A Systematic Evaluation of Dissolved Metals Loss during Water Sample Filtration

Regional Applied Research Effort - Addressing Challenges through Science and Innovation

Project Purpose

This research study examined how water quality collection and filtration approaches, including commonly used capsule and disc syringe filters, may cause losses in the amounts of soluble lead and copper found in a sample. A variety of commercially available filter materials with a pore size of 0.45 micrometers (µm) were tested, including polyvinylidene fluoride, polytetrafluoroethylene, nylon, polypropylene, and mixed cellulose acetate. The effects of important water quality parameters including pH, alkalinity and phosphate on sorption losses were examined. The impact of filtration approach, including flow rate and sequential sampling, was also explored.

Background

EPA method 1669[1] provides specifications for the collection and filtration of water samples prior to the analysis of dissolved and particulate trace elements and metals in surface waters. Filtration apparatus specifications include the use of disposable, tortuous path, capsule, and disc filters with an effective pore size of 0.45 µm and a diameter of 15 millimeter (mm) or larger.

Similar filtration approaches are commonly used to separate soluble and particulate constituents in drinking water samples, bench and pilot-scale water treatment studies, and fundamental drinking water research studies. Syringe filters are typically used in such cases given the ease of utilization. The Texas Commission on Environmental Quality (TCEQ) noted differences in dissolved metal levels in water samples when different filter types were employed for particulate filtration in the field. Specifically, discrepancies were found in dissolved metal fractions between those samples filtered using a low-volume syringe filter and those filtered through an in-line capsulated cartridge filter. Similar inconsistencies have been noted by EPA researchers performing filtration separations during fundamental drinking water research investigations. In the case of TCEQ, many reported considerably higher lead and aluminum values for water samples filtered using syringe filters than those reported when utilizing in-line capsule filters, often within similar water types found in similar geographic areas. This action can confound a state's ability to confirm the relative quality of its data and the subsequent impairment status of its waters. The findings have clear implications on the validity of dissolved metals measurements reported by a state's 303(d)[2] lists, and the reliability of research data reported to have used similar filtration procedures.

Research Approach

Twelve different 25 mm diameter, 0.45 µm filter types were tested (Table 1) and divided into two categories: those with no prefilter (8) and those with a prefilter (4). The prefilters were either a glass microfiber prefilter (PFG) or a polypropylene prefilter (PFP). According to the manufacturers, the prefilters remove larger particles and are designed for high particle loaded samples. Lead sorption filter tests were initially performed using water containing 50 mg C/L dissolved inorganic carbon and 40 µg/L lead at pH 7. The lead concentration is representative of water that exceeds the drinking water regulatory lead action level of 15 µg/L. Average lead concentrations of 15 mL sequential filtered water samples up to a total volume of 90 mL were calculated from two or three separate tests and are shown in Table 2.

Research Goals

1. **Based on EPA method 1669[1], determine if the type of filter material used to separate soluble from particulate fractions creates significant losses of dissolved metals (lead and copper) in water during filtration due to adsorption losses.**
2. **Evaluate the impact of water chemistry and other factors on sorption losses during water filtration.**
3. **Identify the best filtration material and protocol for separating soluble and particulate metals in water samples.**

Research Results

The tendency for filters to adsorb dissolved lead were categorized as (1) *good* (little to no adsorption or total lead lost), (2) *moderate* (some adsorption and/or total lead lost), or (3) *poor* (large amounts of adsorption and/or total lead lost). Lead sorption losses were also presented as the 'total' lead (%Pb mass) loss from the water to the filter over the entire 90 mL filtered volume. The results of this study clearly show that soluble lead sorption losses to filters can be significant and sometimes extreme. Based on this work, any syringe filter with a prefilter should be avoided, especially those with a PFG.

(1) Of the filters evaluated, only the PP and MCE filters were categorized as *good* performers and should be initially considered for use. Under the conditions of this test, the results from using these two filters suggests that there is no need to waste the initial volume filtered. In practice, however, users should consider performing an evaluation to define protocol that is most acceptable for their case.

Table 1. Manufacturer and types of 0.45 µm filters tested.

Manufacturer	Filter Membrane Type
A	Liquid Chromatography (LCR) Hydrophilic Polytetrafluoroethylene (PTFE)
A	Ion Chromatography (IC) Hydrophilic PTFE
A	Nylon
A	Mixed Cellulose Esters (MCE)
B	Nylon
B	Polyvinylidene Fluoride (PVDF)
B	Polypropylene (PP)
B	Polyethersulfone (PES)
B	PFG Nylon
B	PFG Charged Nylon
B	PFG Glass Microfiber (GMF)
B	PFP Nylon
B	PFP PES

(2) The filter materials categorized as *moderate* performers were PES, LCR PTFE, IC PTFE, PVDF, Nylon A, Nylon B, and PFP PES. However, lead losses associated with these filters were generally within the first 30 to 45 mL of water filtered and could possibly be minimized in some cases if an initial volume is wasted in practice.

(3) The filter materials categorized as *poor* performers were the PFG Nylon, PFG Charged Nylon, PFP Nylon, and PFG GMF.

Table 2. Summary of All Testing Results. Values are total percentage of lead lost to filter surface over 90 mL of filtered water.

	Percent Lead Sorbed														
	Lead (24 ug/L) only					Lead (40 ug/L) only					Lead (40 ug/L) and Calcium (100 mg/L)				
	Replicate 1	Replicate 2	Replicate 3	Average	95% C.I.	Replicate 1	Replicate 2	Replicate 3	Average	95% C.I.	Replicate 1	Replicate 2	Replicate 3	Average	95% C.I.
LCR PTFE	-	-	-	-	-	10.79	12.48	9.84	11.04	1.51	-	-	-	-	-
IC PTFE	-	-	-	-	-	16.76	18.10	10.76	15.20	4.42	1.82	5.03	5.29	4.05	2.19
MCE	2.68	3.08	0.50	2.09	1.57	5.80	7.58	1.44	4.94	3.58	0.33	2.80	0.89	1.34	1.46
B Nylon	19.95	9.94	14.45	14.78	5.67	15.27	21.24	13.40	16.64	4.63	5.60	5.52	4.60	5.24	0.63
PVDF	-	-	-	-	-	21.84	17.44	-	19.64	4.31	-	-	-	-	-
PP	1.26	3.94	3.02	2.74	1.54	5.45	8.38	-	6.91	2.87	0.46	1.16	-0.43	0.40	0.90
PES	5.77	11.81	10.57	9.38	3.61	6.20	7.49	5.58	6.42	1.10	4.65	3.70	3.36	3.91	0.76
PFG Nylon	-	-	-	-	-	99.46	99.79	99.52	99.59	0.20	48.06	80.85	86.64	71.85	23.54
A Nylon	-	-	-	-	-	21.44	23.26	30.09	24.93	5.16	-	-	-	-	-
PFG Charged Nylon	-	-	-	-	-	99.67	99.86	99.60	99.71	0.15	-	-	-	-	-
PFP Nylon	-	-	-	-	-	49.00	52.83	43.89	48.57	5.08	-	-	-	-	-
PFP PES	-	-	-	-	-	15.69	15.55	4.22	11.82	7.45	-	-	-	-	-
PFG GMF	-	-	-	-	-	99.40	99.36	99.21	99.32	0.11	-	-	-	-	-

Contacts

Principal Investigators: Darren Lytle, (513) 569-7432, lytle.darren@epa.gov | Keith Kelty, (513) 569-7414, kelty.keith@epa.gov| Maily Pham, (513) 569-7212, pham.maily@epa.gov | Michael Schaub (214) 665-7314, schaub.mike@epa.gov

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[1]*EPA Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels*; U.S. Environmental Protection Agency, 1996.
[2]*Clean Water Act Section 303(d)*; 33 U.S.C. §1251 et seq.; U.S. Environmental Protection Agency, 1972.