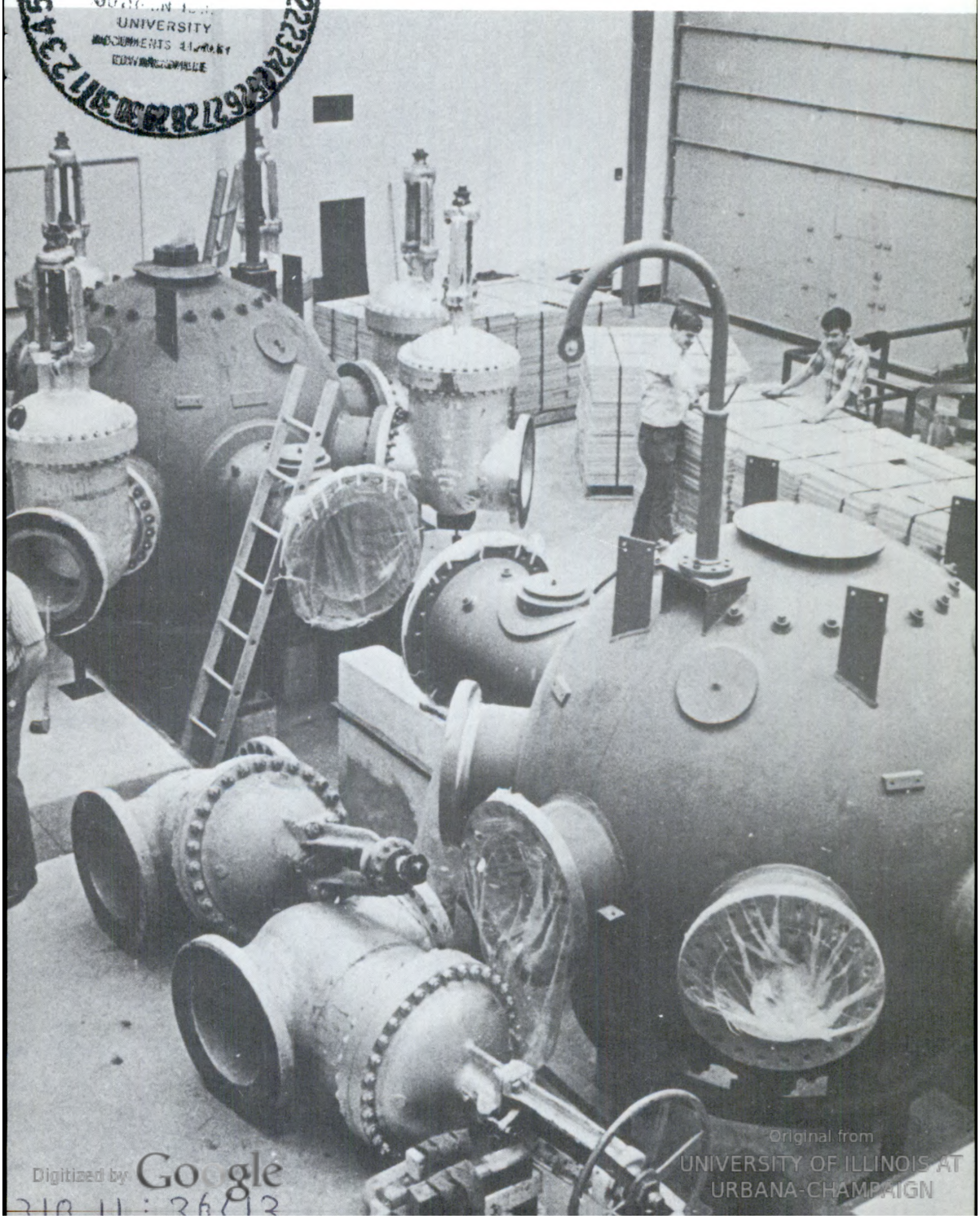
NAVAL RESEARCH

REVIEWS

DECEMBER 1973



PROFILES IN SCIENCE

Professor Herman P. Schwan

Professor Herman P. Schwan has been supported by ONR for the past 20 years. His work for ONR is concerned with the effects of microwaves on biological systems, including tissues, biological macromolecules and man. He is primarily responsible for the standards of safe microwave exposure which are presently in use in many Western countries. His ONR supported work includes: determinations of electrical tissue properties at microwave frequencies, mode of propagation of microwaves through complex tissue structures, relative absorption cross section of man and animals, study of "hot spots" generated by microwaves in brain tissues, theoretical and experimental work on the forces which alternating fields may apply to macromolecules and cells.

Since 1950 Dr. Schwan has been at the University of Pennsylvania as Professor and now Chair man of the Biomedical Electronic Engineering Department.

DDT: An Anomalously Resistant Molecule

F. W. Juengst and M. Alexander*

The Navy shares the concern of all socially responsible institutions for the protection of the environment. The research described here is part of a joint ONR and Naval Facilities Engineering Command effort in pollution abatement and control. This particular effort contributes to our better understanding the mechanisms of biodegradation through an examination of long-lasting materials. Of particular interest, is the popular pesticide DDT which has substantial economic, social, and health benefit, but whose use is being restricted because of its "polluting" effects. The research is expected to add to our understanding of the biodegradation of DDT and provide information which will be useful in promoting biodegradation of other specific substances.

An enormous number of organic chemicals and natural products enter marine waters. Some are discharged directly into the oceans, some are carried by river water or are bound to particulate matter transported by rivers entering the sea, and some originate from the atmosphere. Many of these substances pose no hazard to marine life because they are subject to rapid biodegradation by bacteria, fungi, or other microorganisms, the end result of the decomposition being the complete conversion of the substance to carbon dioxide, water, and other inorganic products. Owing to the enormous catabolic versatility of the marine microflora, few of the incoming chemicals endure for sufficiently long to create a long- or even a short-term hazard.

A few compounds are highly persistent, however. Inasmuch as the total destruction of complex organic molecules in nature results largely, if not solely, from the actions of microorganisms, the longevity of such substances must be attributable to the inability of these microscopic organisms to degrade them. Among the compounds, polluting agents, and natural products that are thus long-lived are plastics of many types, polychlorinated biphenyls (PCBs), a wide array of natural products characteristic of sediments, and the insecticide DDT (Figure 1). The resistance of biodegradation of many plastics is a useful trait because they do not rot, spoil, or undergo changes that might affect their intended use, yet this very resistance may be offensive because of the unsightly

*Dr. Juengst is Research Associate and Dr. Alexander is Professor at Cornell University, Ithaca, New York. Their major fields of interest are environmental microbiology and establishing how microorganisms modify chemicals of environmental importance.

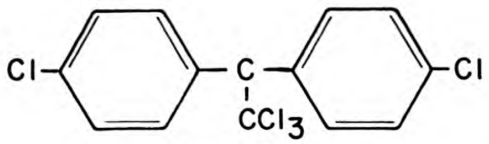


Figure 1 — Chemical formula for DDT

remains. Plastics may create other problems, such as the possible effects of small plastic particles on fish.

A few of these persistent chemicals may be hazardous to some forms of life. Evidence is accumulating, for example, that PCBs are affecting a number of different species, which fact when coupled with the ever growing reliance of society on chemicals result in an environmental problem of considerable and widespread importance. Like the PCBs, DDT is affecting a number of species, and serious question has been raised in many corners of the globe whether this effective and useful insecticide ought to be banned completely on a world-wide basis.

As a pesticide, the phenomenal value of DDT cannot be questioned. Because of its toxicity to *Anopheles* and other mosquitoes, it has saved uncounted human lives and has led to the control of malaria in many countries in the tropics. As a result, it has been said that no chemical ever found or synthesized by man has alleviated as much suffering or been as great a boon to public health as DDT. The insecticide also has been an undisputed blessing to agriculture because of its effectiveness and low cost. Yet its apparent resistance to biological degradation and its concentration through food chains (microorganisms → macroorganisms → small animals or fish → large animals or fish) have been associated with a dramatic decline in the populations of a few animal species, some of which are of economic or esthetic significance or are important species in their natural habitats.

Although the use of DDT has been terminated in the United States, the total world production remains about 150 million pounds per annum (Woodwell *et al.*, 1971). Much of the DDT that is used abroad is designed for the control of insects carrying the causative agents of communicable diseases of humans, such as malaria, or for pests that damage crops essential for countries whose populations are still under- or malnourished. A total cessation in the use of DDT in such nations, in the absence of an effective replacement that can be afforded by these poor countries, would lead to a marked increase in the incidence of human diseases transmitted by susceptible insect vectors and a decline in agricultural production in regions already lacking an adequate food supply.

Not all of the DDT sprayed onto crops and in forests remains on the plants. Some of the applied pesticide falls onto the soil and remains there probably for decades, some is transported with particles of eroding soil into rivers and streams, and a significant portion is volatilized and enters the atmosphere. The DDT, because it is not destroyed readily,

is carried to the ocean with the flow of river water or enters from atmospheric sources.

According to the estimates of Woodwell *et al.* (1971), the total amount accumulated in the marine biota is about 12 million pounds, most associated with algae and higher plants. Sedimentation of organic matter, to which DDT is bound, transports the insecticide or its metabolites to the abyss, where the insecticide may accumulate because the indigenous bacteria exhibit only limited degradative activity (Jannasch *et al.*, 1971). Furthermore, rates of transfer of organic compounds out of the abyss are of the order of hundreds to thousands of years (Woodwell *et al.*, 1971).

Given the large number of reports documenting the slow destruction of DDT in natural habitats, it seemed reasonable to conclude that DDT is not readily attacked microbiologically or that it is not even a substrate for enzymes of bacteria or fungi common in nature. Is it not perfectly plasuible to believe that an extremely persistent molecule owes its longevity to the shortage of organisms containing the enzymes requisite for its breakdown or that no such organisms exist? The conclusion, however, is invalid, and numerous species capable of effecting steps in DDT metabolism have been obtained. Thus, many marine microorganisms have been found that cause some modification in the molecule, modest though the alteration is (Patil *et al.*, 1972).

The results of an experiment designed to show the frequency of aquatic bacteria able to bring about a modification of DDT are shown in Figure 2. The samples were taken from (1) brackish water from the mouth of the Connecticut River at Saybrook, Conn., (2) a salt marsh at Saybrook, (3) and (4) subtidal zone one and two miles, respectively, from the mouth of the Connecticut River in about 2 ft. of water, (5) brackish water from Oyster Creek at Saybrook, (6) subtidal zone at Kelsey Point Beach, Conn. in about 2 ft. of water (fine black sand), and (7) subtidal zone at Sagamore Terrace Beach, Conn. in about 2 ft. of water (course gravel). An alteration in the DDT in these tests is taken as the formation of water-soluble products from the DDT, a compound that is itself highly water-insoluble. The bacteria were chosen at random to obtain a reasonable approximation of their relative abundance in the test samples. The results demonstrate that from less than one-fourth to two-thirds or more of the bacteria from each sample site can convert from 5 to 10% of the DDT, provided to the cultures as the ^{14}C-labelled (radioactively tagged) compound, to water-soluble metabolites. Pfaender and Alexander (1973) found that as many as 90% of the bacteria from polluted water amended with a readily available carbon source could metabolize DDT.

Nevertheless, extensive decomposition of DDT can be brought about by the activities of two bacteria, an action that not only destroys

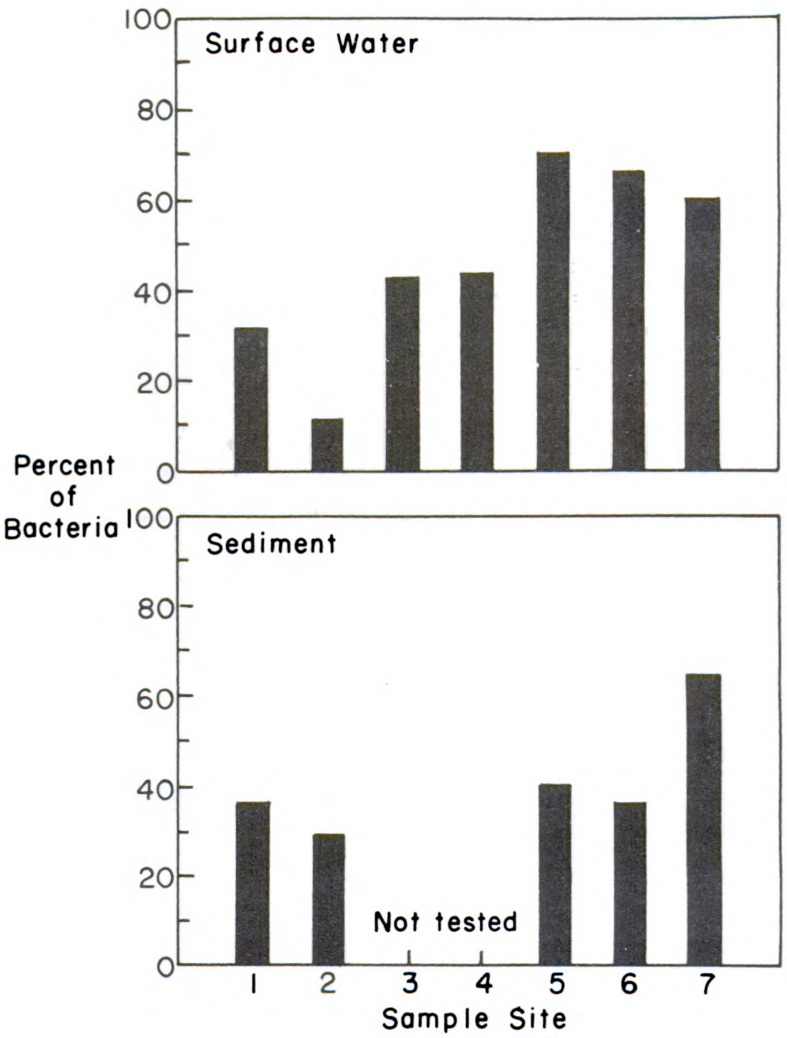


Figure 2 — Graph showing the results of an experiment designed to show the frequency of aquatic bacteria able to bring about a modification of DDT.

the molecule but one that takes place quite rapidly, at least under laboratory conditions. This process, as described by Focht and Alexander (1970a) and Pfaender and Alexander (1972), apparently involves a series of reactions in which changes are brought about in the substituents on one of the carbon atoms lying between the two benzene rings. That carbon atom is then removed from the molecule, and this removal is followed by a cleavage of one of the two benzene rings. The product is *p*-chlorophenylacetic acid (R-CH_2-COOH). This metabolite, in turn, is then degraded by another type of bacteria, yielding thereby a rapid and exhaustive *in vitro* degradation (Figure 3).

The behavior of DDT in natural waters is thus anomalous. On the one hand, it unquestionably fails to succumb rapidly to microbial destruction in the oceans. That is the reason that the insecticide retains its

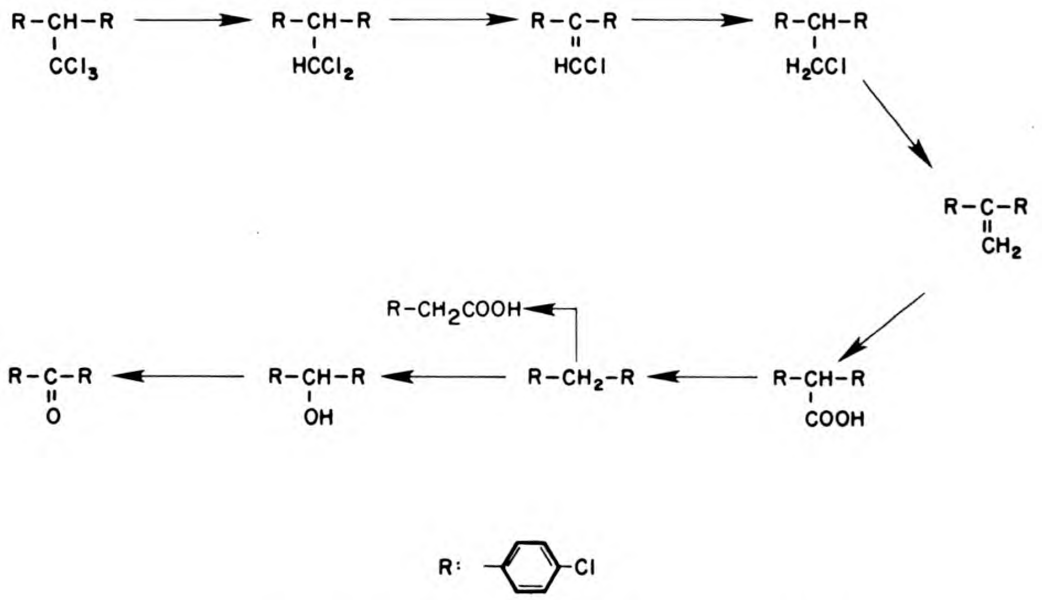


Figure 3 — Initial steps in the microbial degradation of DDT. R is p-*chlorophenyl.*

integrity sufficiently long to appear in cells and tissues of diverse species. On the other hand, the molecule is in fact extensively metabolized by microorganisms under artificial conditions in the laboratory. What is the explanation for this anomaly? That is one of the questions whose answer is currently being sought.

A number of reasons have been proposed recently for the persistence in nature of synthetic compounds and natural products (Alexander, 1972, 1973). Some of these hypotheses have a direct bearing on the persistence of DDT. One possible explanation for its longevity is that the bacteria capable of degrading DDT are physically separated from it. Because the solubility of the insecticide in water is about 0.002 ppm and its solubility in certain lipids is about 100,000 ppm (Reinbold *et al.*, 1972), a large percentage of the DDT may be concentrated in the lipids of living organisms. DDT is known to accumulate in algae, clams, oysters, fish, and other organisms. DDT in lipids or fatty tissues and cells would be inaccessible to microorganisms. In addition, dead algae and other non-living organic material can bind DDT. Bacteria are also capable of binding DDT, as shown by Hicks and Corner (1973). They presented evidence that *Bacillus megaterium* retained about 1.7 μg of DDT/mg cells. About 75% of this DDT was found to be in the bacterial membrane, where a large portion of the cell lipid is located. It is not known whether insecticide bound to bacteria or inanimate substances is less readily available than the unbound chemical, but studies with other compounds have shown that bound, complexed, or sorbed substrates are generally not readily available for decomposition.

Another reason for the persistence of DDT may be that individual microbial populations in the ocean inhibit the species having the capacity to decompose DDT. This hypothesis is supported by a report that DDT decomposition by the fungus *Mucor alternans* is inhibited by other fungi (Anderson and Lichtenstein, 1972). The neighboring species may markedly retard the growth of organisms having the appropriate enzymes or suppress enzymatic activity. Alternatively, inasmuch as one or more of the enzymes may be inducible, conditions in nature inimical to enzyme induction, the absence of an inducer, or a concentration of inducer too low to allow for the appearance of the needed enzymes might explain why we have found that a high percentage of the isolated bacteria have the ability to convert DDT to water-soluble products *in vitro* but yet we have not found any water-soluble products in model communities of marine microorganisms.

Not only is it important to establish why DDT and some of its closely related metabolites are so persistent but it is also essential to identify the products that are generated. These products may be more persistent, more toxic, affect different species, or have different mobilities in nature than the parent molecule. The need for such studies is illustrated by the results of several investigations. For example, 2,4-dichlorophenoxyacetic acid (2,4-D) is metabolized by microorganisms in natural waters, but in the process the pesticide is converted to a product far more resistant to biodegradation and hence more persistent than the original 2,4-D (K. W. Sharpee and M. Alexander, unpublished data). A change in spectrum of toxicity as a compound is transformed is evident in a report that the pesticide thiram is converted to dimethylnitrosamine, a potential human carcinogen, in laboratory models of natural ecosystems (Ayanaba and Alexander, 1973). Thus, establishing the pathway of breakdown and characterizing the products that appear in water assume considerable significance.

It is known that DDT can be converted by bacteria to 4,4′-dichlorodiphenylmethane (Focht and Alexander, 1971; Pfaender and Alexander, 1973). If this compound is indeed produced in nature, removal of the chlorine atoms on the rings would produce diphenylmethane, which can be essentially totally degraded by a number of bacteria (Focht and Alexander, 1970b). Enzymes catalyzing the removal of a chlorine atom from organic compounds are called dehalogenases. Nonspecific dehalogenases might be involved in this conversion; however, dehalogenases appear to be highly specific and generally do not act upon even closely related molecules (Goldman, 1972).

In our search for products of DDT metabolism, the isolation of which may shed light on the pathway of degradation, it was found that *Mucor alternans* produced at least two water-soluble metabolites from DDT. Although these products have not been fully characterized, it is known

that they are not common or postulated DDT metabolites like 4,4′-dichlorodiphenylacetic acid, *p*-chlorophenylacetic acid, or 2-chlorosuccinic acid. It is likely that these water-soluble metabolites, which seem to be organic acids, are probably previously uncharacterized breakdown products. The identities of the compounds are currently being sought.

The presence of a chlorine atom on each of the two benzene rings of DDT may present a major obstacle to functioning of the enzymes which commonly cleave aromatic rings. Dagley (1971) presented evidence that *ortho*-cleaving enzymes (enzymes rupturing the benzene ring between two adjacent hydroxyl groups) are not generally active on rings with substituents, such as chlorine, *etc.*, on them. A typical *ortho*-cleavage sequence would likely produce 2-chlorosuccinic acid or some related compound (Figure 4). The presence of this compound in culture fluids of *M. alternans* was sought, but none was found. The enzymes involved in *meta*-cleavage are generally more active than *ortho*-cleaving enzymes on rings with substituents on them. A *meta*-type of ring cleavage would probably yield a 3-chloro-substituted acid from DDT (Figure 4), as suggested by Focht (1972). The formation of 3-chloro-substituted acids from DDT seems to be plausible, and a search for them in cultures of *M. alternans* and of marine bacteria will be made.

Therefore, despite the long continued applications of DDT, little is known about why it persists and how it is destroyed in marine ecosystems. Furthermore, although DDT use has been terminated or will

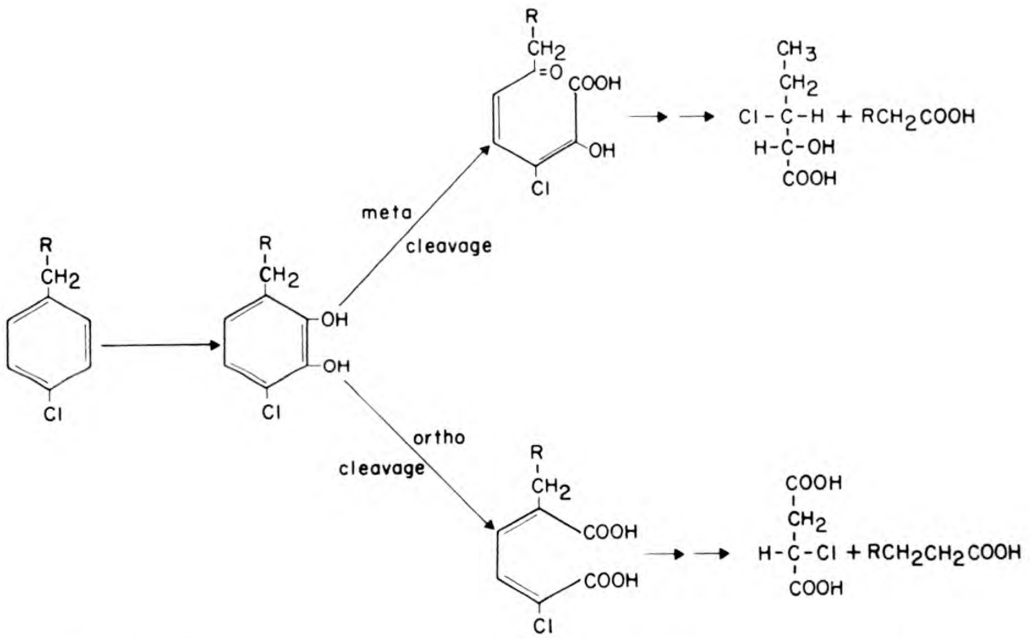


Figure 4 — Postulated pathway for the cleavage of the first benzene ring of DDT. R is p*-chlorophenyl.*

soon be markedly curtailed in the technologically advanced countries, it will endure for many years, and additional amounts of the insecticide will probably be introduced into the oceans owing to its usefulness in public health activities and agricultural operations in the tropics. For these reasons and because the possible hazards of products of DDT transformation in nature only have been partially established, further research is needed to define more adequately the fate and pathway of breakdown of this global and persistent pollutant. In addition, since it is now evident that DDT is in fact biodegradable and not almost wholly resistant to microbial attack, as heretofore believed, it is important to account for its anomalous longevity in nature so that possible means for its destruction or alternative but nonpolluting insecticides may be found.

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Drug Used to Treat Alcoholism May Allow Divers Greater Use of Oxygen

A drug presently used to treat alcoholism holds promise for allowing deep divers to breathe oxygen to a greater extent than is now possible.

The drug is disulfiram, also known as Antabuse. A research program at the University of Kansas directed by Dr. Morris Faiman under an Office of Naval Research contract funded by the Navy's Bureau of Medicine and Surgery has found that the drug provides substantial protection for certain animals against the toxic effects of breathing excess oxygen for extended periods.

A major advantage of oxygen to divers is in reducing the extended decompression time, involving many days or even weeks, that is necessary when ascending from a deep saturation dive. Inert gases, such as helium, that are contained in the breathing gas mixture and accumulate in the tissues of the body must be removed slowly to accomplish decompression safely. This process can be considerably speeded up by breathing oxygen to rapidly replace the inert gas.

Unfortunately, oxygen can have its own adverse effect on the tissues of the body if it is breathed at partial pressures greater than half an atmosphere for more than limited periods. The central nervous system or the lungs, for example, might be affected, depending on the condition of exposure. This may range from temporary effects, such as restriction of vision or pain in breathing, to convulsions or fluid-filled lungs (edema) that may cause death.

At the University of Kansas, when animals were injected with disulfiram before exposure to oxygen, there were significant delays in the times of onset of both convulsions and lung damage. The degree of protection varied in different species and strains of animals.

Further studies are now underway to determine the extent to which protection is complete at all levels in animals and whether the same protection would be provided to humans. The doses of disulfiram given to alcoholics are much lower than those which have been found to protect animals against oxygen toxicity. Studies are also underway to clarify how disulfiram is metabolized in the body, whether the large quantities which afford protection may have toxic effects, and what component or components of the drug provide the protective action.

There is one obvious side effect, which may or may not be desirable. Since the use of disulfiram causes extreme nausea in anyone imbibing alcohol, divers would probably have to avoid alcoholic beverages for about a week after treatment.

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Man-in-the-Sea Project in North Dakota

"There is no other laboratory like this elsewhere in the world that can do the type of research that will be conducted here. This is an automated laboratory in which a computer precisely regulates the atmosphere, including temperature, humidity and pressure as well as recording a variety of physiological data. The animals can be placed in an environment similar to the ocean depths as deep as approximately 1300 feet for days, months or years, with food provided and waste removed by remote control."

The above remarks are from the address made by the Chief of Naval Research, RADM Van Orden at the dedication of the High Pressure Life Laboratory, University of North Dakota, Grand Forks, North Dakota, November 30, 1973 (Figure 1).

Figure 1 – RADM Van Orden examining some experiment results with Dr. Thomas K. Akers, Project Manager of the Man-in-the-Sea Project

The Project

Research into the effects of undersea submersion at depths to 1,300 feet is the prime aim of the Man-in-the-Sea Project at the University of North Dakota. It is one of the largest and most advanced research projects of its kind and has the only high pressure life laboratory in the free world capable of studies of the long-term effects of high pressure on the reproduction, nutritional needs and general health of test animals.

Planning and experimentation for the Man-in-the-Sea Project have been under way since 1968. The research project already has produced nearly 80 scholarly papers, dissertations and theses. It is funded by the U.S. Office of Naval Research, and is staffed by University faculty and student researchers from several academic disciplines.

Man-in-the-Sea research deals with a broad spectrum of problems associated with life under high pressure and has wide-ranging possibilities for application. Knowledge gained from the research will be important in developing ways for man to live under the sea for prolonged periods to recover the food and energy resources found there.

The research also promises to aid medicine by explaining the reaction of the human body to various gases, bacteria, drugs and other substances under pressure. Further application of knowledge derived from the project extends to military and defense needs of the country.

Some areas for expanding research that will result from the Man-in-the-Sea Project were mentioned by RADM Van Orden.

> "We must remember that the ocean is an essential and major part of the world in which we reside. For the Navy it is the environment in which we operate constantly, and we must understand it fully to benefit from the advantages it offers and, at the same time, protect our men from its dangers. Aside from its military use, the ocean offers answers to some of man's most serious problems. Off-shore oil could certainly help in reducing our oil shortages. Some wells are being drilled as deep as 350 feet. Valuable minerals, such as manganese, cobalt and nickel, can be taken from the ocean floor. Even diamonds can be dredged from the ocean bottom and have been for several years off the coast of Africa. Most important of all, the 60 million tons of fish and shellfish now obtained from the sea each year world-wide could be increased to one to two billion tons per year if instead of hunters we became harvesters of the sea through fish farming. Japan already has had some success in doing this."
>
> "Before we can begin to experiment with man himself, however, several biomedical questions must be answered in an animal laboratory such as this one. What is the effect of

breathing inert gases over such long periods of time? Should we continue to use helium or should we switch to the only lighter gas hydrogen? What are the effects of living so long in remote isolation under constant stress? What type of food is needed to provide adequate nutrition?"

The major component of the High Pressure Life Laboratory, measures 40 feet long, 20 feet wide and nine feet high and consists of two seven-foot spheres joined by a passageway with a 24-inch gate valve. Each sphere is surrounded by seven 18-inch animal living chambers separated from the main sphere by 18-inch gate values. To facilitate prolonged study under constant pressure, animals can be transferred to the spheres while the living chambers are decompressed, cleaned and resupplied (Figure 2).

Construction of the test facility became possible when the Office of Naval Research made available as Government Furnished Equipment some 15 pieces of machine tools and welding equipment. The ability of the University to machine and fabricate in-house almost all major components resulted in a laboratory that would cost several million dollars more if constructed by industry. In addition, items of electronic

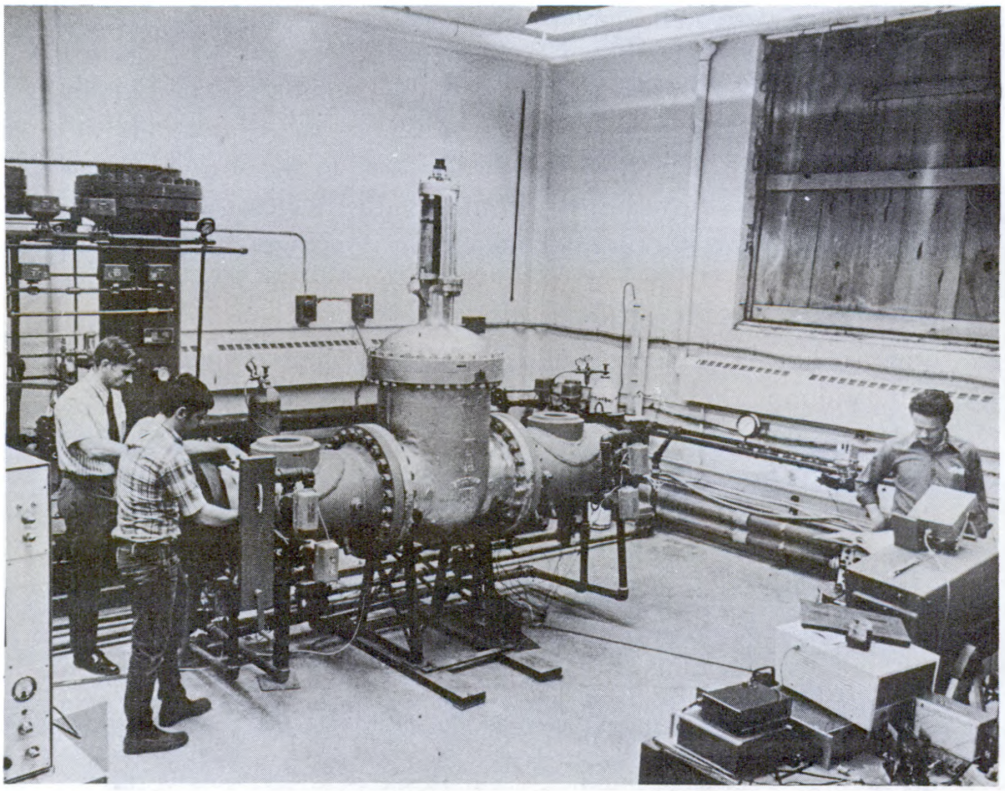

Figure 2 — Fabricating one of the animal living chambers at the machine shop

systems and laboratory instruments obtained from Government excess have greatly facilitated the research program.

Pressures of up to 40 times that experienced on the earth's surface or a depth of 1300 feet can be maintained in the chamber complex. Research calling for exposure to high pressure for prolonged periods of up to two years of more is planned (Figure 3).

The History

In 1967, the Department of Defense initiated Project Themis as a means of developing centers of excellence in universities throughout the country. The University of North Dakota recognized that, following the Navy SEALAB experiments, there were still problems to be solved before man could live at deep depths for very long periods, and the solution to these problems had to start with animal experiments. They formed a team that created a successful proposal that brought about the establishment of the hyperbaric center. Thus began the long collaborative efforts between The University of North Dakota and the Office of Naval Research to develop the High Pressure Life Laboratory.

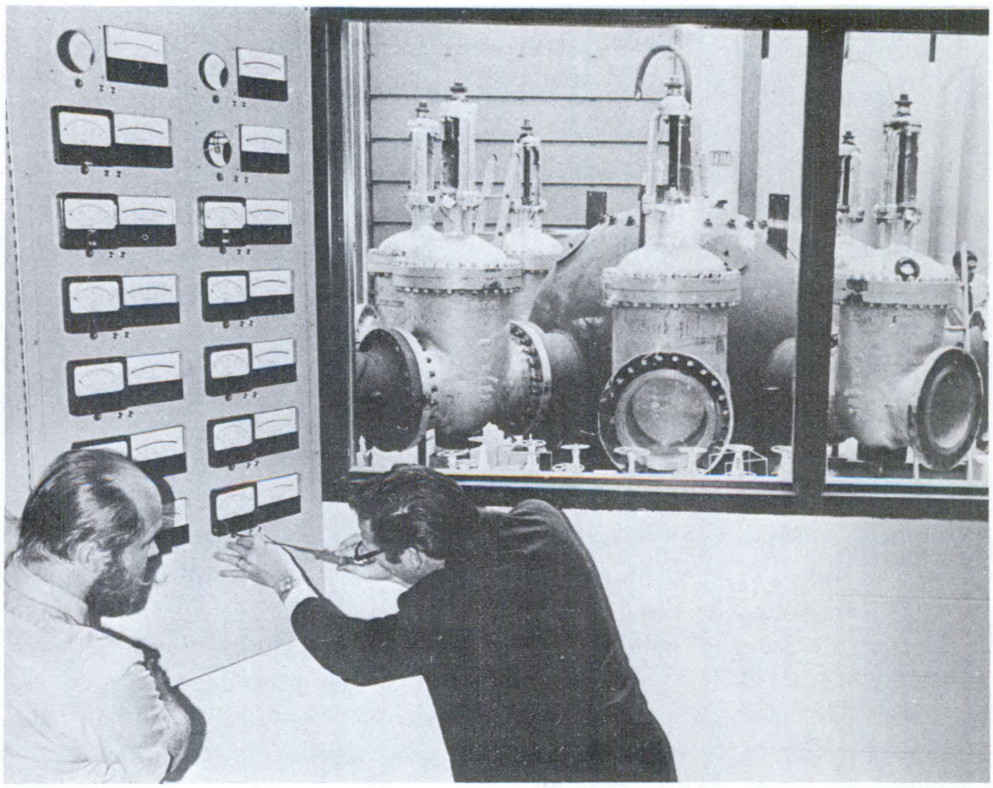

Figure 3 — The computer room looking into the main chamber

During the five years of building the facility, the researchers were not idle. Using the first chambers, the physiologists carried on research in various areas of high pressure life support, and during this time many graduate students received high pressure training while pursuing their advanced degrees. Research studies have been completed in a variety of areas. The toxic effects of various combinations of gases have been studied on rats, mice, guinea pigs and chinchillas, and the effects of pressure stress have been assessed on rats. A series of studies was completed, examining the stressors and their effects during decompression upon brain wave patterns, eye movements induced by vertigo and the learning behavior of small rodents.

The nutrition of animals kept in the bizarre environment found in hyperbaric work has been assessed through a long series of nutrition experiments, and the most appropriate temperature to bring the animals to thermal neutrality in these bizarre gas mixtures has been established. Studies of the microbiological changes that take place in this type of environment have begun. Several pharmacological studies have been carried out and are continuing, using the small chambers that eventually were incorporated in the main facility. Obviously, the majority of this work was done on a short-term (one-to-three day) experimental basis, looking forward to the day when animals can be placed in the hyperbaric chambers, to be maintained from six weeks to two years at pressure.

In concluding his address RADM Van Orden mentioned the long standing partnership between ONR and the universities of the Country.

> "When the Office of Naval Research was established in 1946, it was the first permanent Federal agency designed to support university research. A firm partnership between ONR and the universities was established because the Navy recognized, as it still does today, that we rely on the campus research community to create the new ideas necessary to keep our Navy modern and effective."

FLIP Cruise on Barstur Range

FLIP has just completed a cruise on the BARSTUR Range. Among the objectives of the cruise that were met were studies applicable to fluctuations of sound energy. Explosive shots were fired at various ranges from FLIP. The received energy was detected by hydrophones on FLIP. The variation in the direction of the received energy between the surface duct and bottom bounce paths were measured. The purpose is to determine how much the bearing/deflection values of sonars, depending on the bottom bounce path, may be off from the actual position of the target. Not only did this experiment provide the important information described above, but assistance was provided to the Range for their calibration requirements.

Fluorinated Network Polymers

J. R. Griffith and D. E. Field
Naval Research Laboratory

From fluorinated precursors we have synthesized polymeric materials composed of network molecules such as epoxies and polyurethanes. These tough, chemically resistant substances are, in effect, a new class of materials, inasmuch as the presence of large amounts of fluorine confers properties not commonly found in such polymers. Some applications in coatings are developing, and a broad range of potential applications are in prospect, including the construction of major hardware items of fiber-reinforced plastics or structural plastics.

Introduction

The Navy has used structural plastics and fiber-reinforced plastics in many relatively small applications and in a few large ones such as the Polaris motor case. The possibility of using such materials on a grand scale for the fabrication of large items—hulls and superstructures—has been an R&D consideration for many years (1). The magnitude of a change from metal to plastic construction entails problems too critical to assess except on the basis of experience; and the developing experience has been slow, except for certain specialized applications in which the strength-to-weight ratio of plastic materials has been an overwhelming consideration. Factors such as structural failure due to vibration or buffeting by ocean waves, damage by marine organisms, and fire at sea must be considered with respect to new materials of construction.

One of our programs in the Chemistry Division is an effort to improve polymeric materials with respect to those factors which would make them more suitable for naval applications, particularly, strength and toughness (2), water resistance (3), and nonflammability. Our attention has centered upon epoxides, or epoxies, because of the ease with which they can be fabricated into high-quality, high-strength products; and our recent concern has been to impose a fluorocarbon nature upon liquid epoxy resins through organic synthesis (4). Our intent is to combine as many properties of epoxy resins and polytetrafluoroethylene (PTFE) in one molecular species as possible in the belief that this area of polymer

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chemistry has been neglected and will provide some valuable new and useful polymers (5).

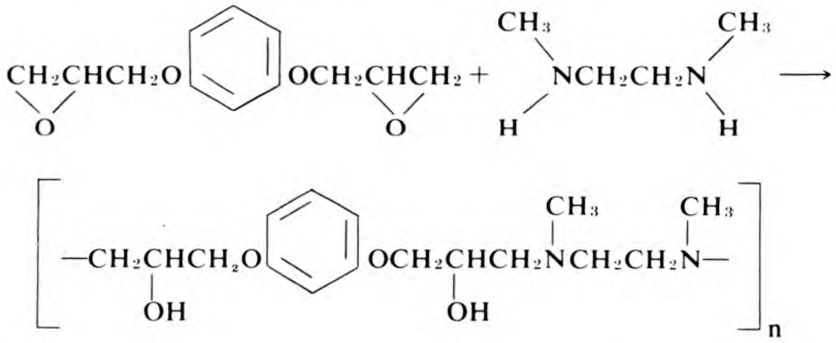
The synthesis of a fluorinated epoxy is tantamount to synthesis of a fluorinated polyurethane since the reaction sequence epoxy → polyol → polyurethane is readily accomplished (6). Epoxies and polyurethanes are quite similar at the molecular level because both usually are network polymers which result from coupling reactions entailing no elimination of small molecules. High-strength, fiber-reinforced plastics are commonly produced from the stronger epoxies, but the polyurethanes are tough, glossy, and abrasion resistant, which factors lead to superior coatings.

General Syntheses

A common type of linear polymerization involves a coupling of difunctional molecules to produce a chain:

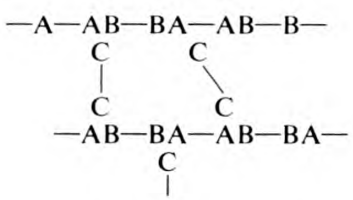
$$A{-}A + B{-}B \longrightarrow {-}A{-}AB{-}BA{-}AB{-}B{-}$$

An example of epoxy polymerization of this type is:



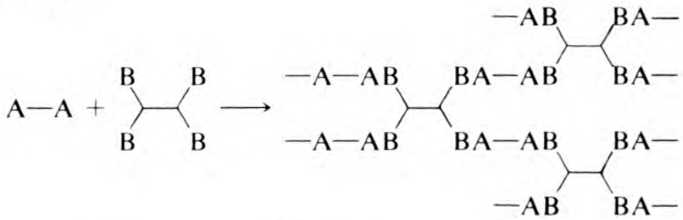
In this reaction an epoxy ring is opened by an amine to effect a coupling of molecules with simultaneous formation of an hydroxyl (OH) group. If n is a small number (< 100), the product may be classified as a "prepolymer" which is capable of further reaction through the hydroxyl group brought about by a "crosslinking" agent:

$$-A-AB-BA-AB-B- + C-C \longrightarrow$$

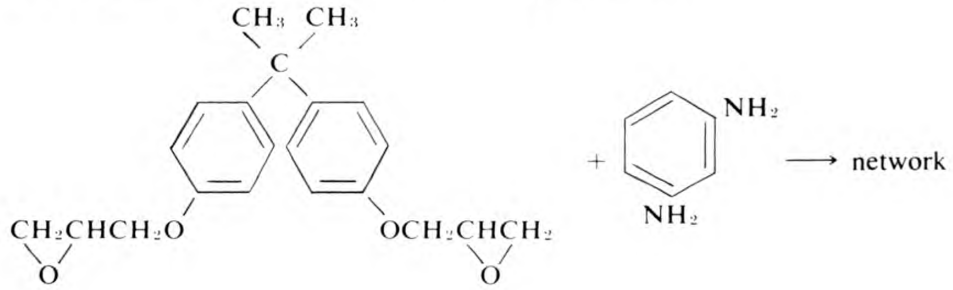


The "prepolymer" approach to a network molecule which extends indefinitely in three dimensions is often used in polyurethane systems.

If one of the initial reactants has more than two reactive groups, the polymerization reaction produces branched chain molecules in the early stages (7), and network formation proceeds without passage through a distinct prepolymer stage. This reaction mode is the basic one for producing epoxy plastics of the types used in casting, laminating, and filament winding.



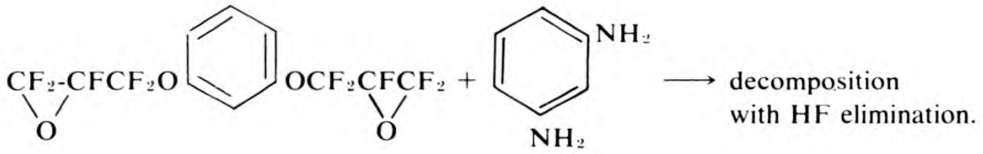
A typical example of reactants for this process is:



This compound, the diglycidyl ether of Bisphenol-A, is the standard of epoxy resin utility. It is difunctional and reacts with the tetrafunctional diamine to produce one of the common epoxy structural plastics.

Introducing substantial quantities of fluorocarbon into such polymers involves a number of considerations, the first of which is the availability of suitable fluorinated starting materials. Since heavily fluorinated organic compounds do not exist in nature as do the petrochemical base stocks of common polymers, each fluorinated precursor must be individually synthesized. Usually the precursor must also bear at least one reactive group (preferably two in our concept), but the commercial supply of such fluorinated components has been rather limited. A second consideration is that fluorine cannot be substituted indiscriminately for hydrogen on an organic molecule (8). For example, if all of the hydrogen on ethanol, CH_3CH_2OH, were replaced by fluorine, one would have CF_3CF_2OF. The -OF group has little in common with -OH and could hardly be expected to behave in an analogous fashion. If the structure were then redrawn to eliminate fluorine on oxygen, the unstable compound CF_3CF_2OH would be obtained, which would quickly decompose to produce hydrogen fluoride (9). The only common members of this series then consist of the β-substituted fluoroethanols, such as CF_3CH_2-OH. This limitation on the location of fluorine is of some consequence in the selection of structures for synthesis of epoxy-derived polymers,

because it eliminates many possibilities which seem attractive upon first consideration. An epoxy resin such as the following may be capable of synthesis, but in the common cure reaction shown it would be quite useless because of hydrogen fluoride generation:

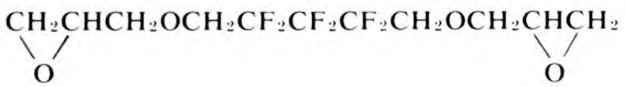


Apart from this difficulty, there is another concern which arises from the use of fluorine to replace hydrogen on an epoxy ring. Fluorine has a powerful attraction for electrons, and it alters the electronic structure of an epoxy ring sufficiently that reaction rates in common use modes are out of the necessary ranges for effective polymer cure (10). Because the reactivity of the common epoxy is convenient in many practical uses, we have kept fluorine at some distance on the molecular structure from the epoxy ring in most syntheses.

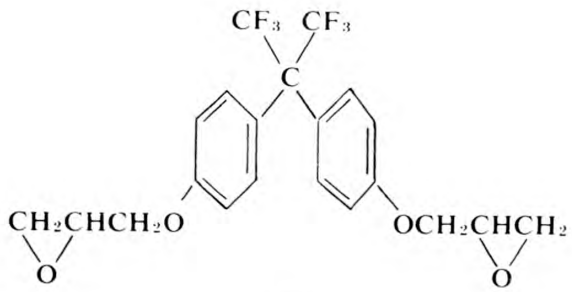
Most practical applications of epoxy materials to date have employed the type of epoxy formulas illustrated above (diglycidyl ether). Once a fluorinated diglycidyl ether is synthesized, it can be used relatively easily as a reactant for the synthesis of an analogous polyurethane.

Fluorinated Diglycidyl Ethers

Prior to 1967, few fluorinated diglycidyl ethers, illustrated below, had been synthesized. Dammont *et al.* (11) reported in 1965 on the synthesis of compound I, and compound II was the first fluorinated epoxy synthesized at NRL, although it had been previously synthesized:

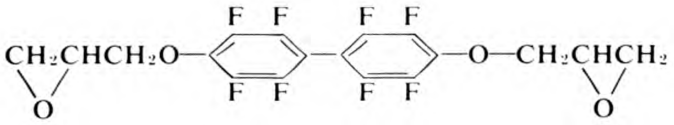


I

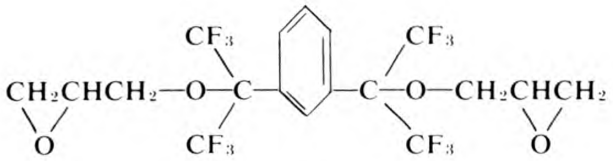


II

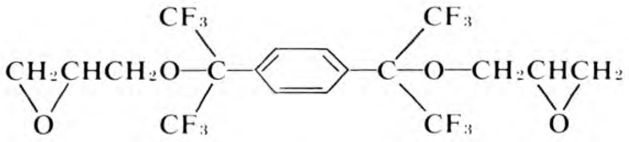
Compound II is the only product of any consequence that resulted from the direct analog approach to fluorinated epoxy resins. It is the diglycidyl ether of Bisphenol-A which has been fluoro-substituted on the central aliphatic portion; however, attempts to introduce more fluorine into this particular structure were not fruitful. The following formulas are listed in the chronological order of synthesis and depict the evolution of fluorinated diglycidyl ethers since 1967:



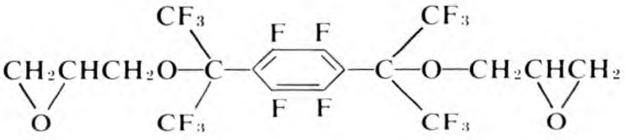
III



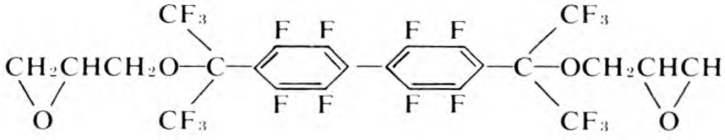
IV



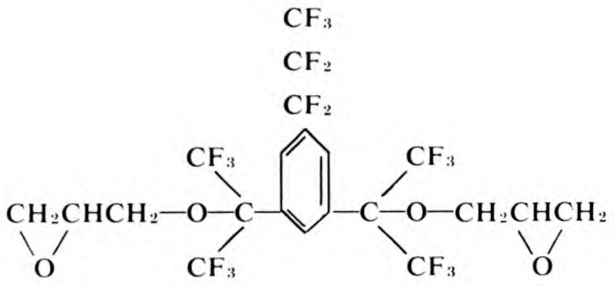
V



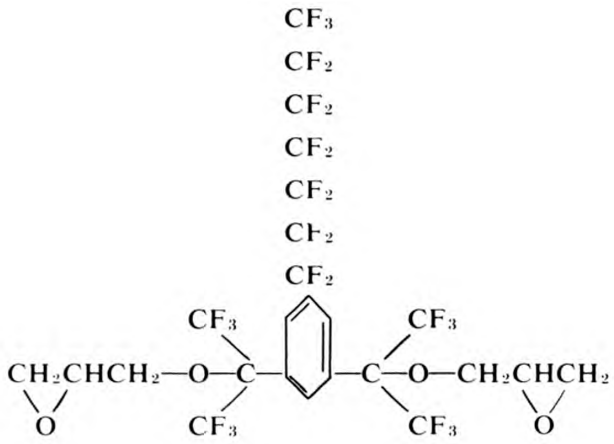
VI



VII



VIII



IX

Of these materials, compounds IV, VIII, and IX are worthy of special note because of certain practical qualities. Calspan Corporation (formerly, Cornell Aeronautical Laboratory), where a large cloud physics chamber was lined with a material such as that shown in Figure 1, has recently made a major application of polyurethane derived from compound IV. The precursor to IV is relatively inexpensive, since it is readily produced from hexafluoroacetone, a cheap reactant as fluorinated compounds go. Compound IV contains four trifluoromethyl groups and a most effective form of fluorocarbon; the unsymmetrical substitution of the rigid, stable benzene nucleus makes this an excellent unit for the building of tough, stable polymers. Although compounds VIII and IX are somewhat more expensive than compound IV, they contain more fluorine and have the special distinction of being liquids in pure form at ordinary temperatures. Colorless, transparent plastics of exceptional clarity can be cast from either compound; their liquid nature also allows them to be used with convenience in special applications such as filament winding and adhesive bonding.

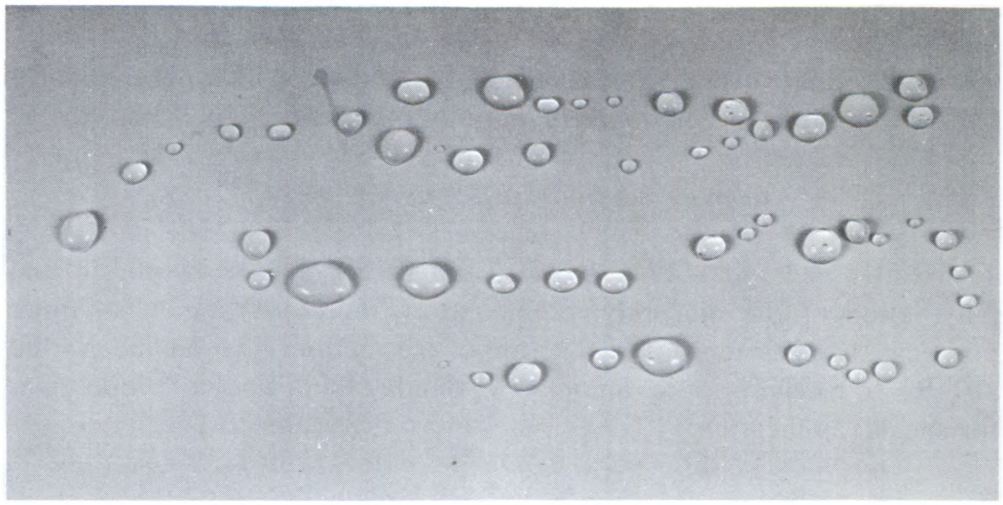
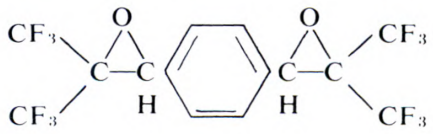


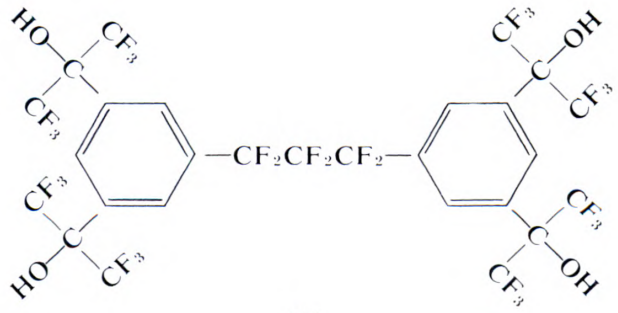
Figure 1 — Water droplets stand at high contact angles on a weathered FEP-fluoropolyurethane coating

Further progress is possible in fluorinated epoxies of nonglycidyl ether types, and one such material, compound X, has been synthesized:



X

Also, one tetra-ol precursor, compound XI, has been synthesized, and it may be of interest to investigate some fluorinated glycidyl ethers of higher functionality:



XI

Fluorinated Polyols and Polyurethanes

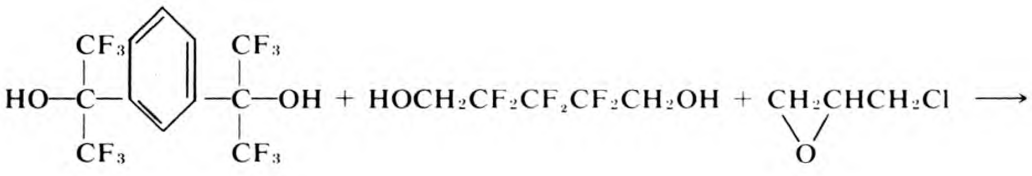
From fluorinated precursors the polyols can be generated in either of two ways. A diglycidyl ether may react with a fluorinated diol to give a regularly alternating polyol of the type

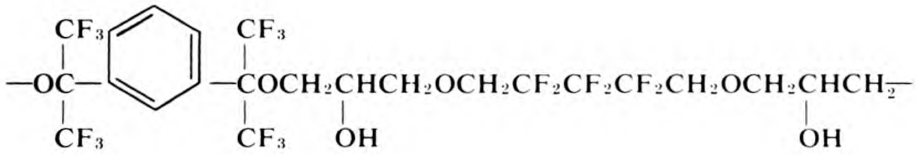
—A—AB—BA—AB—B— *etc.*;

or two fluorinated diols may be reacted simultaneously with epichlorohydrin to produce a polyol with randomly distributed units:

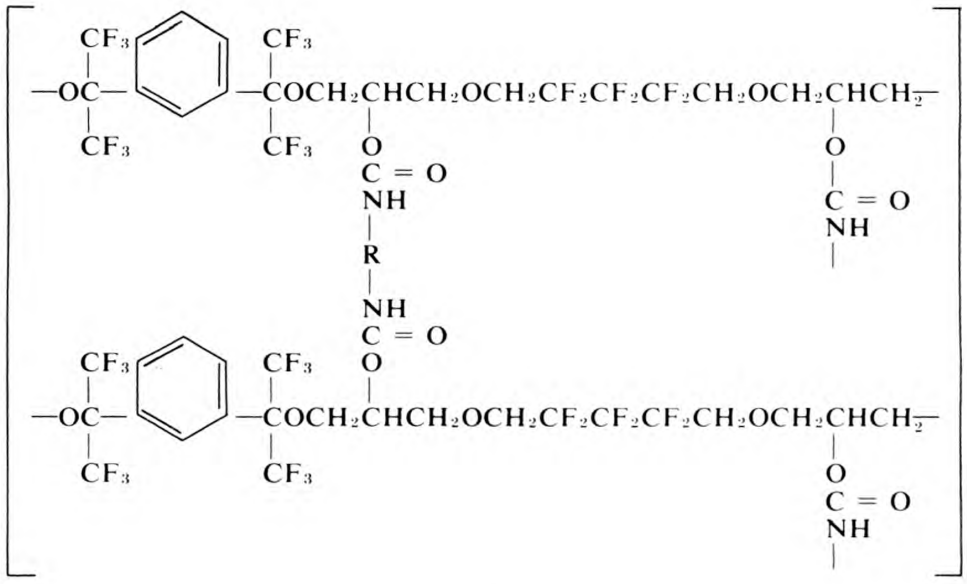
—AB—BB—BA—AB—BA—AA—AA—AB— *etc.*

This latter method avoids the necessity for separate synthesis and purification of the diglycidyl ether and is, therefore, somewhat more practical than the former. A reaction of the random type has been used at NRL to produce approximately 30 pounds of fluoropolyol prepolymer for special applications:





In actual practice, this polyol is dissolved in a solvent and blended shortly before use with an isocyanate crosslinking agent which converts it into a polyurethane network by reacting with the pendant hydroxyl groups:



These reactions are most useful for the production of some unusual coatings, and these particular components have been applied three different ways as illustrated in Figures 1, 2, and 3. Figure 1 illustrates the hydrophobic character of a coating which has been exposed to the weather at a 45° slant facing south in Washington, D.C. for a year. The polyurethane components of this system are sufficiently fluorinated that they wet particles of PTFE rather well, and this coating contained fluorinated ethylene propylene polymer (FEP) particles as an extender pigment. After the years' exposure, the panel was washed free of accumulated dirt, and water would stand at high contact angles on the surface as illustrated.

Figure 2 shows a similar composition of coating formulated for use as a white aircraft finish which also contains titanium dioxide as a pigment. On the half of the panel marked "F," the coating is fluorinated; and on the half marked "E," the coating is a conventional aircraft epoxy. The central portion of the panel was marked with an organic dye in large letters "F" and "E," covered with soot in a candle flame, and subsequently washed with a cleaning solution. Soil release, or cleanability, is the feature of the fluorinated coating which is most striking since the dye-mark "F" and the soot has been completely removed whereas the dye-mark "E" and carbon stain remains in contrast on the conventional epoxy paint.

The interior of the cloud physics chamber at the Calspan Corporation has been coated with a high-glass, unpigmented, fluorinated polyurethane of NRL design and manufacture (see Figure 3). Calspan is using this chamber to investigate atmospheric pollution problems under sponsorship of the Environmental Protection Agency and the Office of Naval Research. Calspan decided to use an experimental material in this application despite the inherent risks involved with any innovative material and the high cost. The essential properties required of the surface were that it be highly resistant to particle generation under soft ultraviolet light and be easily cleaned between experiments. The original

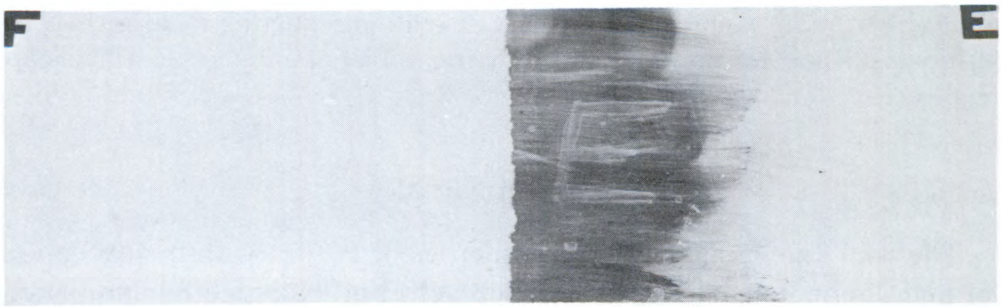


Figure 2 — Comparison of soil release by fluoroepoxy coating "F" and conventional epoxy "E"

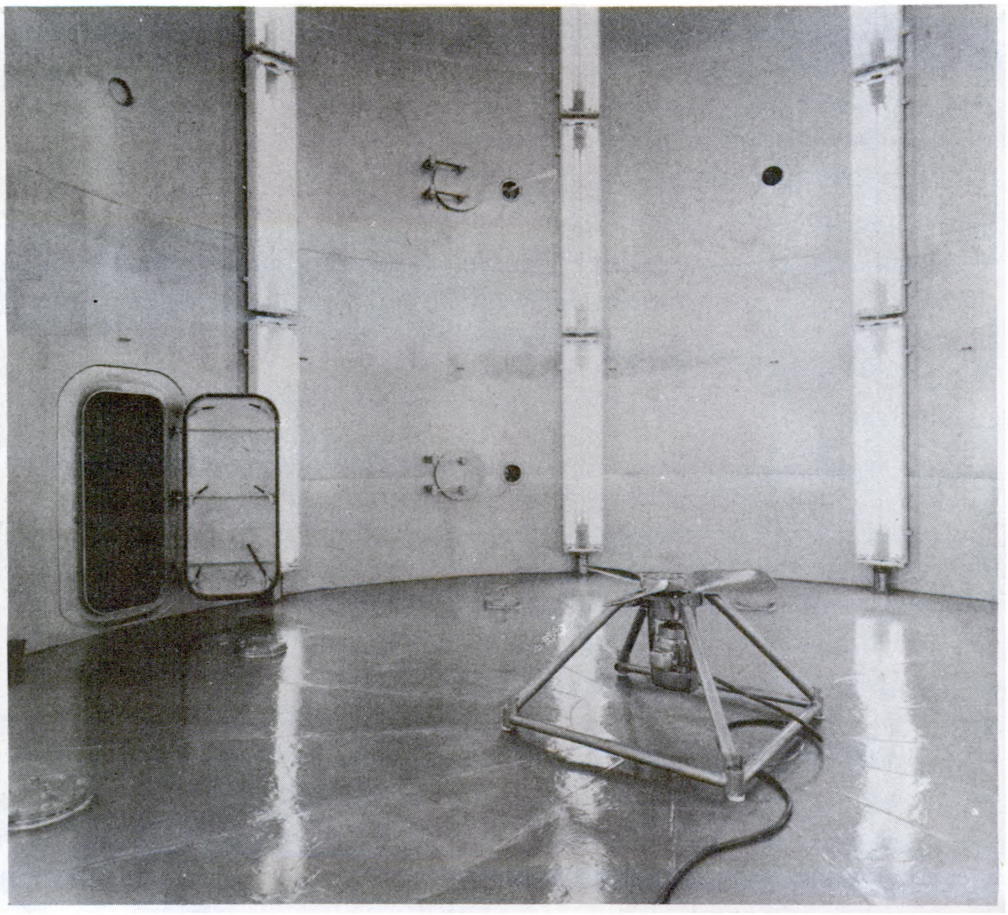


Figure 3 — High gloss, unpigmented NRL fluoropolyurethane coating on the large cloud physics chamber of Calspan

concept was the design of a bag of PTFE to fit this chamber, which is a cylinder 30 feet in diameter and 30 feet in height. Application of a paint-like substance by means of conventional methods (brushing was used) was obviously less expensive and easier than the original plan. A study by Calspan of particle generation in soft ultraviolet light and of the change in critical surface tension (12) with time found (a) that approximately two orders of magnitude less particles were emitted than evolved from an equal surface of a conventional coating and (b) that the critical surface tension remained in the PTFE region after ultraviolet exposure.

Potential Applications

The well-known and unusual properties of PTFE, with the exception of high thermal stability, can be approached or exceeded by fluorinated epoxies and polyurethanes; and in many potential applications of these new materials, they will serve the function of PTFE while being much

easier to use in a practical manner. A number of applications for such materials are developing, among which are the following:

Those Already Mentioned—Structural plastics, special aircraft coatings, and environmental research coatings are presently being evaluated experimentally.

Aircraft Windshield Castings—Clear, tough castings of fluoroepoxy can be formed which are highly hydrophobic and retain this quality throughout the bulk.

Lubricant Barrier Films—Tough, thermally resistant polyurethane or epoxy films to prevent lubricant migration from critical mechanisms are in prospect.

Coatings for Ships and Icebreakers—The low friction characteristics of PTFE-filled fluoroepoxy coatings and the inherent hydrophobicity are of interest.

Dental Prosthetics—As fillings for teeth or the molding of false teeth, the fluoroepoxies hold promise.

Ammunition Coatings—Corrosion protection combined with low friction are promising.

Spacecraft Coatings—Temperature control due to fluoropolymer absorption-emission characteristics is a possibility.

Nonburning Plastics—If the fluorine content is sufficiently high, a form of flame resistance can be obtained.

Solid Propellant Matrix—Liquid fluoroepoxies offer convenient matrix characteristics, including excellent wetting of the oxidizer or motor case wall, and high-energy performance.

Optical Cements—Low refractive index may be of value in some lens applications.

Laser Window Adhesive—An HF laser with calcium fluoride windows and a PTFE coating was recently sealed successfully with fluoroepoxy.

Conclusions

This work has resulted in heavily fluorinated epoxies and polyurethanes which are practical in every respect except cost. The precursors to these materials continue to be specialty items of relatively high cost. Properties can now be obtained in fluorinated network polymers, however, which are quite useful and cannot be realized as satisfactorily with any other materials. We hope that the time is not too distant when large-volume production of fluorinated precursors will bring the costs of these materials within the range of the multitude of applications for which they are suited.

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NRL Research Helps Prevent Fuel Deterioration

Naval Research Laboratory (NRL) scientists are furnishing the Navy with important knowledge on the treatment and handling of a variety of hydrocarbon fuels so as to prevent their deterioration by microorganisms, primarily bacteria and fungi.

These microbial contaminations are known to have caused many problems, particularly in aviation gasoline and in jet fuels, such as JP-5. Commercial airlines use similar fuels, which are also known to be susceptible to microbial growths.

Among the problems encountered have been slow fueling of aircraft because of clogged filters, malfunctioning of quantity gauges, souring of fuels and corrosion. These contaminations invariably occur in association with water because it is required for microbial growth. The quantity of water involved may vary from a few drops of condensate on the bottom of an airplane wing tank to thousands of gallons for displacement of fuel in a storage tank aboard ship. Even small quantities of water, however, support potentally dangerous amounts of growth.

The most promising means of preventing microbial growth is by the addition of inhibitory chemicals. Compounds for controlling microorganisms in hydrocarbon fuels, must be more than good microbial inhibitors; they must be compatible with the fuels, noncorrosive to fuel systems and harmless to marine life if discarded in the ocean.

Research is continuing in an effort to find improved means of controlling microorganisms in all types of hydrogen fuels used by the Navy.

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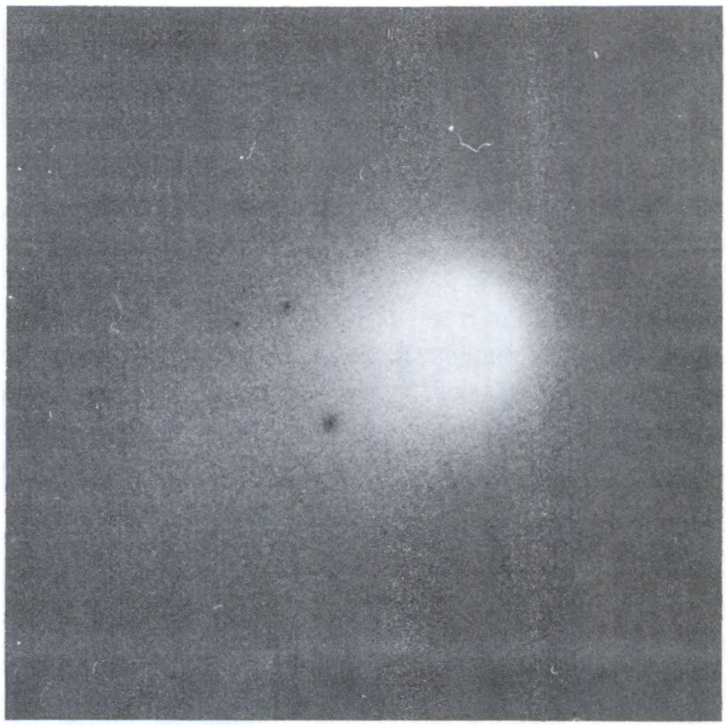
Research Notes

NRL Takes a Look at Comet Kohoutek



An ultraviolet camera designed and built by two scientists at the Naval Research Laboratory (NRL) and carried aloft by a NASA Aerobee Rocket snapped a unique picture of Comet Kohoutek on January 7, 120 miles over White Sands, New Mexico.

After analyzing this and other photos from their 4-camera payload, NRL's Drs. Chet Opal and George Carruthers report that the spherical image in the picture is a huge cloud of hydrogen three times the diameter of the sun that completely envelopes the head and most of the tail of the visual comet.

Some of the hydrogen appears to be blown away by the sun to form a tenuous cloud behind the comet which is many millions of miles across.

The image was obtained by photographing the comet in the light of the Lyman alpha line of atomic hydrogen, using a special electronic ultraviolet camera designed by the two space scientists. A similar camera is also on board SKYLAB and has recorded other images of Kohoutek. These will be available when the SKYLAB astronauts return to earth with the film transports from the camera.

Space scientists believe that the comet consists largely of water ice, in which are embedded dust and other ices. As the water evaporates when the comet nears the sun, the action of sunlight breaks it up into its component hydrogen and oxygen atoms. The lighter hydrogen atoms escape rapidly, producing the extensive cloud as shown.

Recommendations for Solving Some Water Pollution Problems

Charles F. Rowell
Naval Postgraduate School

Introduction

The Navy has been working on the problems of water pollution for some time. As far back as 1966 the Navy conducted research in the area of shipboard sewage. Four years later when the concern over pollution was felt throughout the government, the Navy established the Division of Environmental Protection, which has focused on the application of current techniques to pollution problems. The Division's work with local authorities and with shore facilities has made the Navy the leader in cleaning up the nation's harbors as required by Executive Order Number 11507, 5 March 1970.

The research and development (R and D) work has been in the area of oil and sewage problems aboard ships where the treatment systems are limited by stringent conditions of space and motion. The major work in this field is being carried out by the Naval Ship Research and Development Center, Annapolis Laboratory.

In another area the Navy established a program to monitor all aspects of pollution relating to its installations. NAVFAC undertook pilot studies to establish program requirements for harbors (Pearl Harbor), air rework facilities (North Island), industrial efforts (Crane, Indiana). General coordination of the program was provided by the Naval Civil Engineering Laboratories at Port Hueneme.

The Naval Post Graduate School Study

The question was raised as to whether changes in operating modes or in applying existing equipment would give immediate cost-effective abatement of water pollution from ships. Individuals who had both experience in problems of pollution and existing ship equipment could best answer these questions. Dr. John Huth, Special Assistant to the Director of Navy Laboratories, assembled from the Naval Post Graduate School a group of twelve students from the disciplines of operations research, mechanical engineering, electrical engineering, and management, who working with four faculty members, undertook this study as an interdisciplinary thesis. Much of the research was done in San Diego Harbor because of prior knowledge of the Harbor. The study group searched for methods of installation that could be performed on board a ship by its crew and thus not require delays in a ship yard.

Recommendations

Sewage

It was recommended that a system for grinding and pumping of sewage be installed on Navy ships as a common first step to a number of possible later

strategies of treatment. Such a system was designed and can be used to transfer sewage to bags or barges as well as to pier hookups when they become available. Should R&D produce an on-board treatment system that is satisfactory, this sub-system is a necessary first step toward centralizing the flow and saves piping costs as well by reducing the piping diameters needed.

Oil

The most oil spilled by the Navy is the result of fuel handling. Overflowing tanks are the most significant offenders.

Three complementary approaches were recommended: (1) improved on board indication and control, (2) changes in some regulations and, (3) at least in San Diego, a change in the mode of fueling operation.

The first approach concerns minimizing the number of fuel oil tanks that overflow overboard and implementing effective indication and control measures on these tanks. The recommended measures are accurate level indicators, and an emergency shut-off capability for the fuel transfer pumps from the central monitoring station. The price for this proposal, including labor costs for installation, for all ships of the Cruiser-Destroyer Force will be in the neighborhood of six million dollars. It is projected that the number of overboard oil spills will be reduced by 70-90% (depending on the data basis used).

A second significant change would cost nothing for initial implementation and probably little in use. The recommendation is to lower the maximum fueling level from approximately 95% to 90% and the required refueling level from 85% to 75%. The degradation in readiness is small (at most ~5% of range) and the risk of overfilling is markedly reduced.

The third change in operating procedures involves the elimination of oiler transfer to ships in harbors. The major reason for this recommendation is the notably lower number of spills from fuel pier refuelings. The cost for such a change, which would involve building another fuel pier for San Diego, was found to pay back its initial investment in less than two years by retiring the yard oilers.

Recommendations in the area of spill cleanup were related to questions of equipment needed for an inhouse team and the advantages of contractors *versus* inhouse cleanup. It was shown that the Eleventh Naval District plan for San Diego was very good as to location of spill control crews. The nature of the area to be covered by each crew was examined and recommendations for equipping each crew were made. The study group recommended the use of contractor services for *all* Navy oil cleanup.

International Conference on Stress Corrosion Cracking and Hydrogen Embrittlement of Iron Base Alloys*

The nuclear power industry, the users of modern structural steels, the chemical industry, the pipeline industry, and the oil industry have an important stake in

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controlling the problem(s) of stress corrosion cracking (SCC) and hydrogen embrittlement in steels, as was attested by the introductory lectures of this monumental conference held at Firminy, France from 12-16 June 1973. The lecture on the nuclear power industry for example cited 88 failures due to SCC in approximately 10 years.

There were low-keyed but important shifts in emphasis between earlier conferences on SCC and this one: Only one alloy system was covered; continuum mechanics in the form of fracture mechanics was not only used by numerous authors but was also represented by several prominent specialists in that field; and civilian technology was emphasized rather than either space or military technology. As one example of the attuning of the Conference to timely civilian concerns, it was pointed out that as the world demand for energy experts pressure for drilling ever deeper oil wells—from 20,000 ft in 1973 to 30,000 ft within a few years—the *minimum* yield strength of the drill pipe will have to rise from about 90 ksi to 140 ksi, and the maximum may even go as high as 180 ksi. Many of these deep wells will contain H_2S, so that hydrogen embrittlement cracking of some of these steels is a problem for which containment measures must be found before the wells can be drilled.

The co-chairmen of the organizing committee were Dr. J. Hochmann of Creusot-Loire and Dr. R. W. Staehle of The Ohio State University. The technical program consisted of 78 papers of diverse types. Some were introductory background papers (indicated above), some were reviews, some were descriptions of very recent research, and others represented a re-examination of subject matter which had been largely published before, but here was consolidated and given fresh exposition. There were papers reporting the phenomenology of cracking in various gaseous atmospheres (including hydrogen), in liquid ammonia, in CO-CO_2-H_2O mixtures, in liquid metals, and in concrete. Except for the introductory lectures and the papers describing new phenomenology, most of the program was concerned with fundamentals, particularly models of the cracking processes.

The papers were replete with new facts. As one example, the crack growth rate of a martensitic steel in gaseous hydrogen was observed to decrease three orders of magnitude when the stress intensity (K) was decreased only a few percent. As another example, the acoustic emission in stress corrosion specimens was found to continue some 10 hours after the specimen had been completely severed by SCC. These are only two of the many new observations yet to be explained. The program was devoid of any discussion of engineering test methods and interpretation of test results. Prof. Lacombe's suggestion that "project alloys" be made available generally deserves more than polite applause. Such a measure is essential for a "round robin" exercise which will be needed to establish standard test procedures.

It was evident again at this Conference that bringing the existing knowledge of SCC hazards and control measures to the designer is a matter of much importance, and is one for which no startling progress was apparent.

A special medal of jewel-like workmanship, made of Creusot-Loire's Uranus 50 alloy (stainless steel), was presented to Prof. G. Chaudron at the official dinner. The local arrangements by the French hosts throughout the Conference demonstrated much thoughtfulness, hospitality, and hard work. The Conference

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was sponsored by Creusot-Loire and by the Research Committee of the National Association of Corrosion Engineers

New Reserve Coordinator

CAPT C. H. Bird, Jr., USN, Assistant Chief for Administration and Research Reserve (center) welcomes the new Research Reserve Coordinator, Paul J. Gerdon (left) as CDR John K. Dowhal, USNR, Research Reserve Coordinator from the Branch Office in Chicago looks on. Mr. Gerdon reported to the Office of Naval Research on 24 September. He is a Commander in the Naval Reserve Research Program and actively participates with NRRC 5-8, Washington, D.C. He and his family reside in Springfield, Virginia.

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Cover Caption

The major component of the High Pressure Life Laboratory consists of two seven-foot spheres surrounded by seven animal living chambers.

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