8.2 ADDITIONAL VOLATILE COMPOUND PROCEDURE

8.2.1 Purpose

To qualitatively and/or quantitatively identify volatile substances in submitted evidence by instrumental analysis with headspace gas chromatography/flame ionization (HS-GC/FID) and headspace gas chromatography/mass spectrometry (HS-GC/MS).

8.2.2 Specimen Requirements

Acceptable samples for this analysis include blood, urine, vitreous humor, and other aqueous liquids. For additional samples see Alternative Matrices (section 6.6).

8.2.3 Apparatus and Equipment

Volumetric pipettes and disposable tips
Assorted volumetric glassware
20 mm headspace vials
Crimp caps with septa
20 mm crimper
HS-GC/FID, HS-GC/MS, ChemStation software, compatible computer, and printer

8.2.4 Reagents and Standards

Reference standards (as needed)
0.01667 v/v% n-Propanol (internal standard)
Water (H_2O)

8.2.5 Standard Preparation

The following are examples of how to prepare the standards used in this procedure.

n-Propanol Reference Standard Solution [0.01667 v/v%] (Internal Standard)
Add 166.7 μL of n-propanol and dilute to 1000 mL with H_2O.

8.2.6 Procedure

1. Allow all reference standards and case samples to equilibrate to room temperature before beginning procedure.
2. Label, check, and load/unload all samples in accordance with the “Sample Pipetting Check List” (see Appendix section).
3. Pipette 100 μL of corresponding case sample, calibrator, positive control, or negative control into the appropriately labeled 20 mm headspace vial.

4. Pipette 600 μL of internal standard into each sample to make a final concentration of 0.1 v/v%.
   Note: Smaller sample volumes may be analyzed on a case-by-case basis. The total volume of liquid in the headspace vial must always be equal to 700 μL (e.g., 50 μL sample + 50 μL H_2O + 600 μL internal standard = 700 μL total volume).
5. Seal vial with crimp cap.
6. Analyze and quantitate the samples by HS-GC/FID and confirm by HS-GC/MS (full scan mode).

8.2.7 Reporting

8.2.7.1 Retention times of drugs identified are within ±1% of those of a calibrator or control standard of similar concentration on GC/FID unless otherwise noted in the case file.

8.2.7.2 Mass spectrums of drugs identified are consistent with those of analyzed reference standards

8.2.7.3 Results shall be expressed as “positive”, “no volatiles detected”, etc. and include any clarifying remarks, if applicable.

8.2.7.4 When a definitive conclusion cannot be made, the reason shall be documented on the report (e.g., “insufficient sample for analysis”, “sample unsuitable for analysis”, “results are inconclusive due to sample condition”, etc.).

8.2.8 References

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HS 40XL Automatic Headspace Sampler User’s Guide, Perkin Elmer Co., 1999.

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