

REVIEWED
By tsalazar at 10:32 am, Jan 24, 2019

1/17/2019

Worklist: 2890

<u>LAB CASE</u>	<u>ITEM</u>	<u>TASK ID</u>	<u>DESCRIPTION</u>
C2018-2560	1	137513	AM 28 Blood Multi-Drug Quant Panel 2 by LC-Q



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

Extraction Date: 1/15/19

Analyst: Anne Nord

Plate lot#: 0530533

Plate Expiration: May 21 2019

Mobile phase A: 5mM Amm Form + 0.01% FA
0.5M Ammonium Hydroxide

Mobile phase B: 0.01% Formic Acid in MeOH
Ethyl Acetate 20% Methanol in Water

Blank Blood Lot: amn12518

Column: Agilent 120 EC-C18 (2.1x 100-4um)

LCMS-QQQ ID: 62340

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:

Analytic:

- 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
- 2. Pipette **250µL blood (calibrated pipette) Pipette ID: 1926134** in wells of analytical (standards) plate.
- 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 66759*
- 4. Pipette **250µL 00.5M ammonium hydroxide** in wells of analytical plate.
- 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
- 6. Transfer **300µL of blood+base** 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: 66792
- 8. Wait 5 minutes.
- 9. Add **900uL ethyl acetate**.
- 10. Wait 5 minutes.
- 11. Apply positive pressure for approx. 15 seconds. *(10-15 PSI- Selector to the left)*.
- 12. Add **900uL ethyl acetate**.
- 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: 66819
- 16. Reconstitute in **100µL 20% MeOH** and heat seal plate with foil. Place in autosampler and run worklist.

Post-Analytic

- 1. Create batch and process data.
Worklist path: 2019 Data\011519 mdq Batch Name: mdqp2b
- 2. Make necessary changes to integration limits
- 3. Integration linear and R² 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:

Desipramine, MDA and MDMA were the only compounds evaluated in this run.

Did not extract or run calibrators at the 250 and 500 levels, I had a pipetting error and invalidated these calibrators.



Toxicology AM method 28 panel 2 external prep information
working solution 14706 ng/ml in meoh Doxepin, desipramine, chlordiazepoxide, paroxetine
Stock solution 1mg/ml 150 ul each in 9700 ul meOH
Ppd 8/8/18 Exp: 8/8/19 lot 8819 by AMN

Drug	lot	expiration
Doxepine	fn01281502	2/1/2020
Desipramine	fn10231502	10/1/2020
Chlordiazepoxide	fe07241502	8/1/2020
Paroxetine	fn05111505	6/1/2020

AM 28 control 50 ul working solution lot (8819) in 9950 ul blood lot (18G207D7)
ppd 8/8/18 Exp 8/8/19 by AMN Concentration 73.53 ng/ml each

ISP FORENSICS - Cd'A Instrument # 62340

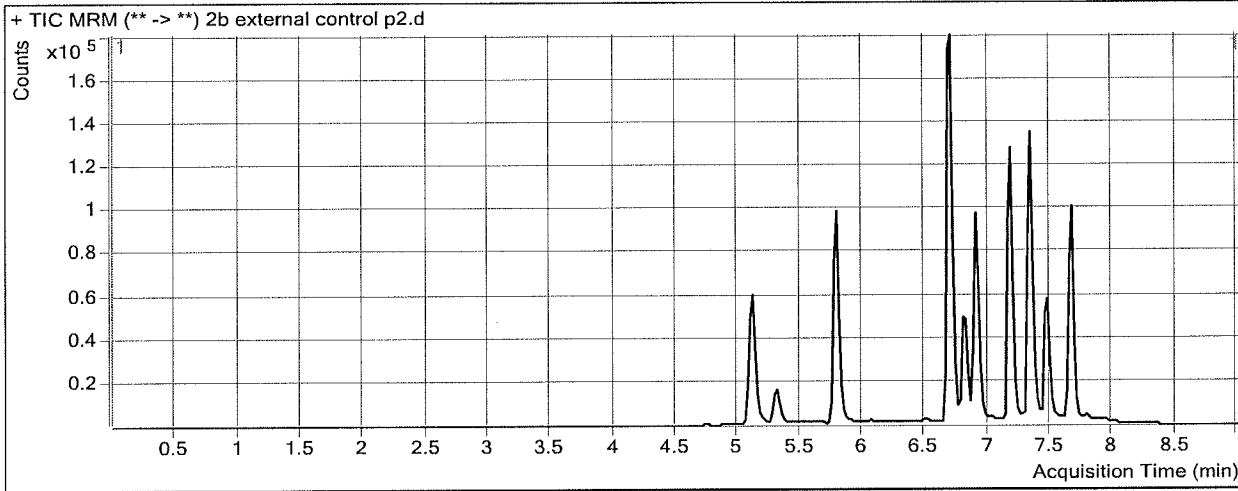
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 04:05 **Data File** 2b external control p2.d
Sample Type Sample **Sample Name** 2b external control p2
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-F6 **Sample Info**
Inj Vol -1 **Comment** AM 28 ~~meperidine 50ng~~ see external control preparation sheet.

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
Desipramine	Desipramine-D3	7.666	77052	56235	1.3702	68.7990

ISP FORENSICS - Cd'A Instrument # 62340

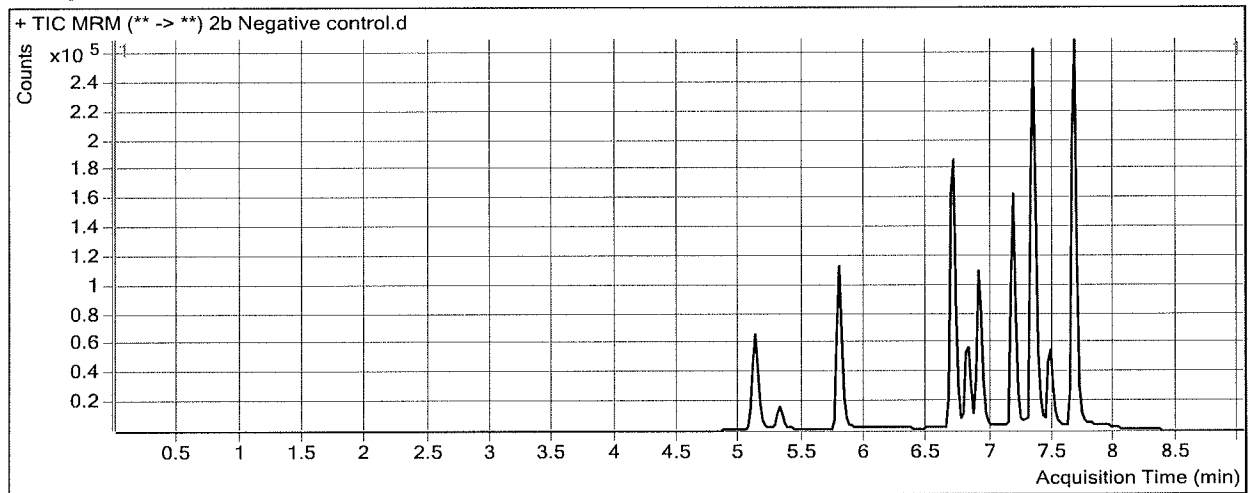
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 03:52 **Data File** 2b Negative control.d
Sample Type Sample **Sample Name** 2b Negative control
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-E6 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



ISP FORENSICS - Cd'A Instrument # 62340

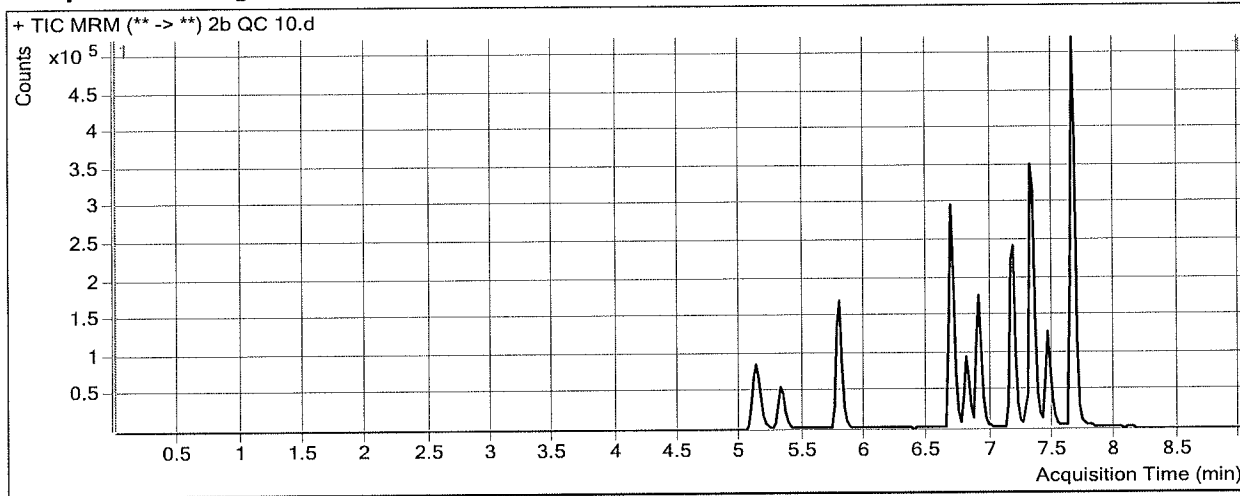
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 04:18 **Data File** 2b QC 10.d
Sample Type QC **Sample Name** 2b QC 10
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-A6 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.139	87994	264243	0.3330	11.2508
MDMA	MDMA-D6	5.334	93079	61576	1.5116	9.6750
Desipramine	Desipramine-D3	7.666	111835	547159	0.2044	10.2534

ISP FORENSICS - Cd'A Instrument # 62340

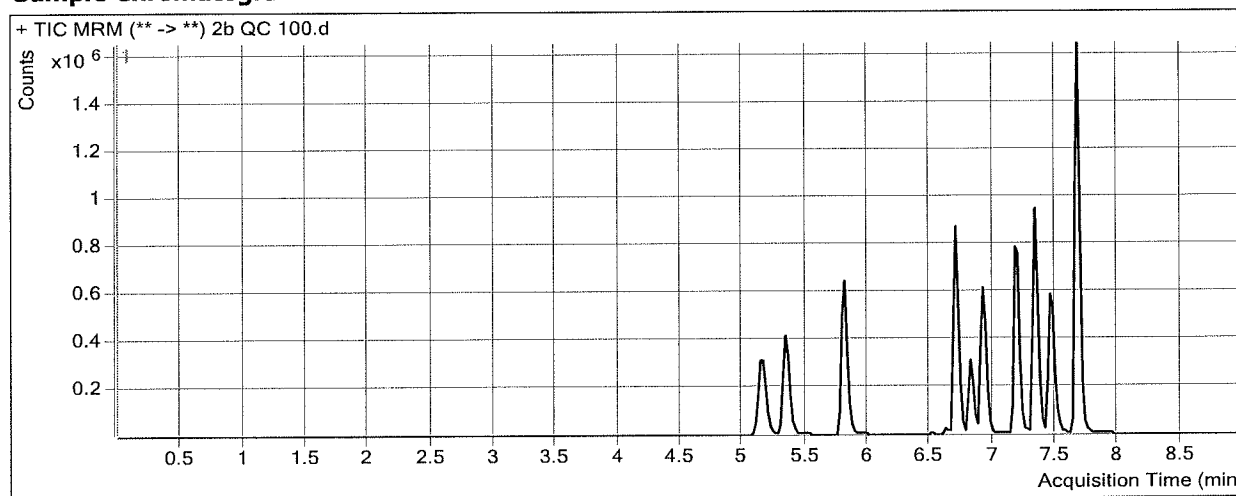
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 04:56 **Data File** 2b QC 100.d
Sample Type QC **Sample Name** 2b QC 100
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-B6 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.159	753683	230420	3.2709	113.4111
MDMA	MDMA-D6	5.334	879169	56000	15.6995	107.8701
Desipramine	Desipramine-D3	7.666	903359	469772	1.9230	96.5608

ISP FORENSICS - Cd'A Instrument # 62340

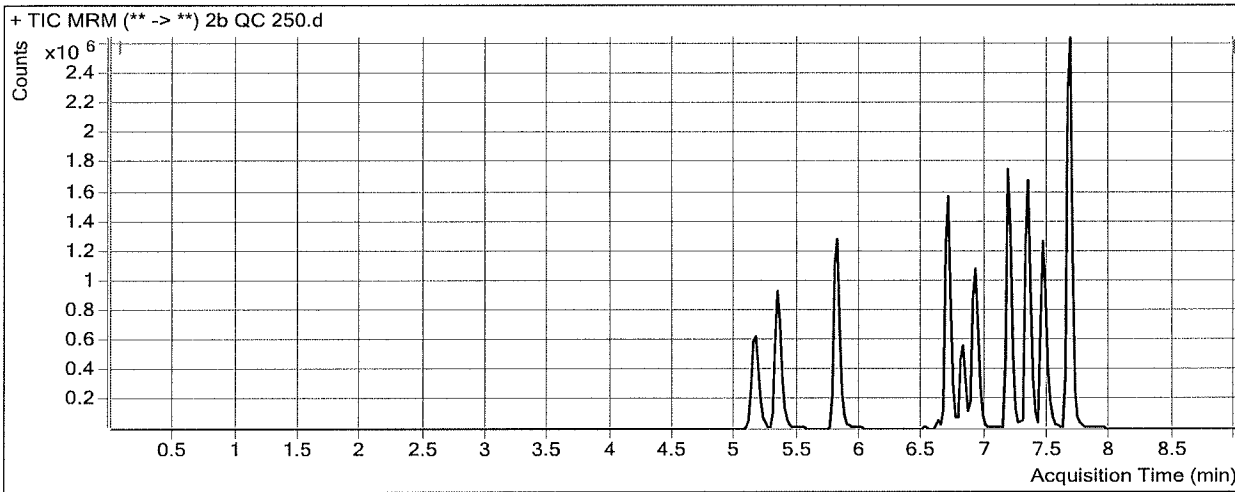
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 05:08 **Data File** 2b QC 250.d
Sample Type QC **Sample Name** 2b QC 250
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-C6 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.159	1546122	205752	7.5145	260.9747
MDMA	MDMA-D6	5.334	1992300	52590	37.8839	261.4101
Desipramine	Desipramine-D3	7.666	1696465	365738	4.6385	232.9335

ISP FORENSICS - Cd'A Instrument # 62340

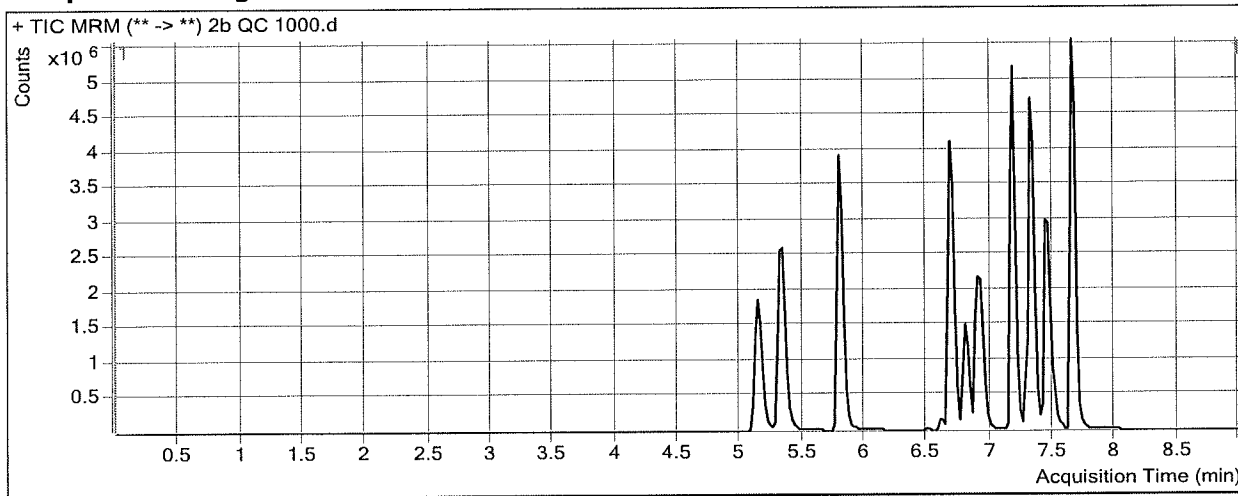
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 05:21 **Data File** 2b QC 1000.d
Sample Type QC **Sample Name** 2b QC 1000
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-D6 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.139	4763047	177421	26.8460	933.1918
MDMA	MDMA-D6	5.334	6625389	46088	143.7543	994.1465
Desipramine	Desipramine-D3	7.666	3996905	200964	19.8887	998.8006

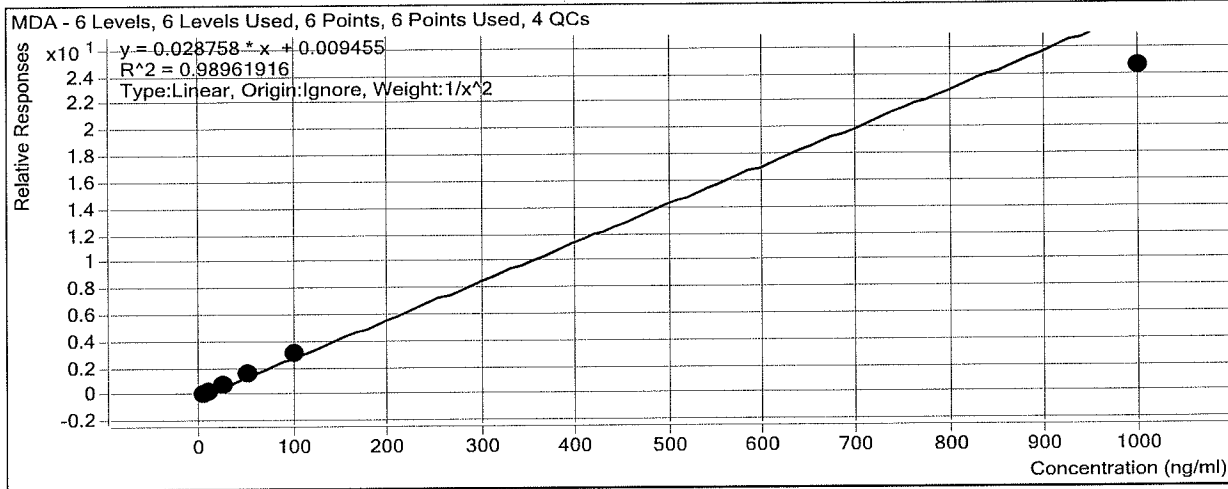
ISP Forensics Calibration Curve Report

Batch Data Path
Last Calib Update

D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
1/16/2019 11:03 AM

Analyst Name ISP TOX

Target Compound *MDA*
Internal Standard *MDA-D5*



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
2b cal 1-5ng	1	<input checked="" type="checkbox"/>	5	4.8	96.0
2b cal 2-10ng	2	<input checked="" type="checkbox"/>	10	10.6	106.3
2b QC 10	2	<input checked="" type="checkbox"/>	10	11.3	112.5
2b cal 3-25ng	3	<input checked="" type="checkbox"/>	25	24.9	99.7
2b cal 4-50ng	4	<input checked="" type="checkbox"/>	50	53.2	106.4
2b cal 5-100ng	5	<input checked="" type="checkbox"/>	100	106.3	106.3
2b QC 100	5	<input checked="" type="checkbox"/>	100	113.4	113.4
2b QC 250	6	<input checked="" type="checkbox"/>	250	261.0	104.4
2b cal 8-1000ng	8	<input checked="" type="checkbox"/>	1000	853.0	85.3
2b QC 1000	8	<input checked="" type="checkbox"/>	1000	933.2	93.3

ISP Forensics Calibration Curve Report

Batch Data Path
Last Calib Update

D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
1/16/2019 11:03 AM

Analyst Name

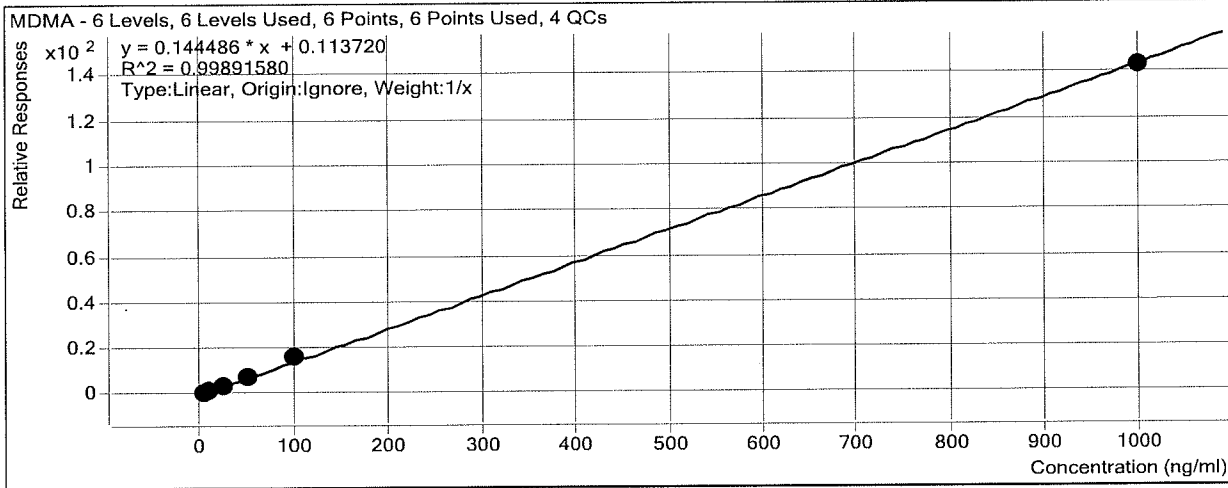
ISP TOX

Target Compound

MDMA

Internal Standard

MDMA-D6



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
2b cal 1-5ng	1	<input checked="" type="checkbox"/>	5	4.3	85.9
2b cal 2-10ng	2	<input checked="" type="checkbox"/>	10	10.1	101.2
2b QC 10	2	<input checked="" type="checkbox"/>	10	9.7	96.7
2b cal 3-25ng	3	<input checked="" type="checkbox"/>	25	24.9	99.6
2b cal 4-50ng	4	<input checked="" type="checkbox"/>	50	52.7	105.3
2b cal 5-100ng	5	<input checked="" type="checkbox"/>	100	109.1	109.1
2b QC 100	5	<input checked="" type="checkbox"/>	100	107.9	107.9
2b QC 250	6	<input checked="" type="checkbox"/>	250	261.4	104.6
2b cal 8-1000ng	8	<input checked="" type="checkbox"/>	1000	989.0	98.9
2b QC 1000	8	<input checked="" type="checkbox"/>	1000	994.1	99.4

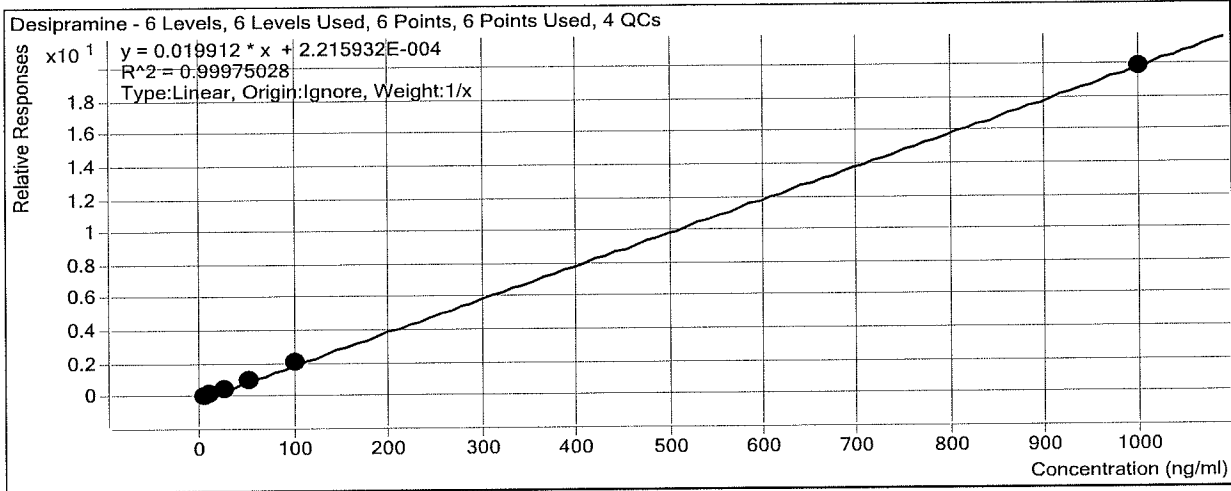
ISP Forensics Calibration Curve Report

Batch Data Path
Last Calib Update

D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
1/16/2019 11:03 AM

Analyst Name ISP TOX

Target Compound *Desipramine*
Internal Standard *Desipramine-D3*



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
2b cal 1-5ng	1	<input checked="" type="checkbox"/>	5	5.0	99.1
2b cal 2-10ng	2	<input checked="" type="checkbox"/>	10	9.5	95.3
2b QC 10	2	<input checked="" type="checkbox"/>	10	10.3	102.5
2b cal 3-25ng	3	<input checked="" type="checkbox"/>	25	24.5	98.2
2b cal 4-50ng	4	<input checked="" type="checkbox"/>	50	52.2	104.4
2b cal 5-100ng	5	<input checked="" type="checkbox"/>	100	103.5	103.5
2b QC 100	5	<input checked="" type="checkbox"/>	100	96.6	96.6
2b QC 250	6	<input checked="" type="checkbox"/>	250	232.9	93.2
2b cal 8-1000ng	8	<input checked="" type="checkbox"/>	1000	995.3	99.5
2b QC 1000	8	<input checked="" type="checkbox"/>	1000	998.8	99.9

ISP FORENSICS - Cd'A Instrument # 62340

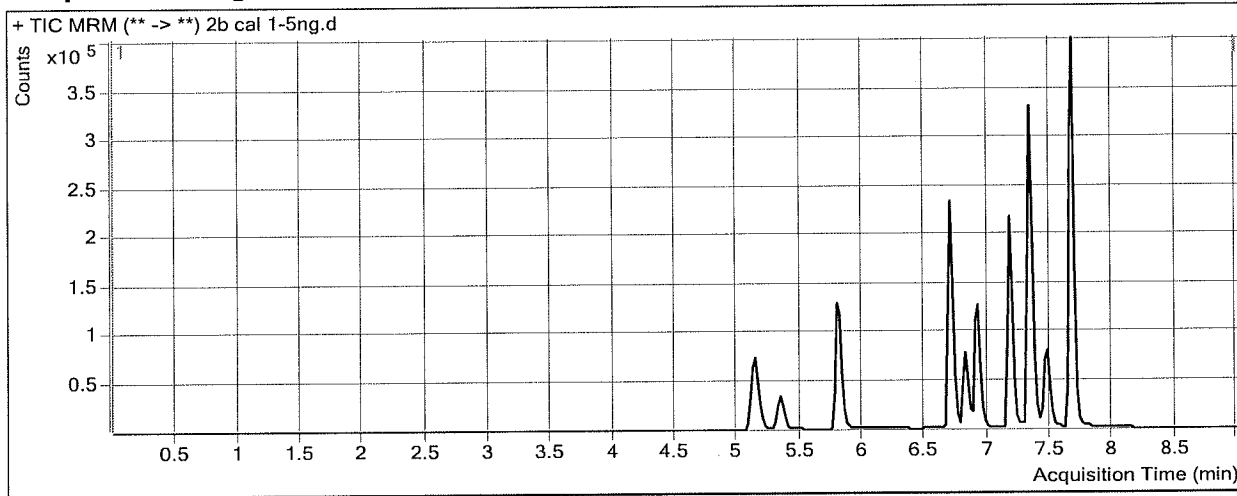
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 02:36 **Data File** 2b cal 1-5ng.d
Sample Type Calibration **Sample Name** 2b cal 1-5ng
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-A5 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.159	40311	273215	0.1475	4.8017
MDMA	MDMA-D6	5.334	44055	60022	0.7340	4.2928
Desipramine	Desipramine-D3	7.666	49202	497800	0.0988	4.9526

ISP FORENSICS - Cd'A Instrument # 62340

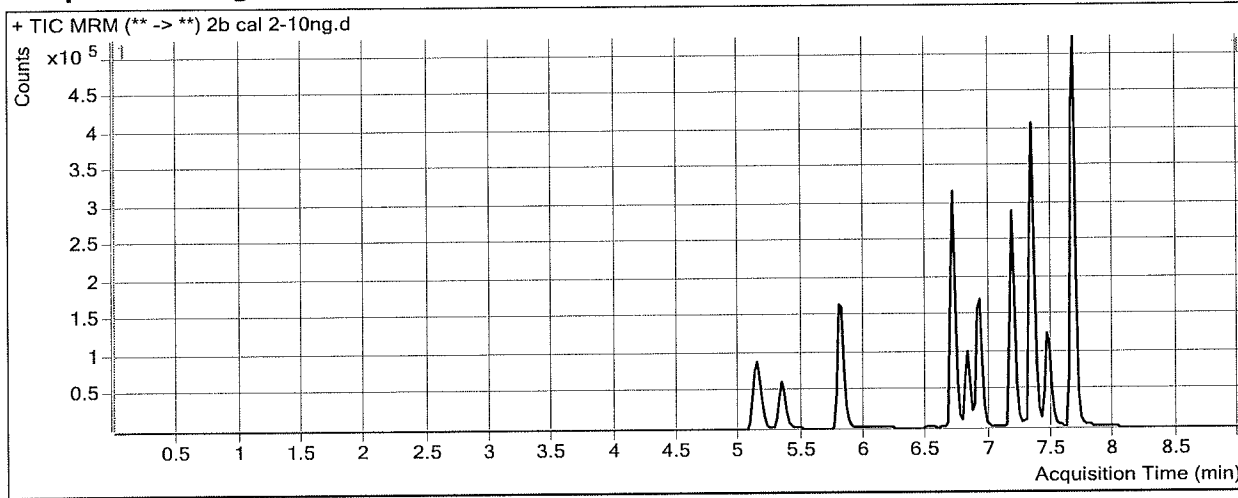
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 02:49 **Data File** 2b cal 2-10ng.d
Sample Type Calibration **Sample Name** 2b cal 2-10ng
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-B5 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.159	92323	292946	0.3152	10.6301
MDMA	MDMA-D6	5.334	104875	66524	1.5765	10.1240
Desipramine	Desipramine-D3	7.666	108166	569209	0.1900	9.5322

ISP FORENSICS - Cd'A Instrument # 62340

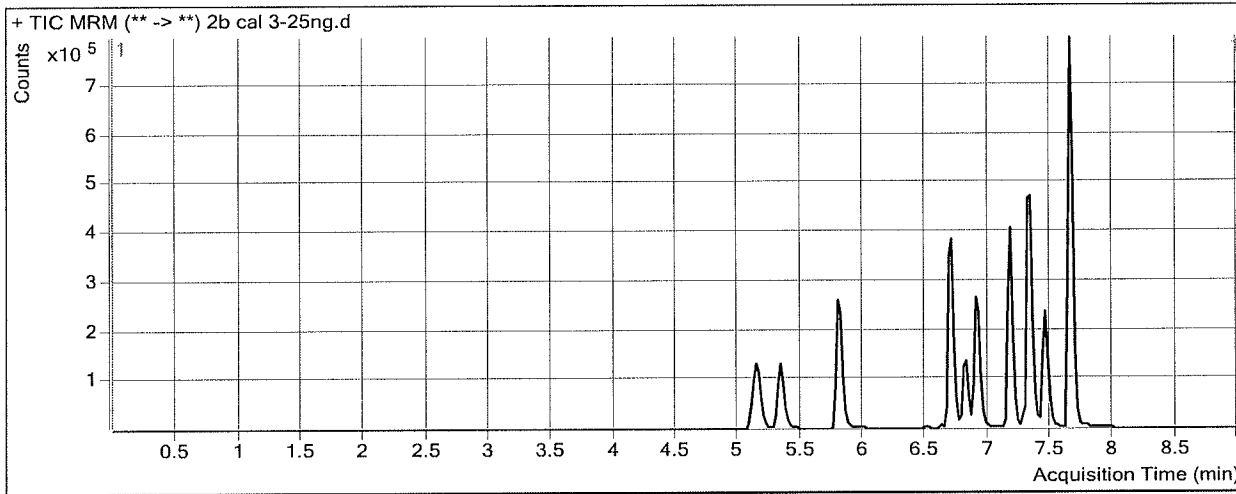
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 03:02 **Data File** 2b cal 3-25ng.d
Sample Type Calibration **Sample Name** 2b cal 3-25ng
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-C5 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.159	213914	294611	0.7261	24.9197
MDMA	MDMA-D6	5.334	250621	67513	3.7122	24.9051
Desipramine	Desipramine-D3	7.666	284190	581243	0.4889	24.5432

ISP FORENSICS - Cd'A Instrument # 62340

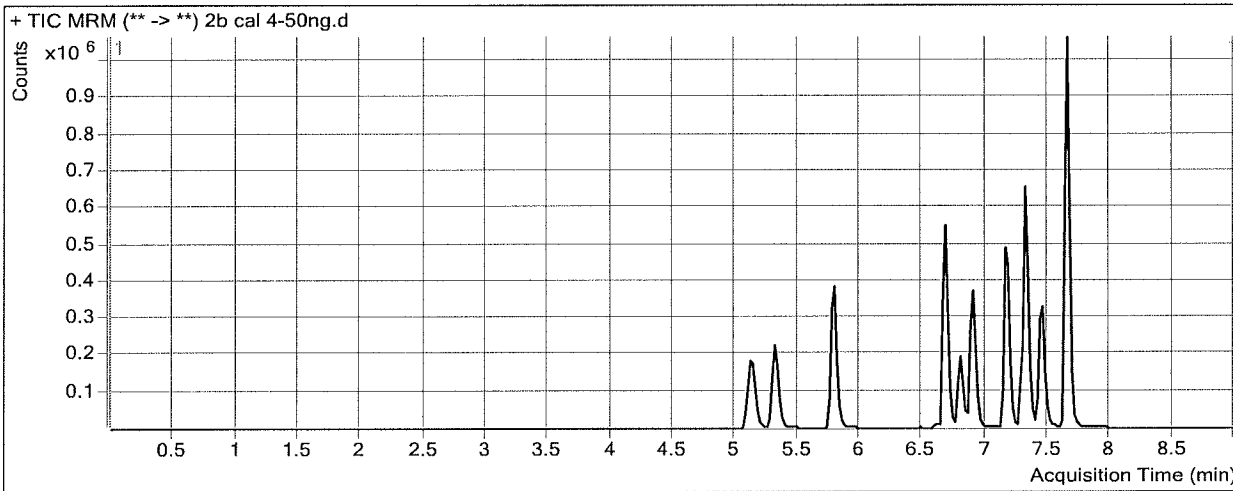
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 03:14 **Data File** 2b cal 4-50ng.d
Sample Type Calibration **Sample Name** 2b cal 4-50ng
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-D5 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.139	380529	247271	1.5389	53.1841
MDMA	MDMA-D6	5.314	454011	58786	7.7232	52.6655
Desipramine	Desipramine-D3	7.646	495675	476556	1.0401	52.2237

ISP FORENSICS - Cd'A Instrument # 62340

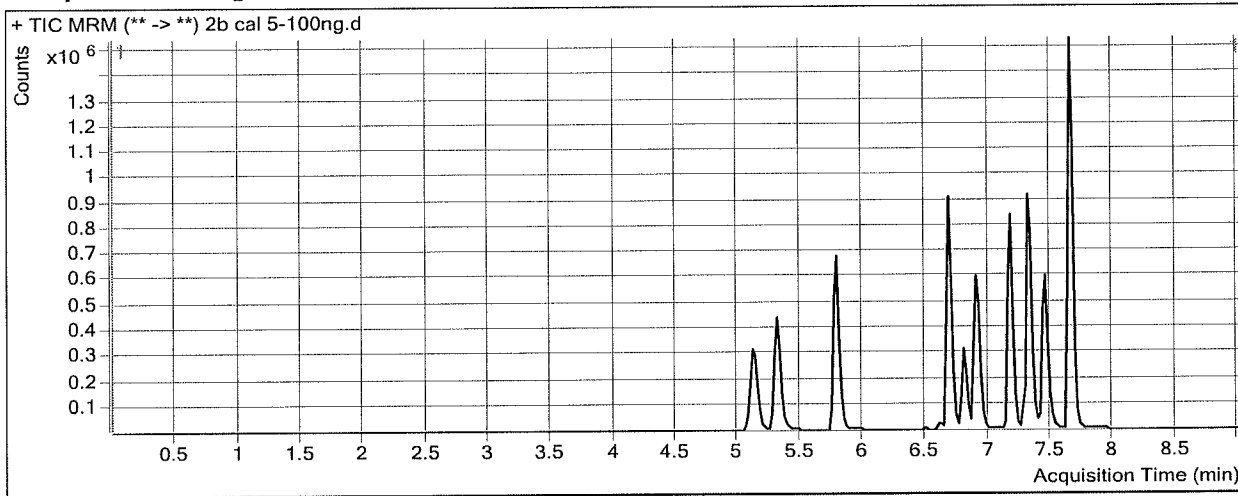
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 03:27 **Data File** 2b cal 5-100ng.d
Sample Type Calibration **Sample Name** 2b cal 5-100ng
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-E5 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.139	729144	237736	3.0670	106.3218
MDMA	MDMA-D6	5.314	920802	58018	15.8710	109.0570
Desipramine	Desipramine-D3	7.666	877172	425660	2.0607	103.4791

ISP FORENSICS - Cd'A Instrument # 62340

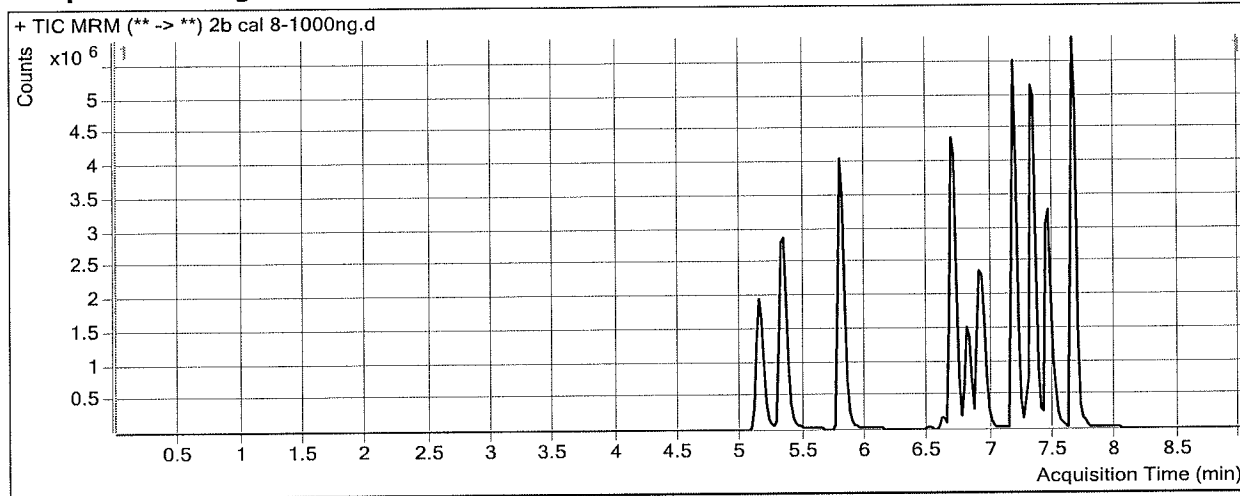
Multi-Drug Confirmatory Analysis Report

Batch Data Path D:\2019 Data\011519 mdq\QuantResults\mdqp2b.batch.bin
Analysis Time 1/16/2019 11:03 AM **Analyst Name** ISP Tox
Report Time 1/16/2019 11:09 AM **Reporter Name** ISP Tox
Last Calib Update 1/16/2019 11:03 AM **Batch State** Processed

Analysis Info

Acq Time 2019-01-16 03:40 **Data File** 2b cal 8-1000ng.d
Sample Type Calibration **Sample Name** 2b cal 8-1000ng
Dilution 1 **Acq Method** 2b mdq 618 .m
Position P2-H5 **Sample Info**
Inj Vol -1 **Comment** AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
MDA	MDA-D5	5.139	4836243	197087	24.5387	852.9588
MDMA	MDMA-D6	5.334	7347648	51381	143.0043	988.9557
Desipramine	Desipramine-D3	7.666	4219772	212922	19.8184	995.2693