REVIEWED
By Anne Nord at 1:32 pm, Oct 13, 2022

TS

9/13/2022

Worklist: 6102

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2022-3438	3	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3493	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3534	4	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3535	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3588	3	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3655	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3658	2	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3659	4	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3676	2	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3734	2	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3741	2	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
M2022-3742	3	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2484	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2484	2	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2580	1	BLOOD	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2582	1	BLOOD	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2588	1	BLOOD	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2588	2	BLOOD	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2639	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2758	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
P2022-2761	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ

Worklist: 6102

TS

	LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
	P2022-2762	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
	P2022-2765	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
	P2022-2782	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
	P2022-2788	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
	P2022-2805	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
	P2022-2808	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
	P2022-2814	2	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ
*	P2022-2824	1	BCK	AM 25/AM 26 Blood MultiDrug/THC Screen by LC-QQQ

*Case P2022-2824 only ran on AM 25 due to limited sample volume.

TS

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 Date: 2/10/2022
Title: Laboratory Manager

Quality Review

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

TS

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

Extraction Date: 09/26/2022 Analyst: Tamara Salazar
Plate lot#: 2220315 Plate Retest Date: 09-15-2022-ok with external control
Mobile phase A: 10mM Amm Form Mobile phase B: 0.1% Formic Acid in MeOH
Blank Blood Lot: Lampire 22B52015-1 Blank Urine Lot: N/A
LCMS-QQQ ID: 069901 Column: Phenomenex Phenyl Hexyl (4.6x50mm, 2.6um)

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 or 250µL hydrolyzed urine in wells of analytical (standards) plate.
Pipette ID: Click here to enter text.
☐ 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: *300uL*
☒ 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.
☒ 17. Add 50µL 1% HCl in MeOH to wells and place plate cover on plate before drying. This step is required for urine samples, but optional for blood samples.
☒ 18. 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: Instrument nitrogen generator indicated a potential maintenance required. Analytical plates were frozen until PEAK analytics could be contacted. Plate was ran on 09/27/2022 after the company indicated that the generator would be functional.

TS

	1	2	3	4	5	6	7	8	9	10	11	12
A	IS + Cal. 1	M2022-3588-3	P2022-2484-1	P2022-2761-1	P2022-2824-1	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample
B	IS + Cal. 1	M2022-3655-1	P2022-2484-2	P2022-2762-1	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample
C	Neg Blood	M2022-3658-2	P2022-2580-1	P2022-2765-1	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample
D	Ext Ctrl	M2022-3659-4	P2022-2582-1	P2022-2782-1	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample
E	M2022-3438-3	M2022-3676-2	P2022-2588-1	P2022-2788-1	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample
F	M2022-3493-1	M2022-3734-2	P2022-2588-2	P2022-2805-1	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample
G	M2022-3534-4	M2022-3741-2	P2022-2639-1	P2022-2808-1	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Cal. 1
H	M2022-3535-1	M2022-3742-3	P2022-2758-1	P2022-2814-2	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Sample	IS + Cal. 1

All wells to contain 60 µl of residual DMSO

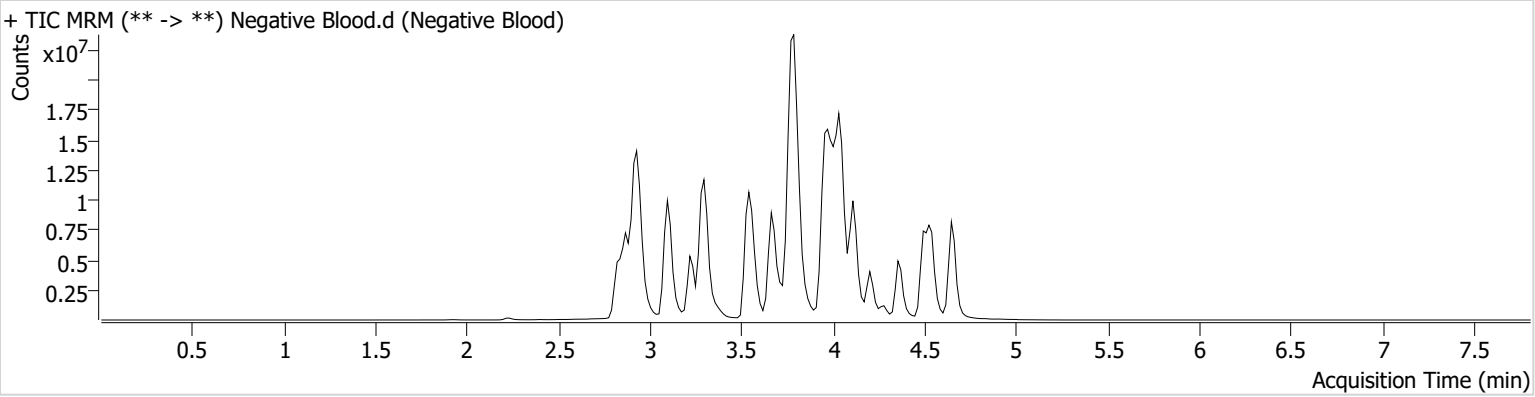
TS

AM #25 Multi-Drug Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 25.batch.bin
Calibration Last Update 10/3/2022 8:08:58 AM

Instrument	Falco (069901)	**Data File**	Negative Blood.d
Type	Sample	**Sample**	Negative Blood
Acq. Method	AM 25 MDS.m	**Operator**	Tamara Salazar
Sample Position	P2-C1	**Comment**	
Injection Volume	5		
Acq. Date-Time	9/27/2022 4:31:22 PM		
Sample Info.			

Sample Chromatogram



TS

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 µL of 1mg/mL stock was added ~~to~~ of 04/03/203 each drug to 9600 µL of LC MeOH. TS

Component	*Source*	*Source Lot Number*	*Expiration Date*
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 µL of methanol external control solution was added to 9800 µL of blood. Approximately 200 ng/mL of each compound.

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

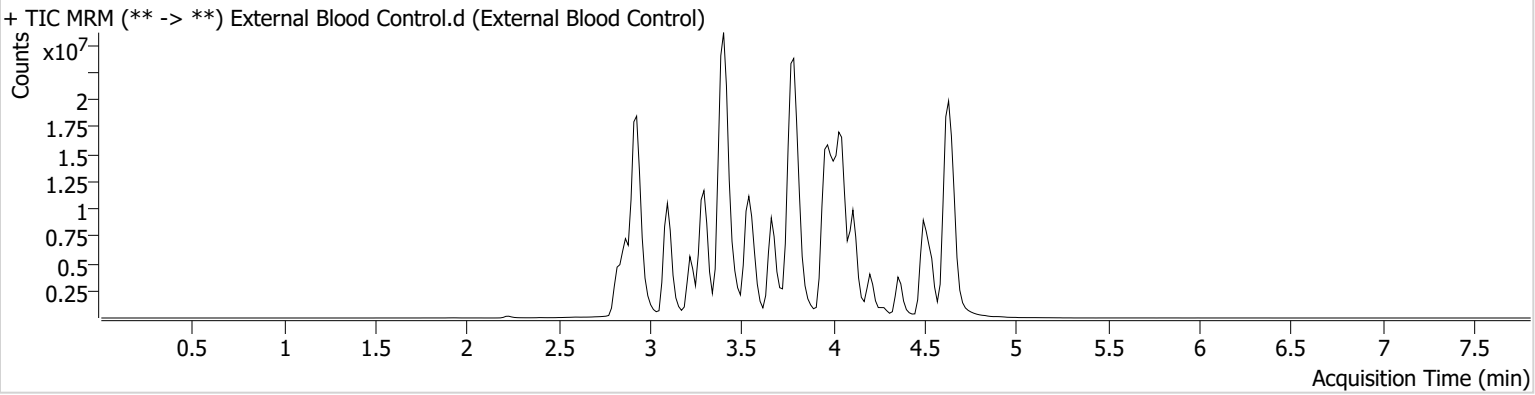
TS

AM #25 Multi-Drug Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 25.batch.bin
Calibration Last Update 10/3/2022 8:08:58 AM

Instrument	Falco (069901)	**Data File**	External Blood Control.d
Type	Sample	**Sample**	External Blood Control
Acq. Method	AM 25 MDS.m	**Operator**	Tamara Salazar
Sample Position	P2-D1	**Comment**	
Injection Volume	5		
Acq. Date-Time	9/27/2022 4:39:47 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
Alprazolam	4.620	27135840	939.98	1295.72	28761276	49.6851
Buprenorphine	4.059	7187960	343984.78	217505.55	3962624	64.7330
Hydrocodone	2.930	10603903	4125.95	384.39	14129159	49.4872
Norbuprenorphine	3.782	25340	26649.61	16473.58	3962624	2.3747
Tramadol	3.407	102564282	∞	463.25	51972121	30.3508

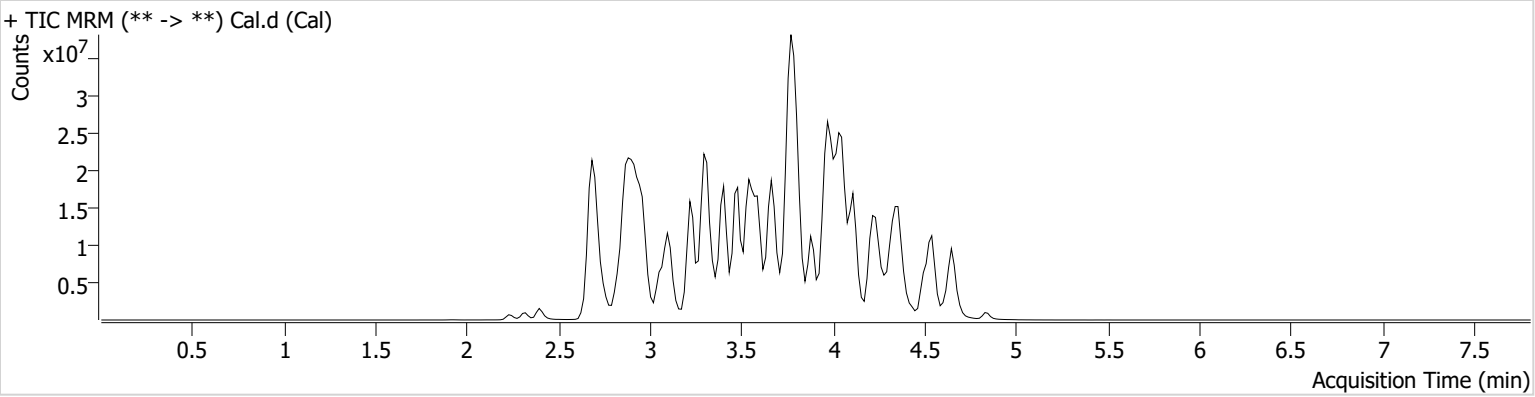
TS

AM #25 Multi-Drug Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 25.batch.bin
Calibration Last Update 10/3/2022 8:08:58 AM

Instrument	Falco (069901)	**Data File**	Cal.d
Type	Cal	**Sample**	Cal
Acq. Method	AM 25 MDS.m	**Operator**	Tamara Salazar
Sample Position	P2-B1	**Comment**	
Injection Volume	5		
Acq. Date-Time	9/27/2022 4:22:45 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
10-OH-Carbamazepine	3.762	4706281	292.44	4526.02	24671801	10.0000
6-MAM	2.850	79949	52877.14	28809.47	1425379	10.0000
7-aminoclonazepam	3.575	1663388	170.41	174.04	6047446	10.0000
7-aminoflunitrazepam	3.790	2648571	1502.09	347.54	6047446	10.0000
9-Hydroxyrisperidone	3.782	11177497	1640.90	360918.09	31718783	10.0000
Acetyl Fentanyl	3.725	543723	230.03	6069.89	40051738	10.0000
Acetyl Norfentanyl	2.888	746275	194.32	167.87	40051738	10.0000
a-hydroxyalprazolam	4.510	114264	4300.23	53630.89	6047446	10.0000
alpha-hydroxymidazolam	4.539	2108208	694.77	753598.78	6047446	10.0000
Alpha-PHP	3.748	6210730	31677.61	495.28	40051738	10.0000
alpha-PVP	3.487	12275078	1480.14	618.43	14568003	10.0000
Alprazolam	4.620	2938694	578.61	439.40	15475517	10.0000
Amitriptyline	4.362	2582750	315.06	336.07	8512467	10.0000
Amphetamine	2.862	7748093	1337.66	545.67	14568003	10.0000
Benzoylecgonine	3.390	383773	1320.56	110.87	603928	10.0000
Brompheniramine	3.987	129347	286.75	876.28	50431635	10.0000
Buprenorphine	4.043	1051376	1401.63	129130.72	3751981	10.0000
Bupropion	3.686	9336837	1139.13	1900.98	27147991	10.0000
Carbamazepine	4.227	17298981	1511.18	1350.04	363925	10.0000
Carisoprodol	4.225	1871977	1713.81	132.74	7532044	10.0000
Chlordiazepoxide	4.652	797893	2731.30	1689.86	15475517	10.0000
Chlorpheniramine	3.899	11576627	970.16	14.33	50431635	10.0000
Chlorpromazine	4.542	2199055	760400.83	2582.55	9343745	10.0000
Citalopram	4.017	5585618	1395.47	647.14	50431635	10.0000
Clomipramine	4.558	3170452	16447.14	10069.57	50431635	10.0000
Clonazepam	4.434	537492	330.63	124.61	15475517	10.0000
Clonazolam	4.369	1254307	642820.26	446680.68	15475517	10.0000
Clozapine	4.109	7821662	3521.65	1857.64	22579369	10.0000
Cocaethylene	3.741	8996299	5441150.56	2509.46	32494310	10.0000
Cocaine	3.542	8330688	3954.42	3120.04	32494310	10.0000
Codeine	2.747	775617	2472.94	4343.52	13703696	10.0000
Cyclobenzaprine	4.285	3641001	1707.90	120.17	8512467	10.0000
Desipramine	4.317	7952132	1603.59	754.42	8512467	10.0000
Dextromethorphan	4.023	3354220	677.65	526.17	17469730	10.0000

TS

AM #25 Multi-Drug Screen Results

Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
Dextrorphan	3.346	4072673	8423889.79	925.73	17469730	10.0000
Diazepam	4.838	1372908	6985.62	9039.53	15475517	10.0000
Dihydrocodeine	2.715	1789969	1271.81	856.30	13703696	10.0000
Diphenhydramine	3.977	15582759	3539.88	1559.83	50431635	10.0000
Doxepin	4.084	3316832	911.19	491.84	24799315	10.0000
Doxylamine	3.591	17753003	1654.47	13829678.65	17469730	10.0000
Duloxetine	4.283	83378	72034.54	12773.06	1276617	10.0000
EDDP	4.052	615753	255.44	108.16	1671176	10.0000
Estazolam	4.529	4900381	761.43	357.57	15475517	10.0000
Etizolam	4.646	485331	524476.52	516741.65	15475517	10.0000
Fentanyl	3.955	341465	467.42	56646.74	20730109	10.0000
Flualprazolam	4.494	934826	462162.57	914514.09	15475517	10.0000
Flunitrazepam	4.558	1194538	819.92	2800317.41	15475517	10.0000
Fluoxetine	4.282	3089898	969.33	195.71	3261865	10.0000
Flurazepam	4.076	4021823	706.44	614.47	15475517	10.0000
Hydrocodone	2.930	2078233	882.98	452.26	13703696	10.0000
Hydromorphone	2.399	2306463	1542.23	18670.30	415982	10.0000
Hydroxyzine	4.384	2904149	1674.03	2089.13	50431635	10.0000
Imipramine	4.331	8646750	2409.37	1169.45	8512467	10.0000
Ketamine	3.317	7799125	2812.97	238.80	17045847	10.0000
Lamotrigine	3.470	580477	460.35	1073.25	50431635	10.0000
Levamisole	2.889	6017790	12521.47	6900.74	32494310	10.0000
Levetiracetam	2.662	2151333	1995.63	881.95	50431635	10.0000
Lorazepam	4.434	346601	204.58	28.30	15475517	10.0000
Maprotiline	4.363	1709648	251.21	407.94	8512467	10.0000
MDA	2.982	3917319	495.16	697.94	36790708	10.0000
MDEA	3.211	6565722	1031.86	811.04	36790708	10.0000
MDMA	3.058	10165737	508892.88	1010.29	36790708	10.0000
Meperidine	3.547	4442325	1215.01	962.86	17469730	10.0000
Meprobamate	3.673	919101	519.20	127.23	7532044	10.0000
Methadone	4.359	9870501	2329.00	785.10	1671176	10.0000
Methamphetamine	2.968	10495684	1027.18	619.06	36790708	10.0000
Methocarbamol	3.578	661572	2114.10	147.88	1671176	10.0000
Methylphenidate	3.487	22338869	1198.69	947.55	30846214	10.0000
Metoprolol	3.422	1332260	510.69	2174.47	17469730	10.0000
Midazolam	4.510	880577	490874.83	290849.54	15475517	10.0000
Mirtazapine	3.653	6032517	5278.24	7694788.53	17469730	10.0000
Mitragynine	4.090	728247	525584.04	1041941.80	17469730	10.0000
Morphine	2.232	475400	306.27	1421.48	415982	10.0000
Norbuprenorphine	3.782	101034	63437.60	267608.33	3751981	10.0000
Nordiazepam	4.686	1282427	661.28	525.91	15475517	10.0000
Norfentanyl	3.318	14276467	882.43	1644.15	40051738	10.0000
Norhydrocodone	2.901	98528	211.42	16032.13	415982	10.0000
Norketamine	3.303	1124964	223.63	6763.33	17045847	10.0000
Normeperidine	3.564	3915858	4747.49	380.17	50431635	10.0000
Noroxycodone	2.868	1897952	∞	357.70	17045847	10.0000
Nortriptyline	4.364	1653838	1052.37	476.52	8512467	10.0000
O-desmethyl-tramadol	2.902	17124237	1635.18	1172.52	50431635	10.0000
O-desmethylvenlafaxine	3.223	3052547	532.87	789004.20	14719726	10.0000
Olanzapine	3.388	3051577	2255972.54	675.43	363925	10.0000
Oxazepam	4.515	1095739	241.19	100.56	6462189	10.0000
Oxycodone	2.882	4762429	1309.34	6390.54	17045847	10.0000
Oxymorphone	2.320	1936542	36.99	219.31	415982	10.0000
Paroxetine	4.294	357686	532.65	10486.83	3261865	10.0000
Phenazepam	4.646	885899	328.91	427.91	15475517	10.0000
Phencyclidine	3.886	10399966	694.45	134.91	17469730	10.0000
Phentermine	3.122	3041729	335.38	33.37	30846214	10.0000
Phenytoin	4.118	834619	1681.50	142.22	363925	10.0000
Primidone	3.473	3160060	69315.12	860.91	363925	10.0000
Promethazine	4.253	13518667	2689.34	1233.46	50431635	10.0000

Cal

TS

AM #25 Multi-Drug Screen Results

Name	RT	Resp.	S/N	S/N	ISTD Resp.	Calc. Conc.
Pseudoephedrine	2.693	58167602	625.43	2243.62	36790708	10.0000
Quetiapine	4.199	6594383	5700.71	3579.13	40453363	10.0000
Risperidone	3.952	9181762	3138.61	30466.21	31718783	10.0000
Sertraline	4.497	683875	9388.14	14253.41	3261865	10.0000
Sufentanil	4.199	239954	203614.64	1282.44	40051738	10.0000
Tapentadol	3.411	8593965	2141.48	601.30	17045847	10.0000
Temazepam	4.668	3192385	1567.03	111.14	15475517	10.0000
Topiramate	3.831	48422	18354.36	22332.24	182231	10.0000
Tramadol	3.407	32791333	2205.65	133.40	50431635	10.0000
Trazodone	4.061	6098082	1077616.62	1469.51	24799315	10.0000
Venlafaxine	3.760	11606286	3980.91	310.28	3261865	10.0000
Zaleplon	4.344	2182335	21946.93	55065.31	40453363	10.0000
Zolpidem	3.803	12734828	10109576.04	1107.59	40453363	10.0000
Zopiclone	3.707	913311	14081.18	243496.10	4041991	10.0000

TS

Idaho State Police
Forensic Services

Request for Departure from an Analytical Method or Quality Standard

Deviation Number (assigned by QM): TOX-22-03

Date of Request:
10/11/2022

Requestor/Discipline:
Tamara Salazar/Toxicology

Analytical Method/Quality Standard, Revision #:
Toxicology AM #26: Blood and Urine THC and Metabolites Screen by LCMS-QQQ, Revision #13

Temporary or Permanent Deviation:

Temporary

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

Deviation is only for this specific instance.

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

Toxicology AM #26, section 4.1.16 specifies: Analyze samples or freeze for future analysis. The plate must be injected within 7 days of the extraction date. The extraction steps 1-6 on were performed on 09/19/2022. Completion of the extraction method was postponed due to the LCMS-QQQ instrument failing checktune. The extraction plate was placed in the freezer until the instrument could be repaired and a passing checktune completed. The extraction procedure was completed on 09/26/2022. Yet, due to an unrelated issue with the nitrogen generator, the extraction plate could not be injected on the instrument until 09/27/2022. This pushed the original extraction steps 1-6 outside of the acceptable 7-day window. The requestor is asking to use the data associated with the AM #26 extraction procedure started on 09/19/2022 and completed on 09/26/2022.

Technical Justification for Analytical Method Deviations:

The extraction was completed and injected within 7 days. If we have an issue with the run, we are allowed to go back to the extraction plate and do another extraction, starting from step 7. The plate must then be injected from 7 days after that point, so this is no

TS

different except that there is no re-extraction. In addition, the data meets the criteria for evaluation as the positive and negative controls are consistent with expected results, and the internal standard responses are adequate.

Technical Review

☒ Departure approved
Comments: This deviation was verbally approved on 09/26/2022, prior to the start of the extraction.

☐ Departure Not Approved
Comments:

Approver: Celena Shrum Date: 10/11/2022
Title: Toxicology Discipline Lead

Quality Review

Quality Approver:
Title: Acting Quality Manager
Date: 10/11/2022

TS

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

Extraction Date: 09/26/2022 Analyst: Tamara Salazar
Plate lot#: 220309 Plate Retest Date: 09/09/2022
Mobile phase A: 10mM Amm Form in LCMS Water Mobile phase B: 0.1% Formic acid in MeOH
Blank Blood Lot: Lampire 22B52015-1 Blank Urine Lot: POC21022
LCMS-QQQ ID: 069901 Column: Phenomenex Phenyl Hexyl (4.6x50mm, 2.6um)

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.
☒ 3. Using a calibrated pipette, pipette 1000µL blood or 1000µL hydrolyzed urine in wells of analytical (standards) plate. Pipette ID: 42
☒ 4. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 5. Add **500µL of 0.1% formic acid in water to blood samples, and 500µL of saturated phosphate buffer to urine samples** in the wells of the analytical plate.
☒ 6. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 7. Transfer **700-800µL of blood+acid or urine+acid** mixture to corresponding wells of SLE+ plate. Amount transferred*: 750 µL*
☒ 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 **2.25mL MTBE. (*Add in 3 increments of 750uL)***
☒ 11. Wait 5 minutes.
☒ 12. Apply positive pressure for approx. 15 seconds. ***(10-15 PSI- Selector to the left).***
☒ 13. Add **2.25mL Hexane. (*Add in 3 increments of 750uL)***
☒ 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.
☒ 17. 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, R^2 values ≥0.98 for each analyte
☒ 3. RT +/- 2% or 0.100 min, whichever is greater
☒ 4. Confirmation testing on case samples with a response for THC and OH-THC of 3ng/mL or greater and/or Carboxy-THC at 10ng/mL or greater (analyst discretion between 5-10ng/mL) may be pursued.
☒ 5. Did all QCs pass for each analyte? (if not, describe in comments section)
☒ 6. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: Extraction analytical steps 1-6 were performed on 09/19/2022. Due to the instrument failing checktune, the extraction procedure was postponed. The analytical plate was placed in the freezer until the instrument could be repaired. Extraction steps 7-17 were completed on 09/26/2022.

Instrument nitrogen generator indicated a potential maintenance required. Analytical plates were frozen until PEAK analytics could be contacted. Plate was ran on 09/27/2022 after the company indicated that the generator would be functional.

Analytical Plate Map

TS

	1	2	3	4	5	6
A	IS + Cal. 1	IS + QC_1	M2022-3658-2	P2022-2580-1	P2022-2765-1	IS + QC_1
B	IS + Cal. 2	Neg Blood	M2022-3659-4	P2022-2582-1	P2022-2782-1	IS + Cal. 7
C	IS + Cal. 3	M2022-3438-3	M2022-3676-2	P2022-2588-1	P2022-2788-1	IS + Cal. 6
D	IS + Cal. 4	M2022-3493-1	M2022-3734-2	P2022-2588-2	P2022-2805-1	IS + Cal. 5
E	IS + Cal. 5	M2022-3534-4	M2022-3741-2	P2022-2639-1	P2022-2808-1	IS + Cal. 4
F	IS + Cal. 6	M2022-3535-1	M2022-3742-3	P2022-2758-1	P2022-2814-2	IS + Cal. 3
G	IS + Cal. 7	M2022-3588-3	P2022-2484-1	P2022-2761-1		IS + Cal. 2
H	IS + QC_1	M2022-3655-1	P2022-2484-2	P2022-2762-1	IS + QC_1	IS + Cal. 1

All wells to contain 100 µl of residual DMSO

SLE Plate Map

TS

	1	2	3	4	5	6
A	IS + Cal. 1	IS + QC_1	M2022-3658-2*	P2022-2580-1	P2022-2765-1*	M2022-3658-2
B	IS + Cal. 2	Neg Blood	M2022-3659-4*	P2022-2582-1	P2022-2782-1	M2022-3659-4
C	IS + Cal. 3	M2022-3438-3	M2022-3676-2	P2022-2588-1	P2022-2788-1	P2022-2765-1
D	IS + Cal. 4	M2022-3493-1	M2022-3734-2	P2022-2588-2	P2022-2805-1	
E	IS + Cal. 5	M2022-3534-4*	M2022-3741-2	P2022-2639-1	P2022-2808-1	
F	IS + Cal. 6	M2022-3535-1	M2022-3742-3	P2022-2758-1	P2022-2814-2	
G	IS + Cal. 7	M2022-3588-3	P2022-2484-1	P2022-2761-1	M2022-3534-4	
H	IS + QC_1	M2022-3655-1	P2022-2484-2	P2022-2762-1*	P2022-2762-1	

*Moved during step 7 of extraction due to blood clot

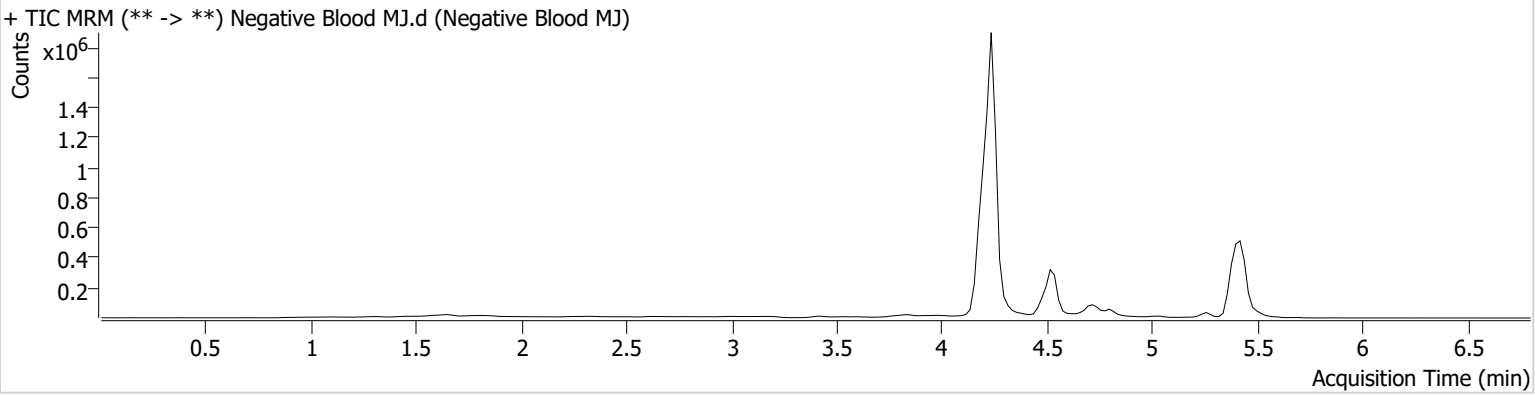
TS

AM #26 Cannabinoids Screen Results

Batch results	D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update	10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	Negative Blood MJ.d
Type	Sample	**Sample**	Negative Blood MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-B2	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 11:36:37 AM		
Sample Info.			

Sample Chromatogram



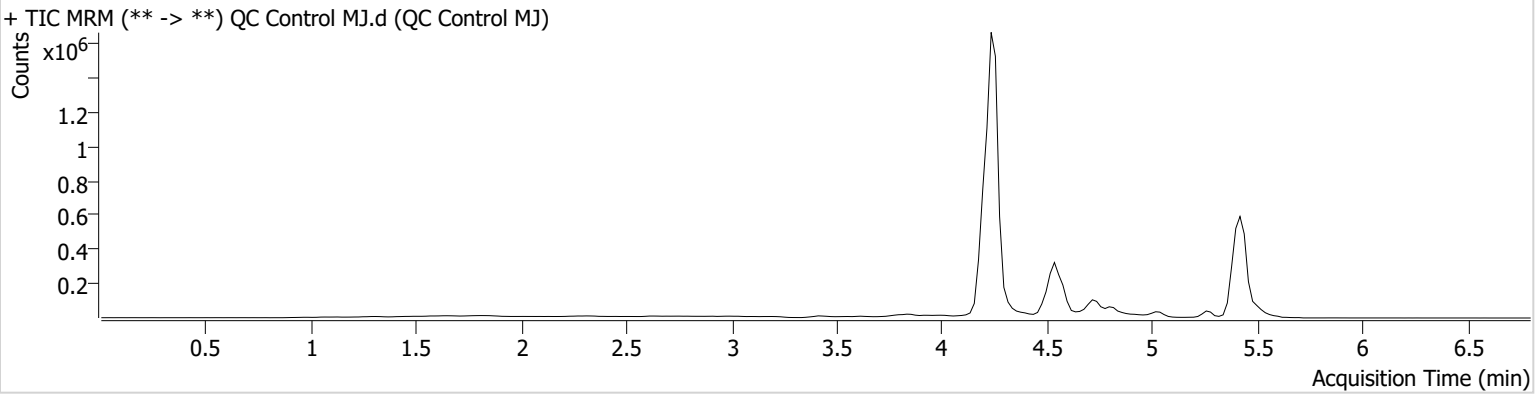
TS

AM #26 Cannabinoids Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update 10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	QC Control MJ.d
Type	QC	**Sample**	QC Control MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 11:21:28 AM		
Sample Info.			

Sample Chromatogram

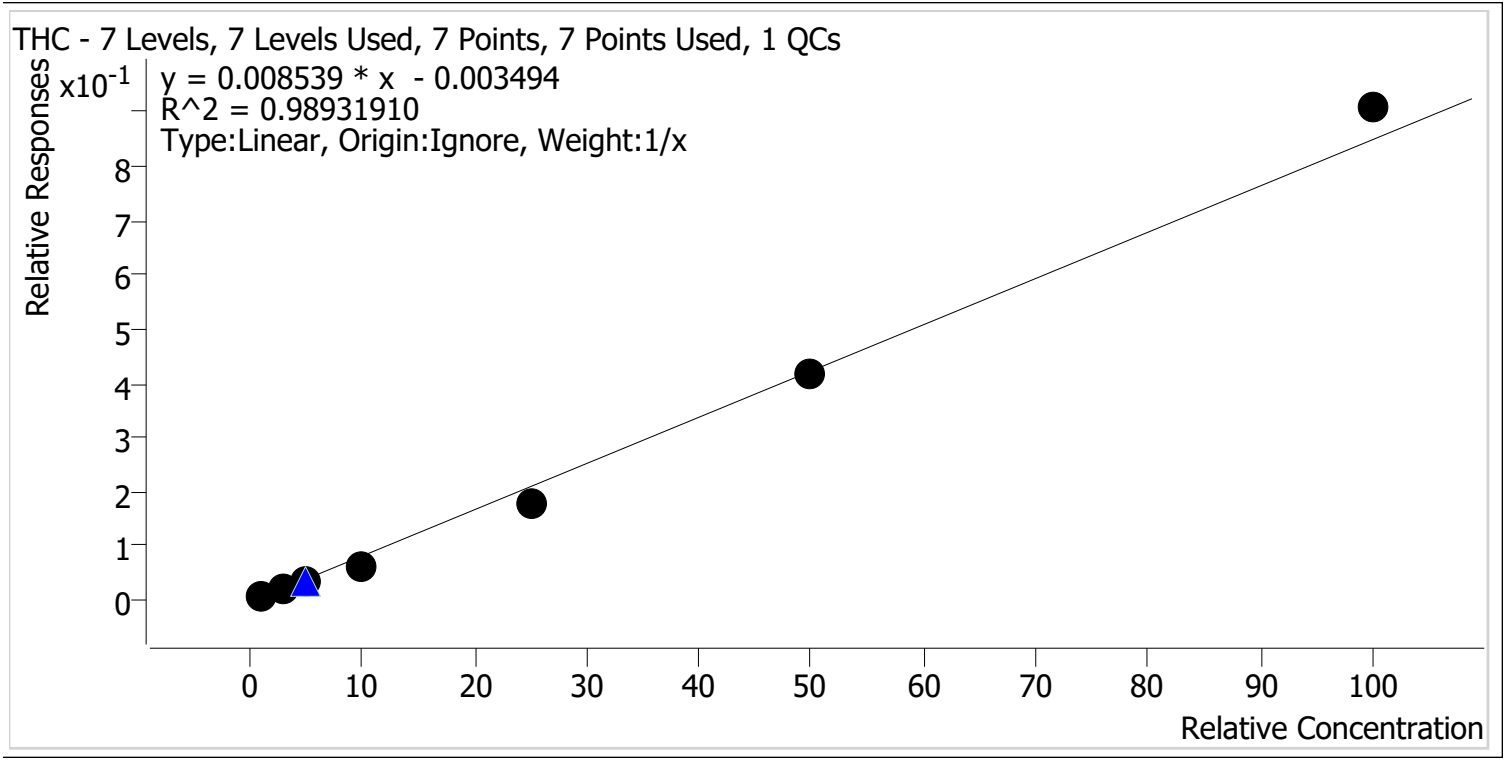


Name	RT	Resp.	ISTD Resp.	Final Conc.	
THC	5.409	11423	308281	4.7488	ng/ml
THC-COOH	4.576	224095	1035735	13.1220	ng/ml
THC-OH	4.262	56257	6990530	4.6418	ng/ml

TS

AM #26 Cannabinoids Screen Calibration Curve Report

Batch results	D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Last Cal. Update	10/2/2022 11:01 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3



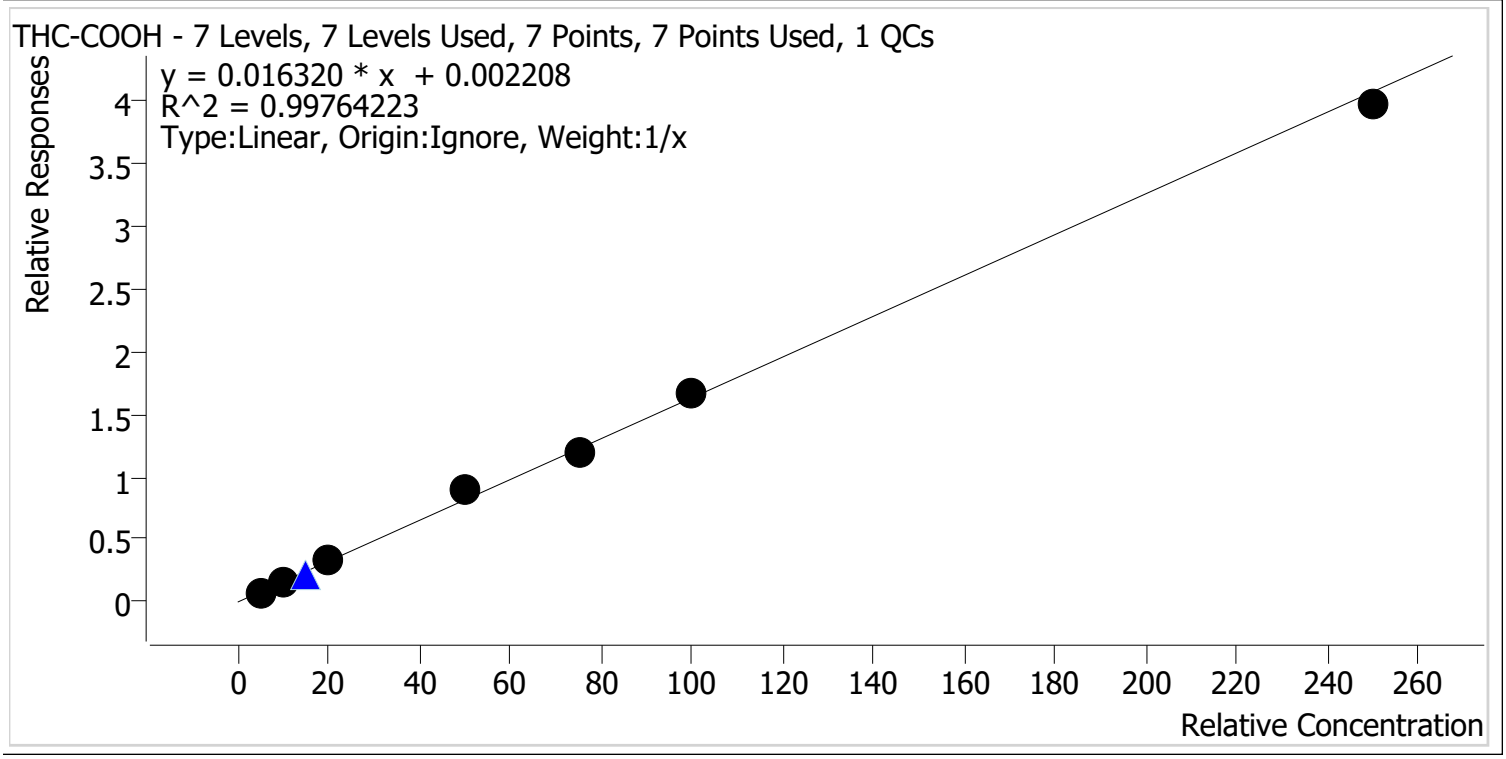
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	1.0	1.4	142.9
Cal 2 MJ	2	✔	3.0	2.9	95.7
Cal 3 MJ	3	✔	5.0	4.8	95.2
Cal 4 MJ	4	✔	10.0	7.5	75.1
Cal 5 MJ	5	✔	25.0	21.6	86.3
Cal 6 MJ	6	✔	50.0	49.0	98.0
Cal 7 MJ	7	✔	100.0	106.9	106.9

Generated at 4:11 PM on 10/11/2022

TS

AM #26 Cannabinoids Screen Calibration Curve Report

Batch results	D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Last Cal. Update	10/2/2022 11:01 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9

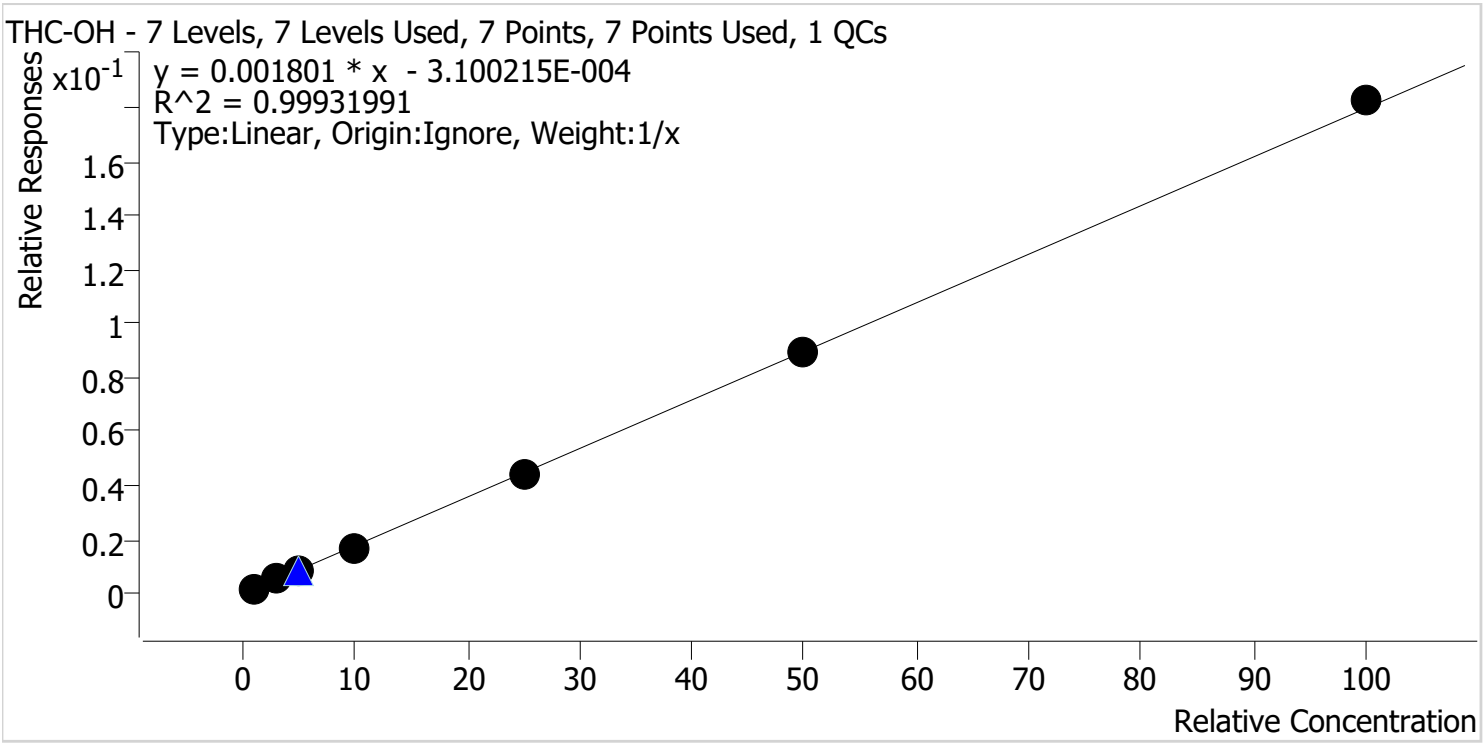


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	5.0	4.5	89.0
Cal 2 MJ	2	✔	10.0	9.9	99.2
Cal 3 MJ	3	✔	20.0	20.6	103.1
Cal 4 MJ	4	✔	50.0	55.4	110.8
Cal 5 MJ	5	✔	75.0	73.5	98.0
Cal 6 MJ	6	✔	100.0	102.5	102.5
Cal 7 MJ	7	✔	250.0	243.6	97.4

TS

AM #26 Cannabinoids Screen Calibration Curve Report

Batch results	D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Last Cal. Update	10/2/2022 11:01 AM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	1.0	1.1	114.0
Cal 2 MJ	2	✔	3.0	3.0	100.0
Cal 3 MJ	3	✔	5.0	4.6	91.1
Cal 4 MJ	4	✔	10.0	9.6	95.9
Cal 5 MJ	5	✔	25.0	24.5	98.1
Cal 6 MJ	6	✔	50.0	49.6	99.2
Cal 7 MJ	7	✔	100.0	101.6	101.6

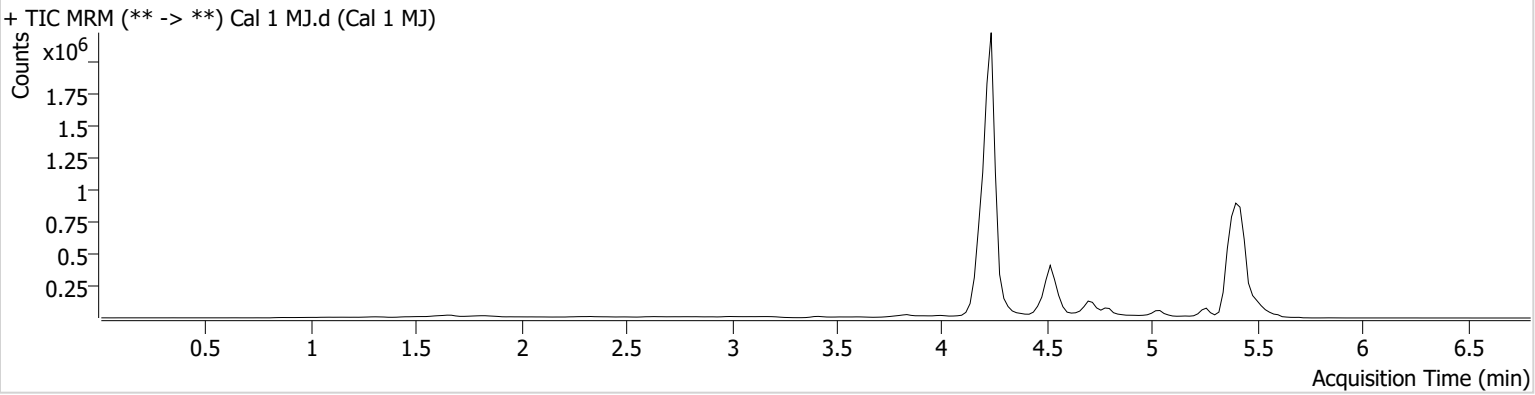
TS

AM #26 Cannabinoids Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update 10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	Cal 1 MJ.d
Type	Cal	**Sample**	Cal 1 MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 10:24:00 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.		
THC	5.449	8571	984702	1.4286	ng/ml	**Low**
THC-COOH	4.556	112492	1502651	4.4518	ng/ml	**Low**
THC-OH	4.242	16358	9383708	1.1404	ng/ml	**Low**

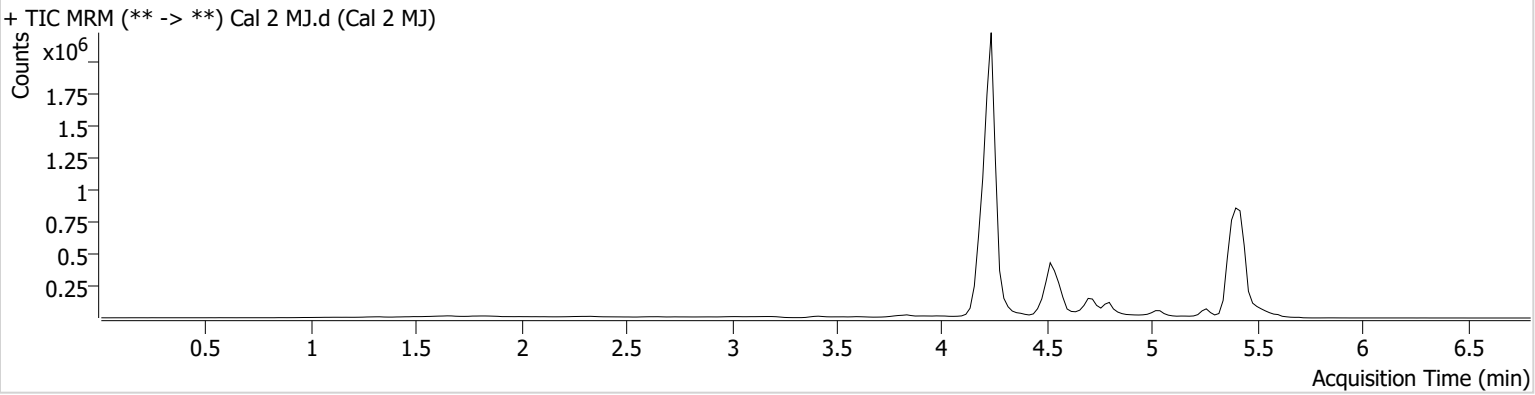
TS

AM #26 Cannabinoids Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update 10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	Cal 2 MJ.d
Type	Cal	**Sample**	Cal 2 MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 10:31:46 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.		
THC	5.389	22149	1054242	2.8697	ng/ml	Low
THC-COOH	4.556	239976	1462930	9.9159	ng/ml	
THC-OH	4.242	45224	8879014	3.0010	ng/ml	

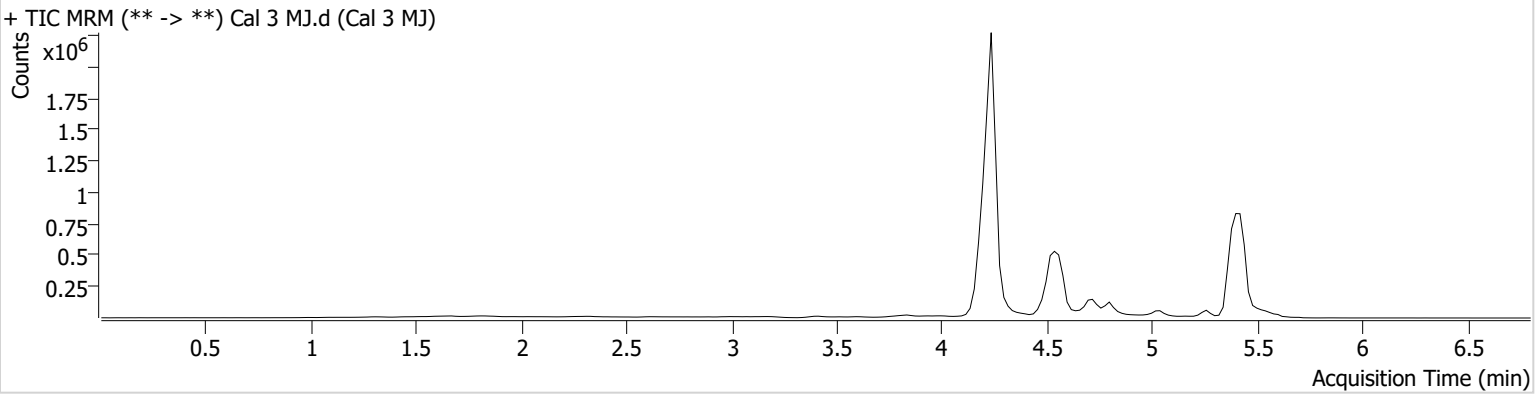
TS

AM #26 Cannabinoids Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update 10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	Cal 3 MJ.d
Type	Cal	**Sample**	Cal 3 MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 10:39:21 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.	
THC	5.389	29372	790274	4.7619	ng/ml
THC-COOH	4.556	527211	1556995	20.6124	ng/ml
THC-OH	4.242	68961	8738807	4.5550	ng/ml

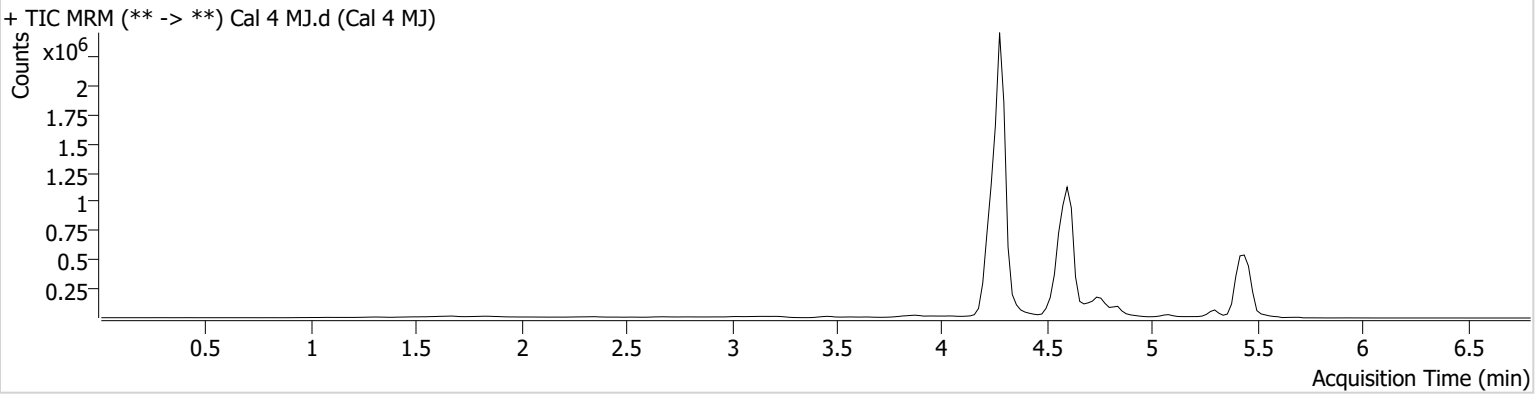
TS

AM #26 Cannabinoids Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update 10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	Cal 4 MJ.d
Type	Cal	**Sample**	Cal 4 MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 10:51:01 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.	
THC	5.429	40119	661346	7.5137	ng/ml
THC-COOH	4.596	1467360	1619313	55.3886	ng/ml
THC-OH	4.302	159753	9421043	9.5900	ng/ml

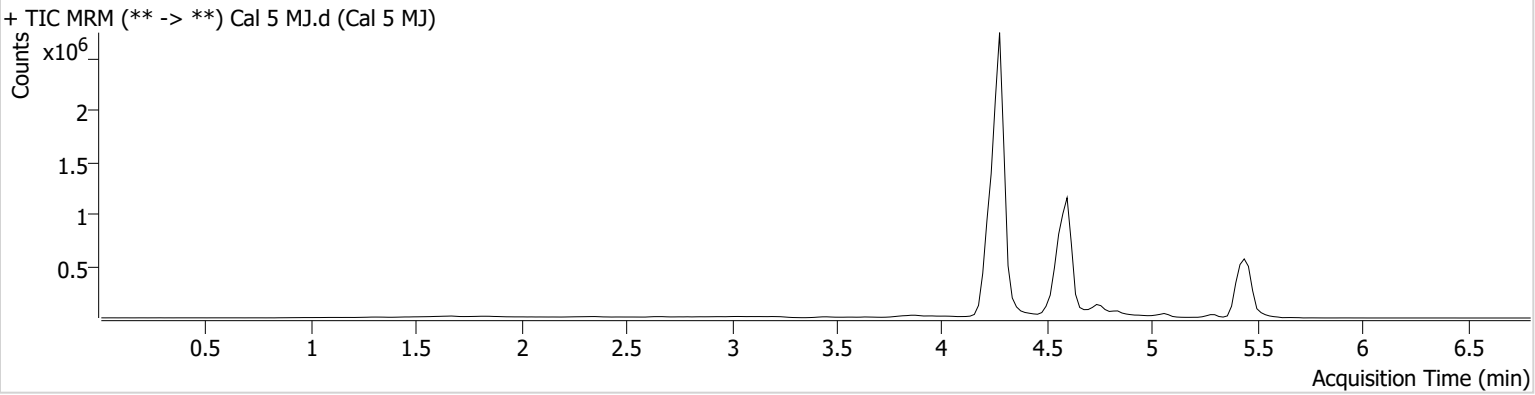
TS

AM #26 Cannabinoids Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update 10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	Cal 5 MJ.d
Type	Cal	**Sample**	Cal 5 MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 10:58:45 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.	
THC	5.429	103503	572862	21.5692	ng/ml
THC-COOH	4.596	1723593	1433788	73.5234	ng/ml
THC-OH	4.282	385775	8793885	24.5365	ng/ml

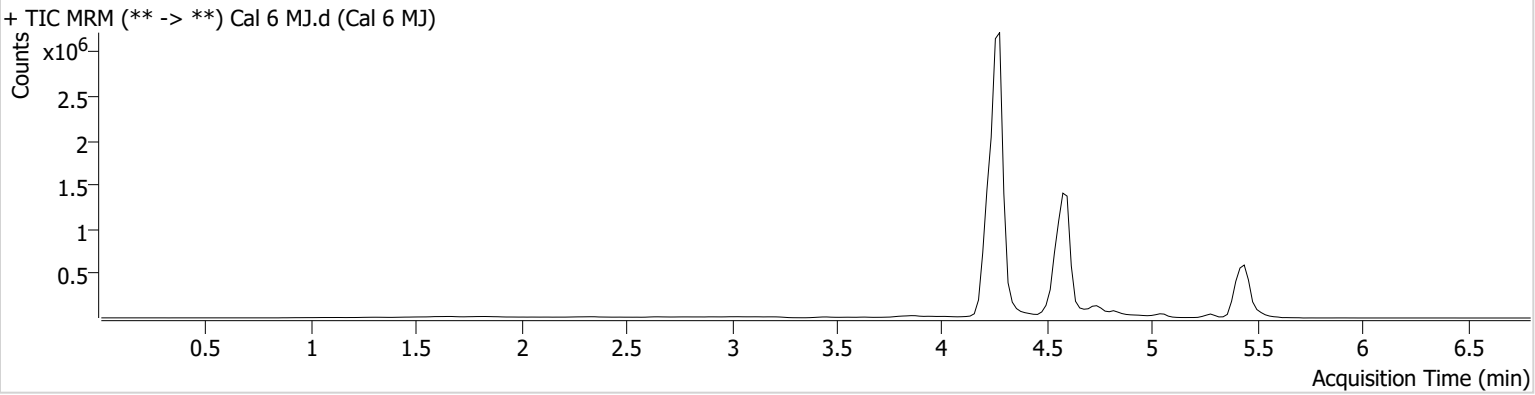
TS

AM #26 Cannabinoids Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update 10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	Cal 6 MJ.d
Type	Cal	**Sample**	Cal 6 MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 11:06:19 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.	
THC	5.429	176605	425859	48.9771	ng/ml
THC-COOH	4.596	2142338	1279166	102.4855	ng/ml
THC-OH	4.282	767593	8623811	49.6069	ng/ml

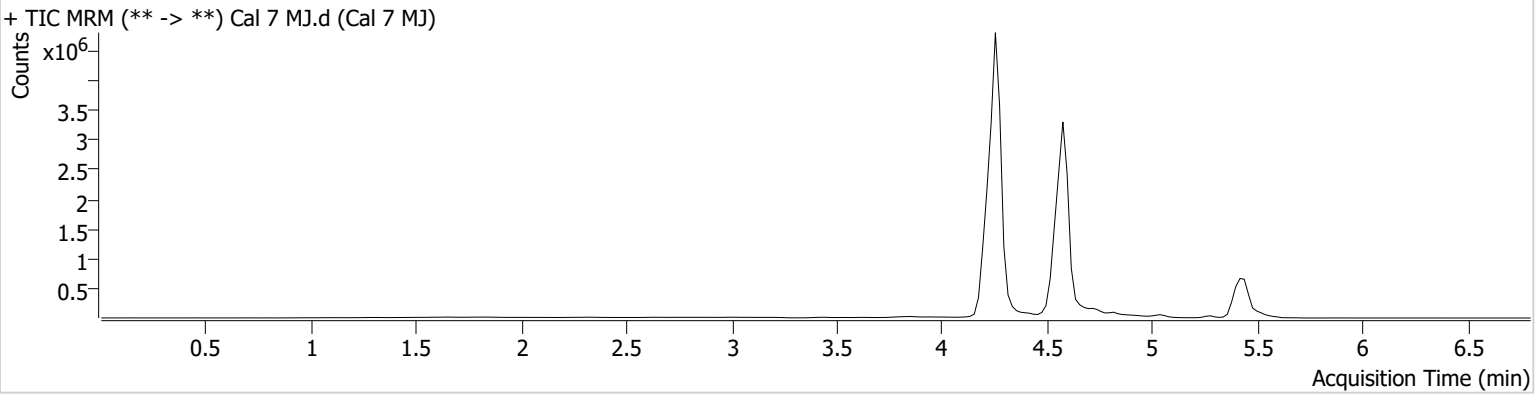
TS

AM #26 Cannabinoids Screen Results

Batch results D:\MassHunter\Data\2022\AM 25-26\092722 AM 25 26\QuantResults\AM 26.batch.bin
Calibration Last Update 10/2/2022 11:01:04 AM

Instrument	Falco (069901)	**Data File**	Cal 7 MJ.d
Type	Cal	**Sample**	Cal 7 MJ
Acq. Method	AM 26 THC.m	**Operator**	Tamara Salazar
Sample Position	P1-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/27/2022 11:13:54 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.	
THC	5.409	263595	289948	106.8798	ng/ml
THC-COOH	4.576	4918073	1236263	243.6224	ng/ml
THC-OH	4.262	1463297	8015004	101.5702	ng/ml