1.0 PRINCIPLE

Blood and urine samples, that require examination for "Weak Acid," Neutral and/or Basic Drugs (WAN/BDS) by GC/MS, are extracted using mixed-bed solid phase extraction columns. Any basic drugs that may be present are extracted into a methylene chloride/isopropanol/ NH4OH mixture, and any Weakly Acidic or Neutral (WAN) drugs are extracted into a hexane/ethyl acetate. After evaporation of the solvents, the extracted drug is analyzed by GC/MS in Scan mode.

Qualitative identification of the WAN and/or Basic drugs by GC/MS is based on retention time and ion ratios for 3 ions compared to the corresponding ion ratios from a calibrator extracted and run in the same batch, or "full-scan" spectra, depending on analyte concentration. Concentrations of WAN and Basic drugs are determined by single point calibration, using cyproheptadine as an internal standard for Basic drugs and Ethinamate for the WAN drugs. Each GC/MS run is separately evaluated using control and blank samples, and is processed by a custom spreadsheet program, on which calibrator, blank and control results are summarized and tabulated, and the batch review process by both the analyst and technical reviewer is documented. Matrix-specific (blood and/or urine) positive and negative controls are extracted and analyzed in each analytical batch. The presence of WAN and Basic drugs may be confirmed in urine, blood or other aqueous fluids.

2.0 SPECIMEN

Specimens requiring confirmation for WAN/BDS are listed by lab case number on the clip board marked "WAN/BDS List" which is maintained in the Toxicology Instrument room. Analysts preparing a batch for analysis should generate their batch sample list from this document (see form 23.4, appended to SOP TX-23).

2.1 All evidence transfers, either between individuals or between an individual and a storage location must be documented on the Chain of Custody for the case, either in the LIMS, or on hard-copy COC document maintained in the Case Jacket.

2.2 When not in the sampling or aliquot process, samples in the Toxicology section must be stored in the secure and locked Toxicology evidence storage room.

2.3 Samples must be maintained in such a manner so that they are protected from contamination or deleterious change. Depending on the nature of the sample, this may mean refrigeration or freezing when not in the analytical process.

2.4 When analysis of samples in the toxicology section is complete, they must be maintained "Under Proper Seal." This is interpreted to mean that the sample, or a container in which the sample is kept, is sealed with tamper-evident tape, with the initials and date of person placing the seal clearly marked on, or proximate to that seal.

2.5 Samples are maintained in the Toxicology Section for 8 weeks after case is completed, in the absence of notification of any legal action, or other reason to maintain the samples. After this period, samples are discarded in the appropriate medical waste disposal container. Sample from fatalities, or cases with requests for retention are maintained by the laboratory.

3.0 EQUIPMENT:

GC/MS and associated data station/computer (HP6890/5973 or equivalent)
General laboratory glassware and equipment
Solid phase extraction manifold and associated vacuum equipment.
Analytical evaporator Zymark Turbovap or equivalent.
Water Bath.
UCT; ZSDAU020 "Clean Screen" solid-phase extraction tubes

4.0 REAGENTS:

4.1 Reagents available as stock items:
Methanol (HPLC grade or equivalent)
Ammonium Hydroxide (Baker Reagent Grade or equivalent)
Ethyl acetate (HPLC grade or equivalent)
Methylene Chloride (Baker Reagent Grade or equivalent)
Isopropanol IPA (Baker Reagent Grade or equivalent)
Blank Blood (may be acquired from Hartford Hospital)
Drug free urine (donated by laboratory personnel)
Target analyte drug Stocks: 1 mL vial 1.0 mg/mL; (Alltech, Sigma, or equivalent)
IS drug stocks; Cyproheptadine, Ethinamate (1 mL vial 1.0 mg/mL; Alltech)
Morphine glucuronide 1 mL vial 1.0 mg/mL (Alltech, Sigma or equivalent)
Deionized water (DIW; Millipore or equivalent In-House supply)
β-Glucuronidase (p. vulgata ; Sigma or equivalent)
Glacial Acetic Acid (Baker Reagent Grade or equivalent)
Sodium Phosphate Dibasic (Baker Reagent Grade or equivalent)
Sodium Phosphate Monobasic (Baker Reagent Grade or equivalent)
Hexane (Baker Reagent Grade or equivalent)

Hydrochloric acid (Baker Reagent Grade or equivalent)

4.2 Reagents prepared in the Toxicology Laboratory:

0.1 M phosphate buffer pH 6.0; Combine 1.7 g Na2HPO4 and 12.14 g NaH2PO4 Q.S. to 1000 mL using DIW.
Methylene chloride/Isopropanol/ammonium hydroxide (39/10/1; volume as needed; Note: Must be prepared fresh each day of use.)
β-Glucuronidase solution; Dissolve 100,000 Fishman units lyophilized powder into 20 mL 0.1 M Acetate buffer pH 5.0.
1 M Acetic acid; Dilute 28.6 mL glacial acetic acid to 500 mL with DIW
0.1 M Acetic acid; Dilute 50 mL 1 M acetic acid to 500 mL with DIW.
Ethyl Acetate/Hexane 1:1 ratio; (volume as needed).
1% methanolic HCl; Dilute 1 mL Concentrated HCl into 100 mL methanol.
Note: Reagent Preparation and Validation is documented in the Toxicology "Reagent Preparation/Validation Logbook" maintained in the Toxicology section. Validation of reagents is addressed in section 4.6, below.

4.3 Internal Standard:

Note: Preparation of all calibrator and control solutions is documented in the "Calibrator and Control Preparation Log" (maintained in the Toxicology Wet Laboratory)

Cyproheptadine internal standard solution for basic fraction: 5.0 mg/L (e.g. 0.5 mL Cyproheptadine, 1.0 mg/L; Q.S. with MeOH to 100 mL)
Ethinamate: Internal standard for acid fraction: 10 mg/L (e.g. 1.0 mL Ethinamate 1.0 mg/L; Q.S. with MeOH to 100 mL)

4.4 Stock Calibrator and Control solutions: Comprised of various drugs as needed; (Alltech or Cerilliant; 1 mg/mL). Composition of the Calibrator (analyte and concentration) will be dependent on the current patterns of observed drugs, and normal levels at which those drugs are observed. (E.g., Phenobarbital cal = 5.0 mg/L, Ketamine cal = 0.5 mg/L)

Note: Preparation of all calibrator and control solutions is documented in the "Calibrator and Control Preparation Log" (maintained in the Toxicology Wet Laboratory)

4.4.1 Calibrator stock solution for drug concentrations at 0.5 mg/L: Add 100 ul of each standard solution and Q.S. to 10 mL in a

volumetric flask.

4.4.2 Calibrator stock solution for drug concentrations at 5.0 mg/L: Add 200 uL of each standard solution and Q.S. to 2 mL in a volumetric flask.

4.4.3 Control stock solution 1 mg/L: Add 100 uL of each standard solution and dilute up to 10 mL in a volumetric flask.

4.4.4. Control stock solution 10 mg/L: Add 200 uL of each standard solution and dilute up to 2 mL in a volumetric flask.

Note: This procedure utilizes controls prepared (spiked) in blank blood and/or urine (as appropriate to batch makeup) as follows: Each run must incorporate a high and low control for each analyte. Blood matrix controls may be used to validate urine results, but not the reverse. Controls are prepared by addition of WAN and Basic drugs from validated stock solutions to blank sample matrix aliquots, prior to extraction, by an individual other than the analyst (details in Procedure below). Acceptable control performance is target value +/- 20%.

4.6 Validation of Reagents: Acceptable performance of all batch control materials and overall batch acceptability (although individual samples may fail) is considered as validation of reagents. Validated reagents are marked with a green dot, detailing the specific procedure for which the reagent was validated, and the batch on which that process was documented. Newly prepared reagents may be evaluated for validity on an analytical batch, prior to any consideration of sample results. Reagents so validated are marked with a green sticker as noted above. Preparation of reagents, and their validation is documented in the Toxicology Section Reagent Preparation Validation Logbook, maintained in the Toxicology laboratory. See SOP #11.

5.0 PROCEDURE

Note: Departure from procedures as specified in this SOP is not anticipated. Should an issue arise that may require such a departure, the issue must be raised with Section Supervisor, Quality Manager and/or the Director. If the proposed change will not present a change of such a magnitude so as to require validation,

the change may be approved, and the Director will modify and re-issue the SOP accordingly.

Any such procedural changes would be subject to the review process afforded by the quality control measures of the analytical scheme described herein. Hence, any modification or change that produces an unexpected deleterious effect on the analytical procedure would be expected to trigger analysis or batch failure in the QC review stages.

5.1 Batch Format: Analytical batches for WAN and Basic drug confirmations should follow the format indicated below: Note that samples are analyzed in duplicate.

5 blank runs used to “condition” the column should be run prior to the calibrator.

Calibrator
Matrix blank
Control High
Control Low
Glucuronide Control (Hydrolysis Batches Only)
Sample 1 rep 1
Sample 1 rep 2
Samples 2-10 Rep 1 and 2
Control High (Final control in the absence of additional samples)
Additional Samples (<10)
Final control (may be high or low)

5.2 Label a 16x100 screw cap culture tube for each sample replicate, blank, calibrator and control, similarly label a SPE tube, place a clean thru tip on each end and load in the manifold.

5.3 Using a validated dispensing pipette, place 1.0 mLs aliquot of each sample replicate into the appropriately labeled tube.

Note: Samples requiring dilution as a function of concentration greater than the high control, should be diluted with 0.1M pH 6.0 Phosphate buffer as appropriate. The initial quantitative values may be used as a guide for the dilution process. The final dilution volume should be greated than the 1.0 ml aliquot that will be taken. The dilution process shall be documented on the batch worksheet, including the pipette(s) used in the process:

Example: Blood sample; 1st run result, ~ 15.8 ug/mL Phenobarbital. A 1:1 dilution would be expected to be adequate, (final result would be ~ 7.9 ug/mL, within the range defined by the low and high controls). Therefore, 600 uL sample is added to a 13 x 100 test tube using a 0 – 1 mL adjustable volume pipette, along 600 uL 0.1 M pH 6.0 Phosphate buffer. (Note; because equal volumes of sample were utilized, pipette precision determines the accuracy of the dilution). The solution is mixed, and aliquotted as per step 5.3. The dilution details, including the dilution factor (Sample Volume: Volume Diluent + Sample Volume) are documented on the batch worksheet.

5.4 Add 1 mL of Negative urine to each control, calibrator and blank to be run if sample matrix is urine. Add 1 mL of negative blood to each control, calibrator and blank to be run if sample matrix is blood.

5.5 Using a validated dispensing pipette, add 20 uL of CHEP and 20 uL of ethinamate internal standard solutions to each test tube of every replicate, blank, calibrator and control.

5.6 Using a validated dispensing pipette, add 50 uL of both 1 mg/L and 10 mg/L drug calibrator standard stock solutions to the Calibrator tube.

5.7 Using a validated dispensing pipette, add 100 uL of both 1 mg/L and 10 mg/L control standard stock solutions to the High Control tube.

5.8 For Hydrolysis batches, 200 uL of 0.1 mg/L morphine 3-β-glucuronide solution to the Hydrolysis Control tube.

5.9 Using a validated dispensing pipette, add 20 uL of both 1 mg/L and 10 mg/L control standard stock solutions to the tube labeled Low Control.

5.8 Add 3 mL of 0.1 M pH 6.0 Phosphate buffer to each replicate, blank, calibrator and control.

NOTE: For urine batches that must be hydrolyzed, add 0.5 mL of β-Glucuronidase solution to each urine calibrator, blank, control and sample replicate and heat in water bath for 3 hours at 60°C.

5.10 Appropriately label a SPE tube for each calibrator, blank, control and sample, place a clean thru tip on each end, and load on the manifold.

5.11 Condition the columns:

1 x 3 mL methanol; drain
1 x 3 mL DIW; drain
1 x 1 mL 100 mM Phosphate buffer; drain
DO NOT LET COLUMN GO DRY!

5.12 Transfer contents of each tube to the appropriately labeled SPE tube, and allow gentle drop wise flow until the level reaches the top of the column bed.

5.13 Wash column:

1 x 2 mL Phosphate buffer; drain
1 x 2 mL 0.1 M Acetic acid; drain
Dry column (5 minutes at > 10 inches Hg)

5.14 Position appropriately labeled 13x100 test tubes under each SPE column, with clean thru tip inside collection test tube.

5.15 Elute Acid analytes:

1 x 1 mL Hexane
1 x 3 mL Ethyl acetate
Collect Eluent at 1 to 2 mL/minute

5.16 Remove receiver tubes and evaporate to dryness at < 40 C using a gentle flow of Nitrogen (Turbovap).

5.17 Reconstitute residue with 150 uL of ethyl acetate and transfer to gc/ms vial with limited volume insert, cap and reserve for GCMS analysis.

5.18 Wash column:
1 x 3 mL methanol; aspirate

5.19 Dry column (15 minutes at > 10 inches Hg)

5.20 Elute Basic analytes: 1 x 3 mL Methylene chloride/IPA/NH4OH (39:10:1) or equivalent ratio. Collect Eluate at 1 to 2 mL/minute.

5.21 Add 1 drop of 1% methanolic HCl to each tube if any SMAs are suspected.

5.22 Remove receiver tubes and evaporate to dryness at < 40 C using a gentle flow of Nitrogen (Turbovap).

5.23 Reconstitute residue with 150 uL of ethyl acetate and transfer to gc/ms vial with limited volume insert, cap and reserve for GCMS Analysis.

6.0 CHROMATOGRAPHY AND MASS SPECTROSCOPY

6.1 Instrument and Setup:

GCMS/Autosampler: (Hewlett-Packard 6890/5973, or equivalent)
Column: 30M RTX-5MS (0.25 mm ID; 0.25 micron film)
Inj. Temp. 250 ^{o} Det. Temp. 80 ^{o}
Oven (init.): 80^{o}, 25 ^{o} /min to 300 ^{o}, (10 min hold).
22.0 min total run time 1 uL inj
Method: Drugsmwa.M; Method Details Appended (Appendix II)

6.2 Injection sequence: Each sample batch is injected in the following sequence:

Calibrator
Matrix blank
Control High
Control Low
Hydrolysis Control (if present)
Sample 1 rep 1
Sample 1 rep 2
Samples 2-10 Rep 1 and 2
Control High (may be final control)
Additional Samples (<10, if included)
Final Control (if additional samples present)

6.3 DETECTION AND IDENTIFICATION

Determination of the presence of WAN and Basic drugs in the sample extract are identified by evaluation of full spectra, and appearance and ratio of the 3 ions characteristic of each species at the appropriate retention time, hence both

retention time (a GC characteristic) and fragmentation pattern and ratio (MS characteristics) are used as the basis of qualitative identification. For a positive identification of WAN and Basic drugs, the retention time must be within 5% of the corresponding analyte in the calibrator injection, and the ion ratios for both qualitative ions must be within 20% of the corresponding ratio calibrator sample. Qualitave identification of each analyte is independent.

Note: Qualitative identification of analytes with abundance < 5% of the I.S. abundance should be considered with great care, and evaluation of significance based on the (presumably) low concentration. Further, those species with expected metabolites/ artifacts should not be identified as present unless those expected metabolites/ artifacts are also present. (Examples, EDDP with methadone, propoxyphene metabolite and propoxyphene artifact with propoxyphene, benzoylecgonine with cocaine, etc.)

6.4 CALIBRATION AND QUANTITATION

6.4.1 Calibration for each batch and fraction is done independently. Hence, no sample analysis conducted under DPS guidelines drug is quantitated based on an historical calibration curve. Calibration is accomplished by the incorporation into the sample procedures of a blank sample of the matrix being analyzed that has known quantities (0.5 mg/L for most Basic drugs and 5.0 mg/L for most Acid drugs) in addition to the internal standard. The response of the system to this calibrator, and the assumption of a 0 response to a 0 concentration, defines a run-specific standard curve that is used as the basis for the quantitative calculation in all controls and samples. The system is therefore “single-point calibration, multi-point control”.

6.4.1 <u>Quantitation</u>

Quantitation is accomplished by the comparison of the response ratio of the analyte in a sample, to the response ratios of the calibrators as expressed as a standard curve. The concentration of the analyte in the sample is then extrapolated from the standard curve, and corrected for any dilution that may have been performed to facilitate the analysis of relatively concentrated samples.

Note: The available quantitative range of this procedure is (in terms of amount of drug injected on the instrument (thereby allowing for appropriate

calculation of diluted samples) is defined for , and validated on each batch by the high and low control, and the acceptable performance of each.

7.0 QUALITY CONTROL AND RUN EVALUATION

7.1 Verification of Vial Sequence: The vial sequence is checked both prior to and after the injection of samples when the auto injector is used. The check after the samples are injected is documented on the run summary sheet.

7.2 Chromatography - Evaluation and Acceptance Criteria: Chromatographic quality is evaluated for each peak. While general guidelines are that a peak should be symmetrical, and be resolved to baseline on at least one side, with 90% resolution on the other side, significant departures from those guidelines may be experienced with forensic samples. In many cases, chromatographic quality will warrant rejection of the chromatographic run, or specific samples, by the operator. Any such action should be clearly documented on the batch summary sheet. Questionable chromatographic peak shape, resolution, or other problems with chromatography can be discussed with the Section Supervisor, Director or the Quality Manager.

7.3 Evaluation of Potential Carryover: Carryover in the chromatogram is evaluated by injection of a blank sample immediately following the calibrator. Carryover of greater than 2% requires batch rejection, and remedial action for the instrument (e.g. replacement of injector insert, new septum and perhaps column trim or even replacement). Demonstrated carryover of less than 2% will require operator consideration with regard to the potential for effects on specific samples, and may require re-extraction of specific samples. Carryover is further evaluated on a per sample basis by the requirement that quantitative results between replicates agree within 20%. Any significant carrover effect should cause the first of the two replicate samples to exceed the second by an amount in excess of the 20% differentials. If not, any carryover may be considered inconsequential. In practice, when a question of potential carryover exists, the potentially affected sample replicates may be repeated at the end of the batch.

7.4 Evaluation of Controls: Control results are documented and evaluated on the batch summary sheet.

7.4.1 Qualitative Results: Both controls must demonstrate the target analyte with acceptable chromatography and spectral characteristics.

7.4.2 Quantitative Results: Both controls must demonstrate the target analyte at a concentration within 20% of the target value

Note: Controls are routinely reported only for analytes detected in samples. However, for batches in which no analytes were demonstrable in samples, controls for at least one analyte will be reported to allow procedural evaluation.

7.5 Internal standard abundance should be at least 500K. For any individual injection to be acceptable, the internal standard abundance must be at least 20% of the corresponding abundance in the calibrator.

7.6 LINEARITY: Linearity of the calibration curve is demonstrable in each batch, for each analyte as a function of quantitative results of control materials.

7.7 SENSITIVITY- LIMITS OF DETECTION (LOD) and QUANTITATION (LOQ) For the purposes of this procedure, the LOD and LOQ are defined as equal to the lowest concentration of the lowest control. Qualitative Identification and/or Quantitative analysis below the concentration of the low control may be accepted on a case by case basis with the concurrence of the analyst, technical reviewer, Director and/or Quality Manager.

7.8 ACCURACY AND PRECISION: Precision of the procedure is evaluated on a yearly basis, by repeat analyses of control or PT materials. Accuracy is expressed as a mean (absolute value) percentage difference between mean quantitative value of 10 reps of the specific control, and the target value. Precision is expressed as the CV of that value

7.9 SPECIFICITY: Specificity is a function of both the resolution of target analyte during the analytical process, and the mass spectral fragmentation that analyte molecules undergo during the instrumental analysis. There has been no report of any material other than WAN and Basic drugs that elutes within 5% if the retention time of known standard materials, and produce the same fragmentation ions and ratios.

8.0 **REPORTING OF RESULTS**: GDS runs are performed as part of GC/MS batches, containing controls and calibrators. The complete batch packets are in the Toxicology Laboratory. This packet contains all run evaluation documentation. Specific chromatograms for each case are filed in the appropriate case file. Results are documented on the “Case Summary” sheet in each case file and include a reference

number for the batch as a whole. If controls do not meet the criteria, the batch can be rejected as a whole or by a case by case basis. The supervisor is notified and proper action is taken to correct any problem. Batches and/or cases shall be repeated as needed.

Procedural Uncertainty is reported with all quantitative results, and is calculated and tabulated annually for each analytical method, (See SOP TX-19 section 6.3).

9.0 **QUALITY ASSURANCE**: Quality Assurance is provided by the multiple layers of checks that are performed both during and after analysis. Specifically:

9.1 The GC/MS run is thoroughly checked by the operator, including vial position on the autosampler, both prior to and following the injection of samples.

9.2 The GC/MS run is reviewed and signed off by a reviewer distinct from the operator, with this review including an evaluation of qualitative and quantitative (where applicable) results, including:

a. Control Results
b. Chromatographic Characteristics
c. Transcription errors

9.3 WAN/BDS runs runs are performed as part of GC/MS batches, containing controls and calibrators. The complete batch packets are in the Toxicology Laboratory. This packet contains all run evaluation documentation. Specific chromatograms for each case are filed in the appropriate case file. Results are documented on the “Batch Summary” sticker on each case file and include a reference number for the batch as a whole. A Batch summary sheet will be produced with each batch. Data on each batch should include fields such as: Sample name, Batch ID (Date of Batch), analysts who generated data, matrix, analyte found (and concentration if applicable), controls run with the batch and results obtained.

9.4 The original run is compared to the Final Report during the Final Director's review, prior to case sign-off.

10.0 **SOURCES OF ERROR**: The utilization of 3-ion SIM methodology, with reference to procedural, controls and calibrators yields qualitative drug identification with essentially no qualitative uncertainty. Urine drug analyses are reported only as qualitative results.

11.0 **REFERENCES:**

Clarke's Isolation and Identification of Drugs. 2nd Edition

UCT United Chemical Technologies: Solid phase extraction methods

Appendix II:
GC/MS temperature program specifications

Parameter			
Initial temp	80° C		
Initial Time	0.00min		
Ramps Rate/final temp/final time	rate	temp	time
	25.0	300	14.00
Post temp	80°c		
Post time	0.00		
Run Time	14.0min		
Front inlet			
Mode	Pulsed splitless		
Initial temp	250°c		
Pressure	8.56 psi		
Pulse pressure	25 psi		
Pulse time	0.50 min		
Total flow	43.4 mL/min		
Gas type	Helium		
Injection volume	1microliter		
Post injection washes Solvent A / Solvent B	Solvent A -3 Solvent B - 3		
Tune file	STUNE		
Acquisition mode	Scan		
Solvent delay	4.00 minutes		
Low mass	40		
High mass	470		
Threshold	250		
MS Quad	150°c max 200		
MS Source	230°c max 250		

Example of controlled WAN batch document TX WAN-1. Batch documents can vary based on nature of batch.

CT DPS; Toxicology Laboratory Batch ID: WAN 4-4-11

WAN GCMS Batch Summary

Autosampler vial postion	Sample	Specimen	Specimen Volume, (ml)	Int. Std. Volume (ul)	Analyte	I.S. abund. (x 1 E4)	Theoretical Concentration (mg/L)	Observed Concentration (mg/L)	Percent Recovery (Acceptable: 80 - 120)	Acceptable, Analyst	Technical Review
36	Calibrator	Water	1.0	20	Ibuprofen 5.05	197	5.00	5.00			
					Meprobamate 5.81		5.00	5.00			
					Secobarbital 5.85		5.00	5.00			
					Topiramate 7.30		1.00	1.00			
37	Blank	Water	1.0	20	BDS: NDD: No drugs detected	123					
38	Control 1	Water	1.0	20	Ibuprofen	196	2.50	0.479	19.1		
					Meprobamate		2.50	2.44	97.6		
					Secobarbital		2.50	2.42	96.8		
					Topiramate		0.500	0.370	74.0		
39	Control 2	Water	1.0	20	Ibuprofen	215	1.00	nd	na		
					Meprobamate		1.00	1.49	149		
					Secobarbital		1.00	1.78	178		
					Topiramate		0.200	0.120	60.0		
	Internal std	meoh			Ethimimate 3.86						
	Ex Standards				CHEP 8.08						
	Ex Standards										
64	TX-11-348	Water	1.0	20	Ibuprofen	347					
65	TX-11-348	Water	1.0	20	Ibuprofen	319					
66	TX-11-349	Water	1.0	20	NDD: Drugs Not Detected	300					
67	TX-11-349	Water	1.0	20	NDD: Drugs Not Detected	287					
38	Control 1	Water	1.0	20	Ibuprofen	220	2.50	0.771	30.8		
					Meprobamate		2.50	2.56	102		
					Secobarbital		2.50	2.54	101		
					Topiramate		0.500	0.483	96.6		

Elution solvent was valid:_____

Samples Extracted by: _____ Date: _____ GCMS Run Date: _____

GCMS #_____

Vial position verified prior to sample removal _____

Analyst Review by: _____ Date: _____ Run Accepted? ___ Yes ___ No

Analyst Comments: _____

Technical Review by: _____ Date: _____ Run Accepted? ___ Yes ___ No

Reviewer Comments: _____

Example of controlled BDS batch document TX BDS-1. Batch documents can vary based on nature of batch.

CT DPS; Toxicology Laboratory Batch ID: BDS 1-7-11

Hydrolyzed BDS GCMS Batch Summary page 1

Autosampler vial postion	Sample	Specimen	Specimen Volume, (ml)	Int. Std. Volume (ul)	Analyte	I.S. abund. (x 1 E 4)	Theoretical Concentration (mg/L)	Observed Concentration (mg/L)	Percent Recovery (Acceptable: 80 - 120)	Acceptable Qual. Analyst	Qual Technical Review
3	Calibrator	URINE	1.0	20	MDA 4.87	299	1.00	1.00			
					MDMA 5.11		1.00	1.00			
					PCP 6.84		1.00	1.00			
4	Blank	URINE	1.0	20	BDS: NDD: No drugs detected	285					
5	Control 1	URINE	1.0	20	MDA	304	0.500	nd	na		
					MDMA		0.500	0.346	69.2		
					PCP		0.500	0.380	76.0		
6	Control 2	URINE	1.0	20	MDA	290	0.200	nd	na		
					MDMA		0.200	0.144	72.0		
					PCP		0.200	0.163	81.5		
	Internal std	meoh			Chep 8.61						
	Ex Standards				DMT 6.304						
					Levamisole 7.19						
21	TX-10-1712	URINE	1.0	20	PCP	370					
					Cocaine						
					Levamisole /Dextramisole						
22	TX-10-1712	URINE	1.0	20	PCP	379					
					Cocaine						
					Levamisole /Dextramisole						
24	TX-10-1744	URINE	1.0	20	MDA	351					
					MDMA						
5	Control 1	URINE	1.0	20	MDA	427	0.500	nd	na		
					MDMA		0.500	0.404	80.8		
					PCP		0.500	0.360	72.0		

Elution solvent was valid:_____

Samples Extracted by: _____ Date: _____ GCMS Run Date: _____

GCMS #_____

Vial position verified prior to sample removal _____

Analyst Review by: _____ Date: _____ Run Accepted? ___ Yes ___ No

Analyst Comments: _____

Technical Review by: _____ Date: _____ Run Accepted? ___ Yes ___ No

Reviewer Comments: _____