## Letters to the Editor—Continued

## **Quality Control**

Sir

The addendum to our paper, "The Trapping, Storing, and Subsequent Analysis of Ethanol in In-Vitro Samples Previously Analyzed by a Nondestructive Technique," which appears on pages 671 through 677 of this issue, makes reference to a quality control problem with the silica gel used in trapping alcohol. We have studied this problem and have located the difficulties.

The original columns contained approximately 250 mg of 20-40 mesh silica gel. After two years of successfully trapping and analyzing alcohol in breath samples, the system seemed to develop difficulties. We found that the silica gel had been changed to a 12-28 mesh size. In an attempt to solve this problem we placed an alcohol solution into a simulator, pumped the vapor into a 4011 AS Intoxilyzer, and produced a 0.47% alcohol reading. A silica gel column was attached to the outlet tube and a second Intoxilyzer was attached in series to this tube. This allowed us to use the second Intoxilyzer to monitor any alcohol breaking through the tube. The 12-28 mesh silica gel did *not* trap the alcohol in the 715-mL vapor sample. Using this procedure and 30-60 mesh silica gel we determined that the minimum amount of silica gel needed to trap all of the alcohol in a 4011 AS cell when using a >0.40% alcohol solution in a simulator was between 150 and 175 mg (Table 1).

Columns were then packed with 172 mg and 200 mg of 35-60 mesh silica gel. The columns were used to trap alcohol samples and then analyzed by gas chromatography (Table 2).

TABLE 1—Determining minimum amount of silica gel required.

Weight of 30-60 Mesh Silica Gel in Column, mg	Concentration of Alcohol Solution Used, %	Average % of Alcohol Breaking Through the Trap
50	0.47	32.1
100	0.47	12.5
150	0.47	3.7
172	0.42	0.0
200	0.47	0.0

TABLE 2—Results of analysis by gas chromatography.

Silica Gel Used in 35-60 Mesh, mg	Intoxilyzer Reading, %	Collected Sample Analyzed by Gas Chromatography, %
172	0.06	0.06
172	0.11	0.11
172	0.16	0.16
172	0.21	0.21
200	0.06	0.06
200	0.27	0.26

The major problem with the silica gel was the particle size coupled with the relatively high flow rate of the Intoxilyzer. Because of the high flow rate of the instrument and its fairly small cell volume, it is important to use a large mesh size and a minimum amount of silica gel. This results in a 100% trapping efficiency and a need for a small volume of liquid to remove the alcohol from the gel. Based on the preceding data our recommendations are as follows:

- 1. The silica gel particle size is an important factor. The 35-60 mesh silica gel produces acceptable results.
- 2. It is recommended that the collection tubes be packed with 175 mg of silica gel. This amount is capable of holding the trapped vapor containing the equivalent of 0.47% alcohol solution. The smaller amount of silica gel facilitates analysis by gas chromatography.
- 3. Each lot of silica gel must be tested to ensure its effectiveness. A sound quality control system must be incorporated in the manufacture of the traps.
- 4. When transferred to a water/n-propanol solution the sample must sit at least 1 h and be carefully mixed before injection.

When using gas chromatography, liquid injection, and flame ionization detection we recommend these procedures:

- 1. Carefully transfer the contents of the tube into 0.6 mL of a 0.2% *n*-propanol solution in a flat-bottom container (which spreads the gel). Gently swirl the container to mix the silica gel and *n*-propanol solution. Do not shake! Allow it to sit for at least 1 h. Swirl again and inject. Use the *n*-propanol as an internal standard.
  - 2. Dilute standards 50/50 v/v with a 0.4% n-propanol solution and inject.
  - 3. Make sure all samples and standards are 0.2% in *n*-propanol.

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