

VERMONT FORENSIC LABORATORY

Alcohol Analysis by Headspace GC-FID

Doc. No.
ALC_P102_v4

Approved by:
Lab Director

Effective Date:
12012013
Status: Archive

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1.0 Purpose and Scope:

- 1.1 This procedure describes the analysis of samples by headspace gas chromatography for ethyl alcohol and related volatile organic compounds.
- 1.2 Samples include calibration standards, whole blood and blood samples which are thought to contain alcohol.
- 1.3 The scope of this SOP includes autosampler, chromatograph and software set-up.

2.0 Responsibility:

- 2.1 All analysts having the responsibility for analysis of blood or other samples for alcohol content are responsible for following this procedure.
- 2.2 This procedure is reviewed periodically by Alcohol Program staff. Necessary revisions are made at that time or when there is an identified need to change this written procedure to be compatible with changing needs in the analytical process.
- 2.3 All analysts performing this procedure for the purpose of reporting analytical results for forensic purposes must be fully trained and demonstrate initial competency in the use of this procedure. All analysts must show ongoing proficiency by successfully analyzing at least one PT sample annually.

3.0 Precautions and Safety Directives:

- 3.1 Prepared samples can be held at room temperature before analysis is begun for a maximum of 48 hours after preparation. Samples are typically analyzed within 24 hours of preparation.

4.0 Procedure

4.1 Principle of Measurement

- 4.1.1 Alcohol and related volatile organic compounds are determined in blood by Headspace Gas Chromatography with Flame Ionization Detection. The sample, a mixture of an internal standard solution, a surrogate and the sample to be analyzed, is heated in a vial sealed with a septum. This is allowed to equilibrate so that proportional amounts of the volatile compound are present in the liquid and headspace. A portion of the vapor above the heated sample is transferred to a sample loop and injected onto the column of the gas chromatograph.
- 4.1.2 In the flame ionization detector (FID), the vaporized alcohol or other volatile compound mixed with hydrogen enters a jet where it is burned in an air atmosphere. The jet itself serves as one electrode and a second electrode is placed above the flame. A potential is applied across these electrodes. When molecules enter the flame, ionization occurs yielding a current flow which after proper amplification, may be displayed on the computer terminal. The FID is a mass-sensitive detector and its response is proportional to the total number of ions

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entering the detector per unit time.

4.2 Equipment & Materials

- 4.2.1 All equipment and materials are located in room 265 unless otherwise stated.
- 4.2.2 Agilent Technologies 6890N Gas Chromatograph with flame ionization detector.
- 4.2.3 Teledyne-Tekmar HT3 Headspace Autosampler.
- 4.2.4 Desktop PC and printer with ChromPerfect Spirit Chromatography and HT3 TekLink Software Packages.
- 4.2.5 Compressed Hydrogen-UHP Grade.
- 4.2.6 Compressed Helium-UHP Grade.
- 4.2.7 Compressed Air.

4.3 Sample and Control Preparation

- 4.3.1 Prepare calibration, control and samples as described in ALC_P101_Sample Preparation for Alcohol Analysis.

4.4 Data System Setup

- 4.4.1 Set up the ChromPerfect Spirit Chromatography data system as described in the Instrument Maintenance Log.
- 4.4.2 Save a copy of the calibration file.

4.5 Chromatograph Setup

- 4.5.1 Assure that the Helium carrier gas is turned on with an appropriate delivery pressure (approximately 45 psi) and that the amount remaining in the supply cylinder is at 500 psi or greater. If not, replace the tank.
- 4.5.2 Assure that the Air tank is turned on with an appropriate delivery pressure (approximately 45 psi) and that the remaining cylinder pressure is at 200 psi or greater. If not, replace the tank.
- 4.5.3 Assure that the Hydrogen fuel tank is turned on with an appropriate delivery pressure (approximately 18 psi) and that the remaining cylinder pressure is at 200 psi or greater. If not, replace the tank.
- 4.5.4 Ensure that the FID flame is lit and the GC status is "Ready for injection".
- 4.5.5 Review all operating parameters and adjust as necessary to assure they agree with the settings outlined in the Instrument Maintenance Log.

4.6 Autosampler Setup

- 4.6.1 Review all operating parameters and adjust as necessary to assure they agree with the settings outlined in the Instrument Maintenance Log.

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5.0 Emergency or High Priority Situations

- 5.1 The Commissioner of Public Safety, Laboratory Director or Alcohol Program Supervisor can designate samples as high priority.
- 5.2 High priority samples are analyzed as soon as possible after successful calibration.
- 5.3 Priority sample results are reviewed and released as soon as they are available, once they pass the quality assurance criteria.

6.0 Quality Control and Corrective Action

- 6.1 All analytical sequences must contain:
 - 6.1.1 An aqueous blank sample.
 - 6.1.2 A Timing Mix sample containing: Acetaldehyde, Methanol, Ethanol, Isopropanol and Acetone.
 - 6.1.3 Five calibration standards having known alcohol concentrations expressed to three decimal places of the following concentrations:
 - 6.1.3.1 STDA ~0.005%
 - 6.1.3.2 STD B ~0.020%
 - 6.1.3.3 STD C ~0.080%
 - 6.1.3.4 STD D ~0.200%
 - 6.1.3.5 STD E ~0.400%
 - 6.1.4 Duplicate samples of a level one (1) whole blood ethanol control obtained from Cliniqua or equivalent NIST traceable control. The mean of the two replicates must be within 10% of the accepted value.
 - 6.1.5 A calibration check sample using STD C, which is analyzed in duplicate after every 10th sample and at the end of every run. The mean of the two replicates must be within 10% of the known value. If not, then all analyses from the last acceptable CCS must be repeated. If one replicate fails due to a preparation error, it may be dropped and the remaining replicate may be used as the check sample.
 - 6.1.6 All samples will be run in duplicate. The two sample analyses must be within 5% of the mean result for that sample. If not, then the analysis must be repeated (two new preparations from the sample) and the two new replicates must be within 5% of the mean result for that sample. The number of samples used to report the result will be determined on a case by case basis.
 - 6.1.7 Calibration standards must be within 10% of their known values except STD A which must be within 20% of its known value.
 - 6.1.8 The correlation coefficient of the calibration line must be 0.99 or greater and the

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average error must not be greater than 10%. If not, the calibration must be repeated.

6.1.9 A calibration curve will be run each day samples are analyzed. Additional samples may be analyzed on a calibration provided that a set of calibration check samples are analyzed before and after samples.

6.1.10 Surrogate compound concentrations must be between 0.900 and 1.100, inclusive, in each sample.

7.0 Preventive Maintenance and Backup

7.1 Agilent 6890N Gas Chromatograph:

7.1.1 Refer to the Agilent 6890N Gas Chromatograph User's Guide located in room 265 for repair and maintenance information.

7.1.2 Gas tanks supplying the gas chromatograph and autosampler will be replaced when the pressures of the tanks reach the low levels defined in Section 4.5.

7.1.3 Monthly, archive the data, alarm, and error log files for the 6890N gas chromatograph.

7.1.4 General maintenance and troubleshooting activities will be documented in the Instrument Maintenance Log.

7.2 Teledyne-Tekmar HT3 Headspace Autosampler

7.2.1 Refer to the Teledyne-Tekmar HT3 Headspace Autosampler User's Guide located in room 265 or the electronic User's Guide found in the TekLink software for repair and maintenance information.

7.2.2 Gas tanks supplying the gas chromatograph and autosampler will be replaced when the pressures of the tanks reach the low levels defined in Section 4.5.

7.2.3 Perform a leak check at least once weekly and after replacing the helium tank.

7.2.4 Monthly, archive the system history by selecting "Trim System History".

7.2.5 General maintenance and troubleshooting activities will be documented in the Instrument Maintenance Log.

7.3 Backup

7.3.1 If the laboratory lacks analytical ability for greater than 10 business days, samples will be sent to a qualified reference lab for analysis.

8.0 References

8.1 ALC_P101_Sample Preparation for Alcohol Analysis.

8.2 Agilent 6890N Gas Chromatograph User's Guide.

