



8.0 Authentication of Reference Material and Matrix Controls: Ethanol

8.1 BACKGROUND

Refer to Analytical Methods 1.0 and 2.0.

8.2 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.

8.3 EQUIPMENT

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

8.3.2 Columns

Restek Rtx[®]-BAC1

Restek Rtx[®]-BAC2

8.3.3 Headspace (HS) vials and Closures

8.3.4 Hand Crimper or Bench Top Crimper

8.3.5 Semi-Automatic Dilutor/Pipetter equipped with sample and reagent syringes capable of dispensing 250 μ L and 2000 μ L, respectively

8.4 REAGENTS

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

8.4.2 Ammonium Sulfate (Certified ACS Grade)

8.5 REFERENCE MATERIAL

Refer to Analytical Methods 1.0 and 2.0.

8.6 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*.

8.7 QUALITY ASSURANCE

Refer to Analytical Methods 1.0 and 2.0.

8.8 AUTHENTICATION OF ETHANOL REFERENCE MATERIALS**8.8.1 General**

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

8.8.1.2 Aqueous reference material used for quantitative purposes must be traceable to NIST standards.

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

8.8.1.4 New lots of ethanol containing aqueous reference material must be authenticated prior to official use.

8.8.1.5 When a certified volatile reference solution contains components in addition to ethanol, only the ethanol concentration needs to be evaluated.

8.8.2 Authentication Analysis

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

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

8.8.3 Qualitative Authentication

8.8.3.1 Calculate the mean retention time for ethanol using the analysis run calibration data.

8.8.3.2 Compare retention times reported for new reference material lot with retention time obtained for calibration data.

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

8.8.4 Quantitative Authentication

8.8.4.1 Obtain the *Certificate of Analysis* for the reference material lot being authenticated.

- 8.8.4.2 Compare the quantitative data from the analysis of a new lot with the *Certificate of Analysis* values.
- 8.8.4.3 The new lot number of ethanol reference material can be accepted if the mean concentration obtained falls within 6% of the target value (assayed) listed on the *Certificate of Analysis*.
- 8.8.4.4 Evaluation of data must be such that compliance with concentration requirements is apparent.

8.9 AUTHENTICATION OF BLOOD MATRIX CONTROLS

8.9.1 General

- 8.9.1.1 The Discipline Leader or designee will characterize a new lot of blood controls with data provided by all available analysts. When an analyst is unavailable to participate in authentication, the explanation must be documented.
- 8.9.1.2 Each ISP-FS laboratory involved in alcohol/volatiles analysis will provide data for the authentication process.
- 8.9.1.3 Blood control *Package Inserts* will be stored centrally.

8.9.2 Blood Control Authentication Run Requirements

- 8.9.2.1 Each laboratory will provide a minimum of 20 samples (40 determinations).
- 8.9.2.2 The samples must be divided into at least two analysis runs.
- 8.9.2.3 At least three bottles of each control level must be sampled.

8.9.3 Blood Control Authentication Evaluation

- 8.9.3.1 The manufacturer's values will be acknowledged, however, the target value and \pm range for blood control lot will be established through a four decimal place truncated mean of all provided determinations.
- 8.9.3.2 The new blood lot number can be accepted if the following requirements are met:

1. The mean relative retention time for the new control is ± 0.10 minutes of the RRT currently established for ethanol.
2. The mean concentration obtained falls within the range provided in the manufacturer's package insert.

8.9.3.3 A 10% and 5% range will be calculated from the mean value of the determinations and used to evaluate accuracy on subsequent analysis. The 5% range will serve as a warning limit.

8.9.3.4 For blood 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 from the previous run. The values must agree within ± 0.10 minutes.

8.10 AUTHENTICATION DOCUMENTATION

8.10.1 Reference Material

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

8.10.2 Matrix Controls

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

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

8.11 REFERENCES AND RECOMMENDED READING

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

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

- 8.11.3 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.
- 8.11.4 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.
- 8.11.5 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.

Property of Idaho State Police Forensic Services
Uncontrolled Internet Copy
OBSOLETE DOCUMENT

Revision History

8.0 Authentication of Reference Material and Matrix Controls: 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.

Property of Idaho State Police Forensic Services
Uncontrolled Internet Copy
OBSOLETE DOCUMENT