

FORENSIC SECTION PROCEDURE MANUAL

FORENSIC ALCOHOL ANALYSIS

QUANTITATIVE ALCOHOL ANALYSIS BY
HEADSPACE GAS CHROMATOGRAPHY

I. Equipment Used:

1. Hewlett Packard 5890 G.C.
2. Hewlett Packard 19395A Autosampler
3. Hewlett Packard 3396A Integrator
4. Micro Lab 400 Series. Auto Dilutor

II. Preparing Dilutor:

1. Check that there is enough internal standard for the analysis.
2. Place dilutor in "Run" mode.
 - A. Set Reagent = 2000 μ l.
 - B. Set Sample = 250 μ l.

III. Preparing Samples:

1. Label each sample vial with Blood Alcohol Number (i.e. CB92-222).
2. Pour approximately 0.5 ml of case sample into a disposable tube.
3. Aspirate and dispense sample into vial. Prepare a separate duplicate sample.
4. Aspirate and dispense water into waste flask.
5. Continue with next sample.

IV. Preparing the Blank and Controls:

1. Prepare the blank (a vial containing room air, internal standard and water).
2. Prepare the controls. The control blood is an independently acquired blood sample containing known

amount of ethyl alcohol and is prepared exactly like a case sample.

V. Preparing Standards:

1. Prepare the standards using the same procedure as the case samples.
2. Use a .10, .20, .30, w/v ethyl alcohol standards and a mixed standard.

VI. Preparing for a Run:

1. Place the vials in the headspace sampler in the following order:
.10, .10, .20, .20, .30, .30, mix, blank, blood control, cases (in duplicate), check standards (.10, .20, .30).

VII. Headspace and GC Parameters:

Headspace analyzer:		GC parameters:	
Headspace method	1	Inlet temp	250 C
Equilibration time	20min	Oven temp	40 C
Bath temp.	40 C	Temp prog	2C/min
Valve/loop temp.	65 C	Detect. temp	250 C
Sampling interval	remote	Final temp	50 C
Last vial number	?		
Method sequence	1		
Vial	1		
Injections/vial	1		
Valve timing	min:sec		
Probe	"01"		
Pressurize	"03"		
Pressurize	"13"		
Vent/fill loop	"14"		

Vent/fill loop	"19"
Inject	"20"
Inject	"30"
Probe	"31"
Carrier flow (He)	1.6 bar
Aux pressure (He)	1.7 bar

VIII. Post Run Analysis:

1. Check analytical results including linearity, accuracy and precision.
2. Record duplicate results of each case sample and report the average of the values.

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I. Equipment:

A. Hewlett Packard Instruments:

1. 5890 Gas Chromatograph
2. 19395A Autosampler
3. 3396A Integrator

B. Micro Lab 400 Series. Auto Dilutor

C. Crimper - Hewlett Packard - Cat. #9301-0720

II. Supplies:

A. Septa - Hewlett Packard - Cat. #9301-0976

B. Crimp Caps - Hewlett Packard - Cat. #9301-0721

C. 20 ml Headspace Vials - Hewlett Packard - Cat. #9301-0717

D. Whole Blood Control - Behring Diagnostics - Cat. #860161

E. Acetonitrile - Fisher Scientific

F. Methanol - Fisher Scientific

G. Acetone - Fisher Scientific

H. Isopropyl Alcohol - Fisher Scientific

I. Acetaldehyde - Fisher Scientific

J. .10, .20, .30, Ethanol Controls - High Purity Chemical, Inc. - Cat. #750-053 through 750-057

K. Mercuric Chloride - Fisher Scientific

L. MEGABORE D-B-WAX 30 meter Column - J & W Scientific Cat. #125-7032

III. Reagent Preparation:

A. Preparation of Internal Standard Solution

1. Prepare 3% V/V acetonitrile stock solution from acetonitrile and deionized water

$$\frac{3\text{ml acetonitrile}}{100\text{ ml soln}} \times 1000\text{ml soln} = 30\text{ml acetonitrile/ liter of soln}$$

Add a pinch of mercuric chloride

2. Prepare 0.012% W/V working internal standard solution

by diluting stock

$$\frac{0.012\text{g acetone} \times 2000\text{ml soln}}{100\text{ ml soln}} \times \frac{100\text{ml stock}}{3\text{ml acetone}} \times \frac{1\text{ml acetone}}{.8\text{g acetone}}$$

= 10 ml stock for 2 liters working internal standard solution. Add a pinch of mercuric chloride

B. Preparation of Mixed Standard

1. Acetaldehyde 0.02% W/V

$$\frac{.02\text{g acetone} \times 1000\text{ml soln}}{100\text{ ml soln}} \times \frac{1\text{ml acetone}}{.8\text{g acetone}} = 0.25\text{ml acetaldehyde}$$

NOTE: Acetaldehyde and pipette must be cold. Boiling point is 20.8 degrees

2. Methanol 0.08% W/V

$$\frac{0.08\text{g MeOH}}{100\text{ml soln}} \times 1000\text{ ml soln} \times \frac{1\text{ml MeOH}}{.8\text{g MeOH}} = 1.00\text{ ml methanol}$$

3. Acetone 0.02% W/V

$$\frac{.02\text{g acetone}}{100\text{ ml soln}} \times 1000\text{ml soln} \times \frac{1\text{ml acetone}}{.8\text{g acetone}} = 0.25\text{ml acetone}$$

4. Isopropanol 0.02% W/V

$$\frac{.02\text{g IPA}}{100\text{ml soln}} \times 1000\text{ml soln} \times \frac{1\text{ml IPA}}{.8\text{g IPA}} = 0.25\text{ml isopropanol}$$

Add deionized water to 1 liter mark
Add a pinch of mercuric chloride

IV. Vial Preparation:

- A. Check internal standard solution to be sure there is enough to complete run
- B. Aspirate sample. Dispense sample into headspace vial. Prepare in duplicate
- C. Tightly crimp cap and septa onto vial
- D. Between each blood sample aspirate water 3 times (3x) and dispense into waste to rinse tubing. It is not necessary to rinse tubing between duplicates.
- E. When finished with pump, rinse tubing.
- F. Specimen vials are loaded in the sample chamber in the following order:

1. Ethanol standards (0.10%, 0.20%, 0.30% w/v)
2. Mixed standard
3. Blank containing deionized water and internal standard solution.
4. Behring blood control in duplicate
5. Cases in duplicate
6. Check standards

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BLOOD ALCOHOL
QUALITY ASSURANCE ADDENDUM

I. Proficiency Testing:

The laboratory voluntarily participates on a continuous basis, in the following blood alcohol proficiency testing programs administered by independent agencies:

- a) U.S. DEPARTMENT OF TRANSPORTATION - NHTSA (National Highway Traffic Safety Administration).

II. Quality Control:

The following rigorous safeguards are employed by each analyst to ensure the validity of their analysis:

- a) Blood alcohol analyses are conducted in DUPLICATE.
- b) Complete calibrations are established at the time of the analysis.
- c) Final reports are reviewed by another Criminalist.
- d) Analytical performance is checked at the time of testing via independently acquired control reference materials.
- e) Specimens, while retained in the laboratory, are refrigerated. A chain of custody is maintained on all items while under the control of the Bureau of Forensic Services.

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