Idaho State Police Forensic Services Toxicology Section

# Section Five Quality Assurance

# 5.1 Pipette Calibration 5.1.2 Option Two: Gravimetric Pipette Calibration

#### 5.1.2.1 SCOPE

The reliability of the volume delivered by piston of plunger operated volumetric apparatus is dependent upon verification of calibration. This manual weighing technique is an option for the verification of a pipette's calibration when larger volumes (≥1mL) are employed.

#### 5.1.2.2 PRINCIPLE

The goal of pipette calibration is to substantiate that the volume delivered by an individual pipet is both accurate and precise. This is accomplished by determining the mass of a volume of liquid of known density that has been delivered into a closed vessel.

#### 5.1.2.3 EQUIPMENT

5.1.2.2.1 Analytical balance

5.1.2.2.2 Thermometer

5.1.2.2.3 Weighing vessels

#### 5.1.2.4 REAGENTS

5.1.2.4.1 Deionized/distilled water

### 5.1.2.5 PROCEDURE

| OIG -        |   |
|--------------|---|
| Frequency of | Calibration Checks                            |
| 5.1.2.5.1.1  | In-house calibration of pipettes by a         |
|              | criminalist or laboratory technician will be  |
|              | valid for four-months. Results and PCS 2™     |
|              | instrument printouts are to be placed in      |
|              | logbook.                                      |
| 5.1.2.5.1.2  | This requirement applies to pipettes          |
|              | employed for quantitative analysis            |
| 5.1.2.5.1.3  | A minimum of 10 data points is to be          |
|              | collected for each pipette calibration.       |
| 5.1.2.5.1.4  | A pipette not in-use need not be calibrated,  |
|              | however, the pipette must be calibrated prior |
|              | to use.                                       |
| 5.1.2.5.1.5  | A calibration check should be performed       |
|              | any time a pipette is serviced.               |
|              |   |

#### 5.1.2.5.2 Initial set-up

All reagents used for the calibration process should be allowed to equilibrate at room temperature for at least one hour.

#### 5.1.2.5.3 Pipette Calibration Verification

- Fill out identifying information on the top 5.1.2.5.3.1 portion of pipette calibration worksheet.
- Determine and record the water temperature 5.1.2.5.3.2 on the worksheet.
- Place a volume of water in the weighing 5.1.2.5.3.3 vessel, which completely covers the bottom of the container, and cap.
- Place the weighing vessel on the balance 5.1.2.5.3.4 and tare.

| 5.1,2.5.3.4               | Place the weighing v                           | essel on the balance                                     |
|---------------------------|--|--|
| 5.1.2.5.3.5               | and tare. With designated pipe                 |  |
|                           | volume of temperatu                            |  |
|                           | into the weighing vess                         |  |
|                           | at least five repetitions                      |  |
| 5.1.2.5.3.6               | For the volume o                               | f water dispensed,                                       |
| `⊗ <sub>x</sub>           | determine the volume                           |  |
| X 21.                     | multiplying the fin                            |  |
| 5, 16,                    | temperature correction                         | factor for water.  |
|                           | / X /  |  |
|                           |  | Competing England  |
| ight office               | Temperature                                    | Correction Factor  |
| Idaho niro k              | Temperature 20                                 | 0.9982   |
| of Idaho nitro F          | <i>Temperature</i> 20 21                       |  |
| of Uncostic L             | Temperature   20   21   22                     | 0.9982   |
| orty of Idaho ortio       | 20<br>21<br>22<br>23                           | 0.9982<br>0.9980   |
| Derty of Idahio ntroll    | Temperature   20   21   22   23   24           | 0.9982<br>0.9980<br>0.9978                               |
| aroperty of Idahoontrolk  | Temperature   20   21   22   23   24   25      | 0.9982<br>0.9980<br>0.9978<br>0.9976                     |
| Property of Idahio ntroll | Temperature   20   21   22   23   24   25   26 | 0.9982<br>0.9980<br>0.9978<br>0.9976<br>0.9974           |
| 5.1.2.5.3.6 P             | 20 21 22 23 24 25 26 27                        | 0.9982<br>0.9980<br>0.9978<br>0.9976<br>0.9974<br>0.9971 |

#### 5.1.2.5.4 Evaporation Check

- Compare the initial weight of the vessel to 5.1.2.5.4.1 the final weight of the vessel, each prior to dispensing the pipet volume.
- The difference in weight between the initial 5.1.2.5.4.2 and final weight is a measure of the evaporation loss (Z).
- Record this weight on worksheet. 5.1.2.5.4.3

#### 5.1.2.5.5 Evaluation of Accuracy

- Calculate and record on worksheet the mean 5.1.2.5.5.1 volume delivered.
- If a loss to evaporation (Z) occurred, add 5.1.2.5.5.2 loss due to evaporation to mean weight. Convert weight of loss to a volume with correction factor as described above. Record adjusted mean volume.
- Determine inaccuracy by calculating the 5.1.2.5.5.3 percent error between the expected (E) and calculated mean(C) volume.

$$% error = \frac{C - E}{E}$$

### 5,1,2,5,6

Evaluation of Precision

5.1.2.5.6.1 Determine the imprecision by calculating the standard deviation for the replicate the staweights.

SDEN

SDEN

SPORTHOLING

STORES

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$$SO = \sum (W_1 \overline{W})^2 (Z)$$
N-1

- Observed weight
- Mean of weights
- Evaporation Loss
- Total number of observed values
- Record SD on worksheet.
- Calculate the relative standard deviation (RSD)/Coefficient of variation (CV)

$$RSD = \frac{100 (SD)}{\overline{W}}$$

Accuracy and Precision Tolerance Limits 5.1.2.5.7 Refer to section 5.1.1 for Acceptance Criteria. Idaho State Police Forensic Services Toxicology Section

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| Section Five-<br>5.1 Pipett | rocedural Mai<br>Quality Assur<br>e Calibration<br>Option Two: |                                     | ration .ce |
| Revision #                  | Issue Date   | History                             | Servi      |
| 0                           | 10/97<br>11-27-01  | Original Issue Reworked/reformatted | COPY       |
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| QC Manage                   | Rick I   | D. Groff                            | Date:      |

Toxicology Program Methods Manual

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Toxicology Procedural Manual Section Five- Quality Assurance

5.1 Pipette Calibration

5.1.2 Option Two: Gravimetric Pipette Calibration

Revision # Issue Date History

0 10/97 1 11-27-01 Original Issue

Reworked/reformatted

Approval

Technical Leader:

S C Williamson

Date: \_//-27-0/

Issuance

QC Manager

Rick D. Groff

Date: /-24-02-

rev. 1
Issued: 11/01
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Toxicology Program Methods Manual

**Idaho State Police Forensic Services Toxicology Section** 

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Toxicology Procedural Manual Section Five- Quality Assurance

Pipette Calibration 5.1

Option One: PCS 2TM Pipette Calibration System

Revision # Issue Date History

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11-27-01

Approval

Technical Leader:

Date: 11-27-01

Issuance

QC Manager:

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Date: 1-25-0

Issued: 11/01

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Idaho State Police Forensic Services Toxicology Section

### Section Five

#### Quality Assurance

### 5.1 Pipette Calibration

### 5.1.1 Option One: PCS 2™ Pipette Calibration System

#### 5.1.1.1 <u>Standard Operating Procedure</u>

Refer to Standard Operating Procedure for the PCS 2<sup>™</sup> Ripette Calibration System. Artel Document #310A2715A, April 1997.

### 5.1.1.2 <u>Frequency of Calibration Checks</u>

- In-house calibration of pipettes by a criminalist or laboratory technician will be valid for four-months.

  Results and PCS 2™ instrument printouts are to be placed in logbook.
- 5.1.2.2.2 This requirement applies to pipettes employed for quantitative analysis
- 5.1.1.2.3 A minimum of 10 data points is to be collected for each pipette calibration.
- A pipette not in-use need not be calibrated, however, the pipette must be calibrated prior to use.
- 5.1.1.2.5 A calibration check should be performed any time a pipette is serviced.

# 5.1.1.3 Manufacturer Data Acceptance Criteria

5.1.1.3.1 Artel recommendations for Piston-stroke Pipette Tolerance

| Pipette Volume | Inaccuracy | Imprecision |
|----------------|------------|-------------|
| 2μL            | 5.0%       | 2.0%        |
| 10uL           | 5.0%       | 2.0%        |
| 20μL           | 5.0%       | 2.0%        |
| 100µL          | 5.0%       | 2.0%        |
| 200µL          | 5.0%       | 2.0%        |
| 1000μL         | 5.0%       | 2.0%        |

# 5.1.1.3.2 Eppendorf recommendations for Piston-stroke Fixed Volume Pipette Tolerance Limits

| Pipette Volume | Inaccuracy | Imprecision |
|----------------|------------|-------------|
| 1µL            | ±2.5%      | ≤1.8%       |
| 2μL            | ±2.0%      | ≤1.2%       |
| 10μL           | ±1.5%      | ≤0.8%       |
| 20μL           | ±1.0%      | ≤0.5%       |
| 100μL          | ±0.8%      | ≤0.3%       |
| 200μL          | ±0.7%      | ≤0.3%       |
| 1000μL         | ±0.6%      | ≤0.2%       |

5.1.1.3.3 Eppendorf recommendations for Repeater Plus Pipette Tolerance Limits

| TOPPIANCE LIMITS                   | Inaccuracy | Imprecision            |
|------------------------------------|------------|------------------------|
| Combitip Plus 0.1mL (beige piston) |            |                        |
| 2μL                                | ±1.6%      | ≤3.0%                  |
| 20μL                               | ±1.0%      | ≤2.0%                  |
| Combitip Plus 0.2mL (blue piston)  |            | 100                    |
|                                    | ±1.3%      | ≤2.0%                  |
| 40μL                               | ±0.8%      | ≤1.5%                  |
| Combitip Plus 0.5mL                |            | 4 - 19 (8 (8 cg - 5)   |
| 10μL                               | ±0.9%      | ≤1.5%                  |
| 100μL                              | ±0.8%      | ≤0.6%                  |
| Combitip Plus 1mL                  |            | a serial de la compani |
| 20μL                               | ±0.9%      | ≤0.9%                  |
| 200μL                              | ₩0.6%      | ≤0.4%                  |
| Combitip Plus 2.5mL                |            |                        |
| 50μL                               | £0.8%      | ≤0.8%                  |
| 500μL                              | ±0.5%      | ≤0.3%                  |
| Combitip Plus 5mL                  |            |                        |
| 100μL                              | ±0.6%      | ≤0.6%                  |
| 1000μL                             | ±0.5%      | ≤0.25%                 |
| Combitip Plus 10mL                 |            |                        |
| 200µL                              | ±0.5%      | ≤0.6%                  |
| 2000μL                             | ±0.5%      | ≤0.25%                 |
| Combitip Plus 25mL                 |            |                        |
| 500pL                              | ±0.4%      | ≤0.6%                  |
| 5000μΙ                             | ±0.3%      | ≤0.25%                 |
| Combitip Plus 50mL                 |            |                        |
| 0 1600peL                          | ±0.3%      | ≤0.5%                  |
| 10000μL                            | ±0.3%      | ≤0.25%                 |

5.1.1.4 <u>User Data Acceptance Criteria</u>

Refer to package insert, for tolerance limits observed by manufacturer for each individual pipette, not listed above.

Initially apply the tolerance limits recommended by ARTEL {5.1.1.2.1}. When a history for an individual pipette is established, the tolerance limits should be fine-tuned and tightened accordingly. Refer to ARTEL publication issue 5 (March 1999) for information regarding

tolerance setting.

| 5.1.1.5 | References<br>5.1.1.5.1<br>5.1.1.5.2 | PCS 2 <sup>™</sup> Pipette Calibration System Procedure Guide<br>ASTM Method E-1154-89 (reapproved 1993), Standard<br>Specification for Piston or Plunger Operated                |
|---------|--------------------------------------|---|
|         | 5.1.1.5.3<br>5.1.1.5.4               | Volumetric Apparatus. Setting Tolerances for Pipette Performance, Artel lab report, issue 5, March 1999. Eppendorf Series 2000 Reference Fixed-Volume Pipettes Instruction Manual |

2

| 5.1.1.5.5 | Eppendorf Series 2000 Reference Adjustable-Volume   |
|-----------|---|
|           | Pipettes Instruction Manual                         |
| 5.1.1.5.6 | Eppendorf Repeater® Plus Pipette Instruction Manual |
| 5.1.1.5.7 | Eppendorf Repeater Pipette Instruction Manual       |
| 5.1.1.5.8 | MLA Macro and macro Selectable Pipette Operator's   |
|           | Manual  |

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#### Ethanol and Other Volatiles

| Revision # | Issue Date | History                               |
|------------|------------|---------------------------------------|
| 0          | 05-30-2000 | Original Issue                        |
| 1          | 12-16-2002 | Updated to comply with Quality Manual |
| 2          | 08-18-2004 | Updated, refined, reformatted.        |

Discipline Leader

Issuance

Quality Assurance Manager

### Idaho State Police Forensic Services Toxicology Discipline

**Section Two** 

#### Ethanol and Other Volatiles

### 2.1 TRAINING OBJECTIVES

This section of the toxicology training manual has many objectives. It is intended to serve as a guide for an Idaho State Police Forensic Services (ISP-FS) analyst training to perform quantitative ethanol and qualitative "other volatiles" analysis, in both biological and non-biological samples. The analysis of these samples is described in SOPs 4.1-Quantitative Analysis for Ethanol and Qualitative Analysis for Other Volatiles in Blood, Vitreous Humor and Urine by Dual Column Headspace Gas Chromatography and 4.2-Analysis of Solutions Containing Ethanol and Common Volatiles. The following subsections address other related issues including administrative issues, the submitted of the sample to the laboratory, collection kit requirements and documentation, instrumental analysis, preparation of laboratory notes, issuance of the analysis report and subsequent courtroom testimony. In order to address questions in court, the analyst must possess knowledge of the pharmacology of ethanol and related compounds, field testing to detect impairment and the associated Idaho Codes. The references cited, or equivalent, should be consulted if the analyst is unfamiliar with the subject matter.

To facilitate the over-all process, training for SOP 4.1 and 4.2 must be pursued concurrently. Answers to questions may be provided verbally and/or in written form. As part of the training process, the Trainee should assist the Trainer with the preparation of samples for analysis as well as perform analysis on blood control samples. Due to the nature of the analysis of biological fluids to detect ethanol and other volatiles, the Trainee should successfully complete the required competency test prior to supervised performance of the SOPs on actual case material.

### 2.2 ADMINISTRATIVE ISSUES

- 2.2.1 The Trainee should be familiar with the Idaho State Policies Manual.
- 2.2.2 The Trainee should be knowledgeable of the content and application of the Forensic Services Quality Manual.
- 2.2.3 The Trainee should be well informed in the content and application of the Forensic Services Health and Safety Manual.

#### 2.3 EVIDENCE HANDLING ISSUES

- 2.3.1 The Trainee should describe the procedures followed for the intake and transfer of specimens submitted for alcohol and/or volatiles analysis.
- 2.3.2 The Trainee should describe the barrier protection measures required when handling biological samples and unknown liquids.
- 2.3.3 The Trainee should describe what IDAPA 11.03.01 mandates as the proper way to collect a blood and urine sample for a forensic ethanol analysis.
- 2.3.4 The Trainee should describe the types and applications of the toxicology collection kits distributed by ISP-FS.
- 2.3.5 The Trainee should discuss how the ISPPS kits comply with IDAPA 11.03.01.
- 2.3.6 The Trainee should discuss the preservative and anticoagulant required for IDAPA compliant blood collection tubes in terms of consequences of using an improper tube.
- 2.3.7 The Trainee should describe the agencies served by their laboratory and the programs involved.
- 2.3.8 References
  - 1. ASTM E1459-92, Standard Guide for Physical Evidence Labeling and Related Documentation.
    - Kippenberger, D.J. and Selavka, C.M. *Training in Specimen Handling* pp. 33-54, *in:* California Association of Toxicologists (CAT) Manual for Analytical Toxicology. 1994.
  - 3. IDAPA 11, Title 03, Chapter 01: Idaho State Forensic Laboratory Rules Governing Alcohol Testing.

#### 2.4 SOLUTION PREPARATION

- 2.4.1 The Trainee should demonstrate the ability to prepare, and record the preparation of, solutions required in the analysis of alcohol and other volatiles.
- 2.4.2 The Trainee should demonstrate a thorough understanding of the nomenclature and calculations involved in the determination of weight percent and volume percent solutions.

#### 2.4.3 References

2. College Chemistry Text, chapter(s) discussing the properties of solutions.

## 2.5 GAS CHROMATOGRAPHY (GC) THEORY AND OPERATION

- 2.5.1 The Trainee should possess a comprehensive background in regards to the principles of GC.
- 2.5.2 The Trainee should provide a brief explanation of GC in terms understandable to a layperson.
- 2.5.3 The Trainee should describe the influence carrier gas flow has on the efficiency of a GC-FID.
- 2.5.4 Define the following terms as they relate to GC.
  - 2.5.4.1 Resolution
  - 2.5.4.2 Area Under the Curve
  - 2.5.4.3 *HETP*
  - 2.5.4.4 Sensitivity versus Specificity
- 2.5.5 Discuss which GC parameters affect resolution. Describe how to approach a lack of resolution.
- 2.5.6 Discuss measures to alleviate peak tailing.
- 2.5.7 The Trainee should possess an understanding of the principles and application of quantitative analysis.
- 2.5.8 The Trainee should describe how amount ratios and response ratios are used to construct a calibration curve.
- The Trainee should discuss the major advantages of using an internal standard method.
- 2.5.10 The Trainee should demonstrate their ability to operate a GC equipped with a flame ionization detector (FID) through both the system software and the instrument touch pad.
- 2.5.11 The Trainee should demonstrate a working knowledge of the GC operating software. The Trainee should have the ability to utilize the system software to develop an analysis method, prepare an analysis sequence, reprocess data, perform a manual calibration, and modify the analysis report format and setting processing parameters to optimize peak detection and integration.

2.5.12 The Trainee should demonstrate their ability to maintain a GC equipped with a flame ionization detector (FID). This includes inlet and detector maintenance, column installation, troubleshooting techniques and the documentation thereof.

#### 2.5.13 References

- 1. Stafford, D.T. *Chromatography*. pp. 93-101, 103-114, *in:* Principles of Forensic Toxicology, edited by Barry Levin, , AACC, 1999.
- 2. Levine, B. Alcohol. pp. 170-184, in: Principles of Forensic Toxicology, edited by Barry Levin, AACC, 1999.

## 2.6 HEADSPACE THEORY AND OPERATION

- 2.6.1 Trainee should possess a working knowledge of the theory and practice of headspace analysis.
- 2.6.2 The Trainee should describe how the proportionality known as Henry's Law, is utilized in headspace analysis.
- 2.6.3 The Trainee should demonstrate their ability to operate a Headspace Analyzer through both the system software and the instrument touch pad.
- The Trainee should be acquainted with how the headspace method parameters such as the GC cycle time, thermostating time, pressurization time, etc., should be optimized.
- 2.6.5 The Trainee should demonstrate their understanding of the system software as it applies to the headspace analyzer including setting up the HS analysis method.
- The Trainee should demonstrate their ability to maintain a headspace analyzer. This includes replacement of seals and sampling needle, transfer line replacement, adjustment of the hand crimper, troubleshooting techniques and the documentation thereof.

#### 2.6.7 References

- 1. Stafford, D.T. *Chromatography*. pp. 93-101, 103-114, *in:* Principles of Forensic Toxicology, edited by Barry Levin, AACC, 1999.
- 2. Saker, E.G. Screening and Quantitation by Headspace Technique of Some of the Vapors Most Commonly Found in Forensic

Toxicology, pp. 1-33, in: Current Approaches in Forensic Toxicology, Chapter 11, SOFT Meeting, 1994.

## 2.7 PIPETTE CALIBRATOR THEORY AND OPERATION

- 2.7.1 The Trainee should have a working knowledge of how to prepare the ARTEL PCS 2<sup>TM</sup> Pipette Calibration System to perform pipette calibration.
- 2.7.2 The Trainee should describe the operating principle of the PCS 2<sup>TM</sup> Pipette Calibration System.
- 2.7.3 The Trainee should demonstrate their ability to operate the PCS 2<sup>TM</sup> Pipette Calibration System through completing a calibration check on the syringes for the sample dilutor.
- 2.7.4 The Trainee should explain the routine maintenance performed on the PCS 2<sup>TM</sup> Pipette Calibration System.
- 2.7.5 The Trainee should outline the requirements for pipette calibration in regards to frequency and acceptance criteria.
- 2.7.6 References
  - 1. PCS 2<sup>TM</sup> Pipette Calibration System Procedure Guide.
  - 2. ISP-FS Standard Operating Procedure 5.1, Pipette Calibration.

2.8 SAMPLE DILUTOR OPERATION

- 2.8.1 The Trainee should have a working knowledge of the Hamilton MICROLAB® dilutor.
- The Trainee should demonstrate the operation of the Hamilton MICROLAB® dilutor.
- 2.8.3 The Trainee should describe the routine maintenance performed on the Hamilton MICROLAB® dilutor.
- 2.8.4 References
  - 1. Hamilton MICROLAB® User's Manual.

### 2.9 STANDARD OPERATING PROCEDURES

2.9.1 SOP 4.1

2.9.1.1 The Trainee should convey their understanding of the analysis protocol in SOP 4.1 for the *Quantitative Analysis for* 

Ethanol and Qualitative Analysis for Other Volatiles in Blood, Vitreous Humor and Urine by Dual Column Headspace Gas Chromatography.

- 2.9.1.2 Trainee should describe the types of samples which qualify for analysis with SOP 4.1.
- 2.9.1.3 Trainee should describe the quality assurance requirements described in SOP 4.1.
- 2.9.1.4 Trainee should describe the acceptance criteria for an analysis run.
- 2.9.1.5 Trainee should describe how quality assurance data is monitored and where it should be stored.
- 2.9.1.6 Trainee should describe the authentication process for both quantitative and qualitative ethanol and other volatiles standards and controls.
- 2.9.1.7 Trainee should describe how blood, urine and vitreous humor alcohol concentrations should be reported.
- 2.9.1.8 Traince should describe how qualitative volatiles should be reported.
- 2.9.1.9 To develop their expertise in using the SOP, the Trainee will practice the SOP on control samples and/or old proficiency test samples.

#### 2.9.1.10 References

- 1. ISP-FS Standard Operating Procedure 4.1, Quantitative Analysis for Ethanol and Qualitative Analysis for Other Volatiles in Blood, Vitreous Humor and Urine by Dual Column Headspace Gas Chromatography.
- 2. Idaho Administration Code, IDAPA 11.03.01, Rules Governing Alcohol Testing.
- 3. 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.
- 4. Restek Applications Note #59598, Dual-Column Confirmational GC Analysis of Blood Alcohols Using

the Rtx<sup>®</sup>-BAC1 and Rtx<sup>®</sup>-BAC2 Columns Optimized for the Perkin-Elmer HS-40 Headspace Autosampler, 1999.

- 5. Stafford, D.T., *Chromatography. in:* Principles of Forensic Toxicology, edited by Barry Levin, pp. 93-101, 103-114, AACC Press, 1999.
- 6. Levine, B., *Alcohol. in:* Principles of Forensic Toxicology, edited by Barry Levine pp. 170-184, AACC Press, 1999.
- 7. 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. Saker, E.G., Screening and Quantitation by Head Space Technique of Some of the Vapors Most Commonly Found in Forensic Toxicology, in: Current Approaches in Forensic Toxicology, Chapter 11, SOFT Meeting, 1994.
- 9. Klaassen, C.D., Inhalants, in: Principles of Forensic Toxicology, edited by Barry Levin, pp. 341-348, AACC Press, 2003.
- 2.9.2 <u>SOR 4.2</u>
- SOR 4.2
  2.92.1 The Trainee should convey their understanding of the analysis protocol in SOP 4.2 for the Analysis of Solutions Containing Ethanol and Common Volatiles.
  - 2.9.2.2 Trainee should describe the types of samples that SOP 4.2 is applied for.
  - 2.9.2.3 Trainee should describe the quality assurance requirements described in SOP 4.2.
  - 2.9.2.4 Trainee should describe the acceptance criteria for an analysis run.
  - 2.9.2.5 Trainee should describe how quality assurance data is monitored and where it should be stored.

- 2.9.2.6 Trainee should describe the authentication process for both quantitative and qualitative ethanol and other volatiles standards and controls.
- 2.9.2.7 The Trainee should discuss the different types of alcoholic beverages and their respective alcohol content.
- 2.9.2.8 Trainee should describe how alcohol concentrations should be reported in alcoholic beverages, simulator solutions and unknown solutions.
- 2.9.2.9 Trainee should describe how qualitative volatiles should be reported.
- 2.9.2.10 To develop their expertise in using the SOP, the Trainee will practice the SOP on control samples and/or old proficiency test samples.

#### 2.9.2.11 References

- 1. ISP-FS Standard Operating Procedure 4.2, Analysis of Solutions Containing Ethanol and Common Volatiles.
- 2. 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.
- Restek Applications Note #59598, Dual-Column Confirmational GC Analysis of Blood Alcohols Using the Rtx<sup>®</sup>-BAC1 and Rtx<sup>®</sup>-BAC2 Columns Optimized for the Perkin-Elmer HS-40 Headspace Autosampler, 1999.
- 4. Stafford, D.T., *Chromatography. in:* Principles of Forensic Toxicology, edited by Barry Levin, pp. 93-101, 103-114, AACC Press, 1999.
- 5. Levine, B., *Alcohol. in:* Principles of Forensic Toxicology, edited by Barry Levin, pp. 170-184, AACC Press, 1999.
- 6. McAnalley, B.H. *Chemistry of Alcoholic Beverages*. pp. 1-27, *in:* Medicolegal Aspects of Alcohol, edited by James C. Garriott, Lawyers & Judges, 1996.

#### 2.10 CASEFILE PREPARATION

- 2.10.1 The Trainee should describe which documents, data and completed worksheets are required to be included in an alcohol/other volatiles analysis casefile.
- 2.10.2 The Trainee should describe requirements for analysis worksheets and data included in casefile.
- 2.10.3 The Trainee should describe requirements for review of casefile and analysis report.

#### 2.10.4 References

1. Idaho State Police Forensic Services Quality Manual, rev. 5, July 2004.

### 2.11 PHARMACOLOGY AND IMPAIRMENT DETECTION

- 2.11.1 The Trainee should demonstrate a working knowledge of the pharmacology of alcohol and other commonly encountered volatiles. This should include an understanding of the factors affecting absorption, distribution and elimination.
- 2.11.2 The Trainee should describe the situation when the alcohol content of arterial blood exceeds that of venous blood.
- 2.11.3 The Trainee should be familiar with the metabolism of ethanol and other commonly encountered volatiles. This should include how metabolism relates toxicity.
- 2.11.4 The Trainee should describe their understanding of the effects of alcohol and other commonly encountered volatiles on the human body. This should include how it contributes to mortality and impairment observed in DUI cases.
- 2.11.5 The Trainee should describe their understanding of postmortem changes and their effect on alcohol concentration.
- 2.11.6 The Trainee should be comfortable with the development, performance and interpretation of Standardized Field Sobriety Tests (SFST) and a Drug Recognition Exam (DRE).

#### 2.11.7 References

1. Levine, B. *Alcohol.* pp. 170-184, *in:* Principles of Forensic Toxicology, edited by Barry Levin, AACC, 1999.

- 2. Kunsman, G.W. *Human Performance Testing*. pp. 170-184, *in*: Principles of Forensic Toxicology, edited by Barry Levin, AACC, 1999.
- 3. Caplan, Y.H. *The Determination of Alcohol in Blood and Breath.* pp. 594-648, *in:* Forensic Science Handbook, edited by Richard Saferstein, New Jersey:Prentice-Hall, 1982.
- 4. Julien, R.M. Central Nervous System Depressants: Alcohol and the Inhalants of Abuse. pp. 64-92, in: Primer of Drug Action, New York:Freeman, 1998.
- 5. Perrine, D.M. Depressants: Alcohol, Benzodiazepines, Barbiturates, pp. 113-129, in: The Chemistry of Mind-Altering Drugs, ACS, Washington, DC, 1996.
- 6. Hobbs, W.R., Rall, T.W. and Verdoorn, T.A. Drugs Acting on the Central Nervous System Hypnotics and Sedatives; Ethanol. pp. 361, 386-393, in: Goodman and Gilman's The Pharmacological Basis of Therapeutics, McGraw-Hill, 1996.
- 7. Garriott, J.C. *Pharmacology and Toxicology of Ethyl Alcohol.* pp. 35-54, *in:* Medicolegal Aspects of Alcohol, edited by James C. Garriott, Lawyers & Judges, 1996.
- 8. Baselt, Randall Disposition of Alcohol in Man. pp. 65-78, in: Medicolegal Aspects of Alcohol, edited by James C. Garriott, Lawyers & Judges, 1996.

Garriott, J.C. Analysis for Alcohol in Postmortem Specimens. pp. 151-163, in: Medicolegal Aspects of Alcohol, edited by James C. Garriott, Lawyers & Judges, 1996.

# 2.12 PREPARATION AND PRESENTATION OF COURTROOM TESTIMONY

- 2.12.1 The analyst should discuss proper demeanor and body language while testifying in court.
  - 2.12.2 The analyst should describe proper attire for court.
  - 2.12.3 The analyst should discuss ways to deal with nervousness while testifying.
  - 2.12.4 The analyst should describe how a casefile should be reviewed in preparation for testimony.

- 2.12.5 The analyst should describe the typical sequence of questions pursued during direct and cross-examination.
- 2.12.6 The analyst should discuss the implications of the following events:
  - 2.12.6.1 Stipulation
  - 2.12.6.2 Objection Over-ruled
  - 2.12.6.3 Objection Sustained
- 2.12.7 The Trainee should discuss sections of Idaho Code where the analysis of biological or unknown samples could be applied.
- 2.12.8 References
  - 1. Osgood, C. Osgood On Speaking. William Morrow: New York, 1988.
  - 2. Weingarten, H. *The Expert Witness: the Toxicologist in Court.* pp. 225-242, *in:* California Association of Toxicologists (CAT) Manual for Analytical Toxicology Training, 1994.
  - 3. Sannito, T. Nonverbal Communication in the Courtroom. Champion, Sept.-Oct., 1985.
  - 4. Idaho Code §18 8002, §18-8004, §18-8006, §23-1333.

2.13 MOCK COURTROOM TESTIMONY

As appropriate for the SOP(s) the Trainee is training for, conduct a mock court trial for the Trainee to provide testimony for a minimum of the following situations.

- 1. DUI blood alcohol analysis with pharmacology questions.
- "Open container violation" including questions about the alcohol concentration of various types of alcoholic beverages.

2.14 COMPETENCY TESTING

Upon the completion of training, the Trainee should complete a competency test consisting of ≥six (6) blood specimens and ≥two (2) non-biological solutions suspected to contain ethanol. The blood specimens should contain a wide range of alcohol concentrations and a minimum of one commonly encountered other volatile.

### 2.15 PERFORMANCE OF ANALYSIS ON CASE MATERIAL

Upon successful completion of competency testing and when possible, proficiency testing, the Trainee should complete no less than 30 case samples under close supervision. The 30 samples must be divided into a minimum of two analysis runs. For purposes of this process, close supervision is at the discretion of the Trainer. The Trainer will cosign these case reports. A listing of the co-signed case samples should be compiled and included in training records.

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|       | ensic Services<br>icology Discipline |                               |
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| Secti | on Two                               |                               |
|       | nol and Other Volatiles              |                               |
| Topi  | c Completion Sign-off                |                               |
| 2.2   | ADMINISTRATIVE ISSU                  | JES                           |
|       | D. t. of Convolution                 | Trainee                       |
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|       |                                      | Trainer                       |
| 2.3   | EVIDENCE HANDLING                    | ISSUES                        |
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|       | Date of Completion                   | ISSUES  Trainer  Trainer      |
| 2.4   | SOLUTION PREPARAT                    | ION                           |
|       | osty Jos                             |                               |
|       | Date of Completion                   | Trainee                       |
|       |                                      | Trainer                       |
| 2.5   | GAS CHROMATOGRAI                     | PHY (GC) THEORY AND OPERATION |
|       |                                      |                               |
|       | Date of Completion                   | Trainee                       |
|       |                                      | Trainer                       |

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| 2.6  | HEADSPACE THEORY AND C                  | OPERATION |  |  |
|------|---|-----------|--|--|
|      | Date of Completion                      | Trainee   |  |  |
|      |   | Trainer   |  |  |
| 2.7  | PIPETTE CALIBRATOR THEORY AND OPERATION |           |  |  |
|      |   | Trainee   |  |  |
|      | Date of Completion                      | Trainee   |  |  |
|      |   | Trainer   |  |  |
| 2.8  | SAMPLE DILUTOR OPERATION                | ION & X   |  |  |
|      |   | Trainer   |  |  |
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|      | "Tho Tho!                               | Trainer   |  |  |
| 2.9  | STANDARD OPERATING PR                   | ØČEDURES  |  |  |
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|      | Date of Completion                      | Trainee   |  |  |
|      |   | Trainer   |  |  |
| 2.10 | CASEFILE PREPARATION                    |           |  |  |
|      |   |           |  |  |
|      | Date of Completion                      | Trainee   |  |  |
|      |   | Trainer   |  |  |

| 2.11 | PHARMACOLOGY AND IMPAIRMENT DETECTION |                                     |  |  |
|------|---------------------------------------|-------------------------------------|--|--|
|      | Date of Completion                    | Trainee                             |  |  |
|      |                                       | Trainer                             |  |  |
| 2.12 | PREPARATION AND                       | PRESENTATION OF COURTROOM TESTIMONY |  |  |
|      |                                       | Trainee Schille                     |  |  |
|      | Date of Completion                    | Trainee                             |  |  |
|      |                                       | Trainer                             |  |  |
| 2.13 | MOCK COURTROO                         | M TESTIMONY                         |  |  |
|      | Date of Completion                    | Trainee Company                     |  |  |
|      | Date of Completion                    | State Trainer                       |  |  |
| 2.14 | COMPETENCY TES                        | тие                                 |  |  |
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|      | Date of Completion                    | Trainee                             |  |  |
|      | 640,                                  | Trainer                             |  |  |
| 2.15 | PERFORMANCE OF                        | ANALYSIS ON CASE MATERIAL           |  |  |
|      |                                       |                                     |  |  |
|      | Date of Completion                    | Trainee                             |  |  |
|      |                                       | Trainer                             |  |  |

| Idaho State Police Forensic Services Toxicology Discipline |               |   |  |
|--|---------------|---|--|
| Section Two  |               |   |  |
| Ethanol and Otl  | ier Volatiles |   |  |
| Revision #   | Issue Date    | History   |  |
| 0  | 05-30-2000    | Original Issue  |  |
| 1  | 12-16-2002    | Updated to comply with Quality Manual   |  |
| 2  | 08-18-2004    | Updated, refined, reformatted.  |  |
| Approval  Discipline Leade                                 | er Si         | Original Issue  Updated to comply with Quality Manual  Updated, refined, reformatted.  Date |  |
| Discipino Louis  | * Idahontic   |   |  |
| Susan C. Willian   | nspir U       | Date  |  |
| Issuance   |               |   |  |
| Quality Assuran  | nce Manager   |   |  |
| Richard D. Grof  | f             |   |  |

Idaho State Police Forensic Services **Toxicology Section** 

Section Five

Quality Assurance

#### 5.8 **Evaluation of Quality Assurance Measures**

#### SCOPE 5.8.1

This SOP deals with the evaluation of quality as confidence in results obtained from toxicology

#### EVALUATION CRITERIA FOR QUAI 5.8.2 **METHODS**

5.8.2.1

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standard, a coversheet providing the information necessary 101 authentication should accompany the GC-MSD printout. The coversheet should, at a minimum, include the lot number, vendor, date of analysis, analyst, and reference used for authentication. The container for the standard will be marked "authenticated" after the authenticity of the standard has been confirmed. The coversheet and MSD print-out will be stored centrally in the laboratory using the standards. It is the responsibility of each analyst to verify that each standard used has been properly authenticated.

Non-extracted Standards (NES)

Standards should be compared to previously analyzed standards along with literature sources. No significant differences should be apparent.

5.8.2.3 Extracted Positive Controls (EPC)

> Positive controls should exhibit proper retention time and mass spectral characteristics for compounds of interest based upon NES.

5.8.2.4 Extracted Negative Control (ENC)

> To verify a lack of contamination in the extraction procedure, the negative control should be examined to satisfy the analyst that it does not contain the compound(s) of interest or interfering

substances. This should be confirmed by extracting appropriate ions in the apposite retention time window.

#### 5.8.2.5 Solvent Blanks

To verify a lack of carryover between samples analyzed on a gas chromatograph equipped with a mass selective detector (GC-MSD), a solvent blank should be run between sample extracts. Ideally no carryover will be observed. If the solvent blank contains a reportable analyte of interest, in order to identify this analyte in the specimen which follows, the corrected area of the analyte peak must be a minimum of 10 times stronger than the corresponding peak in the blank preceding it. If the corrected area does not meet this criterion, the following action must be taken. A new solvent blank should be obtained and the GC-MSD analysis for the sample the blank was intended for, should be repeated. Reportable is defined as a complete fragmentation pattern at the appropriate retention time. Analytes of interest include, but are not limited to, analytes routinely reported.

In addition, it is highly recommended but not required, based upon screening technique(s) samples be run ascending order of concentrations. Samples which are shown by the screen to have the potential of a significant concentration may be diluted prior to extraction.

### 5.8.3 DISTRIBUTION OF QUALITY DATA

5.8.3.1 Documentation of analy

Documentation of analytical standard authentication and originals of casework quality controls (NES, EPC, ENC) will be stored centrally in the file designated for urine toxicology quality data in the laboratory where the analysis was performed. Copies of all quality assurance samples need not be placed in each case file.

5.8.3.2 Copies of analytical standards used to substantiate the identification of each drug compound must be included in each case file if not otherwise indicated in individual SOPs.

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**Toxicology Procedural Manual Section Five** 

Quality Assurance

Evaluation of Quality Assurance Measures 5.8

| Revision #        | <b>Issue Date</b> | History Original Issue  |
|-------------------|-------------------|---|
|                   |                   | CO)   |
| 0.                | 10-18-02          | Original Issue  |
| 1                 | 04-16-03          | Clarifications, Updated.  |
| 2                 | 07-23-03          | Clarification of Authentication Process   |
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|                   | CKOL              | 91.00   |
| Technical Leader: | S.C. Williamson   | Date:   |
| Issuance          | of Jugge          | )   |
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| QC Manager:       |                   | Date:   |
| X Z               | Rick D. Groff     |   |

Toxicology Program Method Manual

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**Toxicology Procedural Manual** 

Section Five

Quality Assurance

5.8 Evaluation of Quality Assurance Measures

| Revision # | Issue Date | History                                  |
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| 0          | 10-18-02   | Original Issue                           |
| 1          | 04-16-03   | Clarifications, Electrication Process.   |
| 2          | 07-23-03   | Clarification of Authentication Process. |

Approval

Technical Leader:

S.C. Williamson

Date: 08/08/03

Issuance

QC Manager:

Rick D. Groff

Date:

rev. 2 Issued: 07/03

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