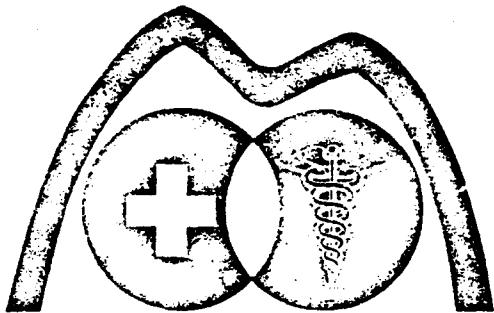


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WATER EXTRACTION OF ADDITIVES FROM PVC PIPE

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WATER EXTRACTION OF ADDITIVES FROM PVC PIPE

INTRODUCTION

Although numerous data has been developed concerning the water extraction of VCM from PVC pipe, no data was found in the current open literature on the extraction of tin stabilizers and other materials used to produce potable water pipe. A limited amount of information was found regarding the extraction of lead based stabilizers that are used for some European potable water pipe service. (1)(2)(3)(4)(5)(6) Therefore, because of our extensive work on extraction of VCM, (7) we decided to initiate a study on other direct pipe compound additives to insure the total safety of PVC potable water pipe.

EXPERIMENTAL

Pipe for this study was extruded using a 2-1/2 inch single screw extruder with typical pipe die and vacuum sizing equipment. Product made for testing was one inch schedule 40 pipe. This type pipe could be used in a variety of applications, including cold water piping systems. The formulation used for producing the pipe for this study is shown in Table 1.

Pipe samples for this extraction testing were prepared by cutting 14 cm. lengths from each pipe compound extruded. Eight lengths of each sample to be tested were placed in a glass jar which was filled with 2.0 liters of deionized water. This surface to volume ratio (pipe surface to water amount) corresponds to 4 ml of water per square inch of pipe surface as has been recommended by the NSF. These jars were then sealed with aluminum lined screw tops.

Duplicate jars were prepared for each pipe sample. One jar was stored at room temperature (25°C) and the other at 50°C. Jars were stored for two weeks. At the end of this initial two week period, a 100 ml sample was withdrawn for trace metal analysis. After sampling, 100 ml of fresh deionized water was added back to each jar and the jar returned to storage. Subsequent analyses at two week intervals were corrected for the dilution effect of the 100 ml of fresh water. A blank sample was run at each temperature.

ANALYTICAL TESTING PROCEDURE

The samples obtained for this testing were analyzed on a Perkin-Elmer Model 503 Atomic Absorption Spectrophotometer operated in the absorbance mode. A single 100 ml aliquot was withdrawn from each sample and blank jar. Samples were preserved between sampling and analysis with 0.1% nitric acid (Baker Ultrex) to prevent loss of metal from solution by precipitation or absorption.

Question

First data point @ 2 weeks.
Any work done to determine if
tin found was truly extract-
ed or if was residues from
extrusion with methyl being
higher due to greater volatility,
than butyl + octyl.

Response

No work done. Do not
know if what was found was
extracted or not.

Tin and titanium were analyzed using the graphite furnace. Calcium and magnesium were analyzed using an air-acetylene flame. For calcium analyses, lanthanum chloride was added to samples and standards at a 20% concentration to suppress calcium ionization.

At least three replicate analyses of each sample were preformed. All results were corrected for blank values.

RESULTS AND DISCUSSION

The first data presented in this study are the tin extraction values. These results (Table 2) show the individual levels extracted on two butyl, two methyl, and one octyl tin stabilized pipe compounds. The stabilizers used are commercially available products. The two butyl and two methyl samples are not duplicates and were made using different stabilizers. The initial test results are reported after two weeks extraction at 25° and 50°C and on through eight weeks extraction at two week intervals.

The data shows that relatively low levels of tin are extracted from these pipe samples with the higher levels occurring after the initial two week testing period. After this point we did not get an increase in the tin extraction level and the values drop off to a relatively constant level and do not go up again.

These tin extraction results were also plotted as shown in Figures I and II. For these plots the extraction values for the methyl and butyl tins were averaged and only one curve is shown for each on both the 25° and 50°C plots. These data show little difference in the methyl and butyl extraction at 25°C, although both show a slight decrease. The octyl is much lower at both extraction temperatures. The data in the 50°C plot (Figure II) shows the same trend with overall extraction levels decreasing with increasing extraction times. The unusual feature of this 50°C plot is the rather high initial extraction value for the methyl tin, followed by a dramatic decrease with time to the point where it is about equivalent to the butyl sample.

A possible explanation for this phenomenon is precipitation of insoluble tin compounds or absorption of extracted tin on the walls of the jar or the pipe. In order to verify this, one methyl tin sample was acidified with concentrated HCl to dissolve insoluble tin compounds. The tin level before acid addition was 0.047 ppm. After addition of acid, the tin level in solution increased to 0.385 ppm, an eight fold increase. This confirms our hypothesis that extracted tin compounds are subsequently converted to an insoluble form and are precipitating from solution or are being absorbed on the walls.

Another important point that can be made from these data is the fact that with increasing chain length, from butyl or octyl, there is a significant decrease

in the tin extraction level. This change in extraction level is not as dramatic as from the methyl to butyl types.

The calcium extraction data is shown in Table III. These results show that calcium extraction level is relatively independent of extraction temperature. The maximum calcium extraction levels generally occur at the two week testing period and then tend to level out.

The titanium extraction data is shown in Table IV. These results indicate that no titanium was detected through six weeks of extraction. The method used for this analysis is a test sensitive to 40 parts per billion.

Magnesium extraction data is shown in Table V. These results indicate that when testing at 25°C, maximum Mg levels are reached after two weeks and stay relatively constant.

The 50°C magnesium extraction data was averaged for the methyl and butyl tin pipe samples and is plotted in Figure III. Magnesium levels show an increase with time through about four weeks at which point the extraction values appear to be leveling out for methyl and butyl but not for octyl.

CONCLUSIONS

This study shows that the extraction levels of metals from PVC pipe are at relatively low levels. The data also indicates that with the exception of

magnesium at 50°C, the extraction levels start to exhibit a leveling out trend between the 2 and 4 week testing periods.

In summary, these results have shown that:

- (1) No titanium was extracted.
- (2) A maximum of calcium extraction levels was reached after two weeks and then essentially a constant level was obtained which was independent of the extraction temperature.
- (3) Tin extraction results show they reach a maximum after two weeks extraction and maintain a rather constant extraction level in four out of five samples. Also, it was shown that as the length of the alkyl chain increases, the tin extractibles decrease.
- (4) Magnesium extraction data shows that at the 25°C extraction temperature, a maximum level occurs within two weeks and remains constant. The 50°C data generally indicates a maximum level is reached in four weeks and then remains constant.

Again, it should be emphasized that the extraction values obtained in this study are at relatively low levels on all metals tested. Another important consideration is that this study was conducted under static conditions and the length of testing is far in excess of normal residence time of water in PVC piping systems.

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TABLE I

FORMULATION FOR EXPERIMENTAL SINGLE SCREW PIPE COMPOUND

	phr
PVC Resin	100
Tin Stabilizer	1.2
Processing Aid	0.5
Titanium Dioxide	1.0
Paraffin Wax	1.0
PE Wax	0.1
Internal Lubricant System	
Calcium Stearate	1.5
Magnesium Stearate	0.25

TABLE II

TIN EXTRACTION DATA

Sample	Stabilizer	PHR Level	Extraction Temp.	Test Results - Extraction Levels (ppm)			
				2 Weeks	4 Weeks	6 Weeks	8 Weeks
1	Methyl	1.2	25°C	0.017	0.011	0.014	0.016
			50°C	0.170	0.077	0.072	0.027
2	Methyl	1.2	25°C	0.033	0.030	0.031	0.029
			50°C	0.037	0.022	0.031	0.024
3	Butyl	1.2	25°C	0.040	0.012	0.031	0.025
			50°C	0.073	0.031	0.044	0.036
4	Butyl	1.2	25°C	0.017	0.007	0.016	0.014
			50°C	0.023	0.009	0.019	0.009
5	Octyl	1.2	25°C	<0.002	0.005	<0.002	<0.002
			50°C	0.004	0.008	0.010	0.011

TABLE III

CALCIUM EXTRACTION DATA

Sample	Extraction Temp.	Test Results - Extraction Levels (ppm)		
		2 Weeks	4 Weeks	6 Weeks
1	25°C	2.20	2.53	2.11
	50°C	2.45	2.94	2.59
2	25°C	1.45	1.66	1.53
	50°C	1.14	1.70	1.58
3	25°C	1.90	2.03	1.71
	50°C	2.38	2.81	2.67
4	25°C	2.00	2.28	2.03
	50°C	2.08	2.59	2.16
5	25°C	2.10	2.30	1.91
	50°C	2.18	2.71	2.44

TABLE IV

TITANIUM EXTRACTION DATA

<u>Sample</u>	<u>Extraction Temp.</u>	<u>Extraction Level (PPM)* After 6 Weeks</u>
1	25°C	< 0.040
	50°C	< 0.040
2	25°C	< 0.040
	50°C	< 0.040
3	25°C	< 0.040
	50°C	< 0.040
4	25°C	< 0.040
	50°C	< 0.040
5	25°C	< 0.040
	50°C	< 0.040

*Test sensitive to 40 parts per billion.

TABLE V

MAGNESIUM EXTRACTION DATA

<u>Sample</u>	<u>Extraction Temp.</u>	<u>Test Results - Extraction Levels (ppm)</u>		
		<u>2 Weeks</u>	<u>4 Weeks</u>	<u>6 Weeks</u>
1	25°C	0.061	0.037	0.037
	50°C	0.096	0.160	0.156
2	25°C	0.027	0.035	0.032
	50°C	0.067	0.129	0.132
3	25°C	0.031	0.030	0.030
	50°C	0.126	0.166	0.132
4	25°C	0.032	0.038	0.032
	50°C	0.082	0.120	0.129
5	25°C	0.036	0.040	0.037
	50°C	0.080	0.144	0.175

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FIGURE I

Tin Extraction vs. Time
@ 25°C Extraction Temperature

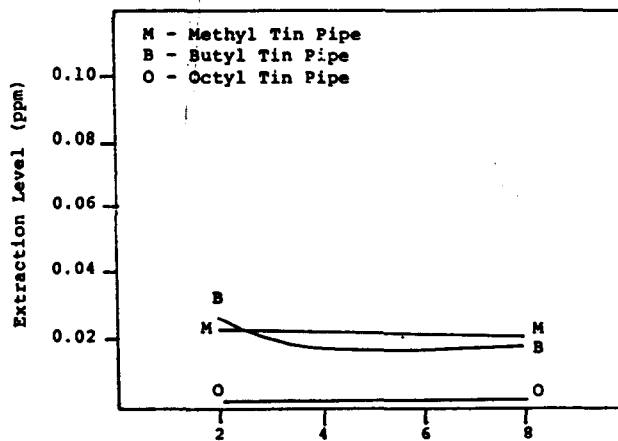
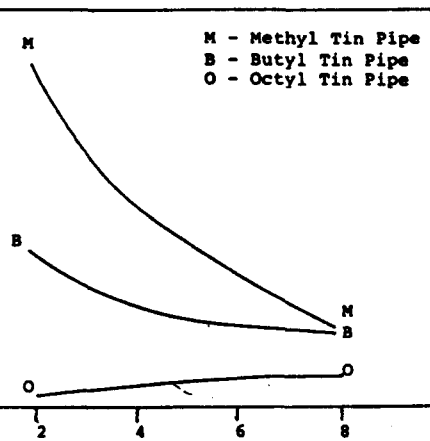


FIGURE II

Tin Extraction vs. Time
@ 50°C Extraction Temperature



Extraction Time (Weeks)

FIGURE III

MAGNESIUM EXTRACTION VS. TIME

Extraction Temperature - 50°C

