A Simple Device for Minimizing Contamination During Automated Ammonia Analysis

W. C. Nickels and G. N. Gaston
Chemical Laboratory, Environmental Measurement Branch, California Department of Water Resources, P.O. Box 388, Sacramento, CA 95802

Many water quality laboratories are engaged in analyzing low level, ambient concentrations of ammonia in water. For this type of analysis, automated procedures are widely used. One problem arises, however, in that samples in tubes or cups are exposed before analysis to the atmosphere which is frequently contaminated with ammonia. This leads to the contamination of samples, blanks and standards. To alleviate this problem, we have developed a sample tube septum cup which is easily constructed, simple to use and inexpensive.

MATERIALS AND METHODS

Analytical Methods. The automatic analyzer and methodology used were essentially the same as the two channel system for total Kjeldahl nitrogen and total phosphorus developed by the EPA (JIRKA 1976), and modified by our laboratory to analyze ammonia and orthophosphate. The ammonia modification utilizes a sampler wash solution of distilled deionized water, a dilution solution of 13 mL conc. H_2SO_4/L, alkaline phenol solution of 50g NaOH/L and 83g phenol/L, 1.40 mL/min flow cell pump tube, wash tube of distilled deionized water between each sample tube, and a 10X range expander.

Routine ammonia procedure consists of analyzing four standards (0.50, 0.37, 0.25, 0.12 mg/L NH_3-N) initially with a 0.25 standard every ten samples. All tubes including the wash tubes are capped immediately after preparation with the septum caps.

Sample Tube Septum Cap. Each cap assembly was constructed from two different size hollow polyethylene stoppers. Nalgene 6190-0010 and 6190-0020. A 2 mm cross section was removed from the bottom of the 0010 size and 9 mm from the 0020 size. A piece of vinylidene polymer plastic, such as Saran, is placed between the two caps. See Figure 1. The sampler probe tip was filed to an acute angle so that it would puncture the plastic septum. Figure 2 illustrates the sampler with the caps in position.
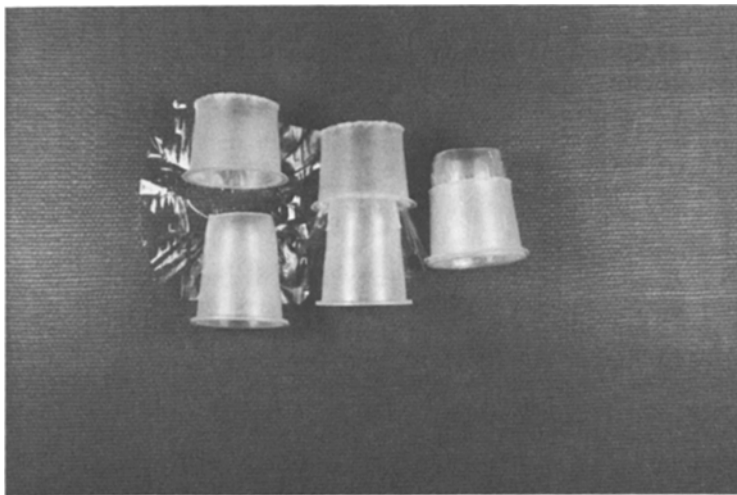
FIGURE 1. DISASSEMBLED, PARTIALLY ASSEMBLED, AND
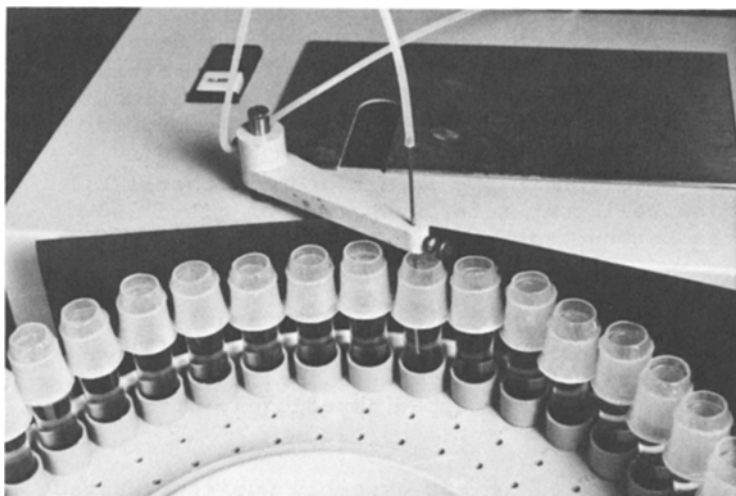   COMPLETED SAMPLE TUBE SEPTUM CAPS.

FIGURE 2. SAMPLER TRAY WITH SAMPLE TUBE SEPTUM
          CAPS IN PLACE.

RESULTS AND DISCUSSION

The sampler tray was loaded at every position with
15 x 85 mm disposable culture tubes containing distilled
deionized water. Sample tube septum caps were placed on
alternating tubes for a total of 10 uncapped tubes and
10 capped tubes. The sampler tray with the 20 tubes was
allowed to stand in the laboratory environment overnight.
This was necessary so that an ammonia contamination
concentration high enough for analysis was obtained. The
laboratory atmosphere prior to this experiment was cleaned
up somewhat by no smoking restrictions, use of non-ammonia
containing floor wax strippers, use of non-ammonia window
cleaners and minimum use of ammonia reagents; also of
benefit, which is inherit in the method, is the small
surface area to sample volume ratio. After the overnight
waiting period, the 20 tubes were analyzed by our routine
ammonia procedure. The results tabulated in Table 1
indicate the effectiveness of the caps.

TABLE 1

Ammonia Analysis of Distilled Deionized Water Samples

Uncapped		Capped	
Sample No.	NH_3-N mg/L	Sample No.	NH_3-N mg/L
1	0.02	2	0.00
3	0.02	4	0.00
5	0.02	6	0.00
7	0.01	8	0.00
9	0.02	10	0.00
11	0.02	12	0.00
13	0.02	14	0.00
15	0.02	16	0.00
17	0.02	18	0.00
19	0.03	20	0.00

As part of the laboratory's quality assurance program, the
routine ammonia procedure was checked for precision and
accuracy utilizing the sample tube septum caps. These data
are presented in Table 2.

TABLE 2

Precision and Accuracy of Automated Ammonia Procedure
for 0.45 u Membrane Water Samples - mg/L NH_3-N

	San Joaquin River	Clear Lake	Clear Lake Bottom	San Luis Obispo Creek	Clear Lake + Spike*	Clear Lake Bottom + Spike*
	0.05	0.12	0.25	0.39	0.23	0.35
	0.05	0.13	0.25	0.38	0.23	0.35
	0.05	0.13	0.26	0.38	0.23	0.35
	0.05	0.14	0.26	0.38	0.23	0.35
	0.05	0.13	0.25	0.38	0.23	0.35
	0.05	0.13	0.26	0.38	0.23	0.34
	0.05	0.14	0.25	0.38	0.23	0.35
S.D.	0.000	0.0051	0.0051	0.0035	0.0000	0.0035
% RSD	0.0	3.8	3.8	0.9	0.0	1.0
Ave. % Recovery					98	99

*Ammonium chloride, 0.10 mg/L NH_3-N

The caps were developed for the 15 x 85 mm sample tubes,
however, the same principle could be used in a scaled down
version to accommodate small sample cups, beakers, etc.

REFERENCE

JIRKA, A.M., CARTER, J.M., MAY, D., and FULLER, F.D.:
Environ. Sci. Technol. 10, 1038 (1976)