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FURTHER EVALUATION OF A DIFFUSION MODEL FOR THE CHARACTERIZATIO--ETC(U)

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FURTHER EVALUATION OF A DIFFUSION MODEL FOR THE CHARACTERIZATION OF THE LEACHING PROPERTIES OF SEVERAL CONVENTIONAL ANTIFOULING COATINGS.

by

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Charles P./Monaghan / Vasant H./Kulkarni ■ Mary L./Good

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FURTHER EVALUATION OF A DIFFUSION MODEL FOR THE CHARACTERIZATION OF  
THE LEACHING PROPERTIES OF SEVERAL CONVENTIONAL ANTIFOULING COATINGS

by

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ABSTRACT

An empirical leaching model based on Fick's First Law of Diffusion has been developed for evaluating and comparing the leaching properties of a series of marine antifouling coatings containing organotin moieties as the active toxicant species. The model has been previously applied to the behavior of a series of coatings containing various triphenyltin and tributyltin compounds blended into a conventional vinyl-type coating. Empirical parameters could be extracted from the data for comparing leaching properties of one coating versus another by analyzing laboratory data from static leach tests. To extend the model and to further verify its utility, the present study was undertaken to apply the model to leaching data in the literature for a variety of coatings containing cuprous oxide, triphenyllead acetate and other triorganotin compounds. The results are encouraging in that the toxicant diffusion curves which are derived for these coatings have the same general form as those observed in the previous study, and the empirical parameters deduced for each coating can be rationalized in terms of the known relative properties of the various coatings.

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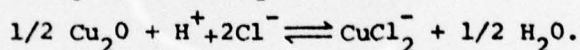
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## INTRODUCTION

For a marine antifouling coating to be effective, the toxicant must leach from the coating fast enough to repel or kill settling organisms. On the other hand, commercial applications require that the leaching be slow enough to insure a long service life for the coating. Leaching models describing the release of  $\text{CuCl}_2^-$  from coatings containing cuprous oxide as toxicant have been described in the literature (1,2,3,4). However, there are several significant differences between coatings containing cuprous oxide as toxicants and those containing organometallic compounds which make these leaching models inadequate for universal employment. A more general model is needed which would permit a quantitative comparison of the properties of antifouling coatings containing cuprous oxide, triphenyllead acetate, tributyltin, or triphenyltin compounds as toxicant.

In coatings containing cuprous oxide, the leaching properties are thought to be determined by the fact that the toxicant particles are dispersed throughout the coating in such a manner that they are in contact. These coatings are known as continuous contact coatings. As the toxicant particles dissolve, pores develop in the coating matrix through which the dissolved toxicant can diffuse to the surface of the coating. The leaching process is thought to occur in several steps as outlined below. Solvent (seawater) diffuses through the exhausted matrix to the leaching zone and dissolves cuprous oxide. Cuprous oxide dissolves in sea water to establish the following chemical equilibrium:



The solvated toxicant molecule diffuses through the porous, exhausted matrix into the quiescent layer of sea water at the surface of the coating and then diffuses out of the quiescent layer into the environment. The rate determining steps are thought to be the diffusion of solvated toxicant through the exhausted matrix and then through the quiescent layer. A stationary state is established where the concentration of dissolved toxicant will be constant throughout any cross section of the exhausted coating and quiescent layer. The flux of dissolved toxicant would then be the same through any unit area in the exhausted matrix and in the quiescent layer (1,4). To characterize the leaching process using these ideas, a thorough study of the matrix is necessary, making the routine application of this type of model in the laboratory somewhat awkward.

Coatings which contain organometallic compounds are different from those containing cuprous oxide in several aspects. The organometallic toxicants, which are of the general formula  $R_3MX$  ( $M=Sn,Pb$ ), are either solids or liquids. The liquids are expected to disperse homogeneously in the coating when mixed with a solvent. The solids may dissolve and disperse homogeneously or they may disperse as fine particles forming a heterogeneous matrix. The usual pigments, carbon, titanium dioxide, and cuprous oxide are insoluble and are also dispersed as fine particles in the coating. Successful antifouling coatings containing organometals have a rather low loading (10%) compared to coatings containing cuprous oxide which may contain as much as forty percent toxicant. Structural differences of the different types of organotin compounds are also thought to be an important factor in the rate of hydrolysis of the organotin compounds (5). Thus the continuous contact leaching models are not appropriate for coatings containing organometallic compounds.

The many types of marine antifouling coating formulations which are being used and the fact that each coating can be either homogeneous or heterogeneous (or both) with respect to the toxicant makes the study of the leaching process a complex problem. An additional complicating factor is that there are many testing and reporting procedures in the literature. The laboratory evaluation of currently used coatings and the development of new coatings require that the leaching process be described quantitatively. Thus, we have undertaken a study to develop a new model which will handle the leach rate data from a variety of coatings, particularly those containing organometallic toxicants. Initial studies for a series of organotin containing coatings have been reported (6). This present study was undertaken to evaluate the utility of the model for analyzing leach rate data in the literature for coatings containing cuprous oxide, triphenyltin, tributyltin, or triphenyllead acetate as toxicants. It was hoped that the derived empirical parameters could be related to the relative effectiveness of the various coatings. In addition, new insights into the leaching process might be gained which would lead to the development of improved matrices and more effective products.

#### THEORY

The empirical diffusion model which has recently been developed describes the leaching process of organotin toxicants from antifouling coatings (6). The model assumes the following leaching mechanisms.

Solvated toxicant molecules diffuse out of the coating in a direction normal to the plane of the coating surface. These molecules diffuse through the thin quiescent layer of solution at the surface and then into the well-mixed environment. The regions of coating surface which are active in the diffusion process depend on the development of a porous matrix as the toxicant and matrix dissolve and leach out of the coating. (see Figure 1 for a pictorial representation of the leaching process). If  $f$  is the fraction of the surface that is active in the diffusion process and if  $A_0$  is the area of the surface plane, then the area,  $A$ , of active surface is given by

$$A = fA_0. \quad (1)$$

Using Fick's First Law of Diffusion and equation (1), the concentration,  $c$ , of toxicant in the leachate is derived to be (details are given in reference 6):

$$c = c_s - c_s \exp \left( -\alpha \frac{A_0}{V} t \right). \quad (2)$$

where  $c_s$  is the concentration of organotin at the coating surface,  $V$  is the volume of the sea water in the experimental container, and  $t$  is time. The parameter  $\alpha$  is  $Df/x$  where  $D$  is the diffusion coefficient of the solvated toxicant molecule and where  $x$  is the thickness of the quiescent layer of solution. The value of  $\alpha$  is characteristic of a coating at a specified temperature and water flow condition at the coating surface. Considering that the concentration of toxicant in a large body of water like a harbor or ocean will always be vanishingly small, the leach rate of toxicant out of the coating at the specified temperature and water flow is given by

$$\frac{dm}{dt} = \alpha A_0 c_s. \quad (3)$$

If this function is normalized with respect to area, it has the form

$$\frac{1}{A_0} \frac{dm}{dt} = \alpha c_s. \quad (4)$$

Thus the leach rate in large bodies of water for explicitly stated conditions can be calculated from parameters determined in the laboratory.

Data reported in the literature are usually leach rate data, and concentration-time values are needed if equation (2) is to be used. To transform literature data into the correct form, leach rate data were fitted with a quadratic equation, and this empirical equation was integrated to obtain calculated concentration-time data. Since equation (2) collapses to

$$c = c_s \quad (5)$$

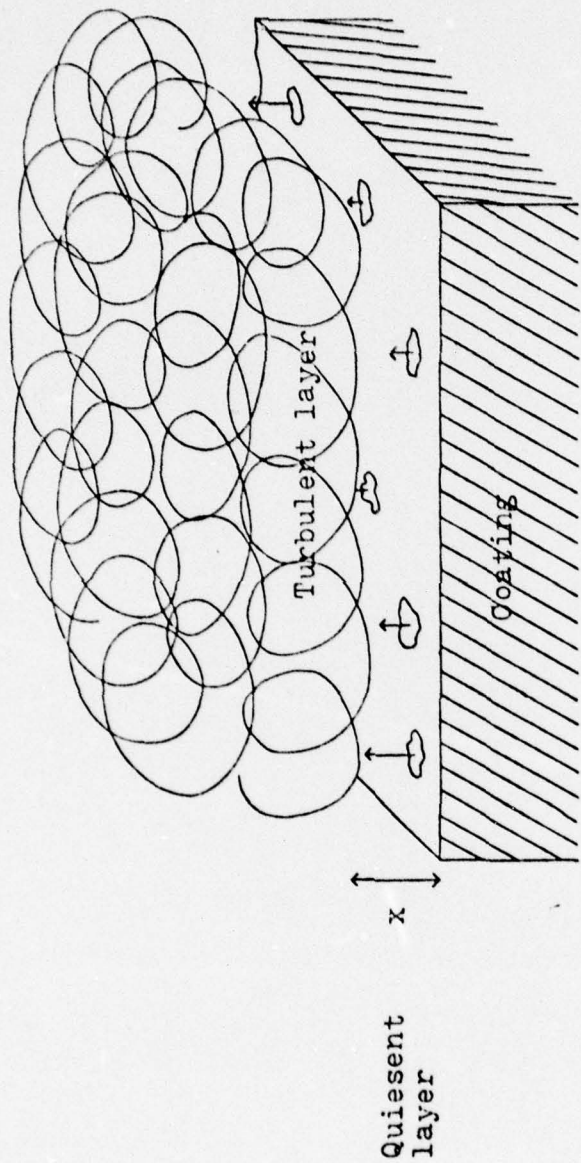


Figure 1. Pictorial representation of the leaching of toxicant molecules from an antifouling coating.

at infinite time, the calculated concentration-time data were plotted and the curve extrapolated to large times in order to estimate  $c_s$ . Using a program called LEACH2, a good value for  $\alpha$  was obtained by first generating data using  $c_s$  and trial values of  $\alpha$  and then comparing the generated data to the concentration-time data. These values for  $c_s$  and  $\alpha$  were further refined by a least squares program called LEACH1 which fit equation (2) to the concentration-time data. Since the concentration-time data has been smoothed by using the integrated function to get the reported data into the correct form, uncertainties in  $c_s$  and  $\alpha$  which were calculated by LEACH1 will not be reported.

#### RESULTS AND DISCUSSION

As can be seen in the normalized leach rate function, equation (4), the rate of release of toxicant into large bodies of water is dependent upon matrix parameters for a particular coating system. The values of  $c_s$  were originally thought to be related to the solubility of organotin toxicant in sea water, but there was considerable variation from solubility data (6). Evidently  $c_s$  is a result of, as yet, poorly characterized interactions between solvent, matrix, and toxicant molecules. The parameter  $\alpha$  must be considered to be completely empirical since there is a lack of information on diffusion constants for many of these compounds and since it is impossible to measure the thickness of the quiescent layer. However, the same diffusion constant would be used for those coatings containing the same type of toxicant compound, and the thickness of the quiescent layer would be the same for similar experiments. Comparison of  $\alpha$  between different coatings containing the same type of toxicant and studies under the same experimental conditions provides a relative measure for comparing the fractions of active surface between coatings. As the fraction of active surface is thought to be linked to the structure of the exhausted matrix, relative values of  $\alpha$  may indicate the relative permeability of various exhausted matrices.

Leach rate data on a series of coatings which contain different amounts of cuprous oxide have been reported in the literature (7). Although scant detail is given on the coating formulation, the paint, which is described as a cold plastic paint, presumably contained cuprous oxide, rosin, and some other polymer. The pigment volume content was held constant by substituting magnesium silicate for cuprous oxide. Glass panels with a surface area of  $155\text{cm}^2$  were coated on both sides, allowed to cure for 1-4 days,

and immersed in clean, aerated sea water. The volume of sea water in the experimental container was 750 ml.

The results of fitting equation (2) to the calculated concentration-time data for the coatings containing different percentages of cuprous oxide can be seen in Figure 2. In Table 1 the values of  $c_s$  are seen to increase as the toxicant content is increased. This trend suggests that the concentration of dissolved toxicant at the surface is related to the toxicant content of the coating. A considerable variation in  $\alpha$  is apparent between a coating which contains a high percentage of cuprous oxide and one which contains a low percentage of cuprous oxide. Since the amount of rosin is presumably the same in all of the coatings and since rosin does dissolve and diffuse out of the coating in an aqueous environment (8), a relatively high ratio of rosin to cuprous oxide may result in a more porous matrix and a more active surface than a coating with a low ratio of rosin to cuprous oxide.

Two types of similar coatings containing  $(C_6H_5)_3PbOAc$  were evaluated to determine the effect of slight composition alterations on leach rates (9,10). Both the Old Type G-4 coating and the New Type G-4 coating contained polyisoprene, chlorosulfonated polyethylene, styrene-butadiene-styrene polymer, and unsaturated polymer resin. The New Type G-4 coating contained twice as much zinc oxide, amorphous silica, and carbon black per 1000 g (wet) as the Old Type G-4 coating. The toxicant,  $(C_6H_5)_3PbOAc$ , was mixed into the elastomeric dispersion before introducing the inorganic material into the New Type G-4 coating; whereas, the  $(C_6H_5)_3PbOAc$  was incorporated into the Old Type G-4 coating at the same time as the inorganic material. Neither the dimensions of the test panels nor the volume of water in the test container were given. The leach rate data were converted into the appropriate form using an assumed area of  $320\text{cm}^2$  and a volume of 500 ml which were values obtained from an earlier publication (9). Values of  $c_s$  will not be correct if the above assumptions of surface area and volume are incorrect, but the relative values will be in the correct order. Values of  $\alpha$  are not dependent on any assumptions of surface area and solution volume.

The magnitudes of  $c_s$  and  $\alpha$  for the Old Type G-4 coating and for the New Type G-4 coating containing 10%  $(C_6H_5)_3PbOAc$  are nearly the same (see Table 1), and plots of the two sets of concentration-time data are nearly

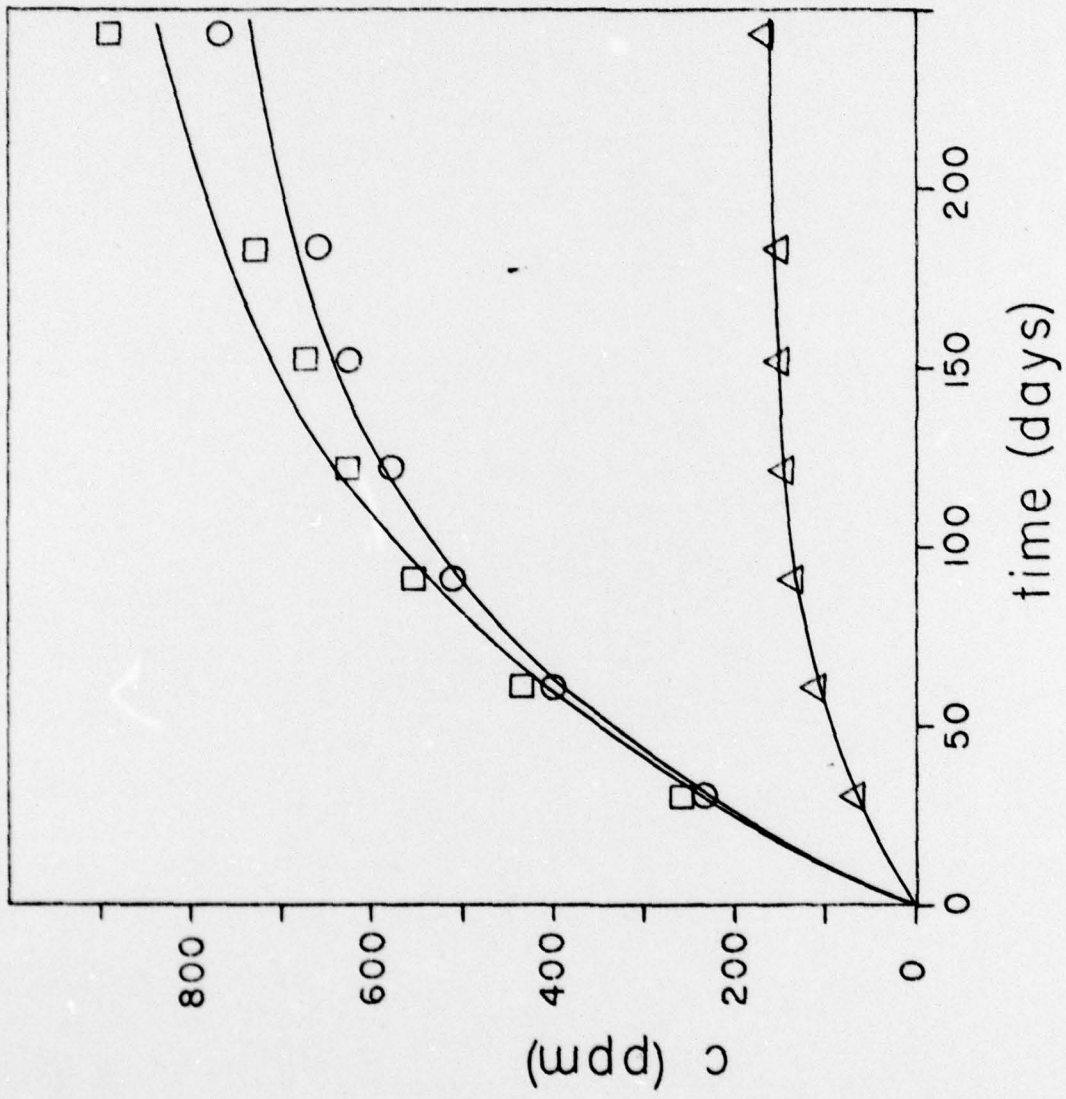


Figure 2. Leaching of toxicant from Cold Plastic Paint containing cuprous oxide.  $\square$  is No. 10,  $\circ$  is No. 8, and  $\Delta$  is No. 4 (See Table 1).

TABLE 1

## EMPIRICAL LEAST SQUARES DIFFUSION PARAMETERS FOR ANTIFOULING COATINGS

COATING	TOXICANT	$c_s$ (ppm)	$\alpha$ (cm day <sup>-1</sup> )
Cold Plastic Paint No.4 (Ref. 7)	Cu <sub>2</sub> O, 4.64%	163	0.086
Cold Plastic Paint No.8 (Ref. 7)	Cu <sub>2</sub> O, 24.8%	792	0.053
Cold Plastic Paint No.10 (Ref.7)	Cu <sub>2</sub> O, 36.0%	929	0.046
New Type G-4 (Ref. 10)	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> PbOAc, 10%	117	0.15
New Type G-4 (Ref. 10)	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> PbOAc, 5%	27.1	0.13
New Type G-4 (Ref. 10)	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> PbOAc, 1.5%	12.3	0.12
Old Type G-4 (Ref. 9,10)	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> PbOAc, 10%	127	0.12
Alum-A-Tox (Ref. 6)	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> SnOAc, 14.2%	7.2	0.16
Alum-A-Tox (Ref. 6)	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> SnCl, 13.4%	2.2	0.18
Alum-A-Tox (Ref. 6)	(C <sub>4</sub> H <sub>9</sub> ) <sub>3</sub> SnOAc, 12.1%	12.8	0.19
Alum-A-Tox (Ref. 6)	[(C <sub>4</sub> H <sub>9</sub> ) <sub>3</sub> Sn] <sub>2</sub> O, 12.3%	9.9	0.17
Alum-A-Tox (Ref. 6)	(C <sub>4</sub> H <sub>9</sub> ) <sub>3</sub> SnCl, 10.9%	3.3	0.45

superimposable as is evident in Figure 3. The conclusions reported in the literature on the study of the G-4 coatings is that the New Type G-4 coating released toxicant at a lower rate than the Old Type G-4 coating. This slower release is attributed to the order in which  $(C_6H_5)_3PbOAc$  is added to the coating mixture. At the 10% toxicant level there is little difference in the leaching properties of the two coatings which is contrary to the conclusion reported. The amount of inorganic material in a coating apparently has little impact on leaching properties.

Results from studies where the amount of  $(C_6H_5)_3PbOAc$  is varied in the New Type G-4 coating are similar to results obtained on coatings containing cuprous oxide. Values of  $c_s$  increase with an increased percentage of  $(C_6H_5)_3PbOAc$  in the coating, but magnitudes of  $\alpha$  are all about the same. The trend in the values of  $c_s$  indicate that the surface concentration of organolead is directly dependent on the loading of toxicant in the coating, as was noted previously for the coatings containing cuprous oxide. The similar values of  $\alpha$  among these coatings suggest that small variations in the toxicant content has little bearing on the fraction of active surface in this type of matrix. As the values of  $\alpha$  are nearly the same, the normalized leach rate is determined primarily by the value of  $c_s$ . This is also apparent in the plots of concentration-time data in Figure 4.

Static leach tests have been performed on panels coated with a vinyl-type coating containing one of several organotin compounds as toxicant (6). "Alum-A-Tox" coating, a commercial vinyl base coating containing titanium dioxide as a pigment, was chosen for the initial studies. One of several  $(C_4H_9)_3SnX$  or  $(C_6H_5)_3SnX$  compounds was incorporated into the coating so that a composition of 3.6% Sn by weight was achieved. Aluminum panels were coated on one side to give a coating surface area of  $77.4\text{cm}^2$ . After the coating was cured, each panel was immersed in 550 ml of artificial sea water.

Considerable variation in  $c_s$  is seen for coatings containing the organotin compounds, but with the exception of the coating containing  $(C_4H_9)_3SnCl$ , values of  $\alpha$  are all about the same. Each curve for the leaching data on the Alum-A-Tox coatings containing a  $(C_6H_5)_3SnX$  or  $(C_4H_9)_3SnX$  compound has the same shape as the curves for coatings containing cuprous oxide or  $(C_6H_5)_3PbOAc$ . One should note in Table 1 that the

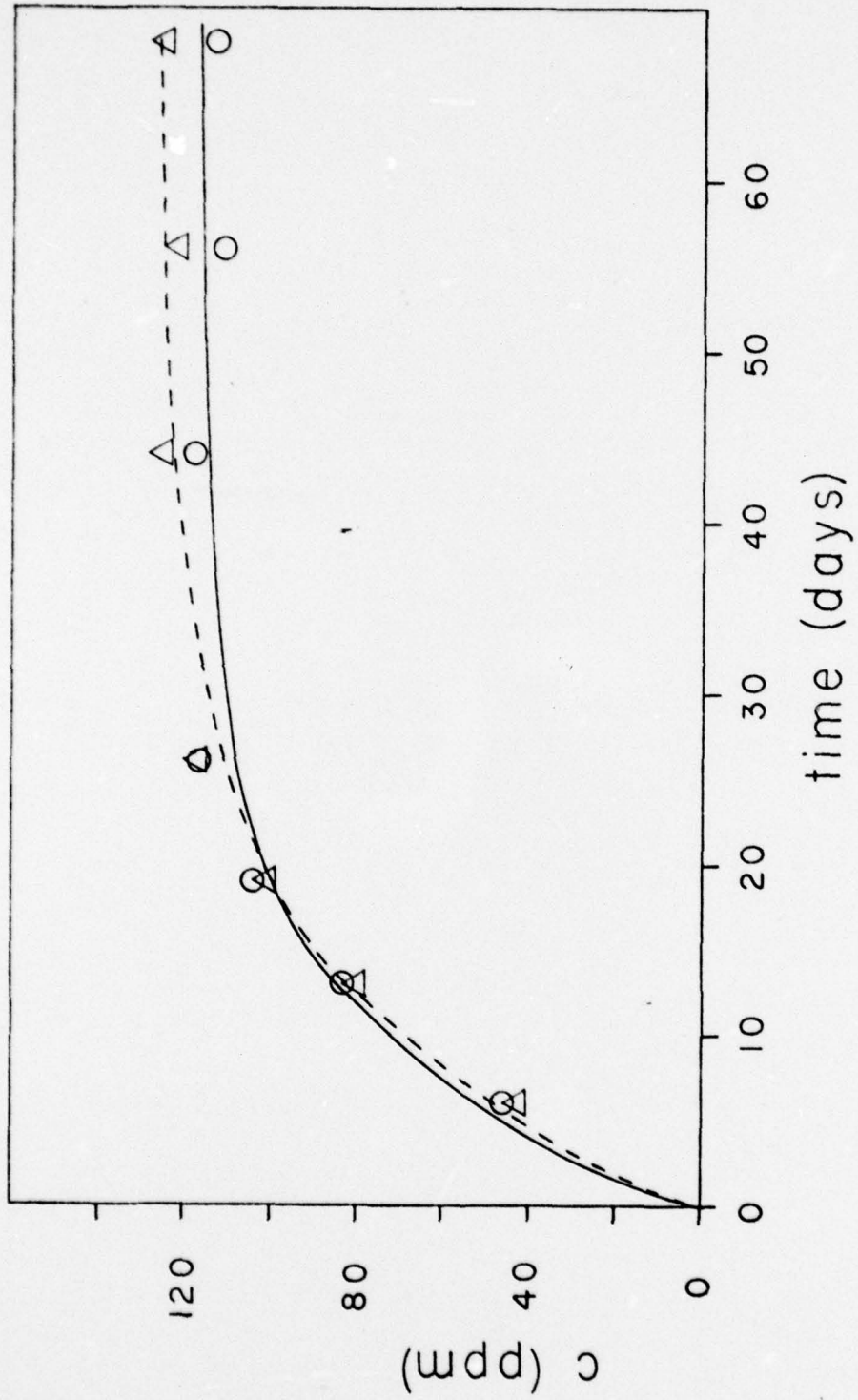


Figure 3. Leaching of  $(C_6H_5)_3PbOAc$  from Old Type G-4 (○) and New Type G-4 (△) coatings.

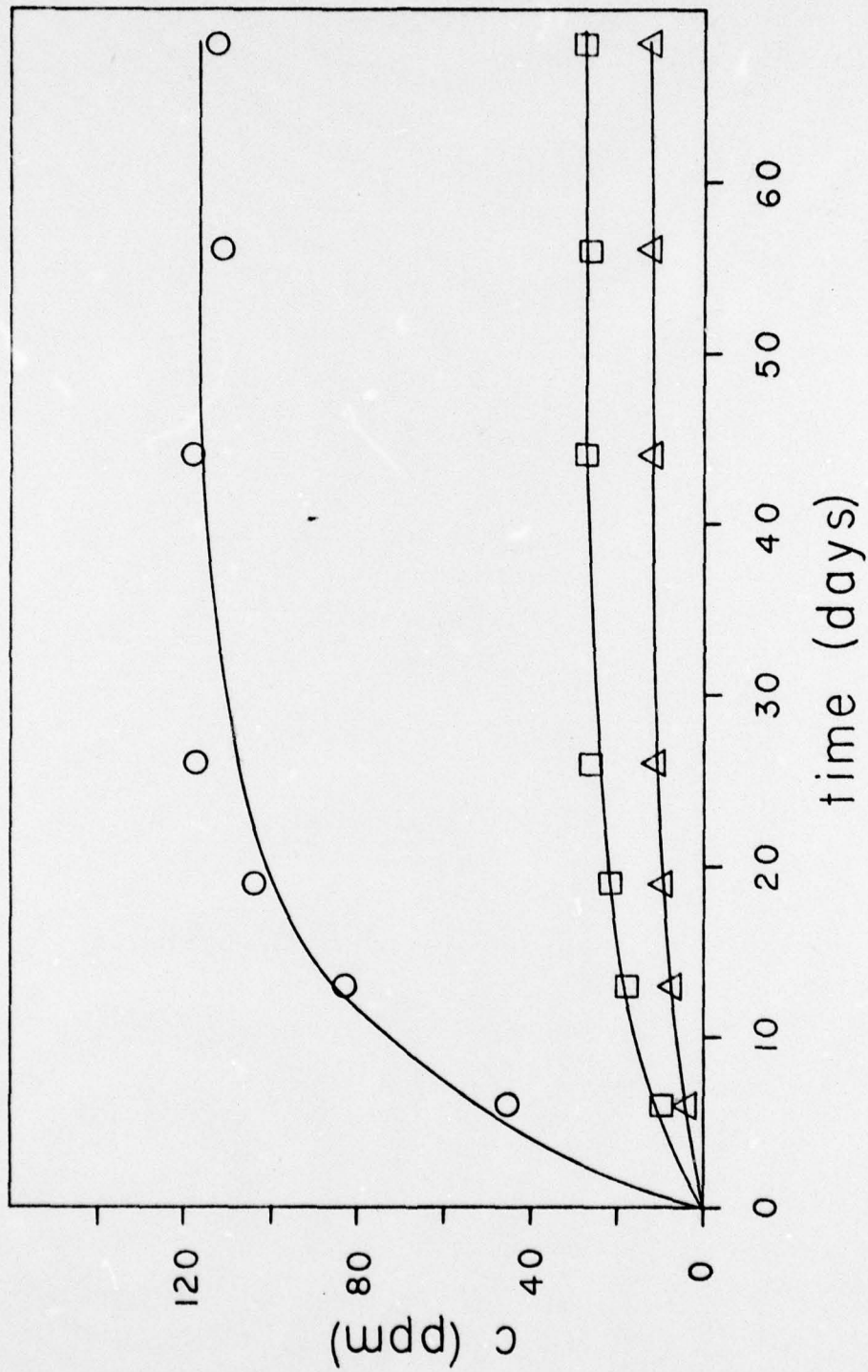


Figure 4. Leaching of  $(C_6H_5)_3PbOAc$  from the New Type G-4 coating.  
 O is 10%,  $\square$  is 5%, and  $\Delta$  is 1.5% (See Table 1).



## CONCLUSION

An empirical diffusion model has been applied to the leaching process of marine antifouling coatings. Leaching of toxicant out of these coatings is characterized by the concentration of toxicant at the coating surface and by the fraction of surface active in the diffusion process. Quantitative laboratory evaluation of a coating is possible and observed trends in  $c_s$  and  $\alpha$  may be discussed in terms of relative porosity of exhausted matrix, quantity of toxicant in the coating, and chemical bonding in the toxicant.

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19. KEY WORDS (Continue on reverse side if necessary and identify by block number) organotin, cuprous oxide, organolead, antifouling coating, controlled toxicant release, diffusion, leaching, antifouling materials		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  An empirical leaching model based on Fick's First Law of Diffusion has been developed for evaluating and comparing the leaching properties of a series of marine antifouling coatings containing organotin moieties as the active toxicant species. The model has been previously applied to the behavior of a series of coatings containing various triphenyltin and tri- butyltin compounds blended into a conventional vinyl-type coating. Empirical parameters could be extracted from the data for comparing leaching		

properties of one coating versus another by analyzing laboratory data from static leach tests. To extend the model and to further verify its utility, the present study was undertaken to apply the model to leaching data in the literature for a variety of coatings containing cuprous oxide, triphenyllead acetate and other triorganotin compounds. The results are encouraging in that the toxicant diffusion curves which are derived for these coatings have the same general form as those observed in the previous study, and the empirical parameters deduced for each coating can be rationalized in terms of the known relative properties of the various coatings.