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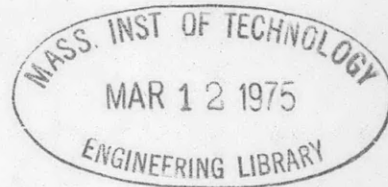
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ANTIFOULING ORGANOMETALLIC STRUCTURAL  
PLASTICS

J. A. Montemarano, et al

Naval Ship Research and Development Center  
Annapolis, Maryland

August 1973



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## ANTIFOULING ORGANOMETALLIC STRUCTURAL PLASTICS

by

J. A. Montemarano and E. J. Dyckman

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**RESEARCH AND DEVELOPMENT REPORT**

August 1973

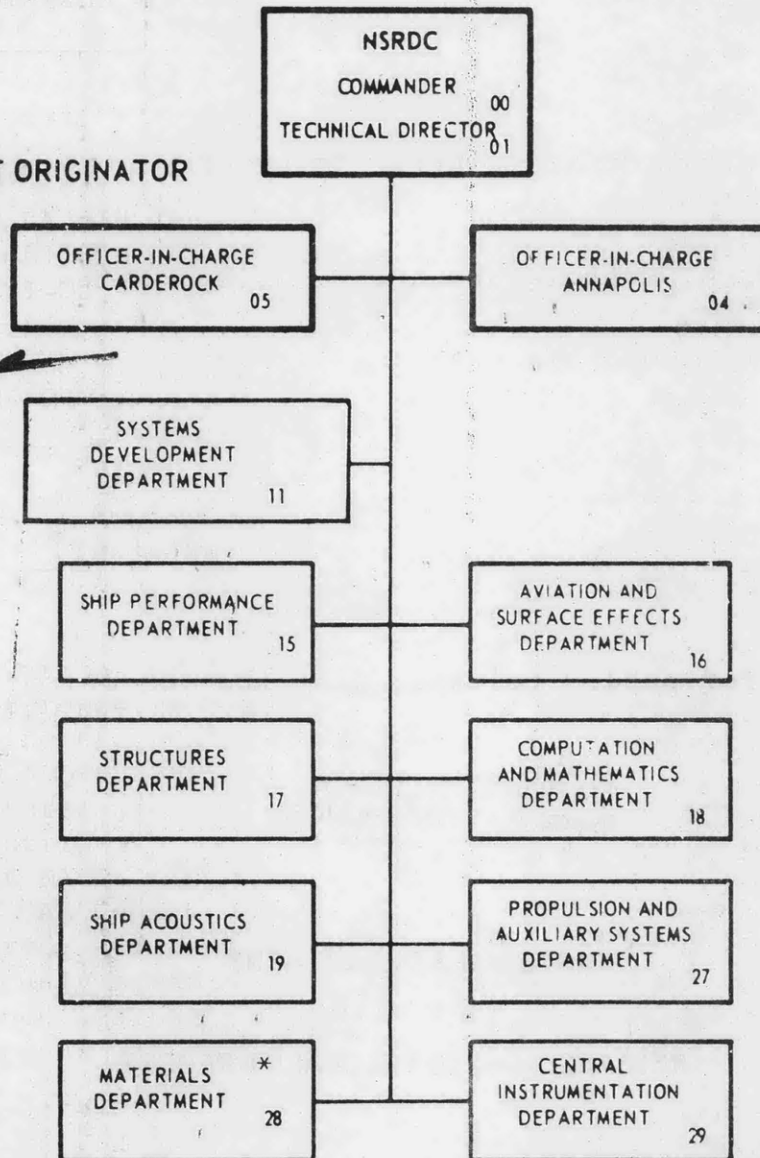
Report 4159

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13. ABSTRACT

Various organometallic plastics such as polymethacrylates, polystyrenes, and polyesters were prepared by chemically incorporating biocidal organometallic compounds on polymeric backbones. Organometallic plastics will be used as coatings and structural plastics where antifouling protection is needed. The antifouling performance of these formulations was evaluated at NAVSHIPYD PEARL, Miami Beach, Florida, and this laboratory. An organometallic curing agent for epoxy resins was prepared. Organometallic polymethacrylates and polyvinyls, previously synthesized, have exhibited 100% antifouling performance against a variety of marine organisms after 15 months of exposure at NAVSHIPYD PEARL.

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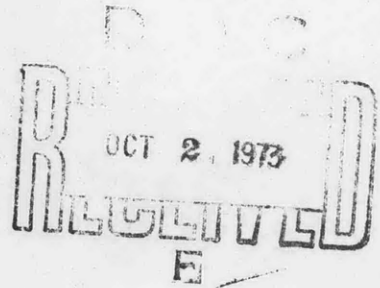
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DEPARTMENT OF THE NAVY  
NAVAL SHIP RESEARCH AND DEVELOPMENT CENTER  
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### ABSTRACT

Various organometallic plastics such as polymethacrylates, polystyrenes, and polyesters were prepared by chemically incorporating biocidal organometallic compounds on polymeric backbones. Organometallic plastics will be used as coatings and structural plastics where antifouling protection is needed. The antifouling performance of these formulations was evaluated at NAVSHIPYD PEARL, Miami Beach, Florida, and this laboratory. An organometallic curing agent for epoxy resins was prepared. Organometallic polymethacrylates and polyvinyls, previously synthesized, have exhibited 100% antifouling performance against a variety of marine organisms after 15 months of exposure at NAVSHIPYD PEARL.

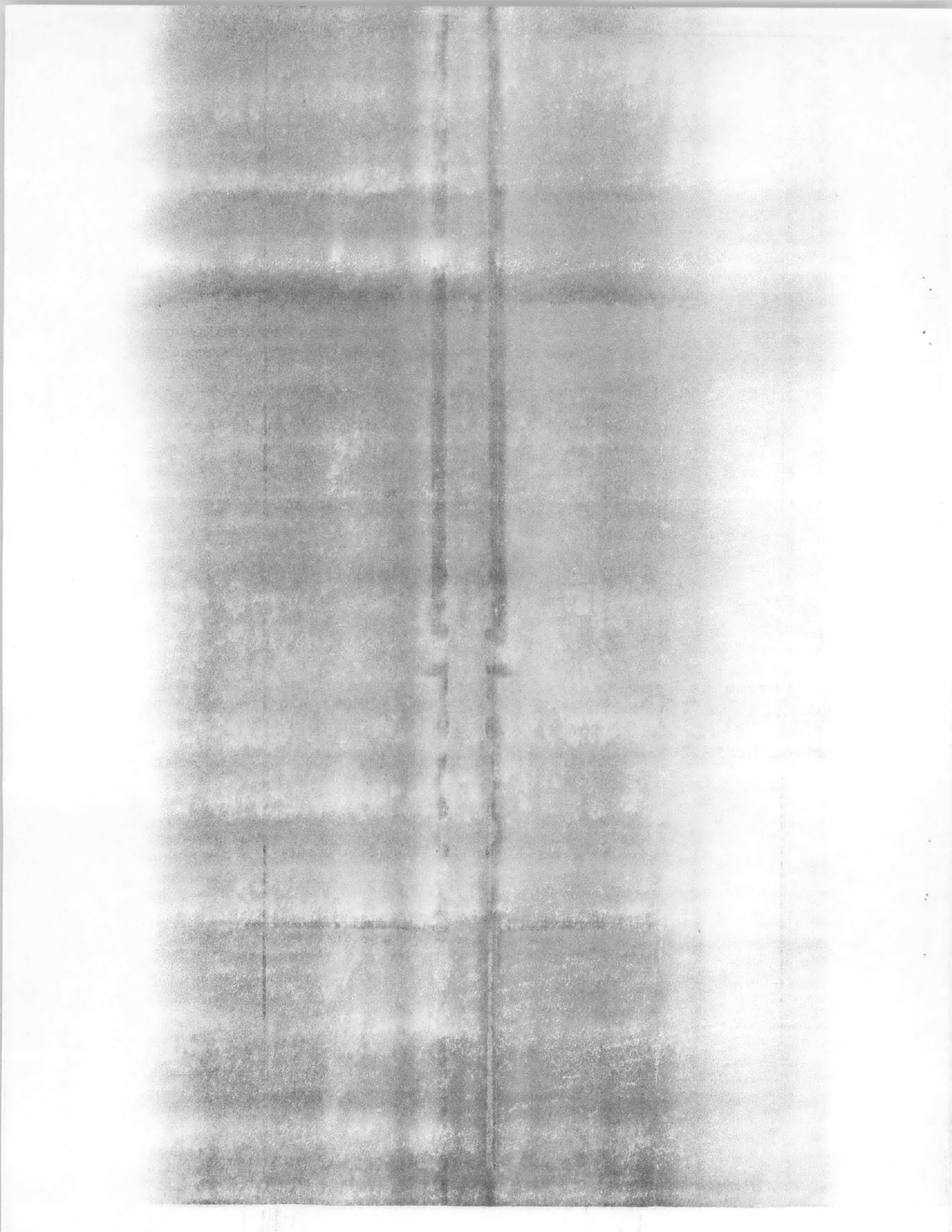
ADMINISTRATIVE INFORMATION

This work is authorized under Program Element 61152N, Task Area ZA614-120-01, Work Unit 1-2853-105 as described in the 1 May 1973 Program Summary.

All work described in this and previous reports is covered under a patent application.

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## INTRODUCTION

As part of the present Navy effort to produce long-lived, low-leaching antifouling coatings, organometallic polymers were developed.<sup>1</sup> It is estimated that these materials will extend the longevity of antifouling systems to 5 years. In addition, transparent, nonwetable, slimicidal films of the organometallic polymers can be used on underwater optical devices. A novel concept in antifouling technology, organometallic polymers are effective because of the chemical incorporation of organometallic biocidal groups, such as tributyltin and tripropyltin, on the polymer backbone. Due to water hydrolysis, these organometallic ions are released from the polymer backbone at a controlled rate at least one order of magnitude less than state-of-the-art Navy and commercial antifouling coatings. As a result, this chemical conservation of the biocidal organometallic agents will provide longer-term antifouling protection for submerged surfaces, while reducing the pollution hazard attributed to presently used antifouling coatings by a factor of ten. After 15 months of exposure at NAVSHIPYD PEARL and after 13 months at this laboratory, 10- by 12-inch panels coated with organometallic polymers have exhibited 100% antifouling effectiveness against bacteria, algae, barnacles, tubeworms, and other members of the fouling community.

Fouling of moored data systems and ship-and-shore facilities by marine organisms impedes Fleet operations and necessitates a large maintenance allocation.<sup>2, 3</sup> Buoys shift due to the excessive weight of fouling organisms.<sup>4</sup> Effective operation of sonars is hampered by the self-noise generated by the fouling of sonar dome surfaces. Wood pilings in berthing facilities undergo structural weakening due to marine borer and fungal attack.<sup>5</sup> The fouling of steel piping, and bronze couplings and fittings in the sea-water intake piping systems of ship-and-shore facilities, leads to reduced flow rates, valve seat damage, and accelerated metal corrosion.<sup>2</sup> Although presently used high-leaching, antifouling hull coatings may provide short-term antifouling protection (3 to 18 months of service life) they undergo premature failure due to poor adhesion, and poor abrasion and erosion resistance. Consequently, this development of organometallic resins as structural plastics for the fabrication of nonfouling buoys, glass reinforced plastic (GRP) sonar domes, hull coatings, sea-water piping systems, and for the treatment of wood and cement shore structures will provide longer periods of protection from fouling for these objects (figure 1) and eliminate costly maintenance.

<sup>1</sup>Superscripts refer to similarly numbered entries in the Technical References at the end of the text.

## MATERIALS AND METHODS

Various organometallic polymers were prepared. Organometallic monomers such as tributyltin methacrylate and tripropyltin methacrylate were copolymerized by solution polymerization with styrene or methyl methacrylate (table 1). The comonomers, styrene and methyl methacrylate, were uninhibited prior to polymerization by vacuum distillation and by neutralization with a weak base, respectively.<sup>6</sup> To control discoloration of the organometallic copolymers of styrene and tributyltin methacrylate, various anti-oxidants (2, 4-dinitrophenylhydrazine, 4-cyclohexylcyclohexanol, and Irganox 1076) and ultraviolet light absorbers (Uvinul 40, salicylaldehyde, and Tinuvin p) were added prior to polymerization.

**TABLE 1**  
**PREPARATION OF ORGANOMETALLIC POLYMETHACRYLATES AND STYRENE-TRIBUTYLTIN METHACRYLATE COPOLYMERS BY SOLUTION POLYMERIZATION**

Organometallic Polymer Synthesized		Reactants																																	
		Organo-metallic Monomers(1)	Comonomer(1)	Molar Ratio of Organo-metallic Monomers to Comonomer	Initiator	Additives	Solvent																												
Polymer Letter	Polymer Name																																		
S1	Poly (tributyltin methacrylate/styrene)	Tributyltin methacrylate (reference 7)	Styrene	1:3.5	Azo initiator(2)	-	Benzene																												
S2				↓		↓		↓	↓	↓																									
S3											↓	↓	↓	↓	↓																				
S4																↓	↓	↓	↓	↓															
S5																					↓	↓	↓	↓	↓										
S6																										↓	↓	↓	↓	↓					
S7																															↓	↓	↓	↓	↓
S8																																			
S9	Poly(styrene)	-	-	1:2	Benzoyl peroxide(4)	Irganox 1076, Tinuvin p(5)	Benzene																												
S10	Poly (tributyltin methacrylate)	Tributyltin methacrylate	-	↓	↓	↓	↓																												
S11	Poly (tributyltin methacrylate/styrene)	↓	Styrene	1:1																															
P50, P51	Poly (tributyltin methacrylate/tri-propyltin methacrylate/methyl methacrylate)	Tributyltin methacrylate, tripropyltin methacrylate	Methyl methacrylate	1:1:1	Benzoyl peroxide (0.5% by weight)	-	↓																												

(1) Preparation of monomers found in reference 7.  
(2) Azo initiator stands for 2, 2'-Azo-bis-(2-methylpropionitrile). The azo initiator was used 0.1% by weight in this reaction.  
(3) The azo initiator was used 0.1% by weight in this reaction.  
(4) The benzoyl peroxide was used 0.1% by weight in this reaction.  
(5) Irganox 1076 was used 0.15% by weight and Tinuvin P was used 0.25% by weight in this reaction.

The organometallic polymethacrylates and styrene-tributyltin methacrylate copolymers were painted on 10- by 12-inch Plexiglas panels and dried for 1 week at 80° C\* under vacuum. (This drying procedure can be altered for industrial use.) These panels were immersed at NAVSHIPYD PEARL, Miami Beach, Florida, and this laboratory to test their antifouling performance.

Unsaturated polyester resins of various polybasic acids and polyhydric alcohols were also synthesized (table 2).<sup>8,9</sup> The acid number of each resin was determined prior to curing.<sup>8</sup> These resins were cured with tributyltin methacrylate either by solution or bulk polymerization. In solution polymerization, tributyltin methacrylate, in a 1:1 molar ratio with the resin, and benzoyl peroxide, 0.5% by weight, were added while the resin was a warm fluid (between 70° and 80° C). The solution was heated for approximately 1 hour until the resin was a hard sticky mass not easily stirred. In bulk polymerization, the curing agent, tributyltin methacrylate, and the initiator, benzoyl peroxide, were added to the resin in the same proportions as above. After the initiator dissolved in the monomer at room temperature, the fluid was poured into a metal mold. The mold comprised two polished stainless-steel plates (treated with Dow Corning release agent XR43130) which were separated from each other by Teflon tubing. The mold was fastened by "C" clamps applied around the steel plates. After the mold had been filled, the ends of the tubing were folded over one another and additional "C" clamps were applied to seal the mold. The resin was then cured at 40° C for 14 hours followed by further curing at 80° C for 6 hours. Organometallic polyesters cured by solution polymerization were painted on 10- by 12-inch Plexiglas panels, dried for 1 week under heat and vacuum, and sent to the three test sites for antifouling exposure. Resins cured by bulk polymerization were primed with an adhesive and attached to test panels which were sent to NAVSHIPYD PEARL.

The preparation of organometallic curing agents for epoxy resins was attempted. Glycine, p-aminobenzoic acid, and 2-acetamidoacrylic acid were used because they contained active hydrogen groups needed to cure an epoxy resin, as well as carboxylic groups which could be esterified to form organometallic esters. The preparation of the tributyltin ester of each acid was based on the reaction of the carboxylic group with bis (tri-n-butyltin) oxide in a 2:1 molar ratio (table 3). The catalyst in each reaction was p-toulenesulfonic acid (used 0.5% by weight).

\*Abbreviations used in this text are from the GPO Style Manual, 1973, unless otherwise noted.

TABLE 2  
UNSATURATED POLYESTER RESINS PREPARED BY CONDENSATION REACTION

Polymer Letter*	Reactants			Solvents
	Polybasic Acids	Polyhydric Alcohols	Molar Ratio of Acids to Alcohol	
P52A <sup>8</sup> P53A <sup>8</sup>	Maleic anhydride	Ethylene glycol, Propylene glycol	1.25:0.35:1.0	Toluene, xylene
P54A <sup>10</sup>	Sebacic acid	2-butene-1,4-diol	1.0:1.0	Xylene
P56A	Maleic anhydride	D-Sorbitol		Dimethyl sulfoxide
P57A <sup>9</sup>	Maleic anhydride, phthalic anhydride	Propylene glycol	1.0:0.63:1.8	Toluene, xylene
P59A <sup>11</sup>	Maleic anhydride,		0.225:0.275:0.50	-
P61A <sup>11</sup>	azelaic acid		0.3:0.2:0.5	

\*Unsaturated polyester resin synthesized. Preparation of each polyester found in reference following the polymer letter. "A" after the polymer letter indicates the resin is uncured.

TABLE 3  
PREPARATION OF AN ORGANOMETALLIC CURING AGENT  
(CATALYST: p-TOLUENESULFONIC ACID, 0.5% BY WEIGHT)

Curing Agent	Ester Synthesis	Reactants			Reaction Temperature °C	Reaction Time, hr
		Acid	Organometallic	Solvent		
E1	Tributyltin glycinate	Glycine	Bistributyltin oxide	Ethanol: H <sub>2</sub> O (1:1)	78	22
E2	Tributyltin p-aminobenzoate	p-amino-benzoic acid	Bistributyltin oxide	Ethyl acetate	82	30
E3	Tributyltin ester of 2-acetamidoacrylic acid	2-acetamidoacrylic acid	Bistributyltin oxide	Dimethylformamide	80	12

### RESULTS AND DISCUSSION

Copolymers of styrene or methyl methacrylate and tributyltin methacrylate, and organometallic polyesters were successfully prepared. Copolymerization of tributyltin methacrylate and styrene resulted in film-forming polymers with slight discolorations (table 4).



TABLE 4  
 PHYSICAL CHARACTERISTICS OF ORGANOMETALLIC POLYMETHACRYLATES  
 AND STYRENE-TRIBUTYLTIN METHACRYLATE COPOLYMERS

Polymer Letter	Organometallic Polymer	Physical Appearance	Tin Content in Polymer,* %	
S1	↓	Poly (tributyltin methacrylate/styrene)	Transparent, orange-tinted film	12.31
S2		Opaque, yellow resin	21.46	
S3		Transparent, orange-tinted elastomer	19.62	
S4		Transparent, yellow-tinted film	16.64	
S5		Transparent, orange-tinted elastomer	20.90	
S6		Tacky, transparent, yellow elastomer	23.73	
S7		Transparent, orange-tinted elastomer	25.43	
S8		Transparent, yellow-tinted elastomer	20.46	
S10		Poly (tributyltin methacrylate)	↓	Transparent, water-white elastomer
S11	Poly (tributyltin methacrylate/styrene)	30.01		
P50	↓	Poly (tributyltin methacrylate/tripropyltin methacrylate/methyl methacrylate)	Translucent, water-white elastomer	22.71
P51		Transparent, yellow elastomer	29.35	
*Samples analyzed at Galbraith Laboratories by atomic absorption spectrophotometry.				

The discoloration of the styrene copolymers was eliminated by the addition of Irganox 1076, an antioxidant, and Tinuvin P, an ultraviolet light absorber. Unsaturated polyester resins cured with tributyltin methacrylate produced elastomers of moderate tin content (table 5).

TABLE 5  
PHYSICAL CHARACTERISTICS OF ORGANOMETALLIC POLYESTERS

Polymer Letter <sup>(1)</sup>	Acid Number <sup>(2)</sup>	Physical Appearance	Tin Content in Polymer, % <sup>(3)</sup>
P52	30	Transparent, yellow elastomer	24.11
P53	73	White granules	20.20
P54	81	Yellow opaque solid	21.11
P56 <sup>(4)</sup>	-	Brown solid	-
P57	91	White oily solid	18.62
P59	65	White waxy solid	15.39
P61	60	Yellow opaque solid	-

(1) The polymer letter without the "A" following it indicates the resin was cured.  
(2) Acid number was determined prior to curing the unsaturated polyester resin.  
(3) Samples analyzed at Galbraith Laboratories by atomic absorption spectrophotometry.  
(4) This acid number was not determined prior to curing.

Casting of organometallic polyester resins yield 1/8- to 3/8-inch-thick cross sections (figure 2), which are being tested for their antifouling performance. An organometallic curing agent, the tributyltin ester of p-aminobenzoic acid (E1) was synthesized. The ester was isolated as white, needle-like crystals from benzene with a melting point of 79° to 80° C and a tin content of 31%. Further analysis of E1 will confirm the structure of the ester. After a cure cycle of 2 hours at 80° C, followed by 2 hours at 150° C, E1 successfully cured Epon 1001, a high molecular weight epoxy resin.

The antifouling performance of the organometallic polymers was evaluated during field exposure at NAVSHIPYD PEARL, Miami Beach, Florida, and this laboratory. Exposed panels were rated for their antifouling performance based on the percentage of surface covered by foulants such as slime, tubeworms, barnacles,

and marine borers. After approximately 1 1/2 years of exposure, organometallic polymethacrylates (figure 3) and polyvinyls (figure 4) exhibit 100% antifouling performance (table 6). After several months of exposure, organometallic polystyrenes (figure 5) and polyesters (figure 6) have performed effectively against fouling organisms (table 6).

TABLE 6 - ANTIFOULING EFFECTIVENESS OF ORGANOMETALLIC POLYMERS

Polymer Letter	Polymer Name	NAVSHIPYD PEARL		Miami Beach Florida		NSRDC/A	
		AP %	Months Exposed	AP %	Months Exposed	AP %	Months Exposed
P43	Poly (tributyltin methacrylate/methyl methacrylate)	100	13				
Mix 1,*P29 & P34	Poly (tributyltin methacrylate) & poly (tripropyltin methacrylate)	100	15				
Mix 4, P36 & P38	Tributyltin & tripropyltin esters of poly (methyl vinyl ether/maleic acid)	100	15				
Mix 5, P37 & P39	Tributyltin & tripropyltin esters of poly (methyl vinyl ether/maleic acid)	100	15				
Mix 3, P20 & P25	Poly (tributyltin acrylate) & poly (tripropyltin acrylate)	80	15				
P42	Poly (tributyltin methacrylate/methyl methacrylate)	100	13				
P44	Poly (tributyltin methacrylate/1H,1H 7H-dodecafluoroheptyl acrylate)	90	13				
P36	Tributyltin ester of poly (methyl vinyl ether/maleic acid)	100	13				
P37	Tributyltin ester of poly (methyl vinyl ether/maleic acid)	100	13			100	10
P1	Poly (tributyltin acrylate)					100	10
P2	Poly (tripropyltin acrylate)					100	10
P4	Poly (tributyltin acrylate/tripropyltin acrylate)	100	10			100	10
P5	Poly (tripropyltin acrylate/tributyltin acrylate)					100	10
Mix 9, P4 & P28	Poly (tributyltin acrylate/tripropyltin acrylate) & poly (triphenyltin acrylate)					100	10
P29	Poly (tributyltin methacrylate)	100	16				
P20	Poly (tributyltin acrylate)	100	13			100	10
P28**	Poly (triphenyltin acrylate)					100	10
P30	Poly (tributyltin methacrylate/methyl methacrylate)					100	10
P41	Poly (tributyltin methacrylate)	100	13				
P25	Poly (tripropyltin acrylate)	100	13				
P45	Poly (tributyltin methacrylate/methyl methacrylate)	100	13				
P13	Poly (tributyltin methacrylate)	100	15				
Mix 10, P4 & P24	Poly (tributyltin acrylate/tripropyltin acrylate) & poly (tribenzyltin acrylate)	100	15				
Mix 11, P4, P24, P16 & P28	Poly (tributyltin acrylate/tripropyltin acrylate), poly (tribenzyltin acrylate), poly (trimethyltin acrylate) & poly (triphenyltin acrylate)	100	15				
S4	Poly (tributyltin methacrylate/styrene)			0	6		
S5	Poly (tributyltin methacrylate/styrene)	80	5	80	6		
S6	Poly (tributyltin methacrylate/styrene)	100	5	88	6		
S7***	Poly (tributyltin methacrylate/styrene)			93	2		
S8***	Poly (tributyltin methacrylate/styrene)			80	2		
S10***	Poly (tributyltin methacrylate/styrene)			100	1		
S11***	Poly (tributyltin methacrylate/styrene)			100	1		
P51***	Poly (tributyltin methacrylate/tripropyltin methacrylate/methyl methacrylate)			100	2		
P52***	Organometallic polyester			100	2		
P53***	Organometallic polyester			100	1		
P54***	Organometallic polyester			100	1		

\*A mix is a physical mixture of two or more organometallic polymers.  
 \*\*In a resin; rosin system (reference 1).  
 \*\*\*Data not back from NAVSHIPYD PEARL.  
 AP - Antifouling Performance.



## CONCLUSION

Organometallic polymers are effective antifouling materials for future application, not only as antifouling coatings but also as nonfouling marine structural plastics in the fabrication of small boat hulls, sonar domes, buoys, sea-water piping, and wood and concrete treatments.

## FUTURE WORK

Additional organometallic polyesters will be synthesized by incorporating the organometallic group into polybasic acids and polyhydric alcohols. The back-bone system of epoxy resins will be altered by chemically attaching organometallic groups to analogs of bisphenol A, and by preparing resins from the curing of tributyltin and glycidyl methacrylate. Further work will be done to prepare more organometallic curing agents for epoxy resins. The development of these organometallic polymers will be application oriented, and will include analysis of the structural properties of the resulting organometallic polymeric materials, as compared to the basic unmodified plastics.

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<b>BENEFITS:</b>	
• ANTIFOULING	• LOW POLLUTION RISK      • OPTICALLY CLEAR
<b>APPLICATIONS:</b>	<b>COATINGS</b>
<u>STRUCTURAL PLASTICS</u>	
<b>USES:</b>	<b>ANTIFOULING PAINT RESINS</b>
• OPTICS — POLYACRYLICS	<b>GEL COATS</b>
• CABLES — POLYALKANES	<b>ADDITIVES</b>
• BUOYS — POLYURETHANES	
• GLASS REINFORCED PLASTICS — POLYESTER	
• HIGH BUOYANCY MATERIALS — EPOXY	

Figure 1

Long-Range Development of Organometallic Polymers  
for Navy Applications

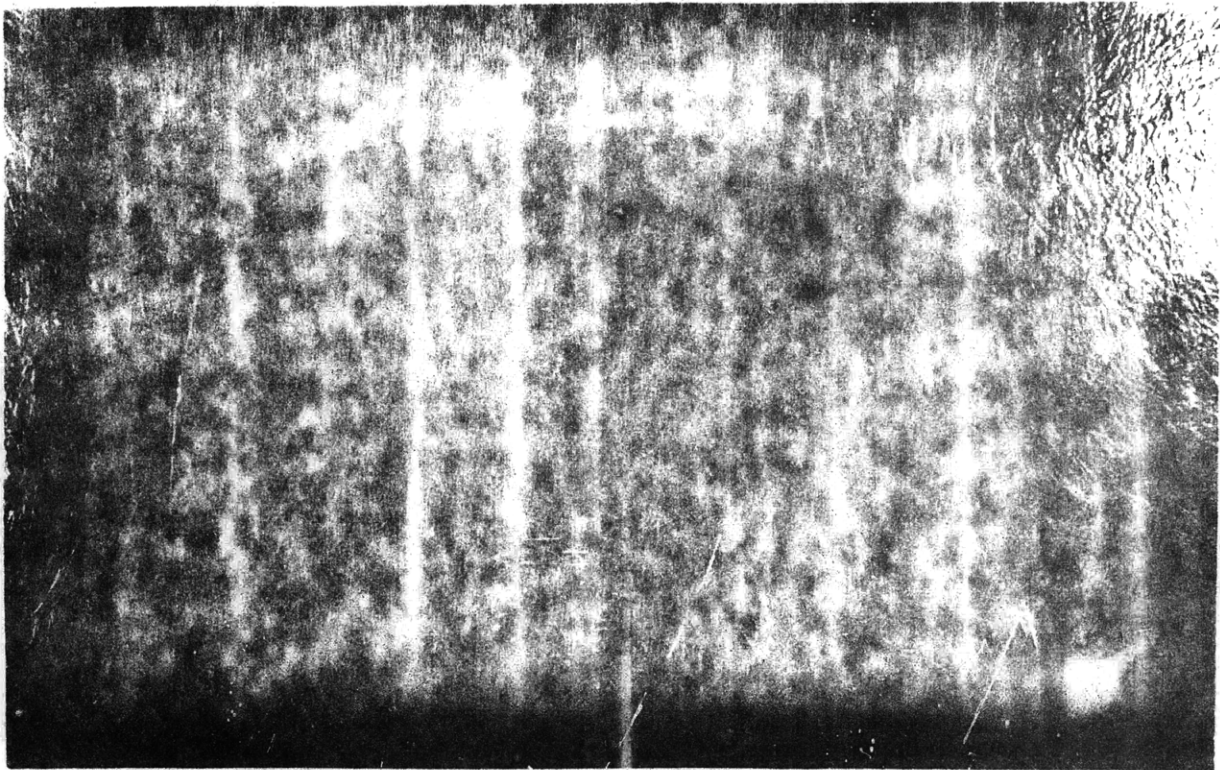
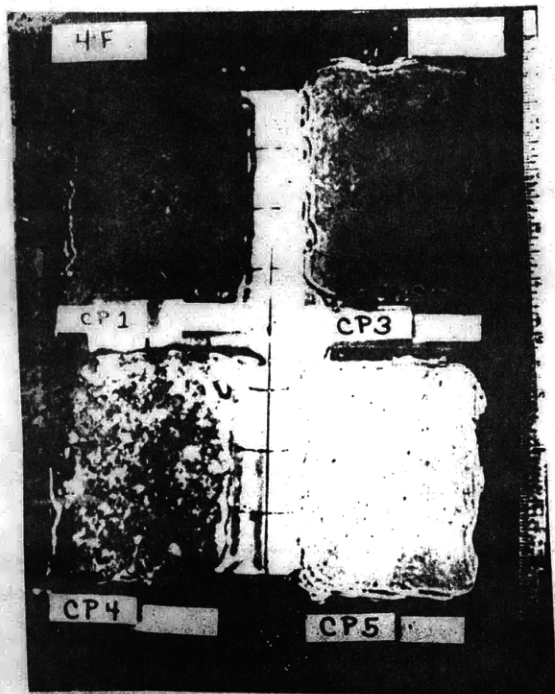


Figure 2

Cast Specimen of Organometallic Polyester



Item (a)  
Before



Item (b)  
After

Figure 3  
Fouling resistance of Oxidometallic Polymethacrylates Before  
and After Exposure for 15 Months at NAVSHEP, PRAGUE



Item (a)  
Before

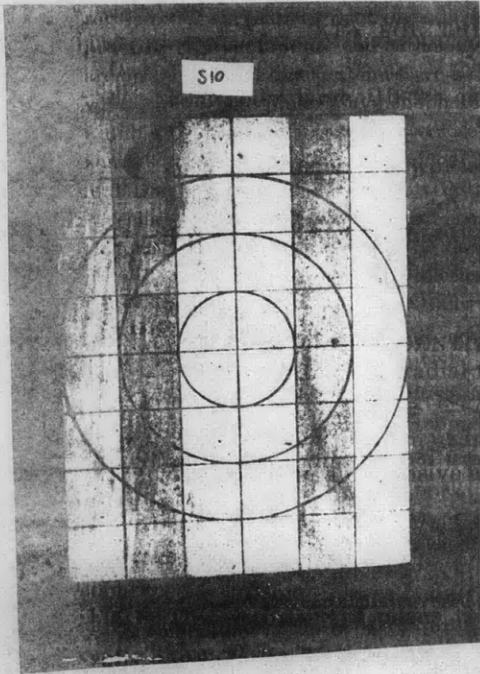


Item (b)  
After

Figure 4  
Fouling Resistance of Organometallic Polyvinyls Before  
and After Exposure for 13 Months at NAVSHIPYD PEARL

28-734





Item (a)  
Before

Item (b)  
After

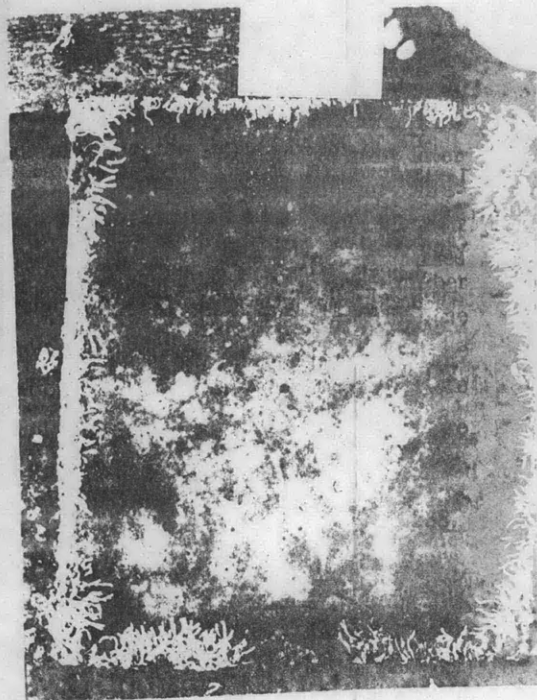
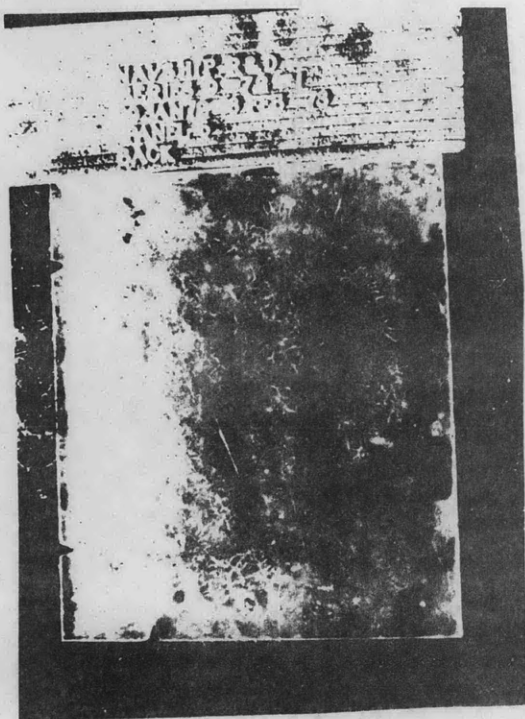


Figure 5  
Fouling Resistance of Organometallic Poly (Styrene/Methacrylate)  
Before and After Exposure for 5 Months at NAVSHEP, PEARL.



Item (a)  
Before



Item (b)  
After

Figure 6  
Fouling Resistance of Organometallic Polyester Before  
and After Exposure for 2 Months at Miami Beach, Florida

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