



Identifying Contamination in Trace Metal Laboratories

Ralph H. Obenauf and Nimi Kocherlakota, SPEX CertiPrep, Inc.

Analytical laboratories are routinely asked to report metals at ppb and ppt levels. However analysis at these levels is not possible unless trace metal contamination is minimized, identified, and if possible reduced to low ppt levels. Contamination can originate in sampling, reagents, reference materials, sample handling and introduction, or in the laboratory environment.

The first step is to identify the level of contamination, usually by running blanks or QC samples that have been prepared and processed the same as the samples to be analyzed. This is not always as easy as one would think since there are so many ways for contamination to gain entry to your results. We present here just two examples.

We all try to keep our labs free from contamination. But unless you work in a “clean lab,” which has little or no exposed metal and is fed with HEPA filtered air, you will have contamination; but at what levels? There is an easy way to determine how clean, or dirty, your lab is with a common laboratory task. Using a hot plate on your lab bench, concentrate a volume of high-

purity nitric acid, in an open beaker. Compare the trace metals in the sample before and after the concentration step as we have done in Table I, below. As seen the “regular” lab in this case introduced an average of more than ten times the contamination as compared with that from the clean lab environment during the ten-fold concentration step. Of course this does not tell you how much contamination was introduced in the “clean lab” sample. Running the pre-concentrate will give you an idea, but just handling the sample has already introduced contamination.

This brings us to another way to determine how one might be contaminating samples by a common laboratory procedure, washing glassware. If you perform volumetric dilutions you probably use Class A pipettes and volumetric flasks and wash them after each use. In Table II we have shown a comparison of the trace metals introduced into a sample simply by passing it through a pipette that had been washed and rinsed in a conventional siphon-type pipette washer versus a high efficiency (HE) washer designed to minimize trace metal contamination. The HE washer first

Table I: Redistilled nitric acid concentrated X10 in a regular laboratory environment versus the same procedure performed in a clean room

Element ppb	Regular Lab	Clean Room	Element ppb	Regular Lab	Clean Room
Ag	0.006	0.01	Mn	1.1	0.1
Al	60	15	Mo	0.8	0.03
As	0.17	0.02	Na	High	25
Ba	1.95	0.25	Nd	0.14	0.025
Ca	>150	100	Pb	0.5	0.4
Cd	0.3	0.003	Rb	0.03	<0.001
Ce	1.5	0.06	Sb	0.04	0.013
Co	0.6	0.008	Sm	0.015	0.003
Cr	2.5	0.4	Sr	0.6	0.3
Cu	1.7	0.23	Th	0.017	0.001
Fe	50	9	Ti	1	0.77
Ge	0.02	<0.01	V	0.35	<0.3
Mg	10	4	Zn	5.5	0.7

Table II: Redistilled nitric acid concentrated X10 in a regular laboratory environment versus the same procedure performed in a clean room

Element ppb	Manual Cleaning	Pipette Washer	Detection Limit
Ag	2.33	<0.01	0.0088
Al	6.42	<0.01	0.13
Be	2.62	<0.01	0.007
Bi	1.07	<0.01	0.0006
Ca	18.8	<0.02	2.9
Co	2.02	<0.01	0.004
Cr	0.91	<0.04	0.28
Fe	1.62	<0.02	0.75
Mg	2.56	<0.01	0.016
Mn	1.72	<0.01	0.012
Na	19.1	<0.01	0.6
Ni	0.96	<0.01	0.18
Pb	5.4	<0.01	0.13
Sn	0.55	<0.01	0.0033
Th	0.24	<0.01	0.0003
Ti	0.56	<0.02	0.003
Tl	1.53	<0.02	0.0075
Zn	9	<0.01	0.4

flushes the pipette with high purity nitric acid, followed by rinsing with ASTM Type I water, and finally drying by pulling air through the pipette with a vacuum. As shown the siphon-type washer introduced more than a hundred fold higher levels of contamination as compared with the HE washer.

There is no way to eliminate trace metal contamination. All one can do is identify the sources, monitor the levels and constantly make improvements in techniques to reduce those levels. We have shown here just two of many sources of contamination that we have encountered and examples of how one can reduce the degree of contamination.

References

1. "Guidance in establishing Trace Metal Clean Rooms in Existing Facilities" :USEPA 821-B-95-001
2. Accuracy in Trace Analysis : NBS Special Edition 422
3. Guide to Environment Analytical Methods :Roy-Keith Smith
4. Clean Manufacturing :A2C2; April 2003
5. Water Environment Laboratory Solutions :April/May 2003

SPEX CertiPrep, Inc.

203 Norcross Avenue
Metuchen, NJ 08840
Tel. (732) 549-7144, Fax. (732) 603-9647
crmsales@spexcsp.com, www.spexcsp.com