REVIEWED

By Amber Gerheart at 10:09 am, Oct 21, 2022

10/19/2022

Worklist: 6137

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
C2022-2126		BCK	AM 28 Blood Multi-Drug Quant Panel 1 by LC-QQQ	
C2022-2220		BCK	AM 28 Blood Multi-Drug Quant Panel 1 by LC-QQQ	
C2022-2239		BCK	AM 28 Blood Multi-Drug Quant Panel 1 by LC-QQQ	
C2022-2244	2	BCK	AM 28 Blood Multi-Drug Quant Panel 1 by LC-QQQ	
C2022-2246		BCK	AM 28 Blood Multi-Drug Quant Panel 1 by LC-QQQ	
C2022-2295		BCK	AM 28 Blood Multi-Drug Quant Panel 1 by LC-QQQ	
C2022-2312		BCK	AM 28 Blood Multi-Drug Quant Panel 1 by LC-QQQ	
C2022-2343		BCK	AM 28 Blood Multi-Drug Quant Panel 1 by LC-QQQ	

AM# 28: Multi-Drug Quantitation by LC-MS/MS

Extraction Date 10/19/22 Analyst: Anne Nord
Plate lot#: 220802 Item: IDP-121-3CDA Plate re-test: 02/02/23

Mobile phase A: 5mM Amm Form + 0.01% FA **Mobile phase B:** 0.01% Formic Acid in MeOH
0.5M Ammonium Hydroxide Ethyl Acetate 20% Methanol in Water
Blank Blood Lot: 22B52016-1 **Urine Blank lot:** blood only run
Column: Agilent 120 EC-C18 (2.1x 100-2.7um)
LCMS-QQQ ID: 69679

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 pipette 250 ul urine in blank well, add 40 ul BG Turbo, add 100 ul 500 mm sodium phosphate buffer, mix for at least 5 minutes at ambient temperature. Pipette **250µL blood (calibrated pipette) Pipette ID: 390993 or 250µL hydrolyzed urine** in wells of analytical (standards) plate.
☒ 3. Pipette **250µL 00.5M ammonium hydroxide** in wells of analytical plate.
☒ 4. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 5. Transfer **300µL of blood+base/urine+base** mixture to corresponding wells of SLE+ plate.
☒ 6. 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: 66792*
☒ 7. Wait 5 minutes.
☒ 8. Add **900uL ethyl acetate.**
☒ 9. Wait 5 minutes.
☒ 10. Apply positive pressure for approx. 15 seconds. ***(10-15 PSI- Selector to the left).***
☒ 11. Add **900uL ethyl acetate.**
☒ 12. Wait 5 minutes.
☒ 13. Apply positive pressure for approx. 15 seconds. ***(10-15 PSI- Selector to the left).***
☒ 14. Remove plate containing eluate. Add 50 ul 1% HCl in MeOH,
☒ 15. Place on SPE Dry and evaporate to dryness at approx. 35°C.
SPE Dry ID: 66819
☒ 16. Reconstitute in **100µL 20% MeOH** and heat seal plate with foil.

Post-Analytic

☒ 1. Create batch and process data.
☒ 2. Make necessary changes to integration limits
☒ 3. Integration linear and R^2 values ≥0.98 for each analyte.
☒ 4. For unknown samples and controls: response ratio within 20% of average of controls and standards, RT within +/- 5% (tramadol RT +/-2%), S/N for primary transition >10 and secondary transitions >5.
☒ 5. Did all QCs pass for each analyte? Yes, see comments Add Control data to QC tracking spreadsheet.
☒ 6. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports.

COMMENTS: Amphetamine, fentanyl, ketamine, methamphetamine, and norfentanyl – were processed and evaluated in this run. Extracts were initialing injected on 10/19/22 at 2.5 ul, due to a couple of the compounds having poor linearity the injection volume was decreased to 0.5 ul and the extracts were re-injected and that set of injections was evaluated.
Curve limits:

amphetamine 5-500 (removed cal 8 due to accuracy)
methamphetamine 10-1000 (removed cal 1 due to accuracy)

Did not evaluate- Norfentanyl due to interfering peak in the qualifier ion.

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

	1	2	3	4	5	6	7	8	9	10	11	12
A					IS + Cal. 1	IS + QC_1	2220-1					
B					IS + Cal. 2	IS + QC_2	2239-1					
C					IS + Cal. 3	IS + QC 3	2244-2					
D					IS + Cal. 4	IS + QC_4	2246-1					
E					IS + Cal. 5	IS + QC_2	2312-1					
F					IS + Cal. 6	negative blood	2343-1					
G					IS + Cal. 7	2126-1						
H					IS + Cal. 8	2295-1						

blank in front

plate position 2

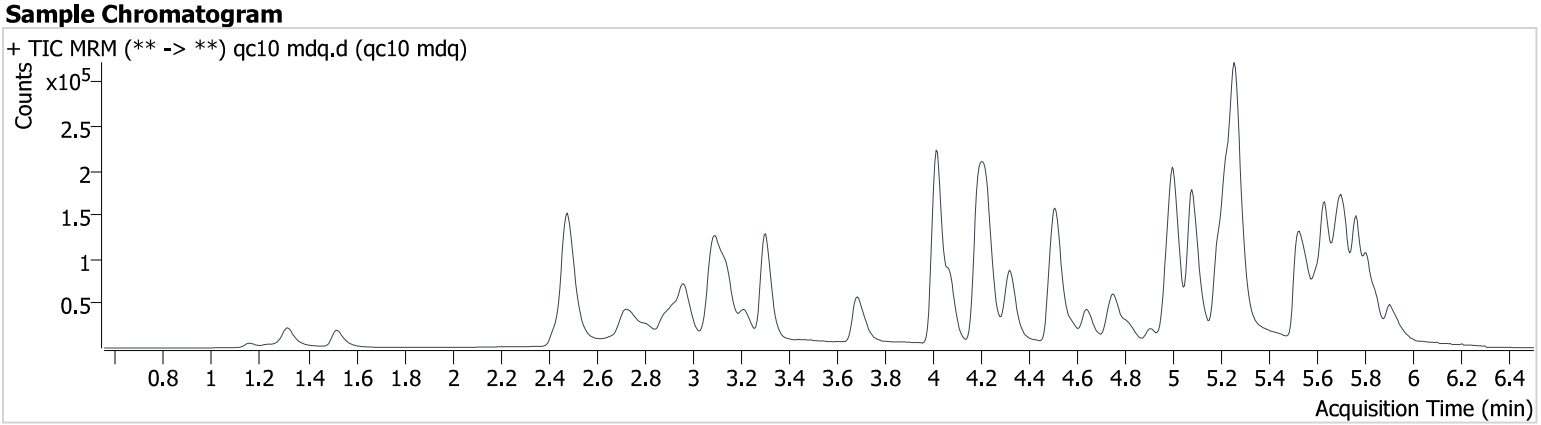
c2022-___-_

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	qc10 mdq.d
Type	QC	**Sample**	qc10 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-A6	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 10:19:48 AM		
Sample Info.			

Sample Chromatogram



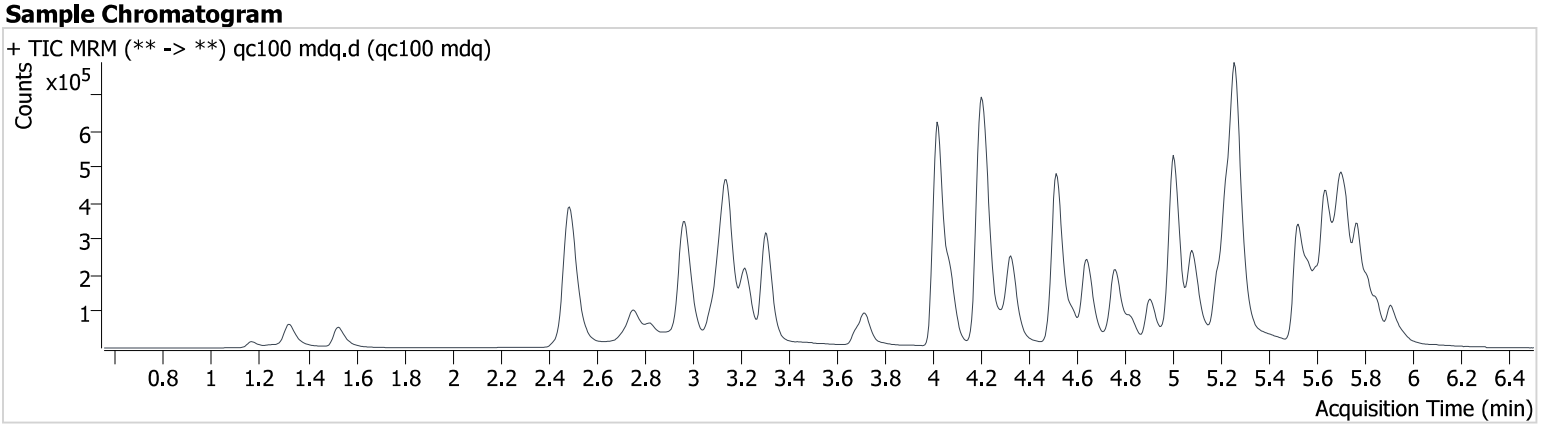
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	38011	860.2	286.1	8957.2	100503	10.470	ng/ml
Fentanyl	5.093	4192	150.9	132.8	∞	181079	0.865	ng/ml
Ketamine	4.025	33588	4655.1	36.4	309.1	138956	10.735	ng/ml
Methamphetamine	3.148	100031	195.9	38.3	648.4	299076	8.190	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	qc100 mdq.d
Type	QC	**Sample**	qc100 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-B6	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 12:52:31 PM		
Sample Info.			

Sample Chromatogram



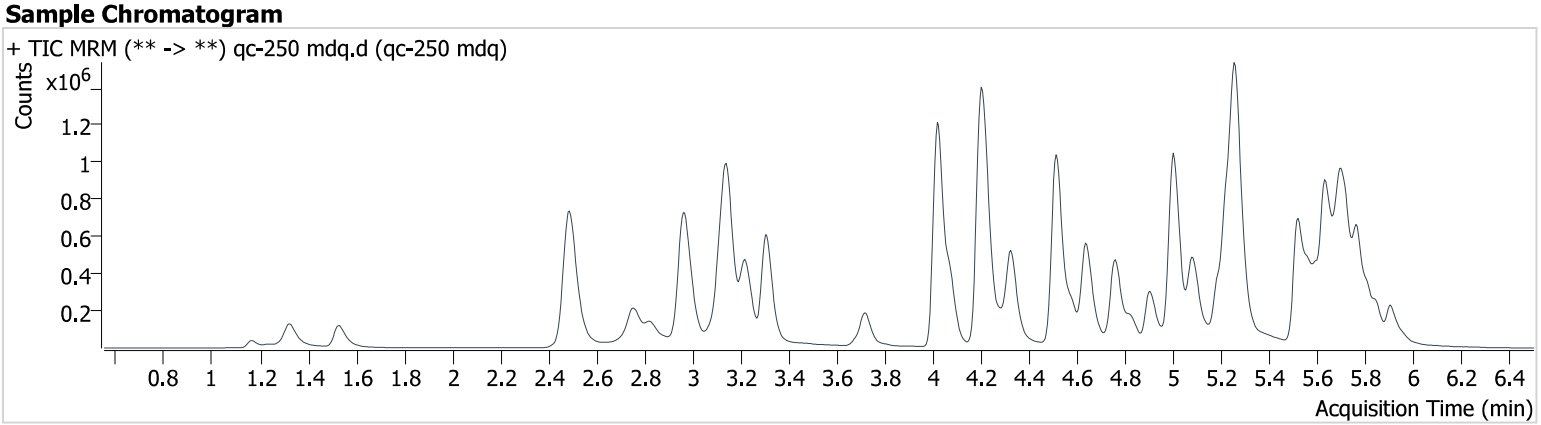
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	289756	13807.3	280.1	18491.0	78893	112.143	ng/ml
Fentanyl	5.093	36411	2312.2	118.3	3558.9	122434	9.943	ng/ml
Ketamine	4.025	259616	59315.9	35.7	4890.4	118941	105.379	ng/ml
Methamphetamine	3.148	639741	∞	37.7	3603.2	258470	106.076	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	qc-250 mdq.d
Type	QC	**Sample**	qc-250 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-C6	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 10:28:49 AM		
Sample Info.			

Sample Chromatogram



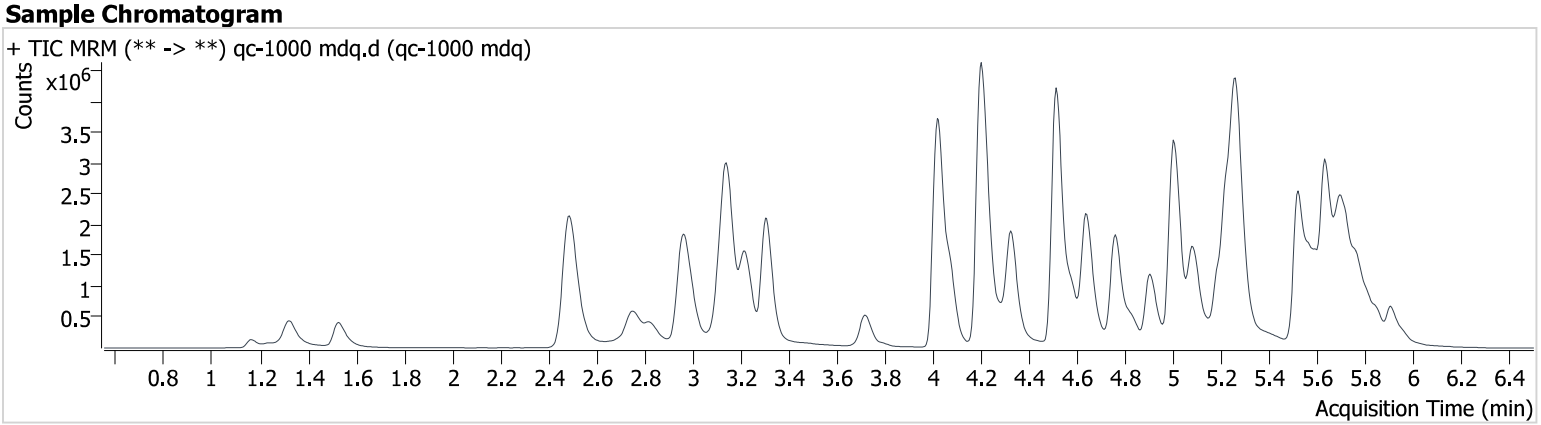
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	665279	27592.0	271.4	31737.4	79663	256.525	ng/ml
Fentanyl	5.093	84011	3871.8	116.5	10453.4	114045	24.484	ng/ml
Ketamine	4.025	568832	17873.6	35.3	6160.4	106430	259.554	ng/ml
Methamphetamine	3.148	1403457	∞	38.6	7775.5	246723	253.009	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	qc-1000 mdq.d
Type	QC	**Sample**	qc-1000 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-D6	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 10:37:49 AM		
Sample Info.			

Sample Chromatogram



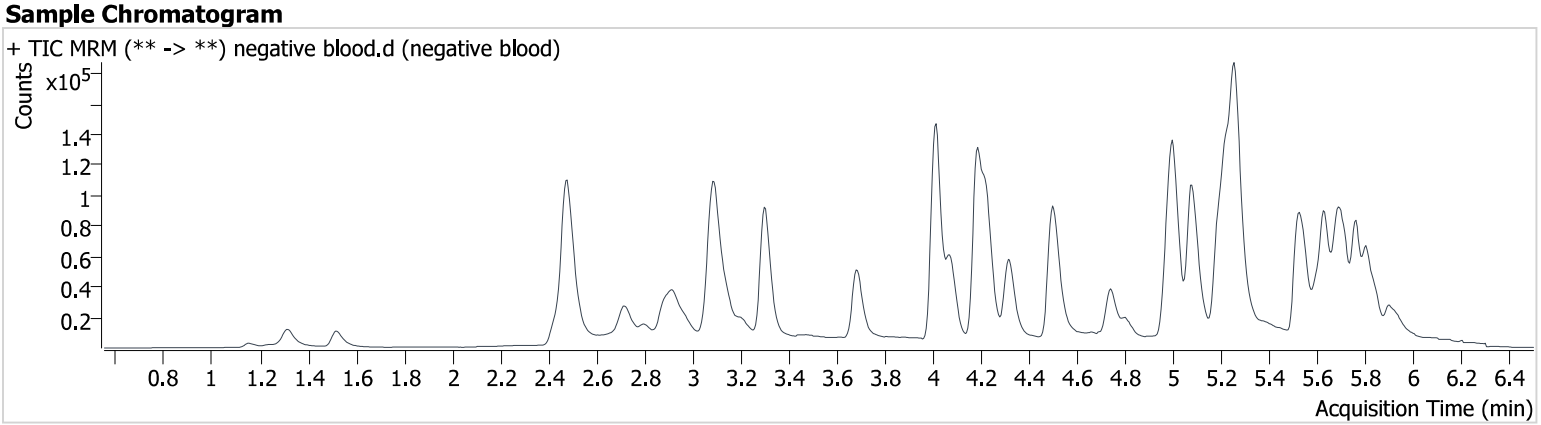
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	1941779	502130.4	259.0	166243.7	81746	731.871	ng/ml
Fentanyl	5.098	322902	22438.2	118.6	623505.3	106244	100.706	ng/ml
Ketamine	4.025	1905450	111097.3	35.4	14632.8	98808	939.254	ng/ml
Methamphetamine	3.148	5222319	∞	37.5	∞	262713	901.878	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

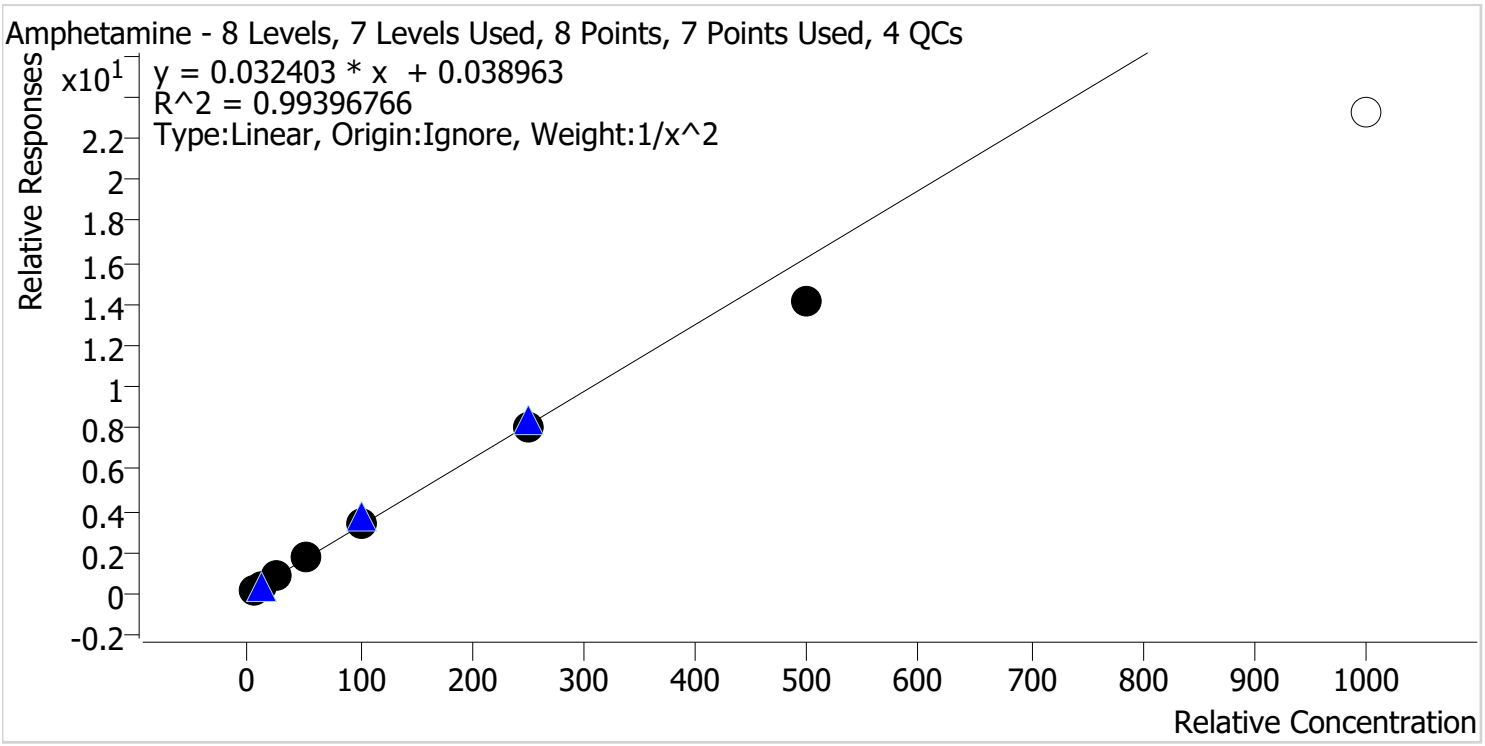
Instrument	69679	**Data File**	negative blood.d
Type	Sample	**Sample**	negative blood
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-F6	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 10:46:48 AM		
Sample Info.			

Sample Chromatogram



Compound Calibration Report

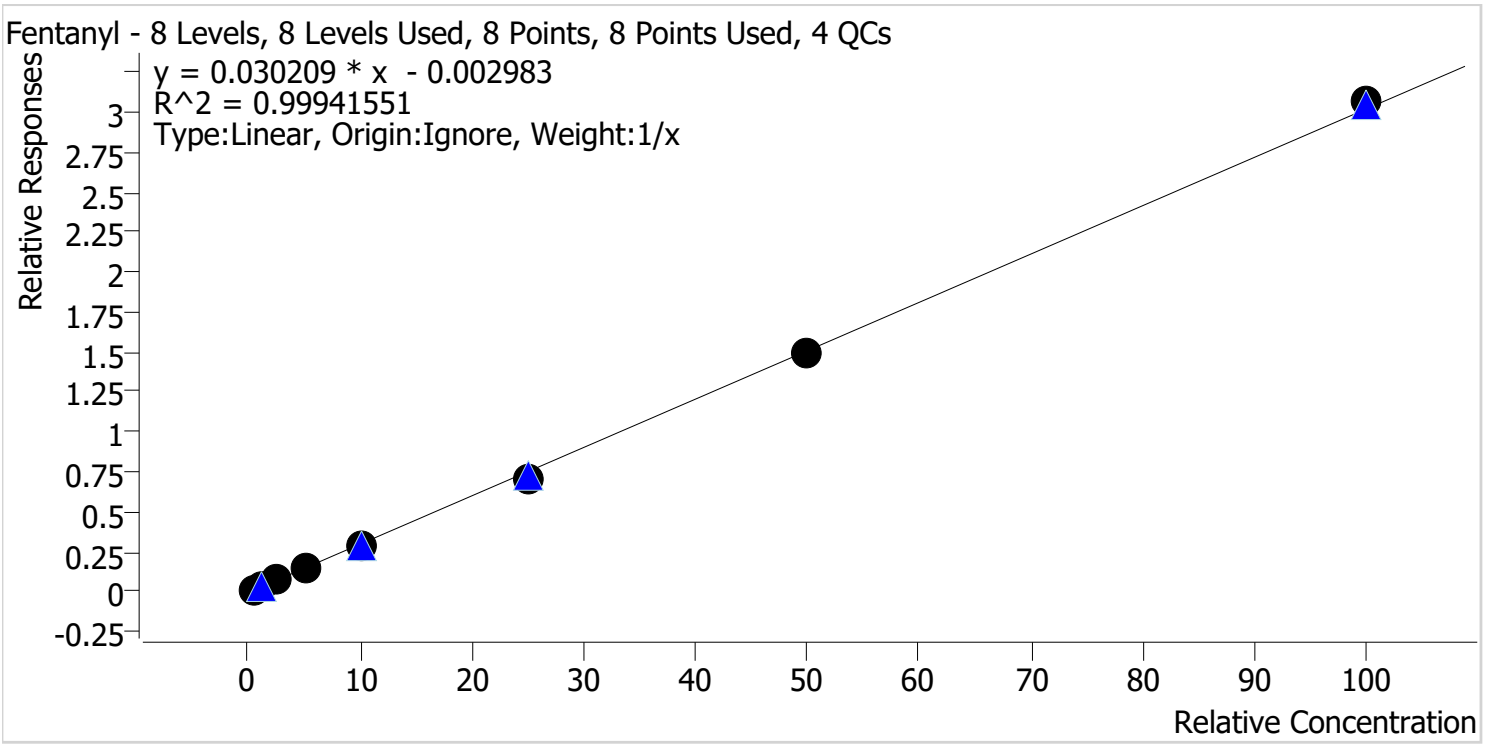
Batch results	D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Last Cal. Update	10/20/2022 1:32 PM
Analyst Name	ISP\datastor
Analyte	Amphetamine
Internal Standard	Amphetamine-D11



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
cal 1 mdq	1	✔	5.0	5.0	99.5
cal 2 mdq	2	✔	10.0	9.8	98.1
cal 3 mdq	3	✔	25.0	25.7	102.8
cal 4 mdq	4	✔	50.0	54.1	108.2
cal 5 mdq	5	✔	100.0	104.7	104.7
cal 6 mdq	6	✔	250.0	248.1	99.2
cal 7 mdq	7	✔	500.0	437.1	87.4
cal 8 mdq	8	✗	1000.0	716.1	71.6

Compound Calibration Report

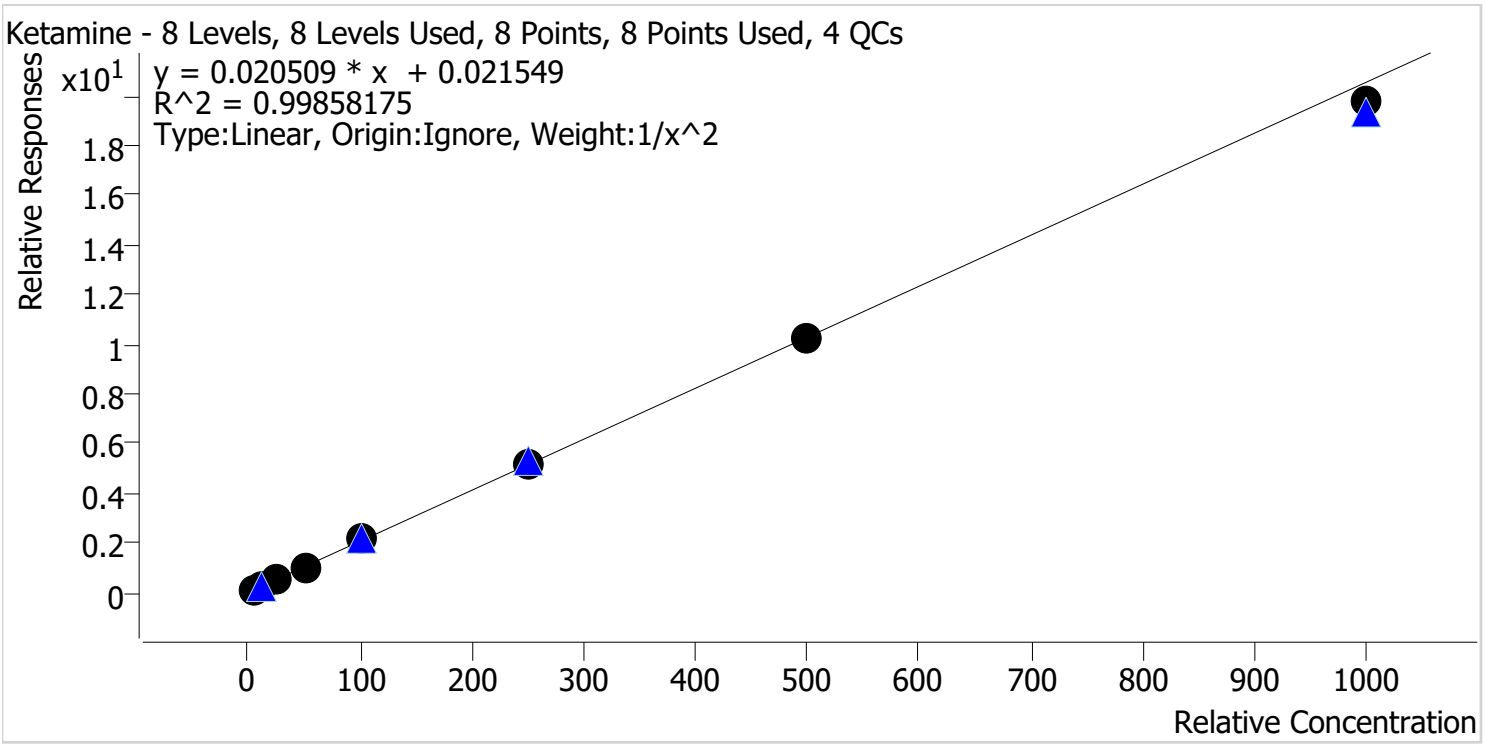
Batch results	D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Last Cal. Update	10/20/2022 1:32 PM
Analyst Name	ISP\datastor
Analyte	Fentanyl
Internal Standard	Fentanyl-D5



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
cal 1 mdq	1	✔	0.5	0.6	114.6
cal 2 mdq	2	✔	1.0	0.9	90.8
cal 3 mdq	3	✔	2.5	2.5	100.2
cal 4 mdq	4	✔	5.0	4.9	98.1
cal 5 mdq	5	✔	10.0	10.0	100.2
cal 6 mdq	6	✔	25.0	23.8	95.2
cal 7 mdq	7	✔	50.0	49.8	99.5
cal 8 mdq	8	✔	100.0	101.5	101.5

Compound Calibration Report

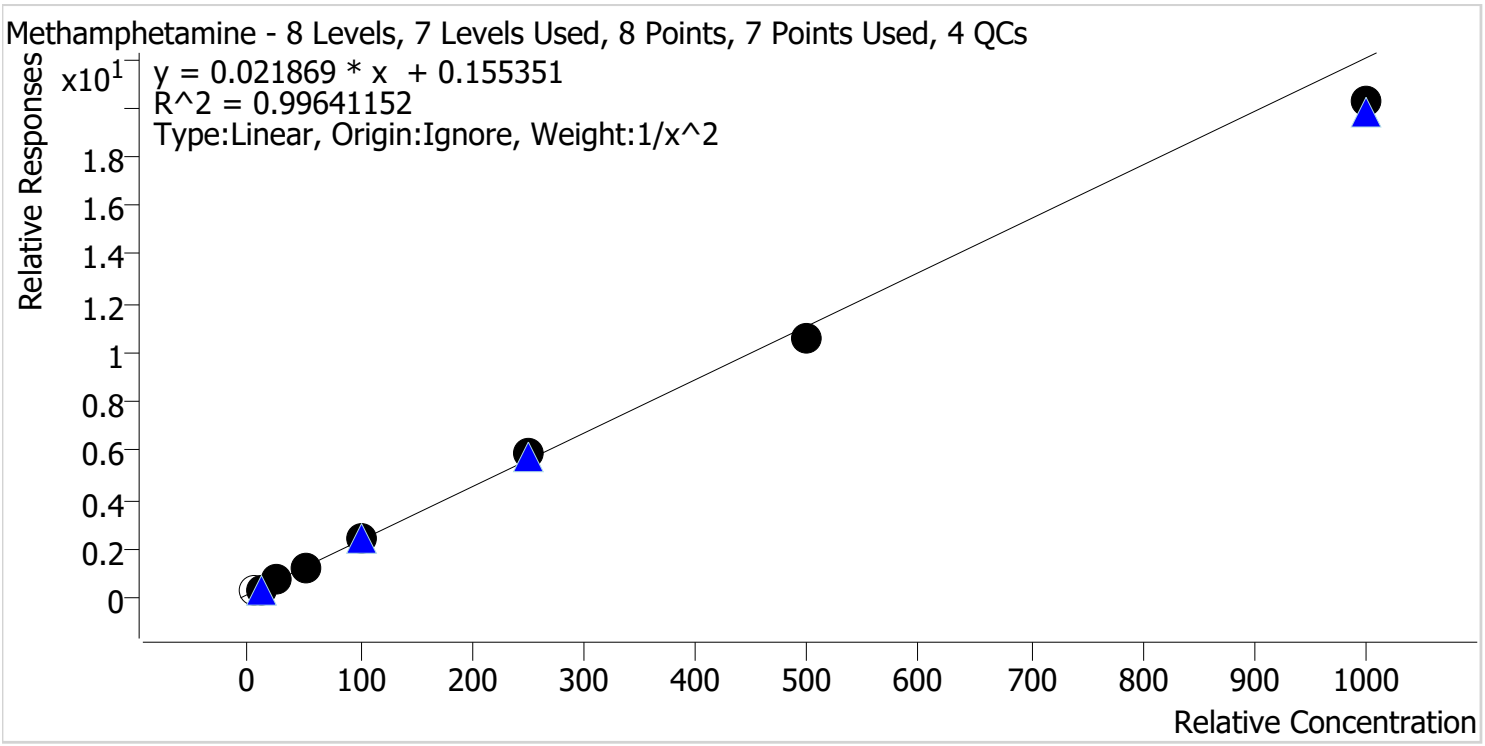
Batch results	D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Last Cal. Update	10/20/2022 1:32 PM
Analyst Name	ISP\datastor
Analyte	Ketamine
Internal Standard	Ketamine-D4



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
cal 1 mdq	1	✔	5.0	4.9	97.2
cal 2 mdq	2	✔	10.0	10.6	106.4
cal 3 mdq	3	✔	25.0	24.5	97.8
cal 4 mdq	4	✔	50.0	50.0	99.9
cal 5 mdq	5	✔	100.0	102.2	102.2
cal 6 mdq	6	✔	250.0	253.1	101.3
cal 7 mdq	7	✔	500.0	495.3	99.1
cal 8 mdq	8	✔	1000.0	962.1	96.2

Compound Calibration Report

Batch results	D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Last Cal. Update	10/20/2022 1:32 PM
Analyst Name	ISP\datastor
Analyte	Methamphetamine
Internal Standard	Methamphetamine-D11



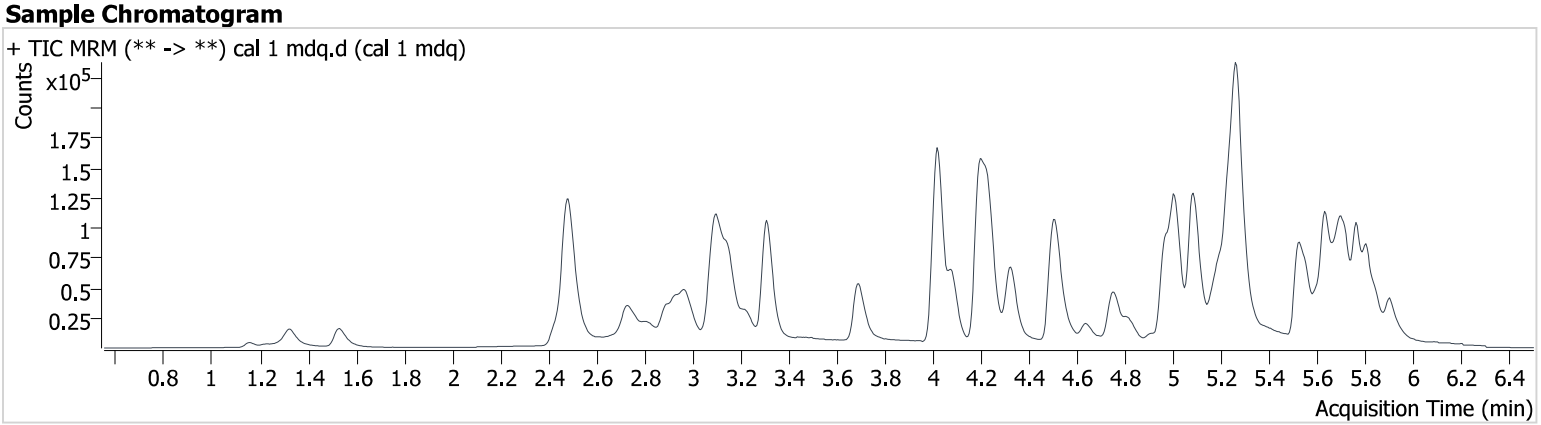
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
cal 1 mdq	1	✗	5.0	9.5	190.0
cal 2 mdq	2	✔	10.0	9.9	99.4
cal 3 mdq	3	✔	25.0	24.6	98.2
cal 4 mdq	4	✔	50.0	51.6	103.3
cal 5 mdq	5	✔	100.0	106.1	106.1
cal 6 mdq	6	✔	250.0	262.7	105.1
cal 7 mdq	7	✔	500.0	480.4	96.1
cal 8 mdq	8	✔	1000.0	918.4	91.8

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	cal 1 mdq.d
Type	Cal	**Sample**	cal 1 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-A5	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 8:58:57 AM		
Sample Info.			

Sample Chromatogram



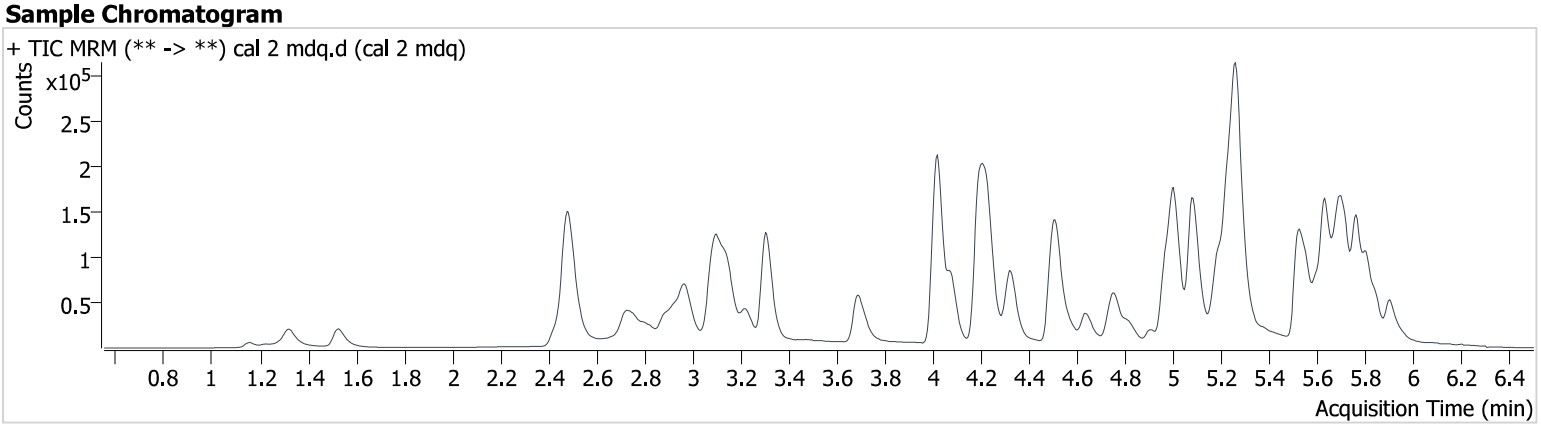
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.974	19171	∞	303.6	576.2	95806	4.973	ng/ml
Fentanyl	5.098	2113	6843.2	105.3	∞	147535	0.573	ng/ml
Ketamine	4.025	15781	2365.4	38.2	148.2	130232	4.858	ng/ml
Methamphetamine	3.158	100526	1930.1	38.1	630.0	276842	9.500	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	cal 2 mdq.d
Type	Cal	**Sample**	cal 2 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-B5	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 9:07:58 AM		
Sample Info.			

Sample Chromatogram



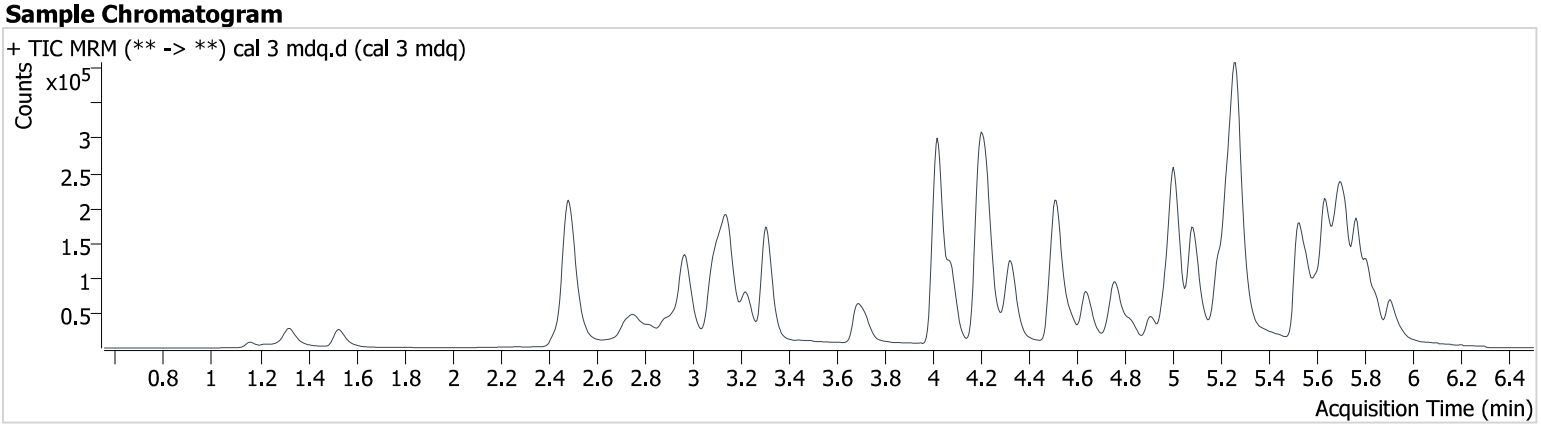
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	37019	1322.6	291.3	1409.2	103715	9.813	ng/ml
Fentanyl	5.093	4432	482.3	122.4	6868.9	181325	0.908	ng/ml
Ketamine	4.025	33598	1815.6	36.3	320.4	140190	10.635	ng/ml
Methamphetamine	3.153	108382	2890.1	36.2	310.1	290789	9.940	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	cal 3 mdq.d
Type	Cal	**Sample**	cal 3 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-C5	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 9:16:58 AM		
Sample Info.			

Sample Chromatogram



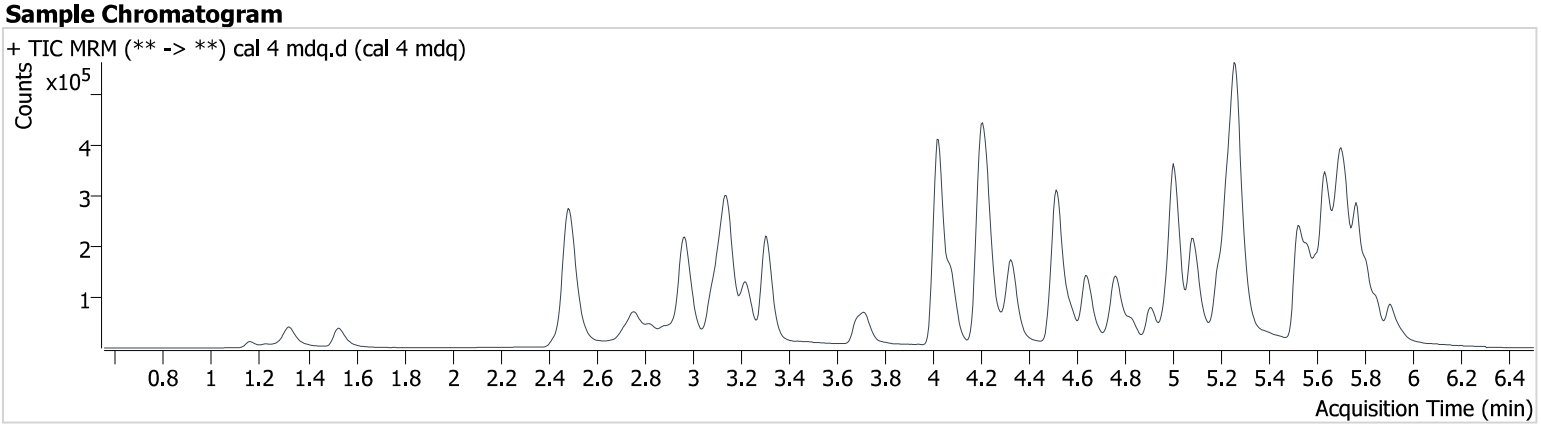
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	95256	4760.6	277.5	6834.0	109261	25.703	ng/ml
Fentanyl	5.093	11202	940.7	115.1	4972.0	154132	2.505	ng/ml
Ketamine	4.025	78343	4262.4	34.8	586.7	149747	24.459	ng/ml
Methamphetamine	3.153	222796	1029.3	37.5	626.7	321763	24.559	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	cal 4 mdq.d
Type	Cal	**Sample**	cal 4 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-D5	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 9:25:57 AM		
Sample Info.			

Sample Chromatogram



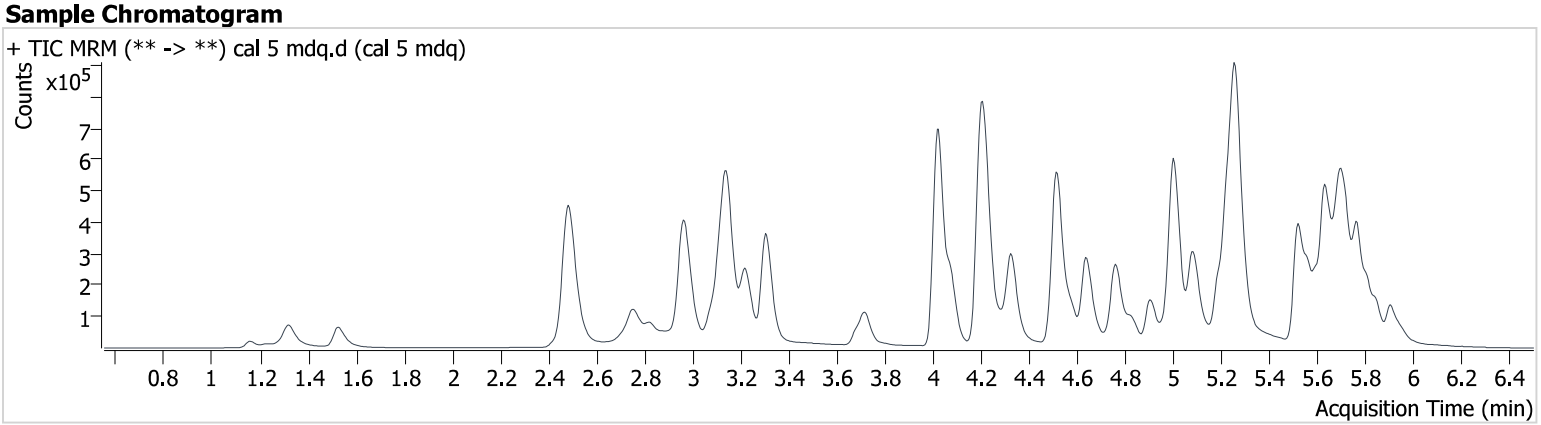
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	173739	3030.2	270.8	959933.0	96951	54.102	ng/ml
Fentanyl	5.093	21314	562.2	115.5	244555.0	146845	4.903	ng/ml
Ketamine	4.025	143483	3877.7	34.8	1338.6	137158	49.958	ng/ml
Methamphetamine	3.148	379274	∞	38.4	2936.3	295191	51.648	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	cal 5 mdq.d
Type	Cal	**Sample**	cal 5 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-E5	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 9:34:56 AM		
Sample Info.			

Sample Chromatogram



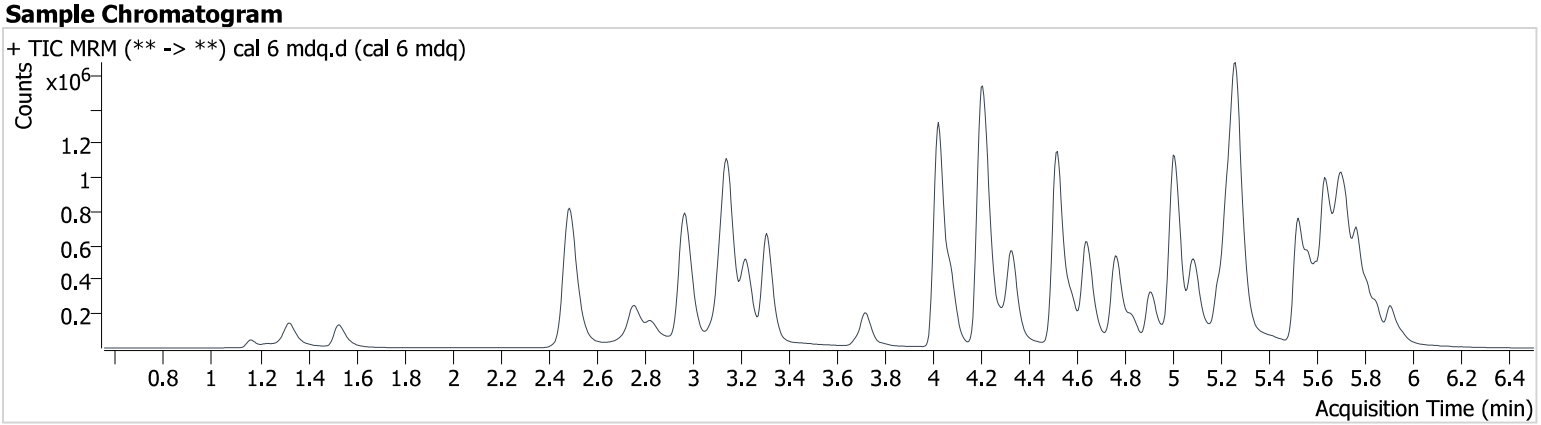
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	346921	18277.7	274.6	21337.5	101067	104.731	ng/ml
Fentanyl	5.093	42160	1264.1	116.7	∞	140730	10.016	ng/ml
Ketamine	4.025	297028	9603.4	34.8	∞	140239	102.223	ng/ml
Methamphetamine	3.148	769228	15275.6	37.6	∞	310774	106.080	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	cal 6 mdq.d
Type	Cal	**Sample**	cal 6 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-F5	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 9:43:56 AM		
Sample Info.			

Sample Chromatogram



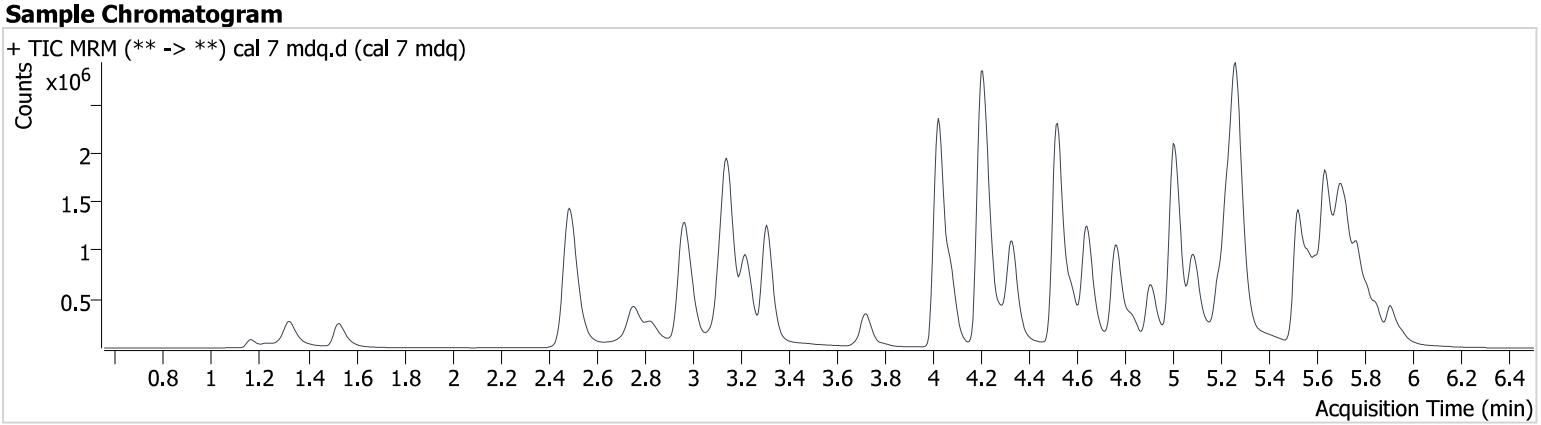
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	716700	22414.6	270.0	106866.7	88714	248.119	ng/ml
Fentanyl	5.098	91410	2533.5	119.4	7784.3	127684	23.797	ng/ml
Ketamine	4.025	617099	48085.1	34.7	7140.4	118377	253.134	ng/ml
Methamphetamine	3.148	1583970	∞	37.9	∞	268474	262.682	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	cal 7 mdq.d
Type	Cal	**Sample**	cal 7 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-G5	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 9:52:55 AM		
Sample Info.			

Sample Chromatogram



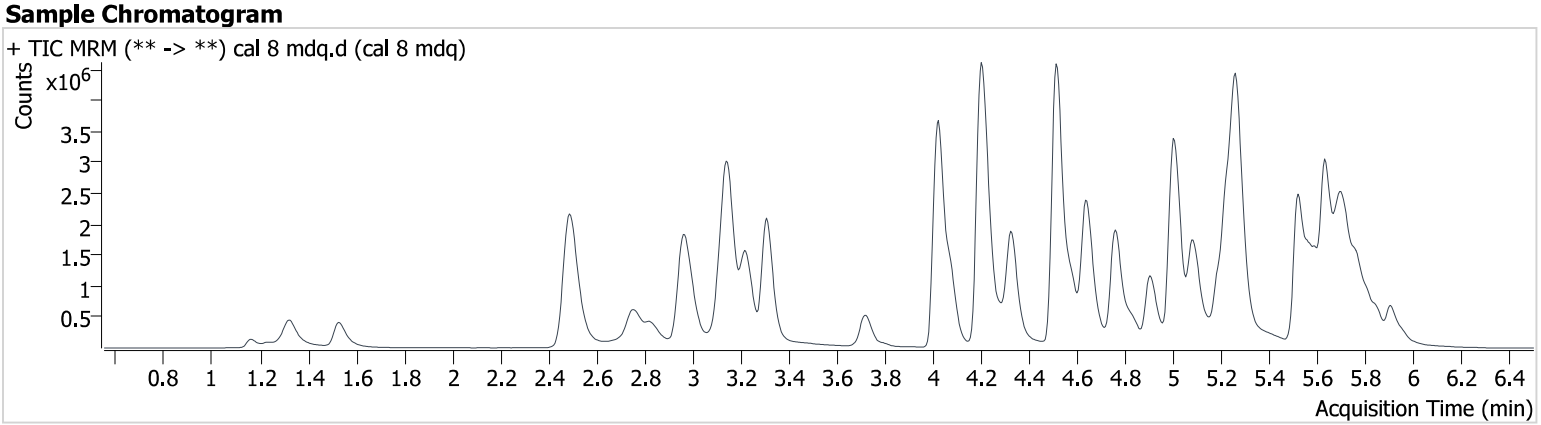
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	1251342	107816.2	262.5	178342.2	88108	437.099	ng/ml
Fentanyl	5.098	181939	34524.9	116.2	∞	121265	49.764	ng/ml
Ketamine	4.025	1174961	206894.6	35.1	20504.0	115431	495.270	ng/ml
Methamphetamine	3.148	2963849	∞	38.8	26185.1	278003	480.403	ng/ml

AM #28 Multi-Drug Quant. Results

Batch results D:\MassHunter\Data\2022\am 27-28\101922 lower volume\QuantResults\mdq.batch.bin
Calibration Last Update 10/20/2022 1:32:57 PM

Instrument	69679	**Data File**	cal 8 mdq.d
Type	Cal	**Sample**	cal 8 mdq
Acq. Method	mdqp1 1-21-21long.m	**Operator**	Anne Nord
Sample Position	P2-H5	**Comment**	
Injection Volume	0.5		
Acq. Date-Time	10/20/2022 10:01:55 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
Amphetamine	2.969	1916418	79926.9	256.6	379474.8	82447	716.142	ng/ml
Fentanyl	5.098	315330	4408.1	118.4	21287.0	102905	101.534	ng/ml
Ketamine	4.025	1905425	203787.3	34.5	13914.3	96464	962.091	ng/ml
Methamphetamine	3.148	5276810	∞	37.6	3648906.9	260718	918.390	ng/ml