2.0 Authentication of Reference Material

2.1 BACKGROUND
Refer to Analytical Methods 1.0.

2.2 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 to establish both the qualitative and quantitative presence of ethanol and other volatiles.

2.3 EQUIPMENT
2.3.1 Refer to the specific analytical method for a listing of equipment.

2.4 REAGENTS
2.4.1 Refer to the specific analytical method for a listing of reagents.

2.5 REFERENCE MATERIAL
Refer to Analytical Methods 1.0.

2.6 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.*

2.7 QUALITY ASSURANCE
Refer to Analytical Methods 1.0.

2.8 AUTHENTICATION OF VOLATILES REFERENCE MATERIALS
2.8.1 General

2.8.1.1 Refer to Analytical Methods 1.0 for GC-HS analysis requirements.

2.8.1.2 Aqueous reference material used to establish the calibration curve must be traceable to NIST standards.

2.8.1.3 All available *Certificates of Analysis* for reference material will be stored centrally.

2.8.1.5 Reference materials without certificates of analysis will be authenticated structurally.

2.8.1.4 New lots of reference material must be authenticated prior to an analyst reporting a conclusion in casework in which that reference material was used.

2.8.2 Authentication Analysis

2.8.2.1 Refer to the AM 1.0 for analysis of the reference materials.

2.8.2.2 Three or more sample vials of the new reference material lot must be prepared and analyzed.

2.8.3 Qualitative Authentication

2.8.3.1 Calculate the mean retention time for the analyte using the analysis run data.

2.8.3.2 Compare volatile retention times reported for new reference material lot with retention time obtained from previous data.

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

2.8.3.4 For analytes of interest that have no previous data for comparison, those substances will be analyzed using structural analysis (GC/MSD, LC/MS, etc). Structural analysis needs to be performed by authorized personnel.

2.8.3.5 *A standard will be considered structurally authenticated when the match (Q) is greater than 85 %, as compared to a library search and the analyst confirms that the spectra matches with no significant differences. If the spectra does not have a library match of 85% or greater the spectra may be authenticated by comparing it to a peer reviewed scientific journal, reference standard compendium or a library match that is less than a 85%. For these three options two analysts trained to use the*

authentication instrumentation must initial the documentation signifying that it is an appropriate match.

2.8.4 Quantitative Authentication

2.8.4.1 Compare the quantitative data from the analysis of a new lot with the *Certificate of Analysis* values.

2.8.4.2 The new lot number of volatile reference material can be accepted if the mean concentration obtained falls within 6% of the target value (assayed) listed on the *Certificate of Analysis*.

2.8.4.3.1 The manufacturer's target value will be used as the target value for the lot.

2.8.4.3 Evaluation of data must be such that compliance with concentration requirements is apparent.

2.8.4.4 When a certified volatile reference solution contains components in addition to ethanol, only the ethanol concentration needs to be quantitatively authenticated. For controls that contain other volatiles (e.g. acetone, methanol, isopropanol) in addition to ethanol, the qualitative determination of the components must be established through the comparison of relative retention times.

2.9 AUTHENTICATION DOCUMENTATION

2.9.1 Reference Material

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

2.9.2 A copy of all data used to authenticate the quantitative reference materials will be maintained by the alcohol discipline leader.

2.10 REFERENCES AND RECOMMENDED READING

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

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

2.10.3 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.

2.10.4 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.

2.10.5 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

2.0 Authentication of Reference Material: Volatiles

Revision #	Issue Date	Revisions
0	09-07-2009	Initial version. Separated from AM 4.1. Language and requirements updated.
0	1-20-2011	Initial version as a volatiles analytical method. Previously a portion of AM 5.14. Language and requirements updated. Major updates: GC-MS analysis for certified reference material (CRM) is no longer required.
1	8-23-2011	Section 8.9 moved to AM 10. Blood matrix controls authentication incorporated into section 8.8. Section 8.8 specific reference to "Aqueous" removed. Modified sections include sections 8.9 (deleted), 8.8.1.4, 8.8.1.5, 8.8.2.2, 8.8.3.1, 8.8.3.2, 8.8.3.3, and 8.8.4.3.1 ***Alcohol methods 8 rev 0 and 9 rev 0 combined and entire method renumbered to 2.X revision 1***