9.0 Authentication of Reference Material and Matrix Controls: Other Volatiles

9.1 BACKGROUND

Refer to Analytical Methods 1.0 and 2.0.

9.2 SCOPE

This method describes the Idaho State Police Forensic Services (ISP-FS) requirements for the authentication of quality assurance material used to provide confidence in the data collected during the analysis of blood, vitreous humor and urine for the presence of volatile compounds.

9.3 EQUIPMENT

9.3.1 Gas Chromatograph (GC) configured with Headspace Sampler (HS) and a Flame Ionization Detector (FID)

9.3.2 HS-GC-FID Columns
Restek Rtx®-BAC1
Restek Rtx®-BAC2

9.3.3 Gas Chromatograph (GC) configured with a Mass Selective Detector (MSD)

9.3.4 GC-MSD Column
HP-5 MS or comparable

9.3.5 Headspace (HS) vials and Closures

9.3.6 Hand Crimper or Bench Top Crimper

9.3.7 Semi-Automatic Dilutor/Pipetter equipped with sample and reagent syringes capable of dispensing 250µL and 2000µL, respectively

9.3.8 Laboratory Oven

9.3.9 Gas tight syringe capable of dispensing 50µL

9.4 REAGENTS

9.4.1 Distilled/Deionized water (free from volatiles of interest)

9.4.2 Ammonium Sulfate (Certified ACS Grade)

9.5 REFERENCE MATERIAL

Refer to Analytical Methods 1.0 and 2.0.

9.6 SAFETY CONCERNS

Biological samples must be processed and chemicals handled according to safety guidelines in the *Idaho State Police Forensic Services Health and Safety Manual.*

9.7 QUALITY ASSURANCE

Refer to Analytical Methods 1.0 and 2.0.

9.8 AUTHENTICATION OF VOLATILE REFERENCE MATERIALS

9.8.1 General

9.8.1.1 Refer to Analytical Methods 1.0 and 2.0 for GC-HS analysis requirements.

9.8.1.2 *Certificates of Analysis* (COA) for certified reference material will be stored centrally.

9.8.1.3 New lots of volatile reference materials, including both aqueous mixed volatiles and single component volatile reference material used to prepare multicomponent mixtures, must be authenticated prior to official use.

9.8.1.4 Certified reference materials (CRM) will be authenticated by GC-HS.

9.8.1.5 Reference materials without certificates of analysis will be authenticated by GC-MSD.

9.8.2 Qualitative Authentication: Certified Reference Materials (CRM)

9.8.2.1 **CRM Authentication Analysis**

9.8.2.1.1 Use Pipetter/Dilutor to dispense 250µL of new volatile reference material lot along with 2000µL of internal standard (ISTD), into labeled headspace vial and apply seal.

9.8.2.1.2 Two or more vials of the new reference material lot must be prepared.

9.8.2.1.3 Retention time data for the compound being authenticated must be available. Ideally, the in-use lot of the compound being authenticated would be included in the authentication analysis run.

9.8.2.1.4 When no retention time is available, as is the case for compounds infrequently or not previously used, the reference material may also be authenticated by GC-MSD. Refer to section 9.8.3 for GC-MSD authentication measures.

9.8.2.2 **CRM Qualitative Authentication**

9.8.2.2.1 Obtain retention time data for the compound being authenticated and calculate the mean retention time.

9.8.2.2.2 Compare retention times reported for new reference material compound with retention time obtained for in-use previously authenticated compound.

9.8.2.2.3 The new lot of compound can be accepted if the mean retention time for the new lot is ± 0.10 minutes.

9.8.3 Qualitative Authentication: Uncertified Reference Materials

9.8.3.1 **Authentication Analysis**

The following volumes and temperatures are recommendations only. Other volumes and temperatures may be used in place of those listed.

9.8.3.1.1 Neat Reference Material (Solvents)

Place ≅10µL of neat reference material into a headspace vial and seal. Manually inject ≅50µL of room temperature vial headspace into GC with an airtight syringe.

9.8.3.1.2 Aqueous Reference Material (Ampules)

Place contents of ampule into a headspace vial, seal and heat at 60°C for approximately 5 minutes. Manually inject ≅50µL of vial headspace into GC with an airtight syringe.

9.8.3.1.3 GC parameters must be optimized for volatile being authenticated with consideration of boiling point. MSD parameters must be set such that appropriate ions are scanned for. The low scan range will most likely need to be lowered from default.

9.8.3.1.4 Library search and/or reference (article, book) must clearly indicate that the appropriate compound(s) are present. The authentication data must be centrally stored.

9.9 AUTHENTICATION OF OTHER VOLATILES IN BLOOD MATRIX CONTROLS

9.9.1 Volatile compounds, in additional to ethanol, in blood matrix controls will be authenticated using the data obtained for ethanol authentication as described in analytical method 8.0.

9.9.2 Use the authentication criteria listed in section 9.8.2.2.

9.10 AUTHENTICATION DOCUMENTATION

9.10.1 Reference Material

Original authentication data and documentation of compliance with acceptance criteria will be maintained in the laboratory performing the authentication.

9.10.2 Matrix Controls

9.10.2.1 Original authentication data will be maintained in the laboratory performing the analysis.

9.10.2.2 A packet containing data from all matrix controls used for authentication and evaluation of the data will be maintained by the discipline leader.

9.11 REFERENCES AND RECOMMENDED READING

9.11.1 Stafford, D.T., *Chromatography. in:* Principles of Forensic Toxicology, edited by Barry Levine, pp. 91-98, 100-108, 114-118, AACC Press, 2006.

9.11.2 Levine, B. and Caplan, Y.H., *Alcohol. in:* Principles of Forensic Toxicology, edited by Barry Levine, pp. 169-184, AACC Press, 2006.

9.11.3 Caplan, Y.H., *The Determination of Alcohol in Blood and Breath. in:* Forensic Science Handbook, edited by Richard Saferstein, pp. 594-648, Prentice-Hall New Jersey, 1982.

9.11.4 Christmore, D.S., Kelly, R.C. and Doshier, L.A. *Improved Recovery and Stability of Ethanol in Automated Headspace Analysis,* J. Forensic Sci. 29(4): 1038-1044; 1984.

9.11.5 Restek Applications Note #59598, Dual-Column Confirmational GC Analysis of Blood Alcohols Using the Rtx®-BAC1 and Rtx®-BAC2 Columns Optimized for the Perkin-Elmer HS-40 Headspace Autosampler, 1999.

Revision History

9.0 Authentication of Reference Material and Matrix Controls: Other Volatiles

Revision #	Issue Date	Revisions
0	09-07-2009	Initial version. Separated from AM 4.1. Language and requirements updated.
0	01-20-2011	Initial version as a volatiles analytical method. Previously a portion of AM 5.14. Language and requirements updated. Major update: GC-MS analysis for certified reference material no longer required. This AM addresses compounds used for only qualitative analysis.

Gamette, Matthew

From: Johnston, Jeremy
Sent: Friday, August 19, 2011 1:04 PM
To: Gamette, Matthew
Subject: rescind

AM 2.0, 3.0, 5.0 and 9.0 are no longer in use or have been incorporated into other AM's and can be archived as no longer in use.

AM 2.0 has been incorporated into AM 1.0
AM 9.0 has been incorporate into AM 8.0
AM 5.0 is redundant to the requirements of Tox or Drug AM's and referenced as such.
AM 3.0 is redundant with Tox AM's

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