reviewed 8/16/18 BWLee

8/9/2018

Worklist: 2628

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
C2018-1548	1	123972	AM 28 Blood Multi-Drug Quant Panel 2 by LC-Q(

C:\inetpub\wwwroot\LIMS reports\ORACLE WORKLIST RPT

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

Extraction Date: 8/8/18 Analyst: Anne Nord
Plate lot#: 0530563 Plate Expiration: May 21 2019

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: 18G207D7 **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: *08082018 mdq* Batch Name: *p2a mdq.*
☒ 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? See comments. Add Control data to QC tracking spreadsheet.
☒ 6. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports.

COMMENTS: *Only Chlordiazepoxide and Doxepin were evaluated in this run.*
1000 QC for chlordiazepoxide was not evaluated the top of the primary peak is missing and the top of the internal standard peak is missing.

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

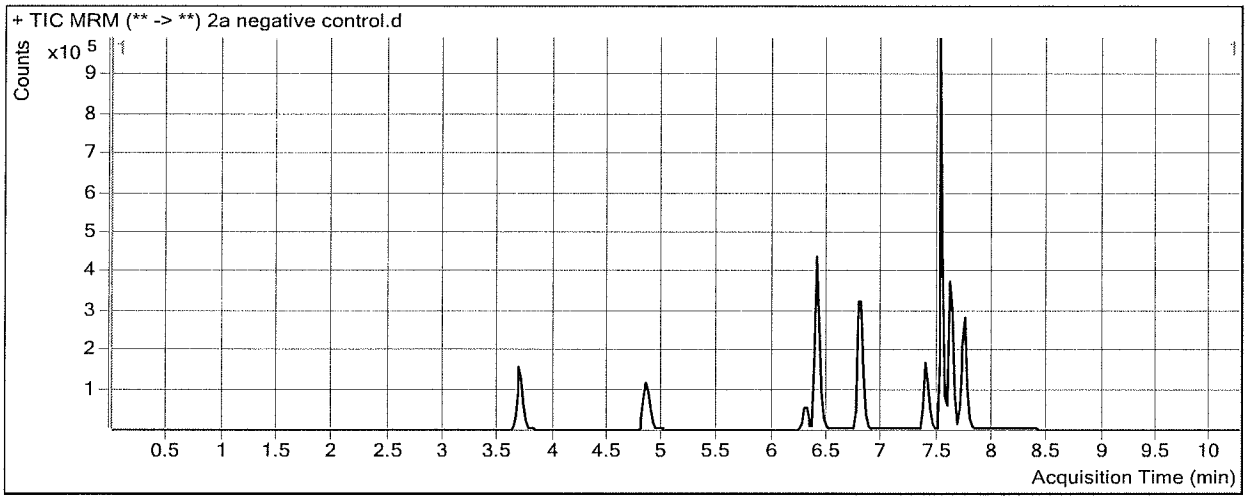
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 03:43	**Data File**	2a negative control.d
Sample Type	Sample	**Sample Name**	2a negative control
Dilution	1	**Acq Method**	2a MDQ Panel 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

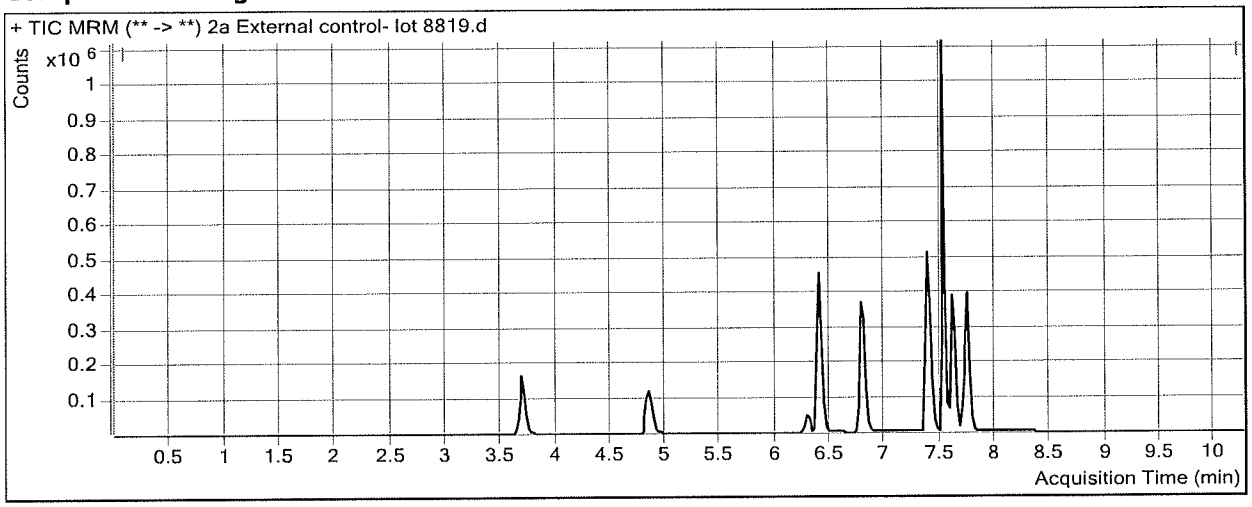
Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 04:08	**Data File**	2a External control- lot 8819.d
Sample Type	Sample	**Sample Name**	2a External control- lot 8819
Dilution	1	**Acq Method**	2a MDQ Panel 618.m
Position	P2-F6	**Sample Info**	
Inj Vol	-1	**Comment**	~~AM-28-mirtazapine 50ng, amitriptyline 50 ng~~

lot 8819 Doxepine, chlordiazepoxide, paroxetine desipramine.

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
Doxepin	Doxepin-D3	7.387	772280	494787	1.5608	66.5916
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	203957	258113	0.7902	75.4142

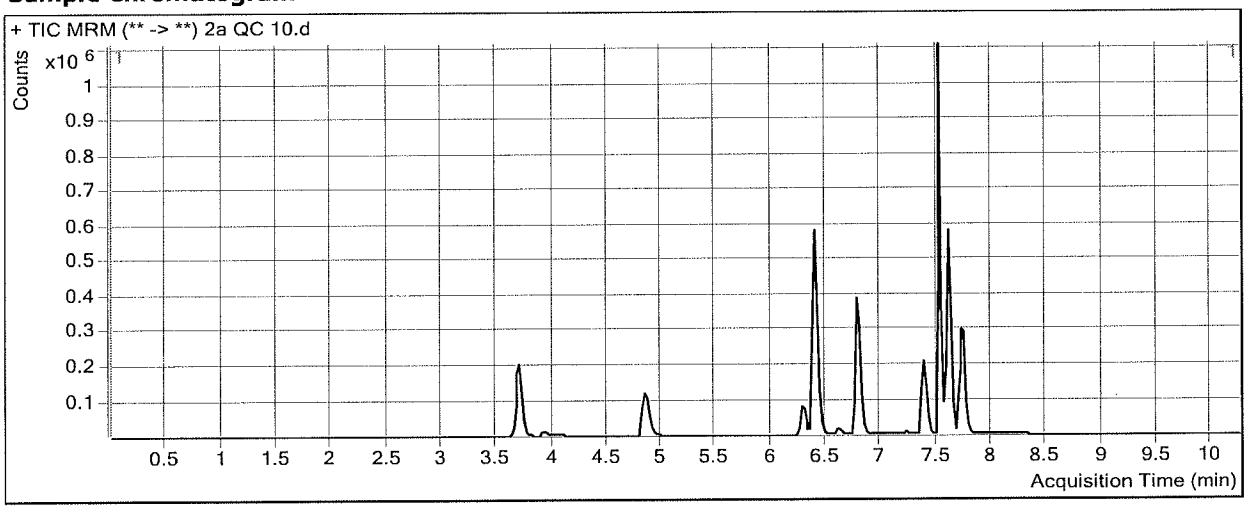
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 04:34	**Data File**	2a QC 10.d
Sample Type	QC	**Sample Name**	2a QC 10
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.387	107924	511015	0.2112	9.1392
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	34778	303365	0.1146	9.1118

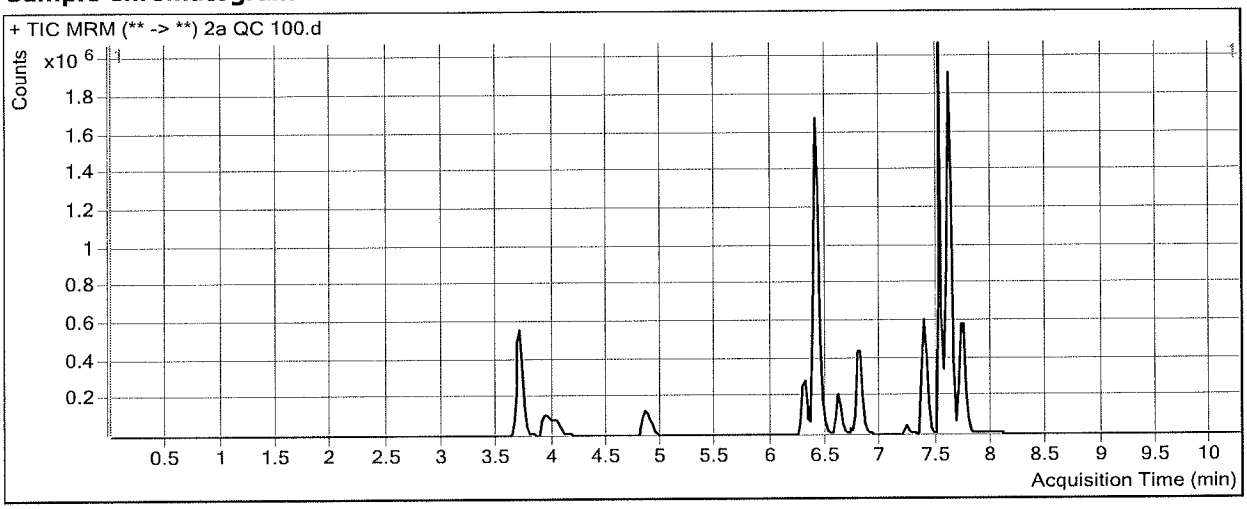
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 05:25	**Data File**	2a QC 100.d
Sample Type	QC	**Sample Name**	2a QC 100
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.387	1051157	480710	2.1867	93.2329
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	244691	234970	1.0414	100.0672

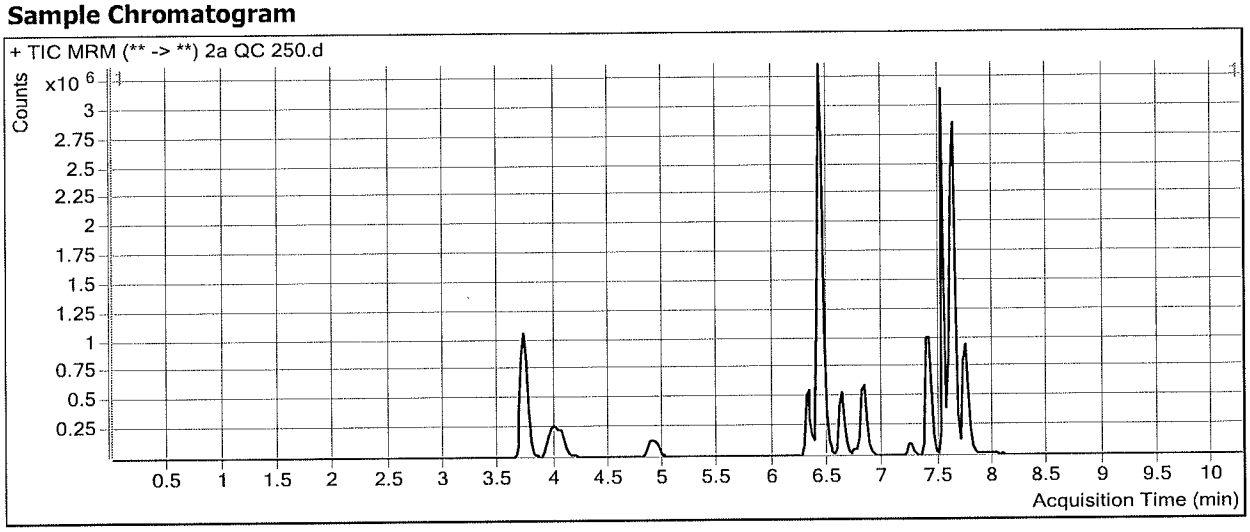
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 05:50	**Data File**	2a QC 250.d
Sample Type	QC	**Sample Name**	2a QC 250
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.408	2305180	403641	5.7110	243.2571
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	486172	160351	3.0319	295.4319

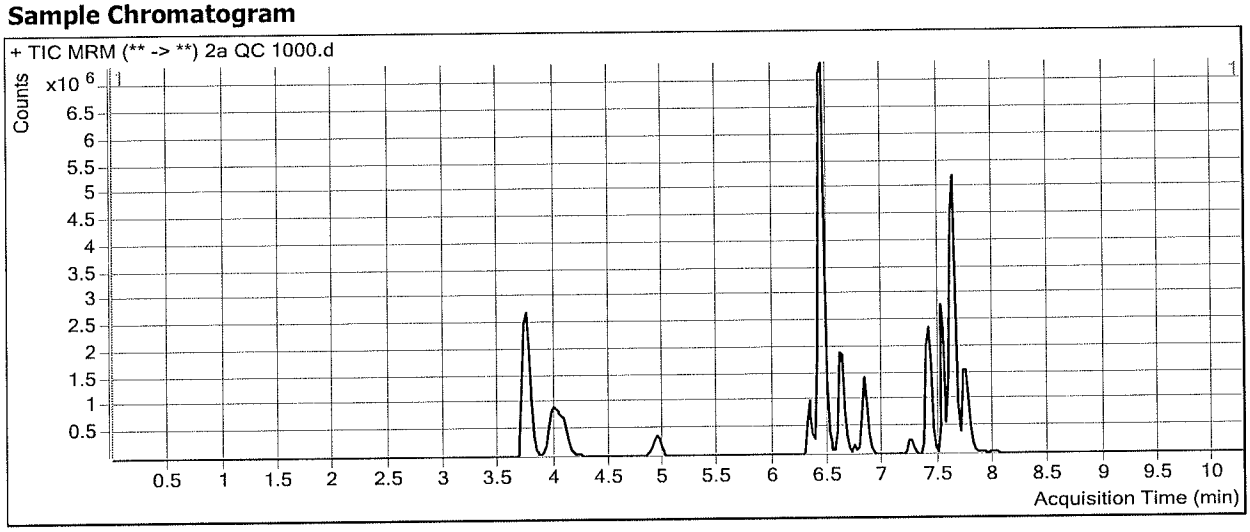
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 06:16	**Data File**	2a QC 1000.d
Sample Type	QC	**Sample Name**	2a QC 1000
Dilution	1	**Acq Method**	2a MDQ Panel 618.m
Position	P2-D6	**Sample Info**	
Inj Vol	-1	**Comment**	AM 28 Panel 2

Sample Chromatogram



Results

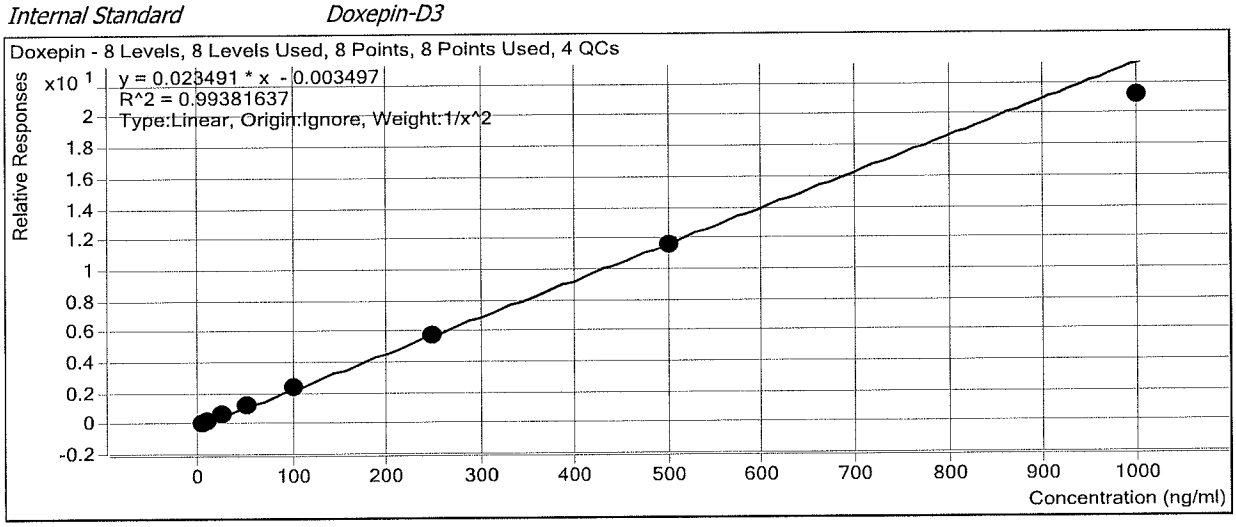
Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
Doxepin	Doxepin-D3	7.408	5853794	279586	20.9374	891.4250
Chlordiazepoxide	Chlordiazepoxide-D5	7.766	743216	75934	9.7876	~~958.4807~~

not evaluated
top of primary peak cut off
IS peak cut-off.

ISP Forensics
Calibration Curve Report

Batch Data Path D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin
Last Calib Update 8/9/2018 10:47 AM **Analyst Name** ISP TOX

Target Compound *Doxepin*
Internal Standard *Doxepin-D3*

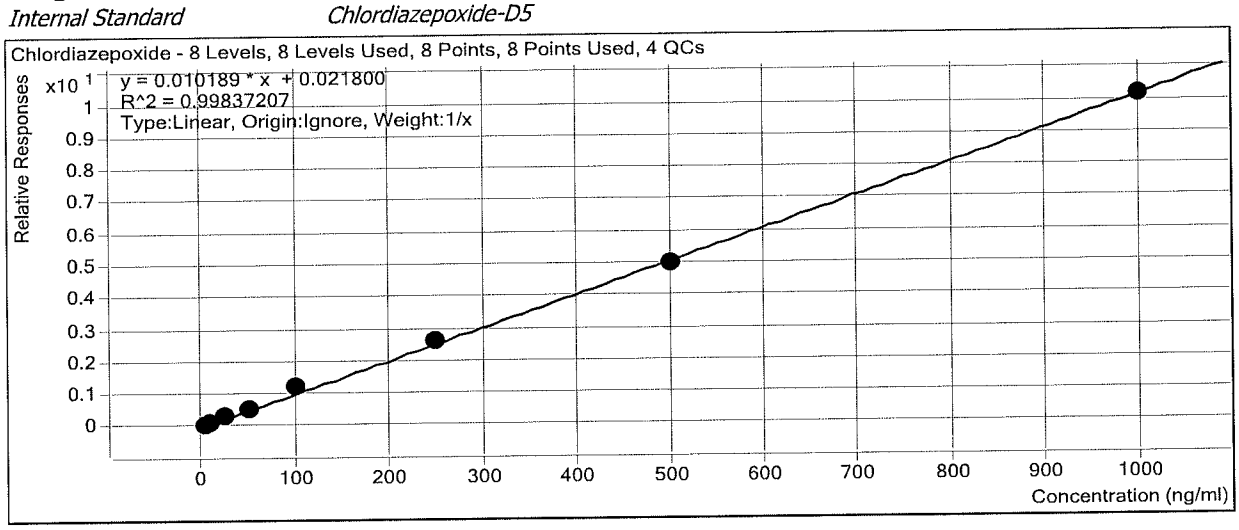


Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
2a cal 1-5ng	1	☑	5	4.7	93.4
2a cal 2-10ng	2	☑	10	11.2	112.0
2a QC 10	2	☑	10	9.1	91.4
2a cal 3-25ng	3	☑	25	25.1	100.3
2a cal 4-50ng	4	☑	50	52.1	104.3
2a cal 5-100ng	5	☑	100	104.2	104.2
2a QC 100	5	☑	100	93.2	93.2
2a cal 6-250ng	6	☑	250	243.5	97.4
2a QC 250	6	☑	250	243.3	97.3
2a cal 7-500ng	7	☑	500	491.6	98.3
2a cal 8-1000ng	8	☑	1000	901.6	90.2
2a QC 1000	8	☑	1000	891.4	89.1

ISP Forensics
Calibration Curve Report

Batch Data Path D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin
Last Calib Update 8/9/2018 10:47 AM **Analyst Name** ISP TOX

Target Compound *Chlordiazepoxide*
Internal Standard *Chlordiazepoxide-D5*



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
2a cal 1-5ng	1	☑	5	4.1	81.8
2a cal 2-10ng	2	☑	10	9.8	98.2
2a QC 10	2	☑	10	9.1	91.1
2a cal 3-25ng	3	☑	25	26.3	105.1
2a cal 4-50ng	4	☑	50	51.0	102.0
2a cal 5-100ng	5	☑	100	115.1	115.1
2a QC 100	5	☑	100	100.1	100.1
2a cal 6-250ng	6	☑	250	252.3	100.9
2a QC 250	6	☑	250	295.4	118.2
2a cal 7-500ng	7	☑	500	487.8	97.6
2a cal 8-1000ng	8	☑	1000	993.7	99.4
2a QC 1000	8	☑	1000	958.5	95.8

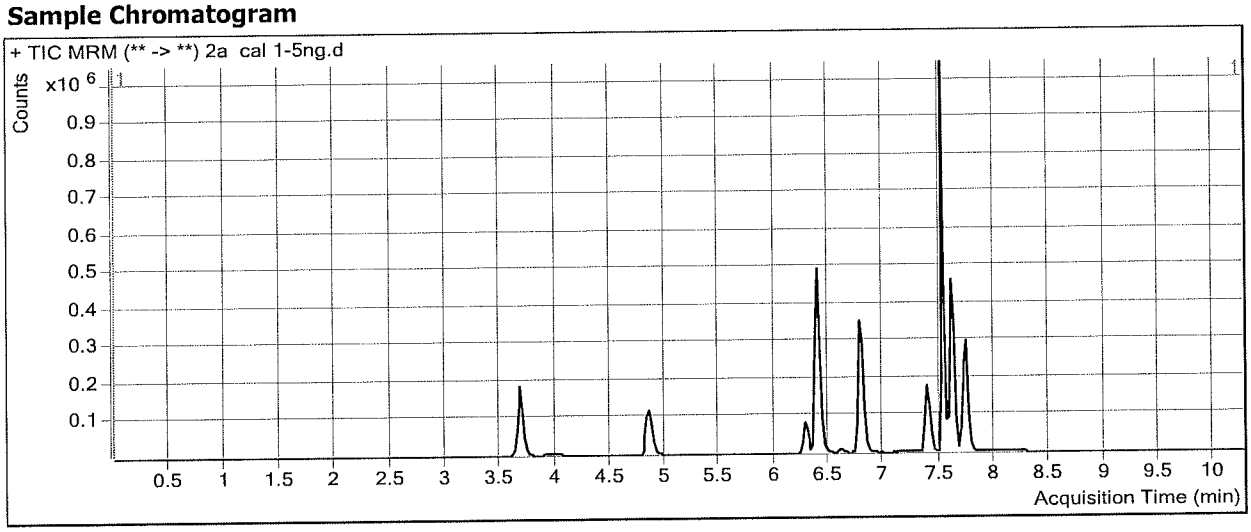
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 00:06	**Data File**	2a cal 1-5ng.d
Sample Type	Calibration	**Sample Name**	2a cal 1-5ng
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.387	46419	436831	0.1063	4.6723
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	17686	278556	0.0635	4.0918

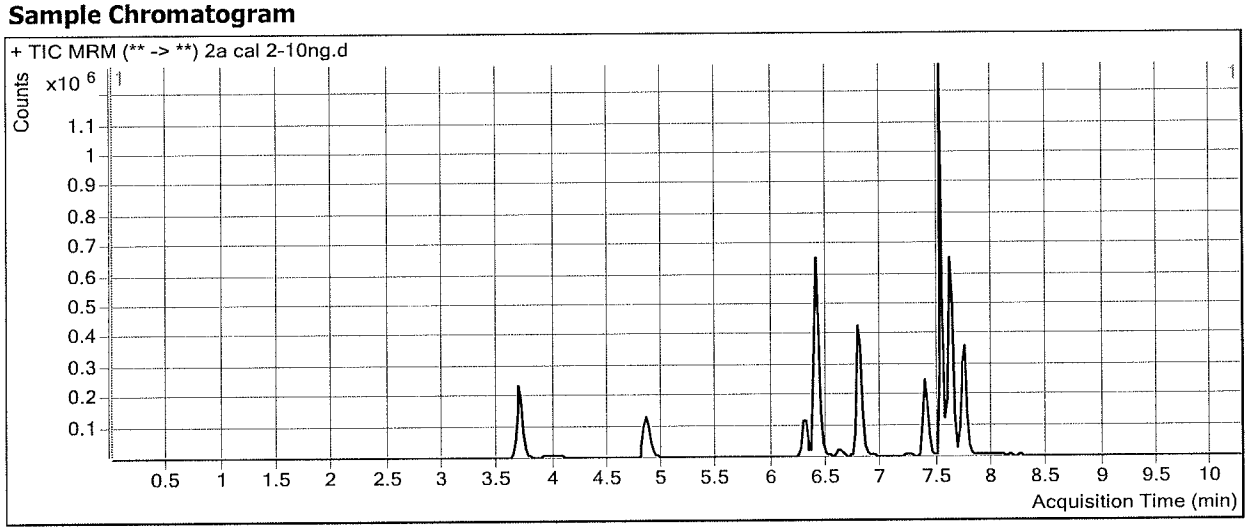
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 00:31	**Data File**	2a cal 2-10ng.d
Sample Type	Calibration	**Sample Name**	2a cal 2-10ng
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.387	132739	511503	0.2595	11.1958
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	40811	334950	0.1218	9.8189

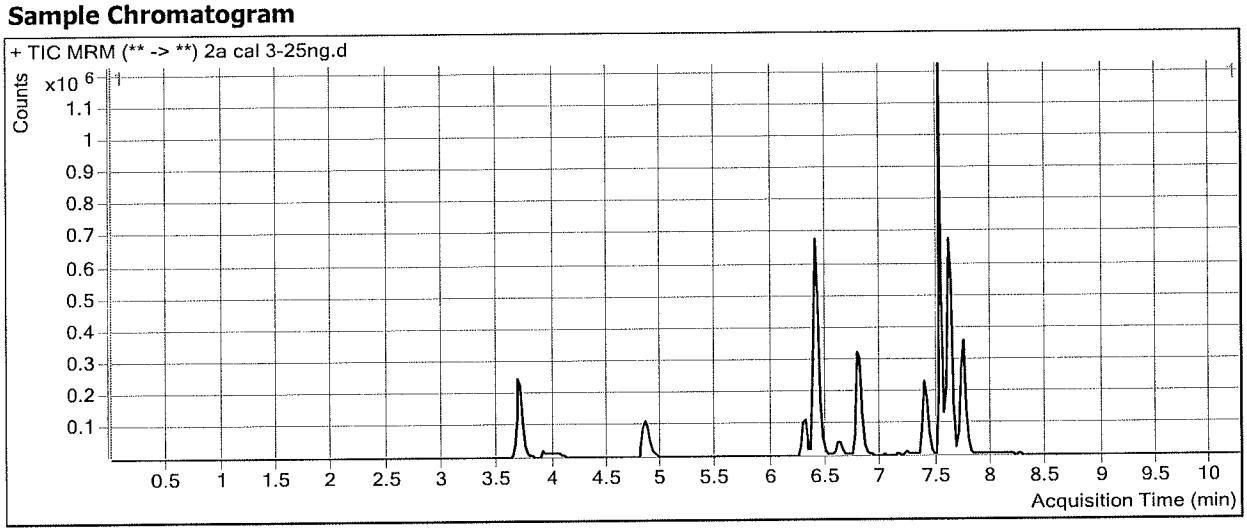
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 00:57	**Data File**	2a cal 3-25ng.d
Sample Type	Calibration	**Sample Name**	2a cal 3-25ng
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.387	222216	379538	0.5855	25.0725
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	76299	263597	0.2895	26.2691

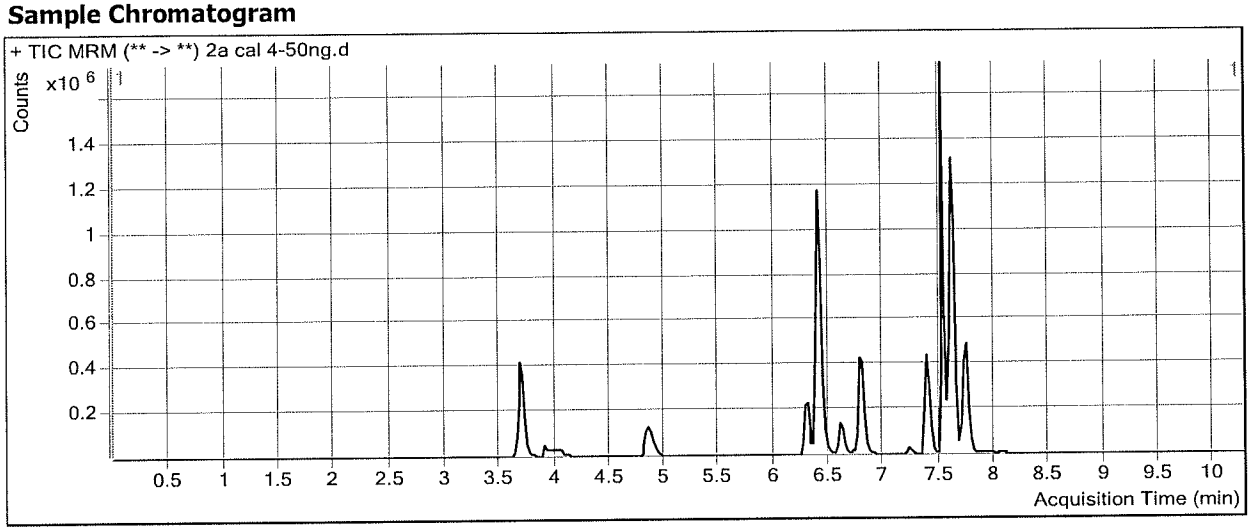
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 01:23	**Data File**	2a cal 4-50ng.d
Sample Type	Calibration	**Sample Name**	2a cal 4-50ng
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.387	588220	481722	1.2211	52.1286
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	149661	276471	0.5413	50.9895

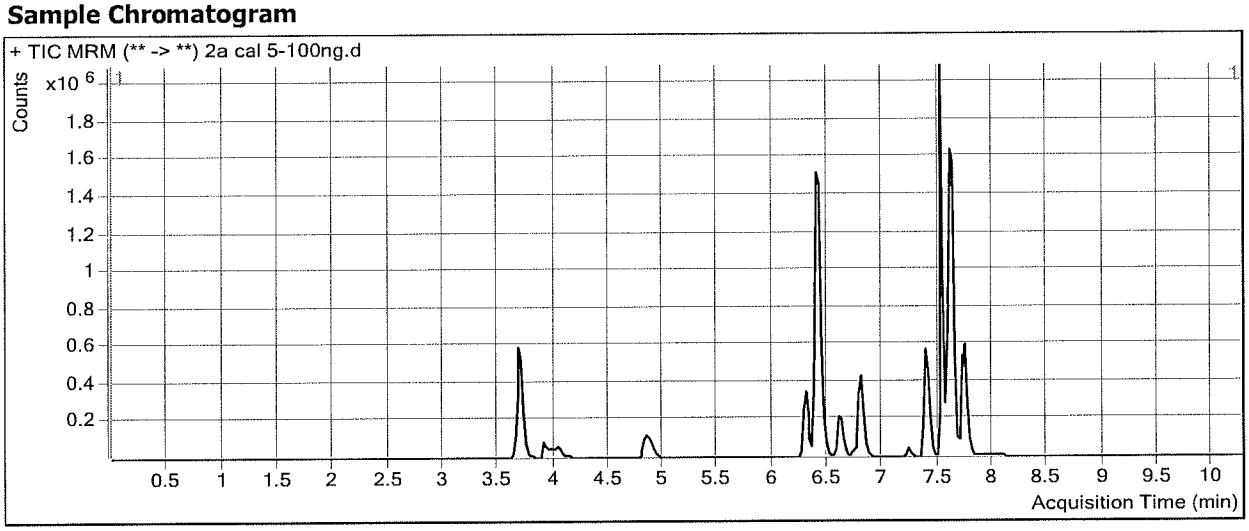
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 01:48	**Data File**	2a cal 5-100ng.d
Sample Type	Calibration	**Sample Name**	2a cal 5-100ng
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.387	984323	402788	2.4438	104.1771
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	231492	193812	1.1944	115.0879

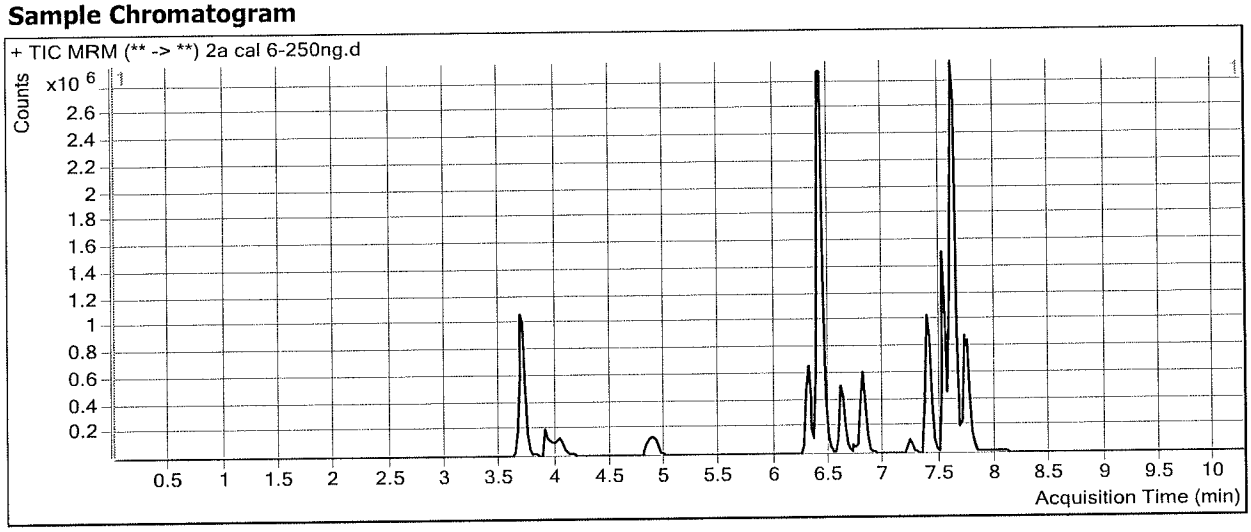
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 02:14	**Data File**	2a cal 6-250ng.d
Sample Type	Calibration	**Sample Name**	2a cal 6-250ng
Dilution	1	**Acq Method**	2a MDQ Panel 618.m
Position	P2-F5	**Sample Info**	
Inj Vol	-1	**Comment**	AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
Doxepin	Doxepin-D3	7.387	2158737	377608	5.7169	243.5085
Chlordiazepoxide	Chlordiazepoxide-D5	7.746	381196	147064	2.5920	252.2597

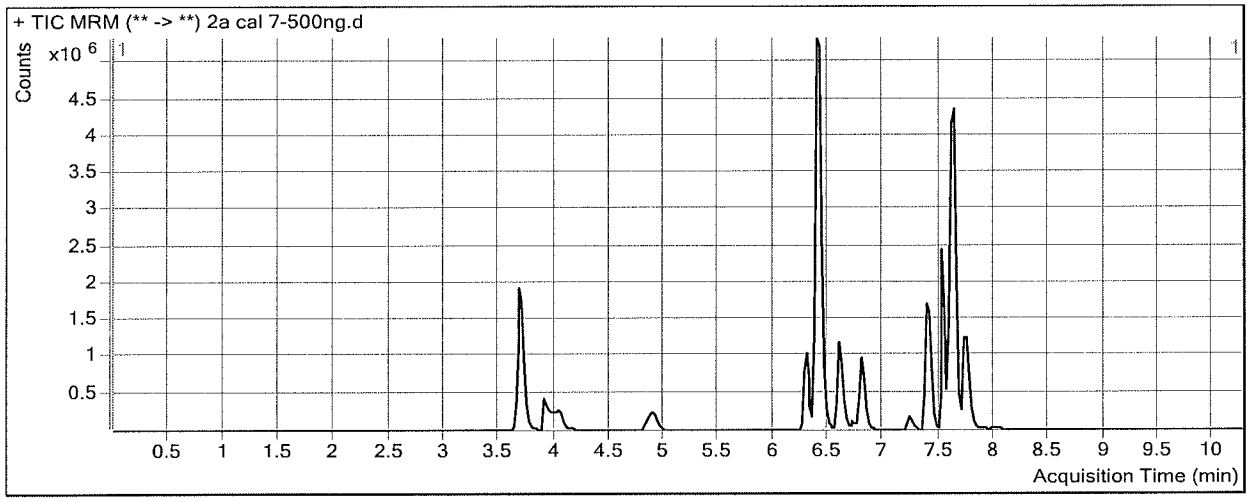
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 02:39	**Data File**	2a cal 7-500ng.d
Sample Type	Calibration	**Sample Name**	2a cal 7-500ng
Dilution	1	**Acq Method**	2a MDQ Panel 618.m
Position	P2-G5	**Sample Info**	
Inj Vol	-1	**Comment**	AM 28 Panel 2

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
Doxepin	Doxepin-D3	7.387	4107808	355839	11.5440	491.5627
Chlordiazepoxide	Chlordiazepoxide-D5	7.766	562809	112742	4.9920	487.8059

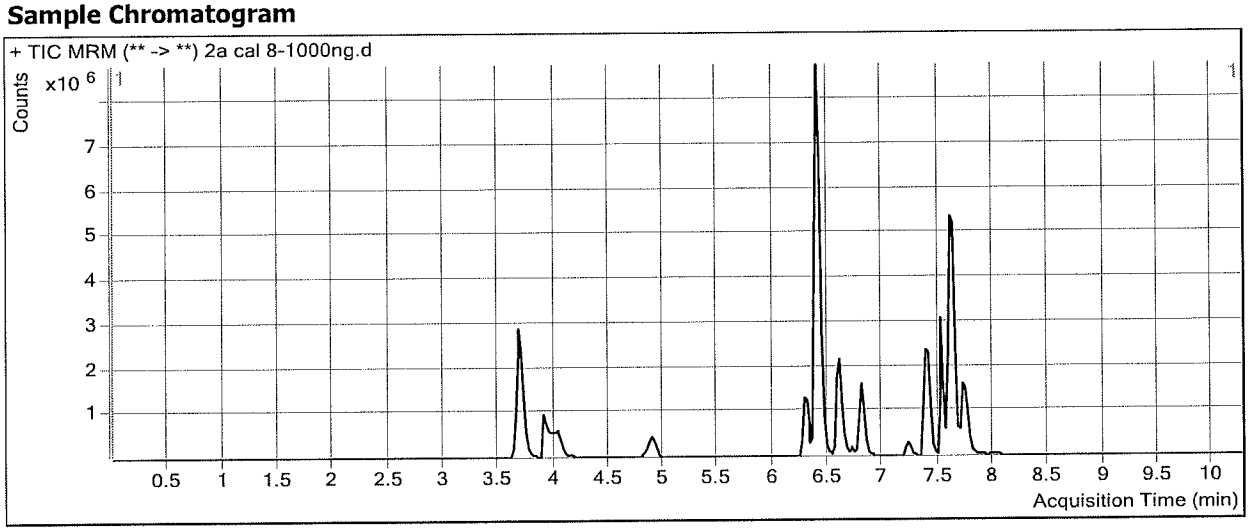
ISP FORENSICS - Cd'A Instrument # 62340
Multi-Drug Confirmatory Analysis Report

Batch Data Path	D:\2018 Data\08082018 mdq\QuantResults\p2a mdq.batch.bin		
Analysis Time	8/9/2018 10:47 AM	**Analyst Name**	ISP Tox
Report Time	8/9/2018 10:47 AM	**Reporter Name**	ISP Tox
Last Calib Update	8/9/2018 10:47 AM	**Batch State**	Processed

Analysis Info

Acq Time	2018-08-09 03:05	**Data File**	2a cal 8-1000ng.d
Sample Type	Calibration	**Sample Name**	2a cal 8-1000ng
Dilution	1	**Acq Method**	2a MDQ Panel 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
Doxepin	Doxepin-D3	7.387	5776467	272792	21.1753	901.5555
Chlordiazepoxide	Chlordiazepoxide-D5	7.766	700085	68999	10.1463	993.6772