K56 A Fast GC/MS Method for the Analysis of Common Selective Serotonin Reuptake Inhibitors

Sumandeep Rana, MS, Wayne B. Ross, MCLS, and Victor P. Uralets, PhD, Redwood Toxicology Laboratory, 3650 Westwind Boulevard, Santa Rosa, CA 95403*

After attending this presentation, attendees will learn about a validated, fast method for routine analysis of common selective serotonin reuptake inhibitors (SSRI's) in human urine.

This presentation will impact the forensic community by demonstrating how choosing the right combination of column and carrier gas results in significant improvement in analytical procedures and improves throughput in a routine testing laboratory.

SSRI's included in this method are: fluoxetine, norfluoxetine (fluoxetine metabolite), sertraline, norsertraline (sertraline metabolite), citalopram, and paroxetine. Deuterated paroxetine (paroxetine-D6) was used as the internal standard.

Urine samples were hydrolyzed using β-glucuronidase from *Escherichia coli,* centrifuged for 5 minutes and then approximately 1 gram of a salt mixture (sodium chloride, sodium carbonate and sodium bicarbonate, 6:1:1 by weight) was added. The alkalinized urine specimens were extracted using liquid-liquid extraction with heptane/ dichloromethane/ dichloroethane/ isopropyl alcohol (10:5:5:1). The organic upper organic layer was separated and dried under air at 40°C. The dried sample extracts were derivatized for 10 min at 65°C with MSTFA/ammonium iodide/ ethanethiol reagent (50 mg/25 mL/75 µL).

GC/MS analysis was performed in electron ionization mode by selective ion monitoring (EI - SIM) using a single quadrupole mass spectrometer with inert ion source. A 230 volt GC oven was used to enable fast temperature programming and hydrogen was used a carrier gas. Separation was performed on a narrow bore column (10 m X 0.15 mm i.d.). All analytes were eluted within 4.5 minutes with Injection-to- injection analytical run time of 7.5 minutes. Three ions for each analyte; paroxetine (249.1, 264.1, 401.2), Fluoxetine (219.0, 262.2, 381.2), norfluoxetine (174.1, 320.1, 439.2), sertraline (274.0, 276.0, 377.1), norsertraline (274.0, 276.0, 320.0), citalopram (238.1, 324.1, 208.1), and two ions for the internal standard; paroxetine- D6 (252.1, 270.2) were monitored.

The procedure was applied to authentic urine specimens and the results showed that hydrolysis is essential to the optimum recovery of most analytes. All analytes were successfully detected in the 4.5 minute run time utilized. The limit of detection for all analytes was 100 ng/mL except citalopram, for which it was 50 ng/mL. The limit of quantitation for citalopram and paroxetine was 100 ng/mL and for all other analytes it was 150 ng/mL. Precision was within 6% and quantitative accuracy was over 94% for all analytes. The method was linear up to 20,000 ng/mL for paroxetine and upto 2000 ng/mL for all other analytes.

A fast and simple GC/MS method was developed for the routine analysis of common SSRI's in urine. This method can easily be used for other body fluids such as blood. The simple sample preparation, combined with short, narrow bore GC column and hydrogen as a carrier gas, drastically decreased sample turnaround time and increased throughput without compromising sensitivity or selectivity.

SSRI's, GC/MS, Hydrolysis

 ** Presenting Author*