Trace Element Contamination
II. Certain pH Electrodes[1]

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INTRODUCTION

A major problem in background-level trace element studies is eliminating or minimizing contamination of the samples. Practices which we employ to minimize sample contamination include water sample collection and storage using materials of colorless plastic construction; pre-analysis preparation under a laminar-flow air filter; and redistillation and solvent extraction to purify chemicals used as preservatives and extractants. Additional contamination sources, such as microlitre pipet tips (BENJAMIN and JENNE 1975), continue to be discovered.

We solvent extract 175- or 1,750-ml water samples prior to atomic absorption analysis in order to eliminate matrix errors and to concentrate the trace elements. The pH is monitored during addition of acid or base by immersing the lower half of a pH electrode in the sample while swirling. For a period of time we experienced contamination of water samples with Pb and Cd. We suspected that a small quantity of paint might occasionally abrade off the uppermost part of the electrode's glass sleeve and fall into the solution, if the painted portion came into contact with the ground glass at the top of the separatory funnel.

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EXPERIMENTAL

An experiment was conducted to establish that the yellow paint was the source of the occasional contamination observed in two sets of analyses. A quantity of paint was scraped off the Sargent-Welch[3/] combination pH electrode, weighed and placed in a 250-ml separatory funnel containing 175 ml deionized water plus 10 ml conc. HNO_3. This was done to estimate the maximum amount of leachable metals in the paint; such low pH values are not normally utilized in the preservation of water samples. The pH was raised to pH 3.0 after a 2-hour reaction time, complexing agents were added, and the trace metal-organic complexes were extracted into ethyl propionate. The pH of the aqueous phase was then adjusted to 4.0, a second extraction was carried out, and the organic extractants were combined for analysis by atomic absorption spectrometry.

RESULTS AND DISCUSSION

To illustrate the magnitude of the contamination's effect on apparent trace-element levels in natural-water samples, a representative comparison of contaminated and uncontaminated samples of Sacramento River water is given in table 1. Concentrations of Cd and Pb are seen to err on the high side by nearly two orders of magnitude, while Fe, Ni, and Zn values are high by a factor of 2 to 4.

The results of the experiments with the electrode paint are shown in table 2. It is obvious from the first set of values that it is possible in a case of severe paint contamination for Ag, Cd, Fe, Cu, Ni, Co, and Pb concentrations all to be in error. In a similar experiment a Cd value of 3.9 μg/ℓ was obtained. The second set of values in table 2 is an estimate of the concentration of trace metals present in the paint in pH 0.1 acid-extractable form, if 2-hour contact is allowed.

Any paint on electrodes used to adjust the pH of trace element solutions should be considered a potential source of contamination. We have solved the contamination problem for our Sargent-Welch electrodes by wrapping the painted portion with Teflon tape which is secured by plastic ties.

[3/] The use of brand names in this report is for identification purposes only and does not imply endorsement by the U.S. Geological Survey.

TABLE 1

Comparison of apparent trace element levels in contaminated and uncontaminated Sacramento River water samples.

Sample	Trace element								
	Ag	Cd	Fe	Cu	Ni	Co	Pb	Mo	Zn
	------------µg/litre of water------------								
Contaminated	0.3	6.2	9.1	3.5	1.9	.05	5.7	.6	7.6
Uncontaminated	0.2	.1	3.5	3.3	.8	<.03	<.4	.7	2.0

TABLE 2

Potential contamination introduced by paint from pH electrode.

Sample	Trace element								
	Ag	Cd	Fe	Cu	Ni	Co	Pb	Mo	Zn
	------------µg/litre of water------------								
Electrode paint	1.7	0.8	48.1	0.5	3.3	3.8	171.6	2.9	18.5
	------------µg/gram of paint------------								
Electrode paint	233	111	6480	70	450	510	23,100	390	2490

REFERENCES

BENJAMIN, M.L. and E.A. JENNE, Interface 4, 25 (1975).