EVALUATION OF PRESERVED BREATH ALCOHOL FOLLOWING ANALYSIS BY INTOXIMETER 3000 AND COLLECTION ON MAGNESIUM PERCHLORATE

E. J. Clary, B.S.*; and C. W. Beheim, B.S.

SYNOPSIS

Court decisions in the State of Alaska have mandated that a portion of the alcohol contained in a breath sample be made available for reanalysis at a later date if requested by the defendant. The nondestructive infrared technique for breath alcohol determination utilized by the Intoximeter 3000 allows for the subsequent trapping by anhydrous magnesium perchlorate of the alcohol contained in the analyzed portion of the breath. Analysis of the preserved breath alcohol specimen is accomplished by headspace gas chromatography. Laboratory results indicate that this system meets the requirements for breath alcohol testing as set forth by the State of Alaska.

INTRODUCTION

In the past 12 years there have been many challenges in the courts across the United States involving the need for some type of reanalysis of breath alcohol findings. These challenges have ranged from attempts to save the Breathalyzer ampoules to requiring that either the breath or its equivalent be preserved for reanalysis at a later time.

In 1982 in the State of Alaska, there occurred a series of court challenges and decisions (Municipality of Anchorage vs Serrano et al. and Cisneros et al., and State vs Brundage et. al.) involving breath preservation. During these hearings various experts in the field of breath testing were called into the state to give their opinions on the feasibility of preserving the breath sample or its equivalent. Based upon the expert testimony that it was technically feasible, the courts directed the State Department of Health and Social Services (the certifying agency for breath alcohol testing) to find such a breath testing system.

In the latter part of 1982, Dr. Harry Colvin of the Alaska Department of Health and Social Services, evaluated four different breath testing instruments that utilized a

* Alaska State Troopers Crime Laboratory, Department of Public Safety, 5700 E. Tudor Road, Anchorage, Alaska 99507, USA.
nondestructive method of analysis. Three manufacturers stated that their instruments had retained-sample capabilities. The instruments were evaluated as to their accuracy, ease of use, absence of radio frequency interference, flexibility, and compliance with existing state laws. Utilizing these criteria the Department chose the Intoximeter 3000 as the instrument most likely to comply with the court's directive. Evaluation of the retained sample itself was begun in January of 1983.

The Intoximeter 3000 employs a nondestructive infrared technique utilizing a dual beam optical system with one source and one detector. The instrument employs a Tagucci semiconductor for detection and quantitation of acetone. All the functions of the instrument are controlled by a Rockwell AIM 65 (Advanced Interactive Microcomputer). The computer provides data storage, monitors continuously instrument parameters, offers step by step prompts to the instrument operator, and controls the many mechanical functions of the analytical portion of the instrument.

Retention of the alcohol contained in the analyzed portion of breath is accomplished by purging room air, supplied by a built-in air pump, through the sample chamber and out through the glass collection tube attached to the retained sample port. The glass collection tube is approximately 16 cm long with a narrow center portion and is threaded on both ends. The ends are closed by means of a threaded cap with a small conical plastic seal. The narrow center portion of the tube contains approximately 1.6g of anhydrous magnesium perchlorate held in place on both ends by a small wad of rayon cotton and a plastic loop made from Tygon tubing.

Magnesium perchlorate has a history of use in breath alcohol determinations that dates back to a paper by Jetter, Moore, and Forrester published in March of 1941. The authors found that anhydrous magnesium perchlorate rapidly and quantitatively absorbed many organic vapors, including ethanol. They also found that the vapors did not go through a chemical change in being absorbed and that they could be quantitatively recovered.

In 1969 a paper by Biasotti and Bradford demonstrated the advantages of ethanol quantitation by headspace vapor analysis on a gas liquid chromatograph after collection on magnesium perchlorate. They showed that with the addition of an internal standard, precision, accuracy, and specificity were acceptable for forensic applications.

Most recently (1981) an unpublished paper by Lucien Haag described the results obtained with the magnesium
perchlorate collection tube used in conjunction with an Intoximeter 3000 and analyzed on an Intoximeter Mark IV gas chromatograph.

METHOD

Investigation of the retained sample system involved several steps, the first of which was to devise a method of analysis suitable to the equipment available. The method used by the State of Alaska for the analysis of the retained breath sample is as follows:

1. The first step in analyzing the preserved breath alcohol specimen involves removing the plastic loop and rayon plugs from both ends of the glass tube. Care should be used when doing this so that no magnesium perchlorate is lost from the tube.

2. After tightly capping one end of the tube, 3.0 ml of an Internal Standard Solution consisting of n-propanol and water are quantitatively added to the tube which should then be quickly and tightly capped.

3. The tube is agitated to ensure that all the magnesium perchlorate goes into solution.

4. The contents of the tube are then transferred to a 30-ml headspace vial containing 1-2 g of granular sodium chloride.

5. The vial is then stoppered, sealed, and placed in a 60° water bath.

6. After the tubes have equilibrated, they are vented and allowed to re-equilibrate; 1.5 ml of headspace vapor are then injected into a gas chromatograph using a pre-heated glass syringe.

The instrument used was a Hewlett-Packard 5711-A Gas Chromatograph equipped with a flame ionization detector and a 6 ft x 1/4 in (18.5 x .64 cm) glass column packed with 5% Carbowax 20M on 60/80 Carbopack B. A Hewlett-Packard 3390-A Integrator was used for quantitation.

The results found in Table 1 were obtained using 3 Smith & Wesson simulators in series connected to a compressed air bottle. The air, at constant pressure and flow, passed into the first simulator containing deionized water for the purpose of raising its temperature and humidity to that more closely approximating human breath.
This humidified warm air then passed into the next simulator containing a water-alcohol solution of the desired concentration. This effluent, in turn, passed into the final simulator of the same alcohol concentration and on into the Intoximeter 3000. The simulator solutions were prepared using an alcohol concentration of .124 g/dl to produce a breath alcohol equivalence of .100 g/210 l. Both the gas-powered tandem simulators and simulator solution concentrations were suggested in a paper by Dubowski (1980).

Table 1

Results Magnesium Perchlorate Preserved Breath Alcohol Simulator Effluents (g/210 l)

<table>
<thead>
<tr>
<th>Simulator Effluent Target</th>
<th>Magnesium Perchlorate Mean</th>
<th>Average Standard Deviation</th>
<th>% Systematic Error</th>
<th>Coefficient of Variation</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>.000</td>
<td>.000</td>
<td>.000</td>
<td>0</td>
<td>0%</td>
<td>0</td>
</tr>
<tr>
<td>.050</td>
<td>.052</td>
<td>+.001</td>
<td>+4.0%</td>
<td>1.9</td>
<td>.051 - .054</td>
</tr>
<tr>
<td>.100</td>
<td>.100</td>
<td>+.001</td>
<td>0</td>
<td>1.0</td>
<td>.098 - .102</td>
</tr>
<tr>
<td>.150</td>
<td>.149</td>
<td>+.004</td>
<td>-0.7%</td>
<td>2.7</td>
<td>.144 - .155</td>
</tr>
<tr>
<td>.200</td>
<td>.195</td>
<td>+.004</td>
<td>-2.5%</td>
<td>2.1</td>
<td>.190 - .202</td>
</tr>
<tr>
<td>.300</td>
<td>.294</td>
<td>+.006</td>
<td>-2.0%</td>
<td>2.0</td>
<td>.286 - .305</td>
</tr>
</tbody>
</table>

The Intoximeter 3000 was used essentially as a collection device for the magnesium perchlorate retained sample tube. Twenty-five tubes were collected at each breath alcohol concentration of .000, .050, .100, .150, .200, and .300 g/210 l.

DISCUSSION

In the course of developing this method several problems with the collection tubes arose. The caps on the tubes supplied with the Intoximeters frequently did not seal the tube due to irregular threaded-end diameters. Also, proper mixing of the perchlorate and internal standard

276
required that the plastic loop and rayon plug be easily removed. Again, the original tubes were unworkable. However, upon making the problems solutions abundantly clear to the original supplier, workable tubes were obtained.

CONCLUSIONS

1. Under laboratory conditions, the correlation between the simulator values and the retained samples is excellent.

2. Perchlorate purity, tube seals, and instrument/tube adapter seals are critical.

ACKNOWLEDGEMENTS

We would like to thank Dr. Harry Colvin and the Alaska Department of Health and Social Services for their support in this work.

REFERENCES


