Idaho State Police
Forensic Services
Toxicology Section

Section Two

2.4 Liquid-Liquid Extraction Methods for GC/MSD Confirmation

2.4.1 General Extraction of Urine Samples for Qualitative Confirmation of Basic and Neutral or Acidic Drugs

2.4.1.1 BACKGROUND

These extraction procedures are extensions of the TOXI-LAB® TOXI-A and TOXI-B thin layer chromatography (TLC) drug detection systems. The samples are extracted as with the TLC system, however, instead of concentrating the extract onto a disc, the solvent extract is concentrated and placed into an automated liquid sampler (ALS) vial for analysis by a gas chromatograph equipped with a mass selective detector (GC/MSD).

2.4.1.2 PRINCIPLE

This procedure describes the extraction of drug compounds from urine. Depending upon the pK_a of a drug compound, either Toxi-A or Toxi-B tubes are used. Basic compounds are extracted with a Toxi-A tube. Addition of urine to the Toxi-A tubes results in the urine becoming alkaline (pH=9) into 1,2-Dichloroethane, dichloromethane, heptane and isopropanol. Acidic compounds are isolated from an acidified solution (pH=4.5) into methylene chloride and heptane with zinc chloride to facilitate the extraction process. The extraction is achieved with an Ansys Toxi-B extraction tube. Either resulting extract is analyzed by full scan GC/MS in EI mode.

2.4.1.3 EQUIPMENT AND SUPPLIES

2.4.1.3.1 Tube Rocker (Fisher Scientific or equivalent)

2.4.1.3.2 Electric Warmer with Omega-12 extraction solvent concentrator (Ansys 118/153)

2.4.1.3.3 Laboratory Centrifuge (Fisher Marathon or equivalent)

2.4.1.3.4 Disposable Aluminum Concentration Cups (Ansys 152)

2.4.1.3.5 Glassware
GC/MS vials (HP 5182-0865 or equivalent)
GC/MS vial microinsert (HP 5183-2088 or equivalent)

2.4.1.3.6 Gas Chromatograph equipped with a mass selective detector (HP 6890/5973) and a HP-5MS Ultra low bleed (5%-Diphenyl-95%-Dimethylsiloxane copolymer) capillary column (25M).

2.4.1.4 REAGENTS

2.4.1.4.1 ANSYS TOXI-TUBES A and B (Ansys 109A-100/ 109B-100)

2.4.1.5 CONTROLS

2.4.1.5.1 Toxi-Control No. 19 – Morphine, amphetamine, imipramine, methadone, propoxyphene, phenobarbital, secobarbital and benzoylecgonine (Ansys 191AB).

2.4.1.5.2 Toxi-Control No. 2 – Amphetamine, methamphetamine, nicotine and cotinine. (Ansys 170B).

2.4.1.6 STANDARDS

2.4.1.6.1 Run necessary analytical standards as indicated by examination of GC/MSD data.

2.4.1.7 PROCEDURE

2.4.1.7.1 Initial set-up
Label TOXI-TUBES A or B, and GC/MS vials with microinserts, with negative control, TC-19 and or TC-2 and appropriate laboratory numbers.

2.4.1.7.2 Extraction Procedure Toxi-A Extraction (Basic or Neutral Compounds)
Transfer 5 mL of urine specimen, negative urine or appropriate Toxi-Control to a TOXI-TUBE A (pH=9).
Rock TOXI-TUBE A for 15 minutes.

2.4.1.7.3 Centrifuge tube at 2500 rpm for 15 minutes.

2.4.1.7.4 Transfer solvent from tube into concentration cup in Omega-12 extraction solvent concentrator. Allow cups to warm prior to the addition of extract.

2.4.1.7.5 Evaporate solvent to approximately 200µL on electric warmer.

2.4.1.7.6 Transfer solvent to labeled GC/MS ALS vial with microinsert.

2.4.1.7.7 Extraction Procedure Toxi-B Extraction (Acidic Compounds)

2.4.1.7.8 Transfer 4.5 mL of urine specimen, negative urine or Toxi-Control 19, to a TOXI-TUBE B (pH=4.5).

2.4.1.7.9 Rock TOXI-TUBE B for 15 minutes.

2.4.1.7.10 Centrifuge tube at 2500 rpm for 15 minutes.

2.4.1.7.11 Transfer solvent from tube into concentration cup in Omega-12 extraction solvent concentrator.

2.4.1.7.12 Evaporate solvent to approximately 200µL on electric warmer.

2.4.1.7.13 Transfer solvent to labeled GC/MS ALS vial with microinsert.

2.4.1.7.4 Gas Chromatography/Mass Spectrometry (GC/MS) Analysis

2.4.1.7.4.1 Inject 1 µL into GC/MS using the ALS.

2.4.1.7.4.2 Analyze sample extract in full scan acquisition. Refer to attached GC/MSD method printout for current analysis parameters.

2.4.1.7.5 Detection and Identification Criteria

2.4.1.7.5.1 The presence of a drug compound can be established if there are no significant differences in the retention time and mass spectra for the sample versus standards.

2.4.1.7.5.2 Acceptable retention time window is ±5%.