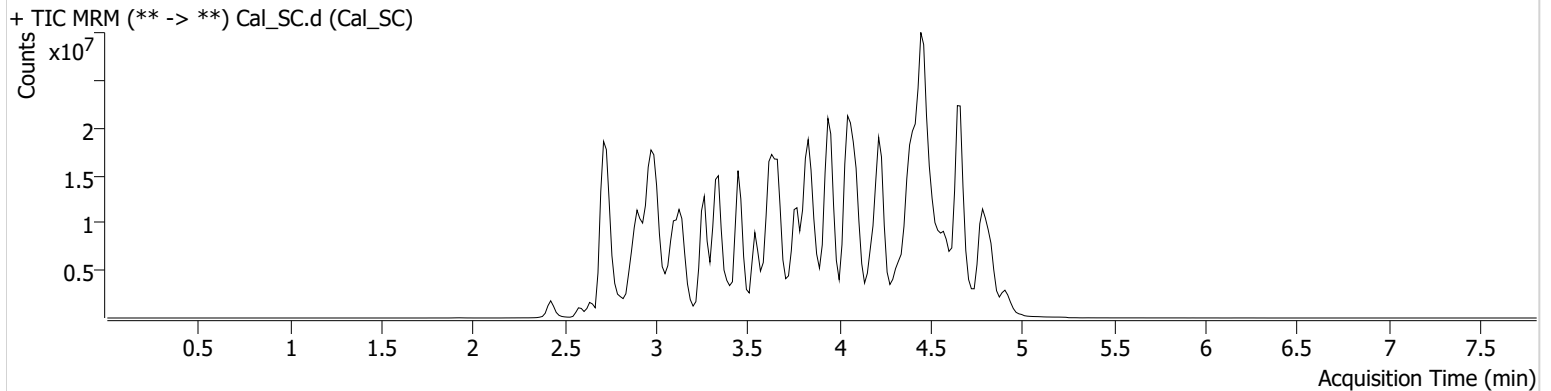


AM #25 Multi-Drug Screen Results

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090922 AM 25 SC reextract\QuantResults\AM 25.batch.bin
Calibration Last Update 9/9/2022 1:26:48 PM

Instrument	Falco (069901)	Data File	Cal_SC.d
Type	Cal	Sample	Cal_SC
Acq. Method	AM 25 MDS.m	Operator	Sarah Collins
Sample Position	P6-H12	Comment	
Injection Volume	5		
Acq. Date-Time	9/9/2022 9:20:24 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
10-OH-Carbamazepine	3.778	4457534	287.93	890.24	23523848	10.0000
6-MAM	3.018	75017	27912.07	31195.83	1617136	10.0000
7-aminoclonazepam	3.605	1383791	390499.36	223.56	5509364	10.0000
7-aminoflunitrazepam	3.805	3193309	525.13	483.34	5509364	10.0000
9-Hydroxyrisperidone	3.951	11841095	4713790.09	556575.81	35653705	10.0000
Acetyl Fentanyl	4.017	621992	703316.23	354765.15	36216736	10.0000
Acetyl Norfentanyl	2.919	601706	3373.07	601.26	36216736	10.0000
a-hydroxyalprazolam	4.525	311044	90.43	2770.15	5509364	10.0000
alpha-hydroxymidazolam	4.600	2688760	216.18	663.52	5509364	10.0000
Alpha-PHP	3.902	4837005	10811.92	4892.77	36216736	10.0000
alpha-PVP	3.626	6145766	1003.44	1440.95	15636180	10.0000
Alprazolam	4.620	2975782	3094.55	265.23	21034281	10.0000
Amitriptyline	4.470	2627938	133.55	171.61	7582455	10.0000
Amphetamine	2.908	6677570	922.14	2505.29	15636180	10.0000
Benzoyllecgonine	3.390	317505	2037.39	488.55	521493	10.0000
Brompheniramine	4.064	153928	1164.08	464.29	47801247	10.0000
Buprenorphine	4.933	1636150	908253.97	237750.67	6394553	10.0000
Bupropion	3.871	7758672	717.30	744.28	24259386	10.0000
Carbamazepine	4.242	12138043	1202.07	1059.14	498119	10.0000
Carisoprodol	4.225	1708857	192887.72	150.87	6817271	10.0000
Chlordiazepoxide	4.729	1247094	118.47	561.71	21034281	10.0000
Chlorpheniramine	3.960	10628201	6253.55	4944.52	47801247	10.0000
Chlorpromazine	4.665	3390658	854.81	531.26	13751648	10.0000
Citalopram	4.079	4802564	583.87	1779519.08	47801247	10.0000
Clomipramine	4.665	4776578	97878.17	1091243.37	47801247	10.0000
Clonazepam	4.434	1187023	2449.69	545.70	21034281	10.0000
Clonazolam	4.369	1847861	3434.94	405210.84	21034281	10.0000
Clozapine	4.463	5950217	1355.41	401.88	18967646	10.0000
Cocaehtylene	3.833	7301727	7136200.37	2005801.43	28814865	10.0000
Cocaine	3.635	6537774	1915775.66	2779.12	28814865	10.0000
Codeine	2.945	547786	490996.61	1412.72	12957661	10.0000
Cyclobenzaprine	4.378	4267392	1831.61	94.77	7582455	10.0000
Desipramine	4.379	9536773	1526.93	794.93	7582455	10.0000
Dextromethorphan	4.101	3001207	373.37	1730.91	15729672	10.0000

Cal_SC

AM #25 Multi-Drug Screen Results

SC



Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
Dextrophan	3.408	3686133	1741.70	508.72	15729672	10.0000
Diazepam	4.853	1884043	361.54	444.61	21034281	10.0000
Dihydrocodeine	2.822	1478776	392.50	312.23	12957661	10.0000
Diphenhydramine	4.054	15222096	48865.30	700.79	47801247	10.0000
Doxepin	4.176	3366999	251.33	66.14	33877760	10.0000
Doxylamine	3.668	17353508	79822.32	306.21	15729672	10.0000
Duloxetine	4.345	96616	22014.47	35489.96	1734507	10.0000
EDDP	4.099	331269	108.94	41.52	863916	10.0000
Estazolam	4.545	8005857	4617.16	1624.90	21034281	10.0000
Etizolam	4.646	349779	202800.74	720342.47	21034281	10.0000
Fentanyl	4.232	463164	103.75	106899.67	25032457	10.0000
Flualprazolam	4.494	1105701	765071.01	1042000.68	21034281	10.0000
Flunitrazepam	4.558	2510271	469.31	3560.42	21034281	10.0000
Fluoxetine	4.328	4543280	1619.69	116.81	7135769	10.0000
Flurazepam	4.306	4720508	1234.90	1369673.97	21034281	10.0000
Hydrocodone	3.128	2094047	736.82	407.07	12957661	10.0000
Hydromorphone	2.581	1568833	5253.90	6147151.57	274013	10.0000
Hydroxyzine	4.583	5189302	1044.05	1716.28	47801247	10.0000
Imipramine	4.423	9044806	710.62	711.56	7582455	10.0000
Ketamine	3.733	5686801	8102330.33	53.90	13889870	10.0000
Lamotrigine	3.639	339085	1040.50	24467.17	47801247	10.0000
Levamisole	3.073	4071471	35096.23	1096.83	28814865	10.0000
Levetiracetam	2.647	1522493	934.96	612.38	47801247	10.0000
Lorazepam	4.434	554118	253.12	24.32	21034281	10.0000
Maprotiline	4.470	1317796	97.58	2340.46	7582455	10.0000
MDA	3.028	3413894	2567.19	590.47	31896652	10.0000
MDEA	3.257	5559820	2818.56	2669.98	31896652	10.0000
MDMA	3.104	7865999	3854716.27	1144.19	31896652	10.0000
Meperidine	3.655	3541691	398.31	4422.19	15729672	10.0000
Meprobamate	3.673	913347	31269.23	80.68	6817271	10.0000
Methadone	4.404	9225868	563.88	531.55	863916	10.0000
Methamphetamine	3.014	9824807	3874.86	530.88	31896652	10.0000
Methocarbamol	3.578	445861	192.27	198903.66	863916	10.0000
Methylphenidate	3.548	19243510	479.10	309.35	23240034	10.0000
Metoprolol	3.453	1175057	433828.39	506.56	15729672	10.0000
Midazolam	4.786	931191	509.48	103244.46	21034281	10.0000
Mirtazapine	4.209	4910294	6595.66	2906.15	15729672	10.0000
Mitragynine	4.306	792236	399200.18	1010831.01	15729672	10.0000
Morphine	2.429	363572	1019.93	308.12	274013	10.0000
Norbuprenorphine	3.859	115671	60698.18	42450.23	6394553	10.0000
Nordiazepam	4.701	2031581	1038.16	351.60	21034281	10.0000
Norfentanyl	3.348	10195338	7744.07	191.93	36216736	10.0000
Norhydrocodone	2.947	156638	325.07	76.79	274013	10.0000
Norketamine	3.857	1042287	401.48	7146.26	13889870	10.0000
Normeperidine	3.610	2356704	205.95	9443.90	47801247	10.0000
Noroxycodone	2.914	2413118	∞	264.04	13889870	10.0000
Nortriptyline	4.425	2285563	1019.60	279.69	7582455	10.0000
O-desmethyl-tramadol	2.933	13226223	13759.07	297.70	47801247	10.0000
O-desmethylvenlafaxine	3.268	2439658	158.99	38.49	11185906	10.0000
Olanzapine	3.926	2056769	641.66	1026.66	498119	10.0000
Oxazepam	4.515	2796877	747.20	420.22	16502253	10.0000
Oxycodone	2.989	3259870	946.51	410.12	13889870	10.0000
Oxymorphone	2.426	3223309	788.39	271.01	274013	10.0000
Paroxetine	4.355	680515	93762.89	303756.17	7135769	10.0000
Phenazepam	4.646	1746172	717432.62	173465.06	21034281	10.0000
Phencyclidine	3.948	8507008	464234.29	1203.63	15729672	10.0000
Phentermine	3.168	2107486	548.98	132.97	23240034	10.0000
Phenytoin	4.133	1016930	819.71	310.12	498119	10.0000
Primidone	3.473	2182255	276334.80	808.27	498119	10.0000
Promethazine	4.392	12537476	690.48	243.06	47801247	10.0000

SC

AM #25 Multi-Drug Screen Results



Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
Pseudoephedrine	2.738	55388920	366.50	907.69	31896652	10.0000
Quetiapine	4.644	6625240	4495687.38	2041.08	39901488	10.0000
Risperidone	4.167	9919001	2916893.15	434.60	35653705	10.0000
Sertraline	4.575	1510315	605332.62	1154.17	7135769	10.0000
Sufentanil	4.644	401245	217650.61	179.70	36216736	10.0000
Tapentadol	3.457	7321347	823.10	1146.85	13889870	10.0000
Temazepam	4.668	5250339	324.01	141.82	21034281	10.0000
Topiramate	3.847	56515	42228.13	18523.84	254182	10.0000
Tramadol	3.453	26715845	∞	261.00	47801247	10.0000
Trazodone	4.813	10159651	830.48	1532.79	33877760	10.0000
Venlafaxine	3.821	10562961	27309.29	251.20	7135769	10.0000
Zaleplon	4.360	3107909	645.40	270.04	39901488	10.0000
Zolpidem	4.467	11715890	5330668.69	5253.64	39901488	10.0000
Zopiclone	4.383	881942	762770.36	1182.12	3770471	10.0000

SC

AM# 26: Screening of THC and Metabolites in Blood and Urine by LC-MS/MS

Extraction Date: 09/07/2022
Plate lot#: IDP-108-3-220309

Analyst: Sarah Collins
Retest Date: 09/09/2022

10mM Ammonium Formate 01/27/2023 SC

0.1% Formic Acid in Methanol 01/27/2023 SC

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

Mobile phase B: ~~0.1% Formic acid in Acetonitrile~~

Blank Blood Lot: Lampire 22B52015-1

Blank Urine Lot: POC021022

Column: Phenomenex Phenyl Hexyl (4.6x50mm, 2.6um)

LCMS-QQQ ID: 069901

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.

Analytic:

- 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
- 2. Urine hydrolysis: add 1.5mL urine to blank plate, add 250µl 1N KOH. Shake and incubate at 40 degrees for 15 minutes.
Using a calibrated pipette, add **1000µl blood and urine (if applicable) (calibrated pipette)** into the appropriate wells of analytical (standards) plate. **Pipette ID:** 3382167
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 4. Pipette **500µL 0.1% formic acid in water blood sample, 500 µL saturated phosphate buffer in urine** in wells of analytical plate.
- 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 6. Transfer **700-800µL of blood+acid or urine+acid** mixture to corresponding wells of SLE+ plate.
Amount transferred: 800 uL
- 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)
- 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.
- 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.
- 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).
- 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? (if not, describe in comments section)
- 6. Enter QCs into control charting.
- 7. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS:

SC

	1	2	3	4	5	6
A	IS + Cal. 1	IS + QC_2 urine	m2022-3517-3	p2022-2786-1		
B	IS + Cal. 2	negative blood	p2022-2327-1			
C	IS + Cal. 3	negative urine	p2022-2328-1			
D	IS + Cal. 4	c2022-1837-1	p2022-2408-1			
E	IS + Cal. 5	m2022-3014-2	p2022-2589-1			
F	IS + Cal. 6	m2022-3132-3	p2022-2597-1			
G	IS + Cal. 7	m2022-3348-3	p2022-2646-1			
H	IS + QC_1 blood	m2022-3493-2	p2022-2668-1			

All wells to contain 100 µl of residual DMSO

SC

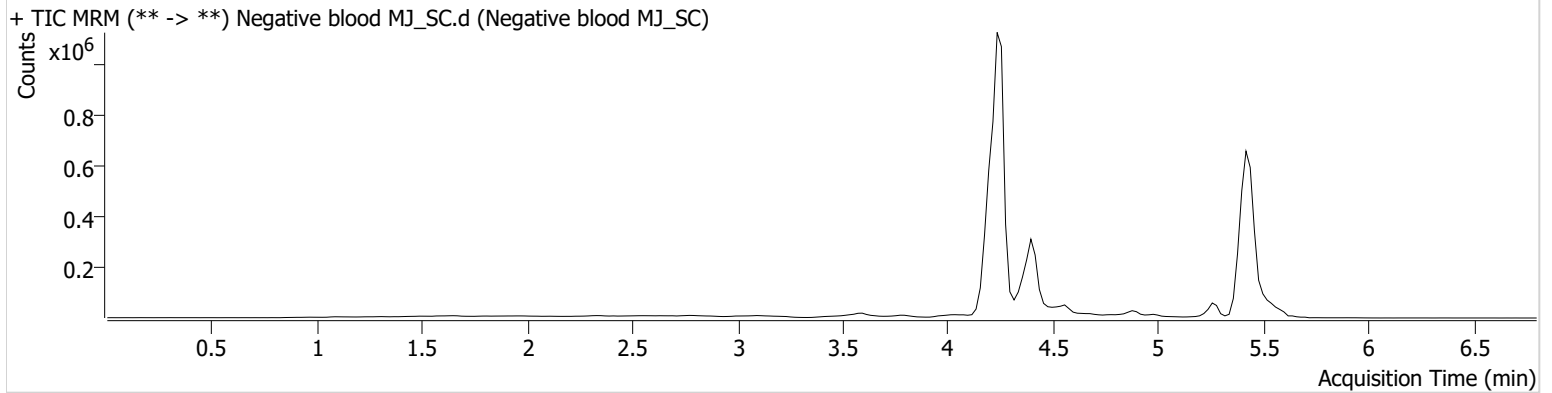


AM #26 Cannabinoids Screen Results

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Negative blood MJ_SC.d
Type	Sample	Sample	Negative blood MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-B2	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 4:14:46 PM		
Sample Info.			

Sample Chromatogram



SC

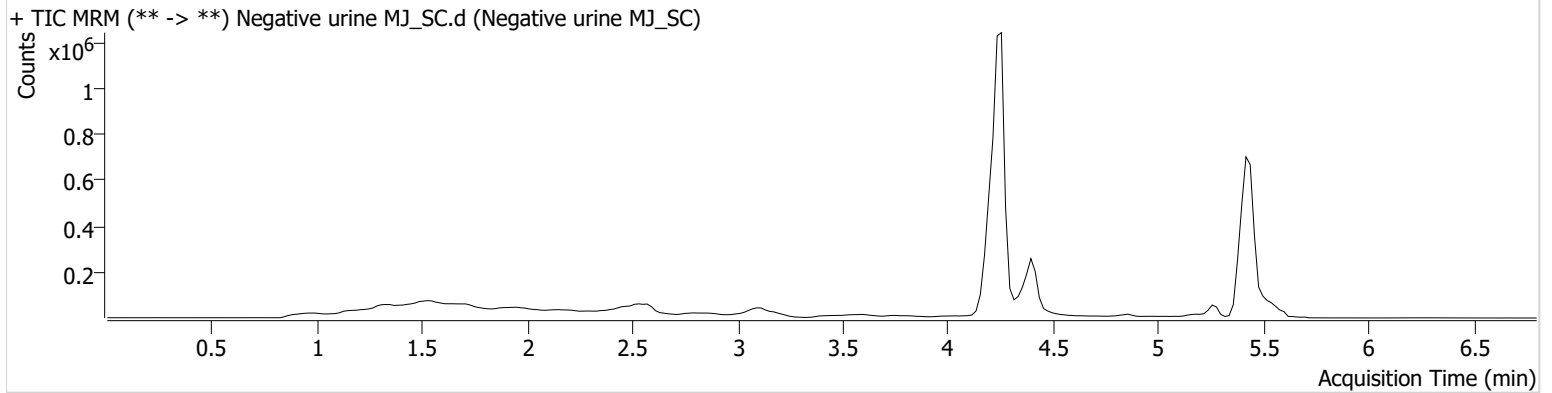


AM #26 Cannabinoids Screen Results

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Negative urine MJ_SC.d
Type	Sample	Sample	Negative urine MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-C2	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 4:29:56 PM		
Sample Info.			

Sample Chromatogram



SC

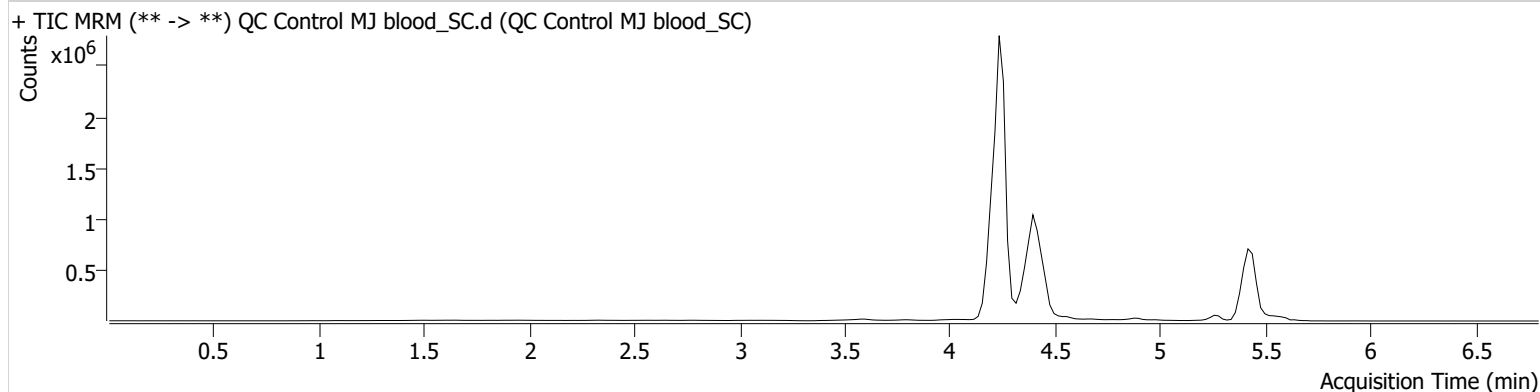
AM #26 Cannabinoids Screen Results



Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	QC Control MJ blood_SC.d
Type	QC	Sample	QC Control MJ blood_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-H1	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 3:59:36 PM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	5.449	9080	237014	4.9201 ng/ml
THC-COOH	4.436	671197	3662601	15.0117 ng/ml
THC-OH	4.262	80396	11357548	4.2541 ng/ml

SC

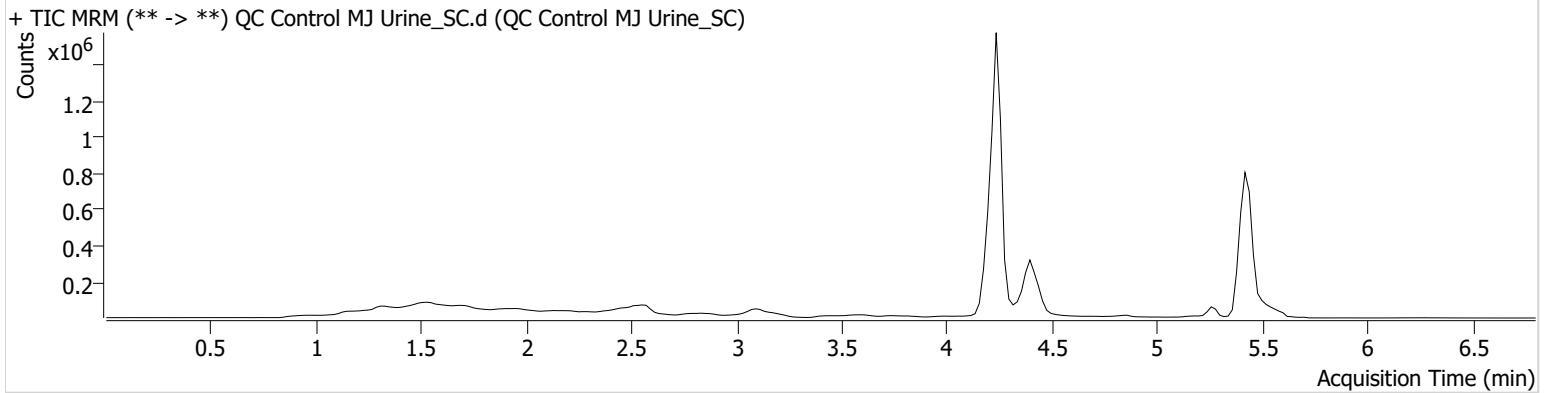


AM #26 Cannabinoids Screen Results

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	QC Control MJ Urine_SC.d
Type	QC	Sample	QC Control MJ Urine_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-A2	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 6:31:02 PM		

Sample Chromatogram



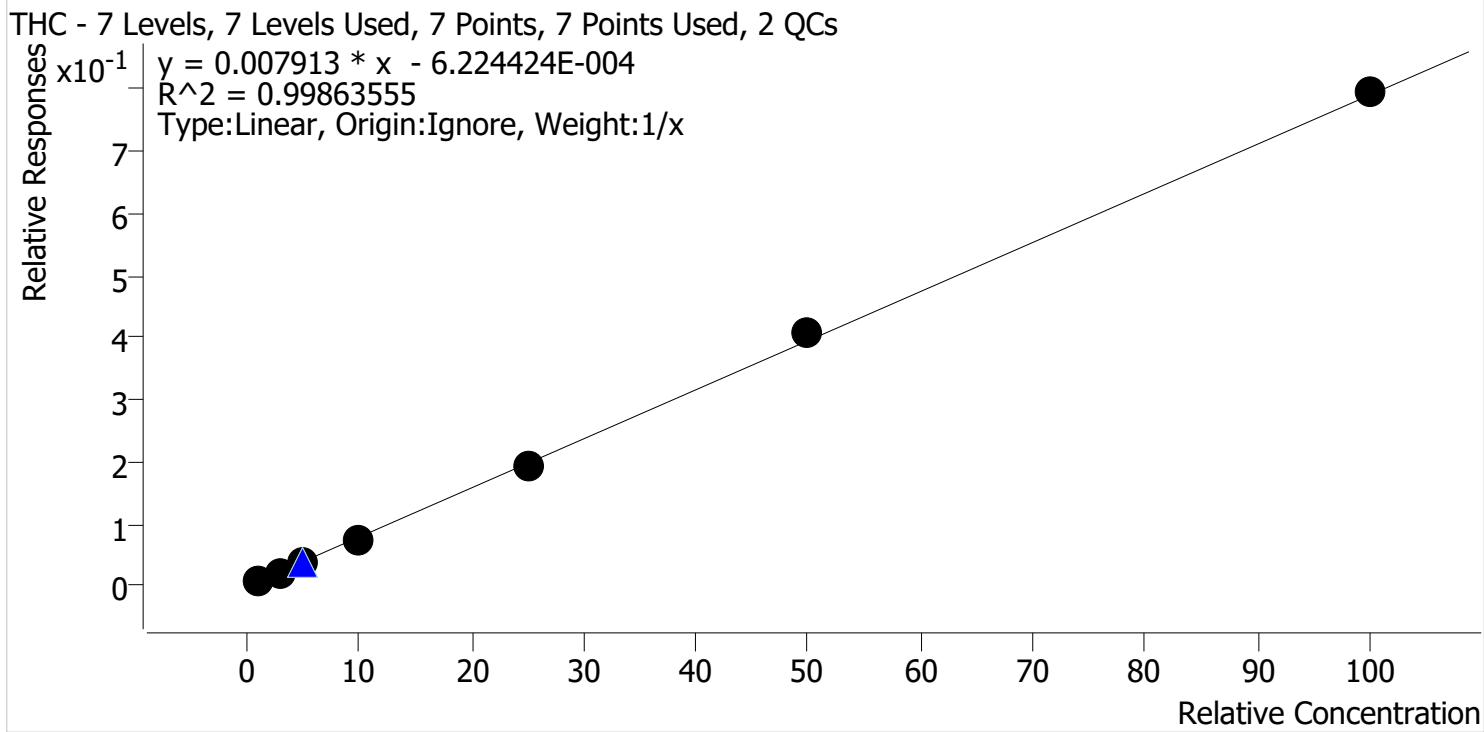
Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	5.409	3293	85779	4.9300 ng/ml
THC-COOH	4.436	173744	900347	15.8463 ng/ml
THC-OH	4.242	40869	5758293	4.2651 ng/ml

SC



AM #26 Cannabinoids Screen Calibration Curve Report

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Last Cal. Update 9/8/2022 7:21 AM
Analyst Name ISP\scollins
Analyte THC **Internal Standard** THC-D3



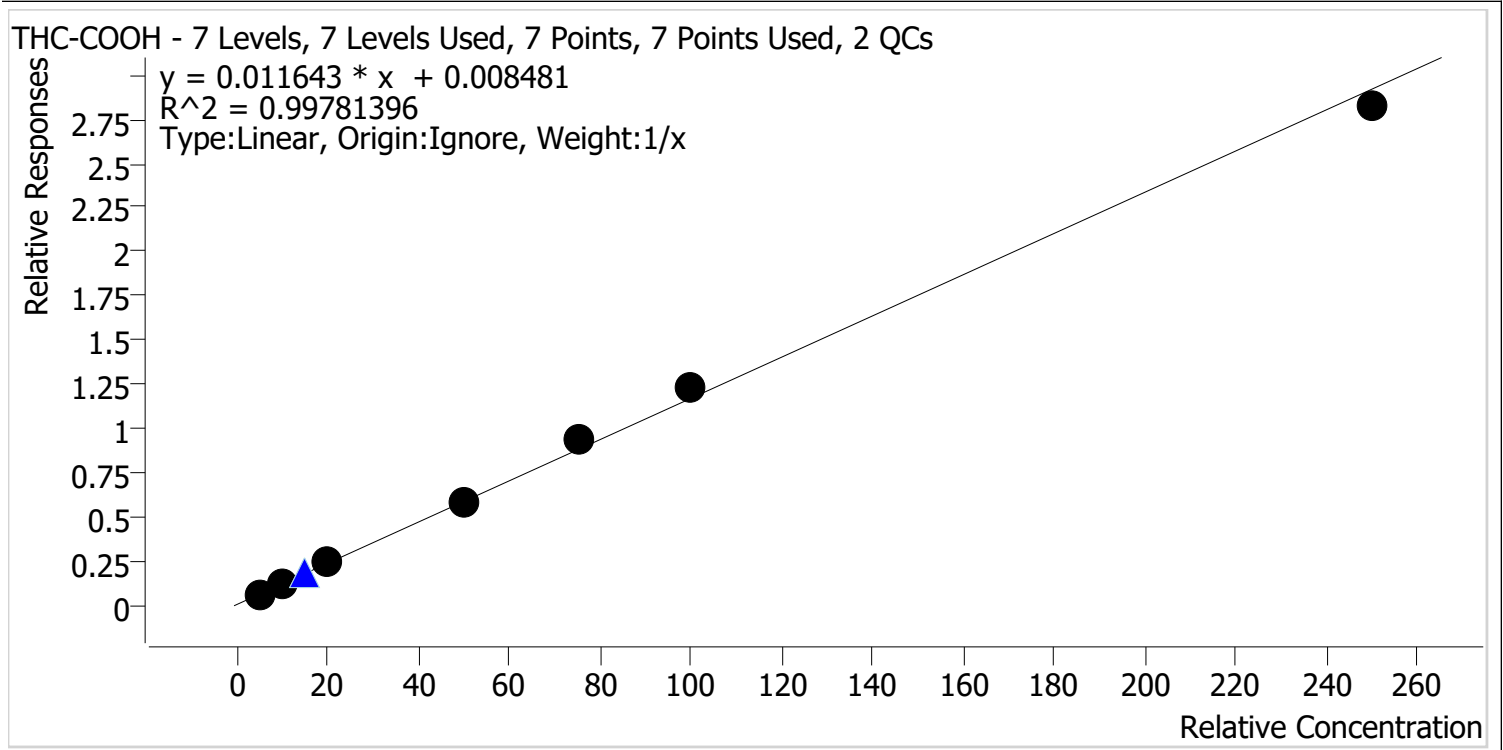
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ_SC	1	✓	1.0	1.2	117.7
Cal 2 MJ_SC	2	✓	3.0	2.7	90.7
Cal 3 MJ_SC	3	✓	5.0	5.1	101.9
Cal 4 MJ_SC	4	✓	10.0	9.0	89.9
Cal 5 MJ_SC	5	✓	25.0	24.2	96.7
Cal 6 MJ_SC	6	✓	50.0	51.3	102.6
Cal 7 MJ_SC	7	✓	100.0	100.5	100.5

SC



AM #26 Cannabinoids Screen Calibration Curve Report

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Last Cal. Update 9/8/2022 7:21 AM
Analyst Name ISP\scollins
Analyte THC-COOH **Internal Standard** THC-COOH-D9



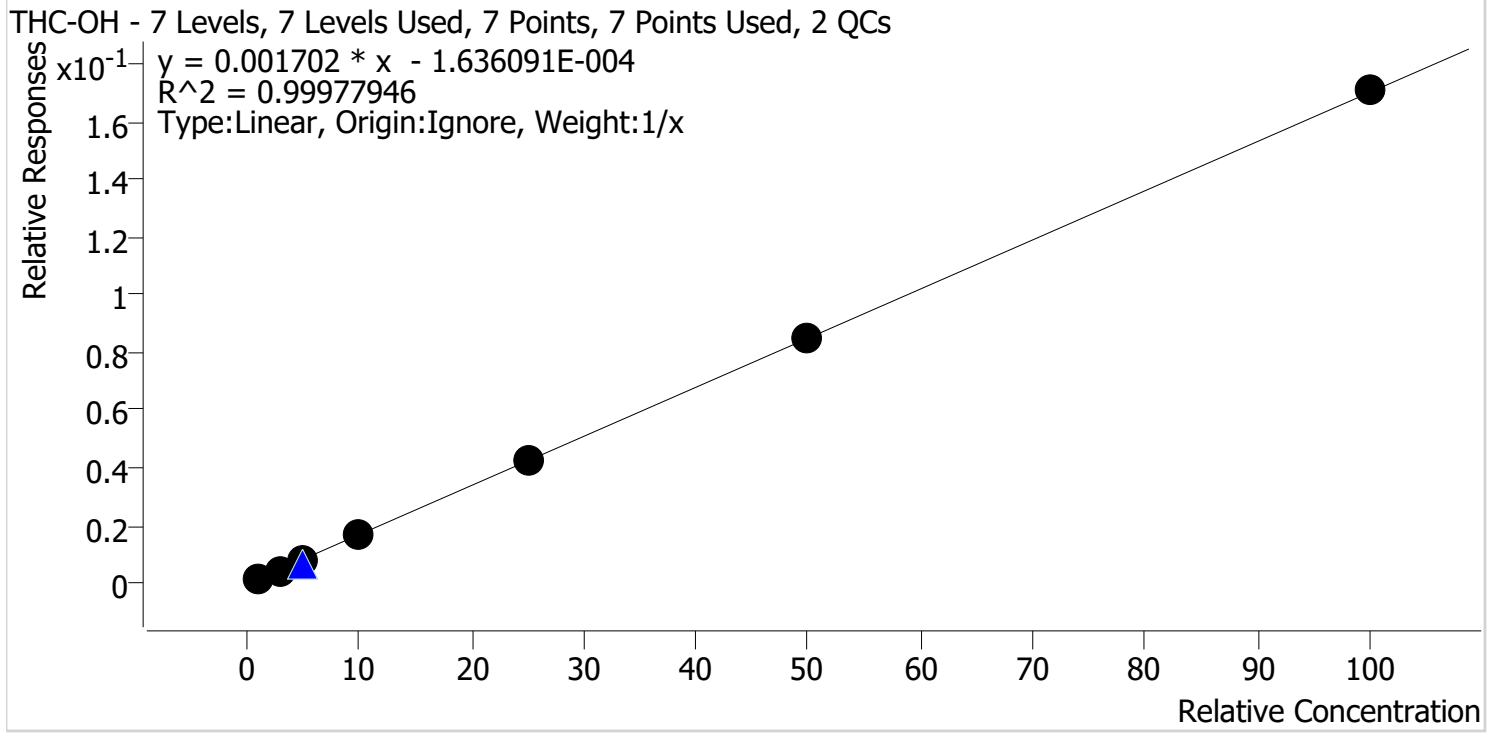
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ_SC	1	✓	5.0	4.8	96.1
Cal 2 MJ_SC	2	✓	10.0	9.7	97.5
Cal 3 MJ_SC	3	✓	20.0	20.1	100.4
Cal 4 MJ_SC	4	✓	50.0	49.1	98.2
Cal 5 MJ_SC	5	✓	75.0	79.7	106.3
Cal 6 MJ_SC	6	✓	100.0	104.9	104.9
Cal 7 MJ_SC	7	✓	250.0	241.6	96.7

SC



AM #26 Cannabinoids Screen Calibration Curve Report

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Last Cal. Update 9/8/2022 7:21 AM
Analyst Name ISP\scollins
Analyte THC-OH **Internal Standard** THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ_SC	1	✓	1.0	1.1	110.6
Cal 2 MJ_SC	2	✓	3.0	2.9	95.3
Cal 3 MJ_SC	3	✓	5.0	4.8	96.4
Cal 4 MJ_SC	4	✓	10.0	9.7	96.8
Cal 5 MJ_SC	5	✓	25.0	25.1	100.4
Cal 6 MJ_SC	6	✓	50.0	50.0	100.1
Cal 7 MJ_SC	7	✓	100.0	100.4	100.4

SC

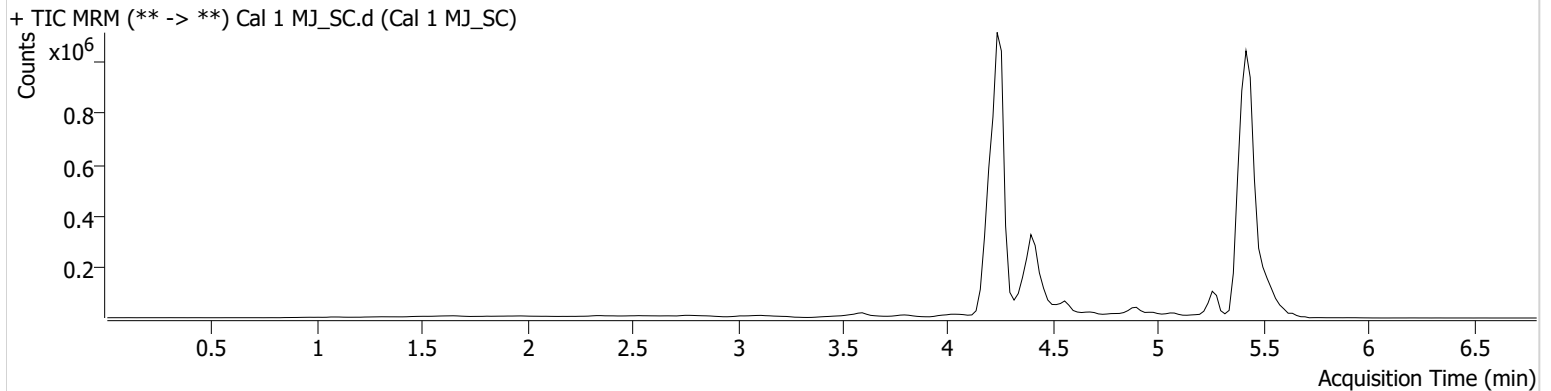


AM #26 Cannabinoids Screen Results

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Cal 1 MJ_SC.d
Type	Cal	Sample	Cal 1 MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-A1	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 3:06:29 PM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.	
THC	5.469	4164	478875	1.1774 ng/ml	Low
THC-COOH	4.436	84191	1307085	4.8039 ng/ml	Low
THC-OH	4.262	9121	5303901	1.1062 ng/ml	Low

SC

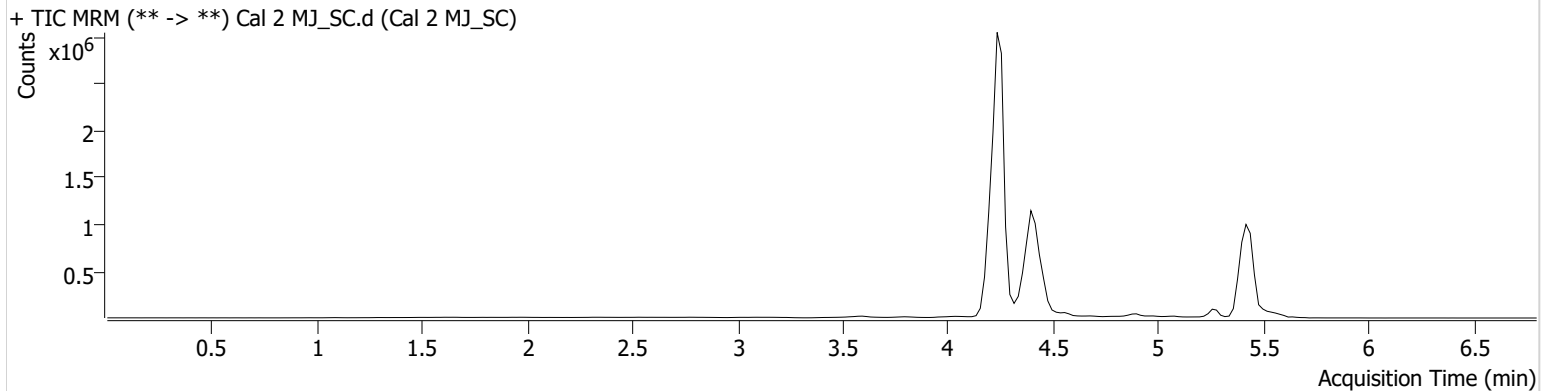


AM #26 Cannabinoids Screen Results

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Cal 2 MJ_SC.d
Type	Cal	Sample	Cal 2 MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-B1	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 3:14:13 PM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.	
THC	5.449	11146	533354	2.7196 ng/ml	Low
THC-COOH	4.436	501933	4115709	9.7464 ng/ml	
THC-OH	4.262	56794	12070747	2.8598 ng/ml	Low

SC

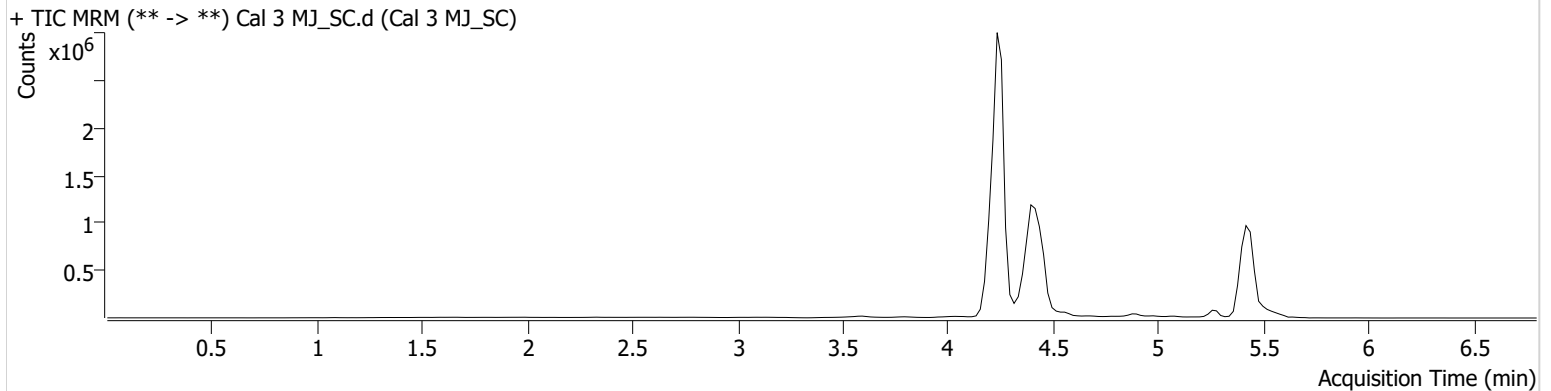


AM #26 Cannabinoids Screen Results

Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Cal 3 MJ_SC.d
Type	Cal	Sample	Cal 3 MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-C1	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 3:21:47 PM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	5.449	17532	441744	5.0942 ng/ml
THC-COOH	4.436	938436	3873524	20.0803 ng/ml
THC-OH	4.262	90239	11225397	4.8181 ng/ml

SC

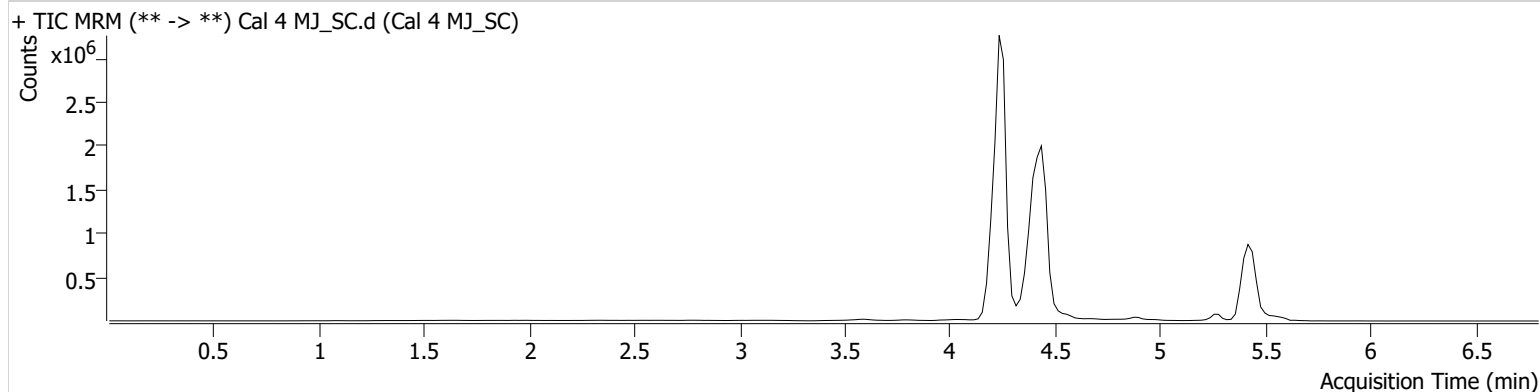
AM #26 Cannabinoids Screen Results



Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Cal 4 MJ_SC.d
Type	Cal	Sample	Cal 4 MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-D1	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 3:29:21 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	5.449	39184	555965	8.9855 ng/ml
THC-COOH	4.436	2511931	4329777	49.1013 ng/ml
THC-OH	4.262	189391	11603447	9.6835 ng/ml

SC

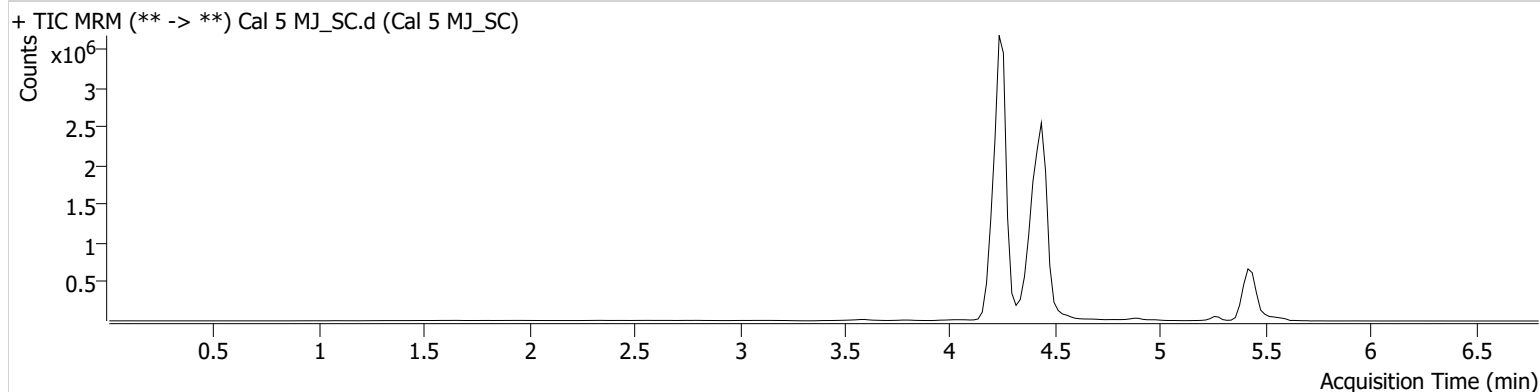
AM #26 Cannabinoids Screen Results



Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Cal 5 MJ_SC.d
Type	Cal	Sample	Cal 5 MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-E1	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 3:36:55 PM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	5.449	63422	332518	24.1825 ng/ml
THC-COOH	4.436	3528581	3767172	79.7226 ng/ml
THC-OH	4.262	476773	11203092	25.0940 ng/ml

SC

AM #26 Cannabinoids Screen Results

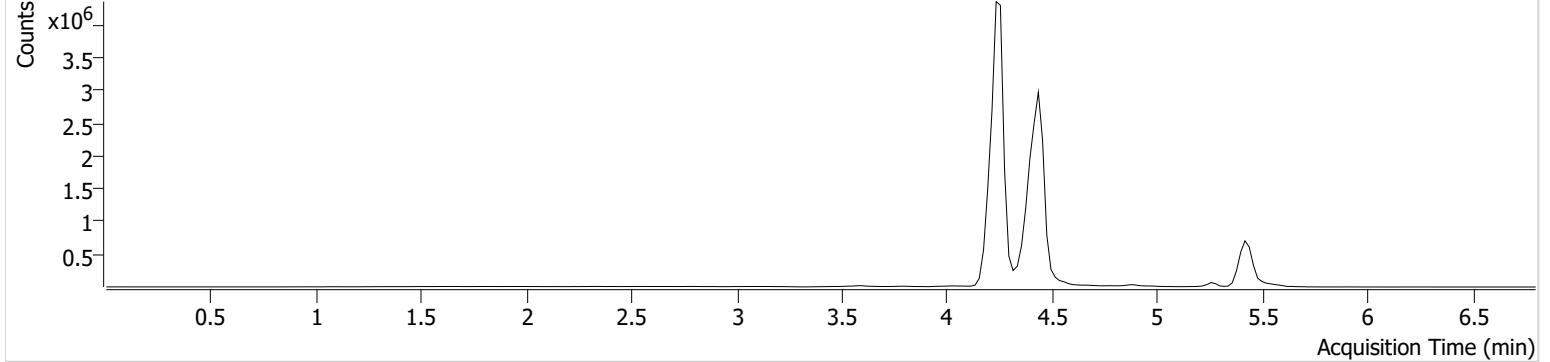


Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Cal 6 MJ_SC.d
Type	Cal	Sample	Cal 6 MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-F1	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 3:44:29 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) Cal 6 MJ_SC.d (Cal 6 MJ_SC)



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	5.449	111823	275903	51.2980 ng/ml
THC-COOH	4.436	4282476	3482279	104.8994 ng/ml
THC-OH	4.262	907878	10677835	50.0390 ng/ml

SC

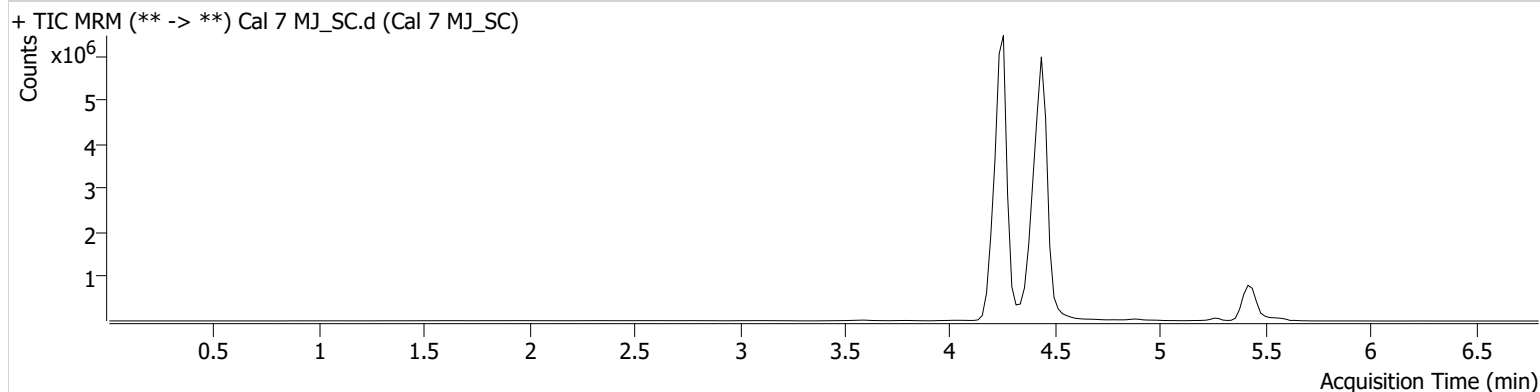
AM #26 Cannabinoids Screen Results



Batch results G:\TOX\Pocatello\Falco\2022\AM 25-26\090722 AM 25 26 CS SC\QuantResults\AM 26 SC.batch.bin
Calibration Last Update 9/8/2022 7:21:43 AM

Instrument	Falco (069901)	Data File	Cal 7 MJ_SC.d
Type	Cal	Sample	Cal 7 MJ_SC
Acq. Method	AM 26 THC.m	Operator	Sarah Collins
Sample Position	P5-G1	Comment	
Injection Volume	10		
Acq. Date-Time	9/7/2022 3:52:03 PM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	5.449	160961	202474	100.5429 ng/ml
THC-COOH	4.436	9123938	3233270	241.6462 ng/ml
THC-OH	4.262	1822812	10674693	100.3995 ng/ml