NSF/ANSI 61 Evaluation of Lead Pipe

Research Methodology and Summary of Findings

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Introduction:

NSF/ANSI 61 was developed to establish minimum requirements for the control of potential adverse human health effects from products that contact drinking water. It does not attempt to include product performance requirements that are currently addressed in the USEPA's ETV program, other NSF standards, or other voluntary consensus standards established by such organizations as the American Water Works Association or the American Society for Testing and Materials.

NSF/ANSI 61, and subsequent product certification against it, has replaced the USEPA Additives Advisory Program for drinking water system components. USEPA terminated its advisory role in April 1990. For more information with regard to USEPA's actions, refer to the July 7, 1988 *Federal Register* (53FR25586).

The standard is intended for use by certifying organizations, utilities, regulatory agencies, and/or manufacturers as a basis of providing assurances that adequate health protection exists for covered products. Product certification issues, including frequency of testing and requirements for follow-up testing, evaluation, enforcement, and other policy issues, are not currently addressed by the Standard.

Assertions though have been made that NSF/ANSI Standard 61 is not sufficiently protective of public health due in part to its purported inability to find an "inline device" holding 6.3 mL and made of pure lead, unacceptable for use in drinking water applications. The assertion was published in the AWWA Journal article "*Lead leaching from inline brass devices: A critical evaluation of the existing standard*" (Dudi et al, 2005) and has been cited in subsequent articles (Triantafyllidou, S. & Edwards, M. 2007, Elfland et al, 2010).

However a complete evaluation of the "inline device" (believed to be a short section of lead pipe) was either not performed, or if performed, not reported. The article claimed that it passed the requirements of Standard 61 based solely on the results of pH 5 testing. Standard 61 requires testing under both pH 5 and pH 10 exposure conditions. The pH 10 water is routinely the exposure condition found to be most aggressive for lead extraction. The purpose of this study was to generate data that shows how lead pipe evaluates under both the required exposure waters: pH 5 and pH 10. Additional work was performed to determine how the pipe would fare using the pH 8 water used when evaluating products to Section 9 of the standard.

The article also asserted that their results demonstrated that the in-line device made of pure lead and evaluated after as few as 2 days of exposure would also pass the standard. The assertion was again made solely on the evaluation using the pH 5 extraction waters. An additional purpose of this study is to generate data that shows how that end use would have been evaluated if both the required exposure waters were used and included the evaluation of Day-2 of the evaluation protocol.

Although the extraction testing under this study was performed in accordance with the requirements of NSF/ANSI 61, the authors of the report want to re-iterate that the findings are not relevant to plumbing products as the use of these leaded materials are strictly prohibited under the standard.

Laboratory Methodology:

Pipe samples

A four foot sample of ¾" lead pipe was provided to NSF for use in this study. The pipe was reported as new, that is had not previously been placed in service, and had no outward indication of prior exposure to water.

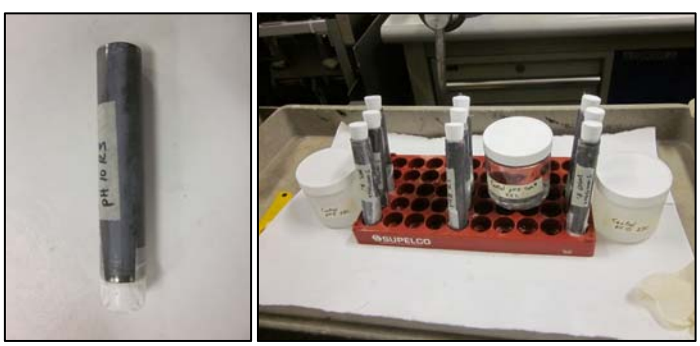
The pipe sample was sectioned into 9, 5" lengths by use of a tubing cutter. Although this minimizes the generation of particulate as compared to use of a hack saw, it does produce a sharp, pinched in area at the opening of the pipe section needing removal. This was accomplished through a few, careful hand turns of a reaming bit followed by a thorough rinsing of the pipe lengths with tap water and bottle brush to minimize inclusion of lead particles in the sample. Following the initial rinsing, the sample was flushed with a minimum of 3 void volumes of deionized water.

Figure 1: Preparation of pipe lengths

Test assemblies

Each 5" length of pipe was used to produce a separate exposure sample. A Teflon-wrapped stopper was tightly fitted into one end and secured in place with tape forming a seal that was not disturbed over the course of the test. A 2nd Teflon wrapped plug was used as the other end seal and was removed daily during water changes.

Figure 2: Test assemblies



Test Waters

The nine test assemblies were randomly divided into three groups of three. One set of the three were exposed using the standard's pH 5 test water; one set of three to the standard's pH 10 test water, and the last set of three to the pH 8 test water of normally used for mechanical plumbing devices (Section 9).

As required by the standard, test waters were made fresh daily. The compositions of test waters are provided in Table 3.1.

Table 1: Test water compositions

Test Water	Composition per liter[1]
pH 5	• 25 mL of 0.1 M NaH_2PO_4 • 25 mL of 0.04 M $MgCl_2$ • 2 mg/L Cl_2 from dilution of NaOCl
pH 10	• 50 mL of 0.1 M NaOH • 50 mL of 0.05 M $Na_2B_4H_7$ • 2 mg/L Cl_2 from dilution of NaOCl
pH 8	• 25 mL 0.4 M $NaHCO_3$ • 2 mg/L Cl_2 from dilution of NaOCl

[1] Diluted to 1 L with "reagent water". Reagent water is defined under the standard as produced through one or more of the following treatment processes: distillation, reverse osmosis, ion exchange, or other equivalent treatment processes and is required to have the following general water characteristics:
– electrical resistivity, minimum 18 MΩ-cm at 25° C (77 °F); and
– total organic carbon (TOC) maximum 100 µg/L.

Exposure protocol

The nine pipe sections were exposure following the procedures outlined in NSF/ANSI 61 for cold water piping products and shown below in Table 2.

Table 2: Exposure protocol

Exposure Day ID	Exposure Duration[1]	Comment
Day-1	24 ± 1 h	Water change
Day-2	24 ± 1 h	Collected for total lead analysis + refilled
Day-3	24 ± 1 h	Water change
Day-4	24 ± 1 h	Collected for total lead analysis + refilled
Day-7	72 ± 1 h	Water change
Day-8	24 ± 1 h	Water change
Day-9	24 ± 1 h	Collected for dissolved and particulate lead analysis +refilled
Day-10	24 ± 1 h	Collected for total lead analysis + refilled
Day-11	24 ± 1 h	Water change
Day-14	72 ± 1 h	Water change
Day-15	24 ± 1 h	Water change
Day-16	24 ± 1 h	Water change
Day-17	16 h	Collected for total lead analysis

[1]At the beginning of each exposure, fresh test water is added to the test assembly which is then allowed to remain static for the duration of the exposure period.

Samplings from Days 2, 4, 10, and 17 for Total lead determination

At the conclusion of each sample exposure from Days 2, 4, 10, and 17, aliquots of the exposed water were decanted into 125 mL polyethylene bottles preserved with 1.2 mL of 1:1 nitric acid.

Samplings from Day 9 for dissolved and particulate lead determination

At the conclusion of the Day 9 exposures a different sampling protocol was established to enable determination of the “dissolved” and “particulate” forms of lead in the samples. For this analysis, the pour-offs from the triplicate exposures were combined to form one sample and partitioned as follows.

- Total Lead: An aliquot of the combined sample was decanted without filtration into a 125 mL polyethylene bottle preserved with 1.2 mL of 1:1 nitric acid.
- Dissolved Lead: The remaining sample was filtered by passed through a 0.45 µm filter and subsequently transferred to a 125 mL polyethylene bottle preserved with 1.2 mL of 1:1 nitric acid.
- Particulate Lead: Was to be determined by subtraction (Total – Dissolved).

Analysis

All analysis for lead was performed in accordance with USEPA Method 200.8 (*Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry*).

Normalization

Test results were normalized in accordance with the formulas of NSF/ANSI 61 which requires the concentration of analytes detected in the extraction water be multiplied by a calculated normalization factor (NF) to account for differences between laboratory and field surface-area-to-volume ratios. The general normalization equation that applies is as follows:

$$NF = N1 \times N2$$

$$N1 = \frac{SA_F}{SA_L} \times \frac{V_L}{V_{F(static)}}$$

where:

SA_F = surface area exposed in the field;
SA_L = surface area exposed in the laboratory;
V_L = volume of extraction water used in the laboratory;
$V_{F(static)}$ = volume of water to which the product is exposed under static conditions; and
N2 = 1 for static normalized results for pipe with a nominal IDs < 4”

As this exposure was performed “in the pipe”, only minor normalization was needed for the Day 17 results as the SA_F and SA_L values are equal and the differences between the V_L and $V_{F(static)}$ is the small amount of water displaced by a portion of the Teflon-wrapped stopper. The NF values for Day 17 ranged from 0.94 to 0.96. The normalization of the remaining days were additionally multiplied by 16/24 to account for the 24 hour period of those exposures versus the 16 hour period of the normal day evaluated (Day 17).

Results:

Provided below are the results of all lead analysis of the research. Table 3 provides a summary of the results from all Day 17 exposures (required evaluation day). Table 4 provides a summary of the remaining days also analyzed for total lead extraction. Table 5 provides a summary of the dissolved lead analysis form Day 9.

The results are reported in four columns.

- The "Sample" value is the result of analysis directly on the test water after exposure to the pipe section. No adjustments are made to this value. These results are simply the concentration of contaminants detected in the exposed water as drawn directly from the exposed test assembly.
- The "Control" value is the result of analysis on the exposure control sample. This sample has been constructed to address potential contaminant contributions that may be an artifact of the test such as from the test water itself or that may be attributed to portions of the test assembly that are not the pipe section under test.
- The "Result" is a calculated value where the "Sample" result has been lessened by any potential artifacts as determined through the "Control" and represents the quantity of contaminant released from the pipe section under test. Only detected Control values are subtracted.
- The "Normalized Result" is a value calculated by multiplying the lab "Result" by a normalization factor to adjust the value to a potential at-the-tap value. This is the value used when comparing the results of testing to the drinking water acceptance criteria in the standard.

Table 3: Lead results from all Day-17 exposures (µg/L)

Test Water	Exposure Day ID	Replicate ID	Sample Value	Control Value	Result Value	Normalized Result
pH 5	17	1	6,000	ND(1)	6,000	5,700
pH 5	17	2	3,700	ND(1)	3,700	3,500
pH 5	17	3	5,400	ND(1)	5,400	5,100
pH 10	17	1	150,000	ND(1)	150,000	150,000
pH 10	17	2	150,000	ND(1)	150,000	150,000
pH 10	17	3	150,000	ND(1)	150,000	140,000
pH 8	17	1	180	1	180	170
pH 8	17	2	130	1	120	120
pH 8	17	3	120	1	120	110

Table 4: Lead results from all non-Day 2, 4, and 10 exposures (µg/L)

Test Water	Exposure Day ID	Replicate ID	Sample Value	Control Value	Result Value	Normalized Result
pH 5	2	1	15,000	2	15,000	9,700
pH 5	2	2	17,000	2	17,000	11,000
pH 5	2	3	17,000	2	17,000	11,000
pH 5	4	1	11,000	1	11,000	7,300
pH 5	4	2	9,600	1	9,600	6,100
pH 5	4	3	11,000	1	11,000	7,000
pH 5	10	1	7,900	ND(1)	7,900	5,000
pH 5	10	2	3,600	ND(1)	3,600	2,300
pH 5	10	3	6,800	ND(1)	6,800	4,300
pH 10	2	1	110,000	2	110,000	68,000
pH 10	2	2	120,000	2	120,000	76,000
pH 10	2	3	110,000	2	110,000	68,000
pH 10	4	1	120,000	2	120,000	75,000
pH 10	4	2	140,000	2	140,000	86,000
pH 10	4	3	170,000	2	170,000	110,000
pH 10	10	1	110,000	1	110,000	70,000
pH 10	10	2	100,000	1	100,000	64,000

pH 10	10	3	110,000	1	110,000	71,000
pH 8	2	1	2,300	ND(1)	2,300	1,400
pH 8	2	2	2,000	ND(1)	2,000	1,300
pH 8	2	3	1,400	ND(1)	1,300	840
pH 8	4	1	280	ND(1)	280	180
pH 8	4	2	330	ND(1)	330	210
pH 8	4	3	280	ND(1)	280	180
pH 8	10	1	210	ND(1)	210	130
pH 8	10	2	200	ND(1)	200	130
pH 8	10	3	240	ND(1)	240	150

Figure 3: Combined graphic of lead results from Days 2, 4, 10, and 17

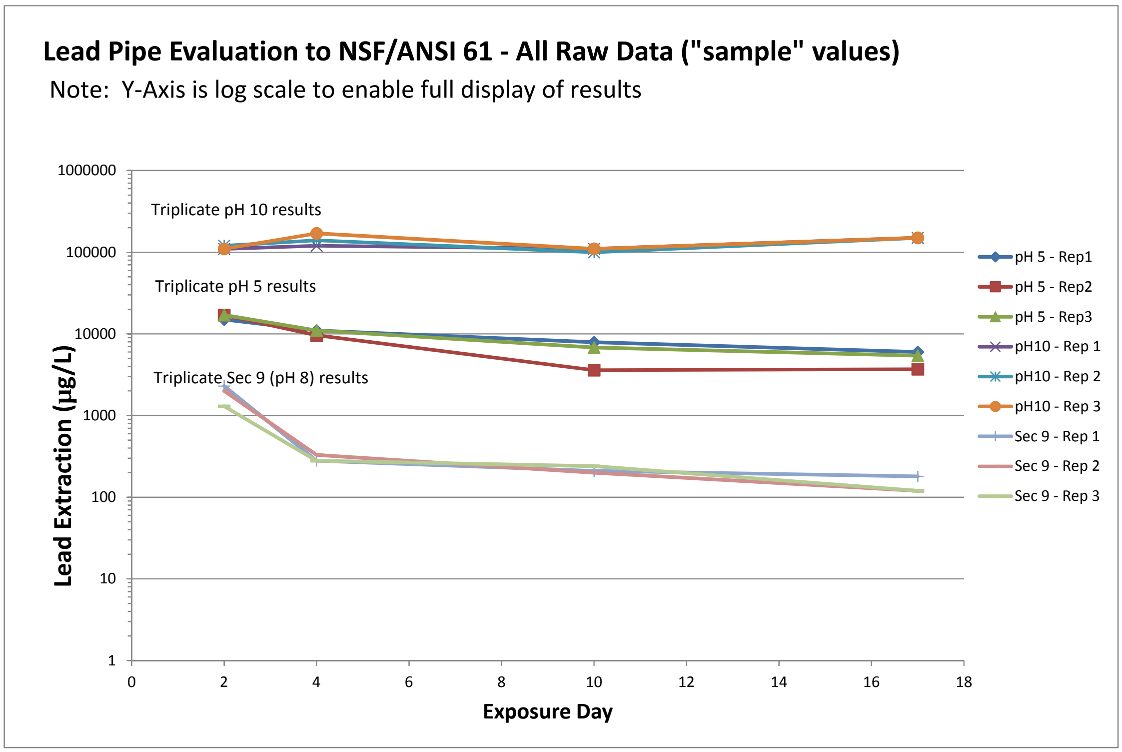


Table 5: Lead analysis on Day 9 exposure (µg/L) [1]

Test Water	Exposure Day ID	Preparation Description	Sample Value	Control Value	Result Value	Normalized Result
pH 5	9	Filtered	10	ND(1)	10	6.0
pH 5	9	Not Filtered[1]	1,900	ND(1)	1,900	1,200
pH 10	9	Filtered	5,600	ND(1)	5,600	3,600
pH 10	9	Not Filtered[1]	120,000	ND(1)	120,000	78,000
pH 8	9	Filtered	70	2	68	43
pH 8	9	Not Filtered[1]	160	2	160	100

[1] Data not to be used as it may not reflect intended research conditions. See report for additional detail.

Methodology issue with "Not Filtered" analysis on Day 9

Some of the results on the non-filtered aliquots are low in comparison to the trends the other days analyzed (2, 4, 10, and 17). This is suspected to have occurred as these samples were not thoroughly mixed prior to removing an aliquot for analysis. Should this work be reproduced, this portion of the research methodology should be modified to ensure a representative aliquot is taken. The results of the non-filtered Day 9 results are not being used in this study as a result. This issue does not affect the "filtered" aliquot as the dissolved lead is relatively uniform in distribution within the sample.

Discussion:

Lead pipe evaluated against NSF/ANSI 61

The results of testing clearly found the lead pipe sample extracting lead far in excess of the NSF/ANSI 61 criterion of 15 µg/L. The average of the Day 17 triplicates for the pH 5, pH 10, and pH 8 Section 9 test waters were 4,800 µg/L, 150,000 µg/L, and 130 µg/L respectively.

Results evaluated as if from a small, pure lead Section 8 device

As there are no pure lead in-line devices known to exist by the authors of this paper, it was hypothesized that a short section of lead pipe was used to simulate "pure lead inline device" in the (Dudi et al, 2004) research. Based on its stated volume of 6.3 mL and the 0.67" ID lead pipe used in the remainder of their study, it's estimated that the wetted surface of the "inline device" was 2.3 in^2.

The surface area to volume ratio of pipe with a ¾" ID is 325 in^2/L. Table 6 provides an analysis of this study's average Day 17 results renormalized to reflect the potential contributions from small Section 8 in-line device. The results from the pH 5 test water are shown to be less than 15 ug/L when normalized to a surface area of only 2.3 in^2. However this does not mean the evaluation would pass NSF/ANSI 61 as the result from the pH 10 exposure is also required to comply. The surface area of exposure would need to be no more than 0.13 in^2 in order for the normalized results to be less than or equal to the 15 µg/L requirement.

*[Example calculation for Table 6: 8.4 µg/L = 4,800 µg/L * (2.3/325) * 0.33 * (12/16)] The 0.33 is the N3 factor normally applied to in-line device test results. The 12/16 represents an adjustment to account for the 12 hr assumption for in-line devices and the 16 hr assumption for pipe the results were first normalized to.]*

Table 7 provides a similar evaluation for the Day 2 results. The analysis also demonstrates that only a very small surface area of exposure would be supported (0.27 in^2) when considering both waters.

Note: Although the Normalized Day 2 results calculate to a lesser value than the Day 17 results, this is due to an over extension of a time correction factor. Lead is known to not extract linearly with respect to extended times of exposure.

Table 6: Day 17 Normalized Lead Extraction Results (µg/L)

Normalization conditions (assumed SA/V ratio)	**3/4" Pb Pipe** (325 in^2/L)	**In-Line Device** (2.3 in^2/L)	**In-Line Device** (0.13 in^2/L)
pH 5 test water	4,800	8.4	0.47
pH 10 test water	150,000	260	15
pH 8 (section 9) test water	130	0.23	0.013

Table 7: Day 2 Normalized Lead Extraction Results (µg/L)

Normalization conditions (assumed SA/V ratio)	**3/4" Pb Pipe** ($325\ in^2/L$)	**In-Line Device** ($2.3\ in^2/L$)	**In-Line Device** ($0.27\ in^2/L$)
pH 5 test water	11,000	18	2.2
pH 10 test water	71,000	120	15
pH 8 (section 9) test water	1,200	2.1	0.25

Additional Observations:

Pipe scales

At the completion of testing, pipe scale was noted having formed on the interior of the some of the pipe sections. A description of the scale is provided Table 8 and photographs in Figure 4. No analysis of the scale composition was attempted.

Table 8: Description of pipe scales formed during testing

Test Water	**Scale Description**
pH 5	White patchy scale appeared in clumps on a small fraction of the exposed area (estimated at less than <5%)
pH 10	Pronounced yellowish brown scale formed across all exposed surfaces.
pH 8	No distinguishable scale was found on the interior of the specimens. Interior had a dull finish.

Figure 4: Pipe sections after exposure

[pH 5 specimens] [pH 10 specimens] [pH 8 specimens]

<u>pH 5 test water</u>: More pronounced scales of lead phosphate compounds were expected to have formed on the pH 5 test assemblies given the high level of orthophosphate in the test water (approximately 20x what's used during corrosion control by water utilities). As these scale were readily friable (broken apart by light mechanical action), and tended to appear at slightly higher frequency toward the bottom of the test assembly, a conjecture was made that the scale formed did not have time over the course of the normal Standard 61 exposure protocol to develop and firmly attached to the pipe surfaces. Rather, the lead phosphate compounds formed may have readily been poured out of the test assembly with the exposed water and included in the total lead analysis as would likely happen with any Standard 61 evaluation of a similar material.

pH 10 test water: The level of scale formed on the interior of the pipe specimens exposed to the pH 10 water was not anticipated. This test water is buffered to a pH of 10 by the use of a high concentration of sodium borate. The scale was not readily friable and appeared securely in place across the full wetted area of the pipe samples. Given the extremely high lead extraction results on these samples, it is reasoned that the lead corrosion is not being bound into the scale attached to the pipe.

pH 8 (Section 9) test water: The results from the pH 8 test water exposures found significantly less lead leached from lead pipe than the both the pH 5 and pH 10 test waters. This result is contrary to that found in separate research on lead extraction from brass, especially in the comparison to the pH 5 test waters. As pure lead materials are excluded from use in drinking water products under the standard, this finding further demonstrates the irrelevance of any analysis of pure lead materials to standard as the results are not representative of how the standard performs on brass, the only material allowed to contain any lead as an intentional additive.

References:

Dudi, A.; Schock, M.; Murray, N; & Edwards, M., 2005. Lead Leaching From Inline Brass Devices. *Jour*. AWWA, 97:8:66.

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Triantafyllidou, S. & Edwards, M. 2007. Critical Evaluation of NSF 61 Section 9 Test Water for Lead. *Jour*. AWWA, 99:9:133.