Proposal No. 69-857C Purchase Request No. NAOO-9-12

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PHYSIO-CHEMICAL STUDY OF FIRE RESISTANT MATERIALS FOR AIRCRAFT CABINS

Part I - Technical

Federal Aviation Administration (DOT)

Proposal No. 69-857C

Purchase Request No. NA00-9-12

PHYSIO-CHEMICAL STUDY OF FIRE RESISTANT MATERIALS FOR AIRCRAFT CABINS

Part I - Technical

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PHYSIO-CHEMICAL STUDY OF FIRE RESISTANT MATERIALS FOR AIRCRAFT CABINS

INTRODUCTION

This proposal is in response to RFP No. NAOO-9-12 (PRN-9-195) by the Federal Aviation Administration, Atlantic City, New Jersey. A multidisciplinary approach to the problem of flame resistance and smoke prevention of materials for aircraft cabins is envisioned, involving IITRI's Physical Chemistry Group, the Polymer, Heat Transfer and Fine Particles sections.

Emphasis will be placed on physio-chemical characterization of pertinent materials, and identification of smoke and poison producing ingredients in materials used in aircraft cabins. To this end, a combination of experimental methods will be proposed which may eventually lead to the recognition of those functional groups in the polymer compositions which inherently produce smoke and/or emit gases and vapors poisonous to humans.

Since thermal analysis is a fast and convenient method most pertinent to the problem, it will be used as the basic instrumental technique to characterize a variety of synthetic materials. In combination with simultaneous gas chromatographic and/or mass spectrometric analysis, it will serve to analyze at pertinent stages of thermal decomposition the volatile reaction products. A most unique method is our fine particle thermal analysis instrumentation where smoke particulates may simultaneously be analyzed with respect to particle size, shape and distribution during thermal decomposition. The results

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from the identification phase of this program (Phase I) will be correlated to pertinent models, and improved formulations proposed and produced by our Polymer group (Phase II).

Simulated in-field testing of newly formulated materials will eventually be conducted by IITRI's Heat Transfer and Fine Particles sections, (Phase III) and compared to presently available materials.

In the following section, a discussion of the overall problem is given from the point of view of fire and smoke hazards, recognition of functional groups in synthetic materials which are potentially toxic, possible methods of identification and characterization of these groups, and the most promising approach towards improved formulations.

In the third section a detailed working statement is given which outlines our planned approach.

DISCUSSION OF PROBLEM

A. Behavior of Fires

Any attempt to identify all of the factors which bear on the hazard of a fire must necessarily be preceded by an investigation and understanding of the fundamental behavior of fires. Various aspects of this subject have been considered by numerous investigators and an extensive literature search has been performed by IIT Research Institute.¹ It involves a review of 1000 pertinent papers dealing with the ignition and combustion phenomena, as well as heat transfer mechanisms. Since the discussion of all fundamental aspects of fire behavior would be

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a repetition of this work and would require a prohibitive amount of space, only some highlights pertaining to ignition and combustion phenomena are considered below.

According to a definition by Graf,² ignition temperature is that temperature at which the rate of heating in the substance exceeds the rate of heating induced by an external source of heat and has visible combustion in the form of a glow or flame as an end result. For a specimen of solid fuel in heated air, for example, two basic types of ignition can be considered possible; pilot ignition and self ignition. Pilot ignition occurs at some temperature where the concentration of evolved combustible gases and vapors (thermo-decomposition products) becomes just sufficient that they may be ignited by a pilot flame, spark, or other source. Self-ignition takes place in the absence of a pilot source, and at a somewhat higher temperature. Over the years, many methods of determining ignition temperature have been devised and it is apparent that the ignition temperatures of gases, vapors, and solids are not constants, but are functions of the systems in which the fuels are situated. Thus, such conditions as concentrations of reactants, initial temperature and pressure, catalytic and other actions of a heated surface, area of heated surface, and rate of heating all can influence the reaction. For example, rapid heating of cellulosic materials through the range of active pyrolysis tends to produce little charcoal, much tar, and highly flammable gases rich in hydrogen, carbon monoxide,

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and hydrocarbons; slow heating tends to produce much charcoal, little tar, and less flammable gases in which there is much water and carbon dioxide.³

In the early stages of a fire, the shape and size of the combustible material can be very important to fire growth or extinction. In the area of solid fuels, much effort has been devoted to the study of the effect of particle size, size distribution, moisture content, and fuel bed size and shape on the rate of combustion; and it has been concluded that the combustion rate is governed by the rate of oxygen transfer to the surface.

B. Hazards of Fire

The hazards of various products of combustion to human occupants present a serious problem. Various studies of accidental fires have shown that smoke and toxic gases appear to be the primary life hazards in most fires, although heat, oxygen deficiency, panic, and emitional shock may become primary hazards under special conditions.

1. Smoke, Solid Particulates in Smoke

Smoke is commonly considered to be the product of burning organic materials rendered visible as aerosols by the presence of small particles of carbon or other solids both combustible and non-combustible in the gas phase. The first effect of smoke on humans is obscuration of vision. In addition to reducing the visibility, smoke can also be injurious to the human

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respiratory tract. Until quite recently, the visible portion of smoke was not considered hazardous. However, recent investigations⁴ point out that this may not be the case.

Complete characterization of smoke is a difficult task due to the many parameters involved. These include particle size, distribution and concentration, particle shape and distribution, and chemical composition.

There are a variety of optical techniques for assessing, measuring and characterization of smokes. Among these are the Ringelmann Smoke Chart, where variable areas of black are printed in a continuous geometrical pattern on white opaque cards, and compared to the blackness of the smoke (characterization ranges from 0, all white, to 5, all black), photometric measurement of smoke, where the ratio of luminesce of the smoke to the background is measured with a photoelectric telephotometer⁵, and by transmissometry (attenuation of light shining through the smoke is measured photoelectrically).

These are all optical methods, and are only measures of gross physical features of smoke.

A more quantitative assessment of the physical and chemical properties of smoke is, however, much more involved. It requires sampling and subsequent analysis of the airborne combustion products. Due to the very fine particulates found in smoke (0.001 to 0.1 microns), it is necessary to use special techniques of sampling. Typical sampling equipment includes

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thermal and electrostatic precipitations and, in particular, high efficiency filters.

After sampling, the following analytical data of the particulates may be determined:

a. Particle size, size distribution and particle shapes by means of microscopy, electron microscopy and the IITRI developed Aerosoloscope. The latter also permits determination of particulate concentrations.

b. Chemical composition by routine analytical procedures in combination with spectroscopic techniques.

2. Volatile Combustion Products

The gaseous products from a fire of synthetic polymeric materials are a mixture of vaporized and thermal decomposition products. The bibliography of the "Fire Gas Research Report" of the National Fire Protection Association Committee on Fire Gas Research⁶ lists numerous publications dealing with the gaseous products of a fire. The effect of these products on humans has been reviewed by Dufour⁷ and Bieberdorf and Yuill.⁸ Some of the findings are presented below.

Almost any incomplete combustion of organic materials will produce carbon monoxide. This gas is considered by numerous researchers to be the foremost danger to life. Prolonged exposure to carbon monoxide is considered dangerous at a threshold concentration of 100 ppm. Concentrations of 1500 ppm are considered dangerous for 30-60 minutes exposures

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and exposure to 12,800 ppm will produce death in 2 to 3 minutes. There is much variation reported in the literature on the actual tolerance limits of humans to carbon monoxide. Also, doubts have been expressed by many professional people as to the accuracy of presently employed analytical methods for the determination of carbon monoxide in air or blood.

Ordinarily carbon dioxide is not considered to be a toxic gas. It is inert and toxic only in relatively large concentration. Inhalation of 7 to 8 percent of carbon dioxide may be fatal in a short period. Certain synergystic effects have been noted such as an increased respiratory rate due to ∞_2 which, in turn, enhances the damage caused by the presence of CO or other toxic or irritating gases.

Several other gases may be encountered in fires. These are: hydrogen cyanide (HCN) which is one of the most acute poisons; oxides of nitrogen which are very toxic; hydrogen sulphide (H S) which at concentrations above one percent is 2 fatal; and others such as sulphur dioxide, hydrogen chloride, and other hydrogen halogens.

Also, there exists a number of organic gases and vapors that act as irritants. Acrolein, a pyrolysis product of some polymers, is typical of this group of gases that cause an excessive flow of tears and a burning sensation in both the eyes and respiratory passages. These reactions can effectively interfere with reasoned judgment and normal vision and respiratory resistance.

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C. Flammability Test Methods

The inherent difficulty in definining the total fire hazard is perhaps partially responsible for the existence of so many different test methods for evaluating the flammability behavior of materials. Many different methods have been developed by governmental agencies and private groups, both in this country and elsewhere, for use on specified classes of materials or for specified applications. Unfortunately, these test methods differ as to sample size, geometric configuration, type and severity of fire exposure, ventilation, type of data recorded, etc., and consequently the results obtained from the various tests are not comparable and cannot be reduced to a common basis. The American Society for Testing and Materials alone lists 17 test methods under the category of "Flammability". Some of these are specifically intended for evaluating the ignition and flammability characteristics of treated and untreated textile materials, or thin plastic sheet materials. The situation is further complicated by the fact that a given test method is frequently assigned different designations by different groups or agencies, and the identity cannot be recognized except by a detailed study of each method. Marcy⁹, for example, lists eight different additional domestic and foreign designations for the relatively simple method known as Method 5902 of Fed. Spec. CCC-T-191b, and the list is not claimed to be complete. Also, at times

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A review of pertinent test methods was carried out recently at IITRI in conjunction with a program, which is currently in progress, concerning the flammability characteristics of vehicle interior materials, sponsored by the National Highway Safety Bureau (Contract No. FH-11-6892). IIT RESEARCH INSTITUTE

there are some small differences between methods with different identifications, and the effects of such differences on the results obtained are not always apparent.

The majority of the test methods involve small-scale, benchtype laboratory experiments. The most significant distinctions between them are the orientation of the test specimen (relative to gravity), and the time of exposure to the igntion flame. The orientation obviously is extremely important because of the preheating and heating reinforcement that the unburned section of the sample experiences from the ignition flame and/or from the combustion gases in the case where the specimen is not horizontal. In some test methods, the time of exposure to the ignition source is so short that the test serves primarily as a measure of ease of ignition rather than of the rate of flame propagation. That is, if the specimen fails to ignite during the brief period of exposure, the rate of flame spread is never measured.

Each test method prescribes the measurements that are to be made during a test -- usually burned length and time -- but no criteria for the accepability of materials are given. Some test methods prescribe class rangings of materials that depend on the rate of flame spread, or define a numerical index, but the performance specifications for given applications are presumably left to the user, or to code-making bodies.

It is important to note that none of the existing test methods is even remotely concerned with the potential (or driving

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force) that the burning material has for transferring heat to an adjacent or contacting surface. The nearest approach is the radiant panel method developed by the National Bureau of Standards¹⁰ which provides for a relative measurement of the rate of heat liberation by the burning material, but nothing concerning heat transfer to another body.

D. Physio-Chemical Aspects of Smoke and Other Combustion Products of Polymeric Materials

After the problems of fire hazards have been discussed in more general terms, it is quite in order to dwell more elaborately on the nature of plastic materials, their possible combustion products, and the most suitable way to establish physio-chemical parameters for identification.

In general, two approaches are possible for materials to be made flame retardant.

1. Polymers which are Inherently Flame Retardant

We may cautiously generalize and state that the elements which impart flame retardancy on a material are phosphorus, on which most commercial retardants are based, and halogens. Other elements are antimony and boron.

Unplasticized polyvinylchloride has good inherent flame retardancy because of its high level of chlorine contained in the polymer chain. Commercial plasticized PVC, however, has a chlorine level which is too low, and supplementary flame retardant additives are required unless a plasticizer is used which acts simultaneously as a flame retardant.

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Halogenated polyhydrocarbons such as Teflon are flame retardant and do not require the use of additives. Polycarbonates are also flame retardant polymers. Unlike PVC, Teflon, and halogenated polyhydrocarbons, however, where the flame retardancy is due to the presence of halogens in the polymer chain, flame retardancy in polycarbonates is produced by the evolution of ∞_2 during thermal decomposition. Their flame retardancy can be even further enhanced by their retardant additives.

2. Additives Imparting Flame Retardancy

Flame retardant additives are either incorporated in the basic resin by the manufacturer, or added to a general purpose resin by the fabricator during processing. Flame retardant additives are usually based on halogenated organic compounds (brominated or chlorinated) in combination with phosphorus containing organic compounds or antimony trioxide. A synergistic effect is obtained, as a result of which the amount of additive required to achieve the desired degree of flame retardancy is reduced. The use of antimony trioxide is limited to opaque systems. Phosphorus, in the form os phosphates or phosphonates has proven very effective when used singly or in combination with halogenated organics. Boron has been recognized as an important contributor to flame resistance. Technical problems, however, have generally limited its use to the production of flame retardant coatings and as a treatment for wood.

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A good solution to the problem of flame retardancy of plastics can be achieved by using a plasticizer or combination of plasticizers which are also flame retardants. This has been accomplished in the case of PVC: tricresyl phosphate, chlorinated bi- and tri-phenyls, cresyl diphenyl phosphate, 2-ethylhexyl diphenyl phosphate used singly or in combination with dioctylphthalate are generally required.

As far as polyethylene and polypropylene are concerned, chlorinated hydrocarbons and chlorinated synthetic polymers used with antimony trioxide have found application as flame retardants.

Flame resistance of polystyrene foams has been achieved by using brominated flame retardants, such as tris(dibromopropyl) phosphate. Flame retardant expandable beads of polystyrene are available to fabricators from many polystyrene manufacturers. Addition of flame retardants to general purpose polystyrene beads prior to molding is not generally practiced.

3. Flame Retardant Comonomers

For some resins, the problem of fire retardancy has been solved by using reactive intermediates which impart flame retardancy to the basic resin by copolymerization. In this case, the fire retardant additive is built into the polymer structure as a comonomer. This approach has found commercial application in the case of polyesters, epoxies, and polyurethane foams. In the case of polyesters, flame retardant comonomers commercially available are dicarboxylic acids or dianhydrides

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the same time high levels of halogens which upon decomposition are p otentially toxic, they might be released as toxic gases such as hydrogen halogens and/or halogens per se. The final goal of any physio-chemical characterization of these materials should then be to define properly those ingredients which are smoke and poison producing, try to minimize or perhaps eliminate completely these effects without sacrificing the fire resistivity by searching for materials which - based on the physio-chemical analysis - meet the double criterium of being fire resistant and less or even non-toxic. In the following work statement a research program is outlined which (a) will most efficiently aid to characterize polymeric materials used in aircraft interiors with respect to smoke producing and toxic combustion products, (b) will lead to a prediction of improved formulations, where these effects are minimized or even eliminated without sacrifice of flame retardancy, (c) eventually, new formulations will be synthesized and tested with standard methods $\frac{11}{45}$ to their capabilities of fire resistance, smoke reduction and minimization in toxic vapor production. PROPOSED RESEARCH PROGRAM

Phase I Physio-Chemical Investigation

A most pertinent method to investigate combustion, smoke and toxic properties of plastics is thermal analysis. This in combination with analytical tools such as gas chromatograph, mass spectrometry, NMR and IR spectrometry should be a potential approach to investigate the problems outlined in Discussion of Problem.

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The author and his colaborators at IITRI have extensive experience in investigations of thermal decompositions of organic (lubricants, polymeric), and inorganic materials by means of combined techniques such as thermal analysis and mass spectrometry, thermal analysis and gas chromatography, or thermal and conductometric analysis. Particular pertinent are their studies on lubricants and lubricant additives and on epoxy resins.

In general, thermal analysis involves the heating of a sample under investigation at a constant heating rate and observation of parameter such as temperature differential between the sample and an inert reference, or the change in sample weight. In differential thermal analysis (DTA) the temperature difference between sample and reference is obtained as a function of time and/or temperature. Chemical and physical interactions become observable, if the temperaturedifference during these interactions is sufficiently large to be sensed by thermocouples or other temperature sensing devices. Thus, exothermal solid or liquid state reactions become visible as upward bands, and endothermal interactions such as melting, dehydration or certain decomposition reactions as downward bands in the DTA curves. In thermogravimetric analysis (TGA) the experimental parameter in question is the weight loss (decomposition, dehydration, etc) or weight gain (oxidation, carbonation, hydration, etc.) of the sample as a

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function of temperature and/or time. A supplementary refinement to this technique is derivative thermogravimetric analysis (DTGA).

Each material (e.g., polymer) has its own thermal characteristic curve and can therefore be used for its analyzation and identification. Curve parameters such as peak position (temperature of peaks) band shape, intensity, width, and area (in DTA and DTGA) characterize each substance.

Changes in ambient conditions such as heating rates, gas atmospheres, and pressure may enable the investigator to deduce conclusions on combustibility, oxidative degradation, etc., of the materials. For example, thermal degradation of certain lubricants in argon is, less pronounced than in oxygen and different lubricants may easily be identified by their thermal characteristic curves (Figure 1).

Stepwise thermal analysis to various temperatures enables us to study the reaction products and solid residues at intermediate stages. This in combination with simultaneous effluent gas analysis (EGA), gas chromatography, and mass spectrometric analysis represents a powerful technique to identify not only the combustion products but also to unravel the combustion mechanism for a better understanding of the reaction. In this context, the Freeman-Carroll equation might be mentioned which is frequently used in thermal analysis to obtain essential reaction parameters such as activation energy and reaction order from TGA and DTGA curves. (Dr. Freeman is the Assistant Director

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Fig. 1 DIFFE HE/ mg. A $l_2 O_3$

of Chemistry Research at IITRI and will act as advisor to the program).

With respect to smoke evolution during thermal decomposition, fine particle thermal analysis has direct bearing to the problem of fire hazards with insufficient oxygen supply. (Figs. 2&3) In this case the materials under investigation will be subjected to thermal analysis under a variety of atmospheric conditions, where oxygen is mixed with nitrogen in ratios between 21/79 (normal case) and o/100 (complete lack of oxygen). During these runs the produced particulates in the smoke will be subject simultaneously to our fine particle analyzer. These experiments should yield informations on smoke production at various stages of decomposition under pertinent atmospheric conditions.

We shall identify among existing plastics the ones that, because of their intrinsic properties, are potentially useful as materials for aircraft interiors. These plastics should meet the following three requirements: (1) being inherently flame resistant, (2) emitting minimum quantities of smoke by combustion, and (3) producing little if no toxic combustion products. The literature available on this subject will be reviewed. Pyrolysis data and composition of the volatile degradation products are available for a number of plastics. Even though these data may have been obtained in pyrolysis experiments which are not typical of a fire environment, the information available may be quite useful for a preliminary screening of suitable plastics. The plastics chosen for study

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Six-Channel Readout 1-8 µ À Aerosol Sumpler Sampler Probe See Schenatic in Figure 4 Glass Tube 1 14 1/min Argon Dilution 1 Gas Cylinder Apparatus DTA

FINE PARTICLE THERMAL ANALYSIS SYSTEM

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Figure

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will be tested according to ASTM STP 422 test procedure. Subsequently, thermogravimetric analyses, in atmospheres of various oxygen content, gas chromatograph studies of the volatile decomposition products, mass spectrometry, infrared spectroscopy and NMR will be performed. This study should provide a basic understanding of the emission of smokes as related to molecular parameters such as structure, presence of particular chemical functionalities, etc., of the volatile portions. Emitted particulates will then be collected, characterized as to shape, size distribution, and analyzed as to their chemical composition.

The analytical results will be tabulated in terms of smoke production, toxicity of the combustion products, and reduction of these unwanted effects as dependent on certain function groups in the synthetics. It is planned that correlation of the analytical data are used as guidelines during Phase II of this program for new plastic formulations which are less flammable and produce less toxic vapor during thermal decomposition than existing materials.

Phase II Search for Improved Formulations of Synthetic Materials

In the second phase, we shall study the modification of existing plastics in order to improve their flame resistance and mimimize smoke formation by combustion. We propose to extend the approach of plastic modification by using reactive intermediates (comonomers). This approach has found successful application in the case of polyesters, epoxies and

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polyurethane foams. The copolymerization approach has some advantages over the use of flame retardant additives. In fact, fairly high concentrations of additives in the resin are necessary to impart flame resistance; such additives may have a negative effect on the basic properties of the resin. Unlike flame retardant additives, when a flame retardant comonomer is used which has the same basic structure as the main monomer of the resin, no change in overall properties of the resin is observed. Furthermore, the high temperature characteristics of the resin may be improved by proper selection of the reactive flame retardant comonomer (i.e., chemical functionalities, structure, etc). Of course, the formulation of the comonomer should take into account not only the problem of flame retardancy but also smoke minimization and toxicity. Commerically available flame retardant additives and flame retardant comonomers will be tested in combustion experiments and the nature of the decomposition products will be determined. As another step, we shall consider a new approach to the problem: polymer blending before fabrication. Α flame retardant polymer will be blended with a general purpose plastic before molding. By proper selection of the two components, which should be compatible (soluble) in the molten state, the resulting blend may exhibit entirely new properties. We plan to select phosphorus based polymers for this purpose. The phosphorus based fire retardant polymer will be blended with the basic resin at the desired level and the solution of the

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two polymers in the molten state will be processed using standard plastic technology. The basic resins used will be the ones that in the first part of this study exhibited minimum amount of smokes and minimum smoke toxicity. To these basic properties, the phosphorus based polymer will add fire retardancy yielding a product with a new combination of properties.

IITRI has extensive experience in the area of synthesis and processing of high temperature plastics; a recent in-house project conducted at IITRI was aimed at the study of new high temperature polymers and led to the synthesis of new aromatic linear copolymers and terpolymers containing carbonate linkages. These polymers will be developed further with respect to their potential use as fire retardant interior aircraft materials.

Phase III Simulated Infield Testing of New Formulation

During this phase a selected number of improved formulations will be compared in a smoke chamber test with the "best" synthetics already available. The gross effects of smoke insofar as they may contribute to the total hazard when people are exposed to an accidental fire situation will be investigated. Several test methods have been developed for this purpose, and the most widely known are the Rohm and Haas XP2 smoke density test,¹¹ and the test method developed by the National Bureau of Standards.¹² Most smoke production tests are based on measuring the attenuation of light by the smoke produced from a burning or thermall grading specimen.

The Rohm and Haas method consists of measuring the light obscuring properties of smoke generated by the test materials IIT RESEARCH INSTITUTE

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in a specified chamber. It involves a cabinet, $12 \times 12 \times 30$ in. high, which is closed and unventilated except for a 1-in. high opening around the bottom. The test specimen is $1 \times 1 \times 1/4$ in., and is heated by a propane-air burner. A photoelectric cell is used to measure the light transmission, from a 3-watt light source, across a 12-in. horizontal path. This light beam path is located approximately 11 in. from the top of the chamber.

The NBS method involves a tightly closed chamber which is 3 x 2 x 3 ft high, and a test specimen which is 3 x 3 in. The light path is arranged vertically (to reduce errors resulting from stratification), with a 30-watt light source at the top of the chamber, and a photometer located directly below the light source at the bottom of the chamber. A radiant source is used to impose an irradiance level of 2.5 watts/cm² at the surface of the specimen. This test provides for both nonflaming (smoldering) tests, and for flaming tests. For the latter, a small gas burner provides pilot ignition.

Either method will be used under a varying degree of oxygen concentration, temperature of ignition and smoke production to obtain quasi infield data on the new formulations. FACILITIES

All equipment and facilities required for this program are available at the IIT Research Institute.

TIME AND COST

The program will require twelve months experimental work, plus three months for writing the final report. The cost estimated is included separately.

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PROGRAM MANAGEMENT

Constitution of the project team is shown in Figure 4. The program will be administered by the IITRI Chemistry Research Division and will be under the administrative supervision of Dr. Eli S. Freeman, Assistant Director of Chemistry Research.

In view of the interdisciplinary nature of the proposed program, the study will be conducted within the framework of a project team organized for the purpose of this project from different divisions. The project leader, in accordance with IITRI policy, will be able to fully utilize personnel from other divisions and associated organizations to take full advantage of individual expertise and skills. Work will be centered in the Physical Chemistry Research group which has all the research capabilities and experience required for this type of program.

Dr. W. K. Rudloff will be the project leader and will be responsible for the technical program. He will be assisted by Mr. Brent Boldt who will essentially handle the physio-chemical experimentation required for this program. Drs. Robert Scholz and Jack O'Neill of our Organic Section will advise during the mass spectrometric and gas chromatographic studies of Phase I. Dr. C. Giori of our Polymer group will be the principal investigator in Phase II and will have responsibility of developing new polymer formulas. He will be assisted by Mr. G. David and advised by Mr. K. Gutfreund, and Dr. A. Stake. Dr. E. Aynsley and Mr. A. Goldsmith will provide guidance during the simulated in-field testing with the smoke chamber.

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Business Administration

T. Plonis, Admin. Sup.

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The team as constituted has particular capabilities in:

- Thermal analysis of polymers
- Gas chromatography and mass spectrometry
- Heat transfer and fire control
- Aerosol and fine particle analysis
- Polymer synthesis

The interrlationship of the individual team member with the total program is shown in a network activity chart (Figure 5). An indication of the estimated man-power assignments to the individual phases and expected duration are presented. Although the chart represents the current estimates and assignments, it is possible that these may be altered and modified, with approval from NAFE^C in order to meet changes in project direction as the research proceeds.

The man-power estimate for the conduct of the research phases are outlined below. The total cost of the program is presented separately in Part II, Cost Estimate.

PERTINENT EXPERIENCE

The IIT Research Institute has a wide variety of experience applicable to the proposed program. Typical examples of related projects are given below:

NAS9-343, NASA Ames Research Center, "Thermochemical Characterization of Model Polymers".

DA-18-035-AMC-341(A) Edgewood Arsenal, "The Catalytic Activity of Metal Oxides on Thermal Decomposition Reactions".

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Man-Months Period



TOTAL PROJECT**

18.5 15

* f = fractional man-months **Rounded estimates and overlaps not included

Figure 5

NETWORK ACTIVITY CHART OF THE PROJECT TEAM

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AF33(615)-3688, AF Aero Propulsion Lab., "Feasibility Study to Increase Activity of Oxides with Oxygen and to Lower the Ignition Temperature Slurry Fuel Metals".

F33-615-67-C-1020, AF Aero Propulsion Lab., "Development of Batch Control Techniques for Synthetic Lubricants by Using Thermoanalysis".

DAAA21-67-C-0798, Picatinny Arsenal, "Mechanisms of Solid-State Catalytic Processes Related to Combustion Reactions".

F33-615-68-C-1092, AF Aero Propulsion Lab., "Development of Batch Control Techniques for Synthetic Lubricants".

14-01-0001-1445, Office of Saline Water, "Preparation of Reverse Osmosis Membranes by Surface Modification of Cellulose and Related Materials".

PH 43-67-108, National Institutes of Health, "Optimized Cellulose Membranes for Artificial Kidney Dialysis Applications".

950746, Jet Propulsion Laboratory "Stable White Coatings."

RP-47, Edison Electric Institute, "Polymers Designed for Extra High Voltage Applications".

NI78-7879, U.S. Naval Weapons Laboratory, "High Temperature Solid Propellants".

In-House IITRI Project Cl099, "Synthesis of High-Temperature Resistant Mixed Polycondensates Containing Carbonate Groups", was related to thesynthesis of new aromatic high temperature polymers, with particular reference to structures containing the carbonate group.

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Feasibility Study on the Use Of A Fiber-Optic Probe to Monitor the Uniformity of Pyrotechnic Mixtures DAAA 21-69-C-0343

In this program the composition of a two component powder mixture was determined from examination of the light reflected from the surface of the powder. High reproducibility was obtained and resolutions better than 1% were readily achieved.

Contrail Visibility Control Contract AF19(604)-1815

Nucleating chemicals were found which reduced the visibility of aircraft condensation trails. The principle involved was that of reducing the particle size of the condensate to the point where scattering of light was not detectable.

Aerosol Sampling and Analysis Contract No. N123(60530)33228A

This was a research study to provide information regarding the most efficient methods for gathering and recording data.

Investigation of Light Scattering in Highly Reflecting Pigmented Coatings Contract No. NASr 65(07)

An experimental and theoretical study of paint films was undertaken to define the controlling parameters which determine the suitability of a paint for thermal control use. A mathematical model was constructed which was used to predict several well known and recorded phenomena. The model was then used to show the role extenders play in enhansing the reflectivity of paints.

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Development of Space-Stable, Low Solar Absortance, Pigmented Thermal Control Coatings Contract No. NAS1-8166 (Langley)

The objectives in this program are to develop a general theoretical model of the optical degradation of materials exposed to the space environment, and to obtain experimental data on the effects of particle size on spectral reflectance. The general model actually consists of three sub-models, one which describes the interaction of space radiation with materials and predicts the rate at which defects are formed in it; a second in which the optical effect of these defects is calculated; and a third one in which the spectral reflectance change resulting from the induced absorption is calculated. The experimental work consists mainly of particle geometry characterization and both optical and ESR spectroscopy.

<u>Prototype Fly Ash Monitor For Incinerator Stacks</u> American Public Works Association

An automatic sampling instrument was developed and tested to determine the quantity of fly ash emitted by municipal incinerator smoke stacks. The instrument was capable of isokinetic and anisokentic sampling. The mass of the fly ash deposited on a filter was determined by the beta attenuation as measured by a geiger tube.

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Hypersonic Reconnaissance Investigations Contract No. AF 33(616)-6322

A study has been made of the deterioration in image quality that occurs when photographing through a turbulent boundary layer. Such a situation usually exists in aerial reconnaissance and is, in part, the reason why the photographic resolution in aerial photographs is much lower than that obtained when the aerial camera equipment is tested on the ground. Investigations of the effect of hypersonic, Mach 7, and supersonic, Mach 3, turbulent boundary layers on photographic image quality were made.

In conjunction with this work the change in refractive index of hot air due to dissociation and ionization was studied in a shock tube using an interferometric technique. This is of importance when photographing from a hypersonic vehicle through a shock wave since the refractive index of the shocked air will affect the photographic distortion.

A further effect considered was the reduction in contrast of an aerial photograph by radiation emitted from the shocked air surrounding a hypersonic vehicle. This radiation could be sufficiently intense to fog the photographic film. To study this effect the radiation emitted from a long hypersonic projectile was measured as a function of distance along its length at a ballistic range facility.

Coherence Limitations in Microdensitometry IITRI Sponsored

The objective of this in-house program was the analysis of optical coherence effects which are encountered in special analysis of photographic images when using very small sampling areas of the order of a few microns. When the effective sampling dimensions approach a few wavelengths of light being used one can no longer consider the case to be purely geometric. With different instruments and different settings the effects of coherence will vary and account for some of the lack of agreement

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found in the field. This work was aimed in particular at providing a better understanding of the characteristics of a recently purchased David Mann type 1140 Microdensitometer.

Image Analysis IITRI Sponsored

This project is a continuation of a previous in-house Project No. AllOO. This project is to develop a better understanding of the various ways which are used in the technical community for the analysis of photographic imagery. From this background we can better analyze the results of other workers in comparison to our work. In particular this program is still devoted largely to separating instrumental influences encountered in the analysis of micro spot sizes.

Studies of Cigarette Smoke Industrial Sponsored

Smoke from several brands of cigarettes was sampled and size and concentration determined. The extremely high concentration of particles involved considerable study of aerosol handling, and the volatile nature of the particles required development of special darkfield microscopy techniques. Rough size fractionation for chemical studies was accomplished with the use of specially constructed impinger.

Aerosol Sampling System Development Contract No. DA 42-007-AMC-139(R)

Design, development, and construction of a multipoint sampling system and information center was carried out under this contract. An instrument capable of automatic isokinetic sampling of aerosols with subsequent analysis of light scattering properties was developed. A remote read-out monitor with choice of time or flow controlled limits was included.

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Clean Room Particle Monitoring System NAS8-20531

A light scattering particle monitoring system was fabricated for use in a clean room complex. Three aerosol sampling sensing systems are multiplexed to a single data handling station. Particles in the size range 0.5 to 32μ will be analyzed at all three stations. Data are stored and printed in terms of station location, particle size distribution, and concentration.

Critical Evalution Of A Proposed Method For Assessing Paint Quality National Paint, Varnish & Lacquer Assn. & National Lumber Mfgrs. Assn.

The object of this program was to develop and validate a laboratory test method based on selected mechanical parameters describing the behavior of house paint with quality ratings established by conventional outdoor exposure tests.

<u>Wind Tunnel Experiments - Supersonic Vehicle</u> <u>Induced Turbulence Effects on Reconnaissance Photography</u> Contract No. AF 33(657)-11322

The objective of the work carried out under this contract was to measure the image degradation produced by the passage of the image forming light through a turbulent boundary layer. A large number experimental measurements were made through supersonic turbulent boundary layers at two different wind tunnel facilities under various flow conditions. A Mach number range of 1.5 to 5 and density altitudes from sea level to 138,000 ft. were covered.

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Both photoelectric and photographic measurements were made. In the former the distribution of intensity in the image of an edge or the line spread function were measured and the optical transfer function calculated by means of two computer programs. Alternatively, the transfer function could be measured directly. In the photographic measurements the image of an edge was recorded on SO243 film, and the optical density across the edge image was subsequently measured with a microdensitometer. Making use of the H & D curve and the computer program, the optical transfer function was calculated. Resolution chart data were also obtained.

Aerosoloscope Design and Fabrication Contract No. DA-18-064(CML 1904)

The program involved air handling desgin, electronic logic design, and optical design to detect and classify airborne particles in the size range of 0.5 to 60 microns diameter. Several instruments in this general category have been designed and constructed at IIT Research Institute for a number of government agencies as well as private industry.

Exhaust Control Device for Internal Combustion Engine Industrial Sponsored

A proprietary exhaust control device was evaluated for its capability to reduce particulate and gaseous emissions from a dynamometer mounted internal combustion engine.

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<u>Air-Fuel Mixing and Smoke Formation</u> Institutional Sponsor

The American Petroleum Institute sponsored this research investigation to determine quantitatively the effects of airfuel mixing and recirculation of product gases, to determine the advantages of recirculation of product gases, to define the physical requirements of systems employing optimum combinations of initial mixing and recirculation patterns and to establish the boundary conditions for smoke formation.

Controlling Gasoline Combustion in Diesel Engine by Variable Drop Size Contract No. DA-11-022-ORD-3658

A program sponsored by the Ordnance Tank-Automotive Command to study the feasibility of controlling the ignition lag of gasoline and diesel fuels in compression ignition engines. Based on available theoretical and experimental information, analysis was made to determine the required drop size variation for controlling ignition lag. Physical means of achieving the desired fuel droplet size were evaluated. The second phase of the program involved the actual design, construction of breadboard model, and testing in the laboratory.

Experimental Evaluation of Non-Catalytic Aferburners Industrial Sponsor

An investigation was conducted into the feasibility of reducing the hydrocarbon content of automotive exhaust gases by injecting air into the exhaust manifold. A valve-in-head six cylinder engine was equipped with an air injection system and analyses of exhaust gas samples collected during idle and deceleration. indicated substantial reduction in hydrocarbon and carbon monoxide content. Although this study did not progress beyond the laboratory stage, it is of interest to note that several years later, the General Motors air injection

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system and Ford's Thermactor system operate on the same principle. Development of a Natural Gas Combustion Chamber for Large Power Diesel Engines Industrial Sponsor

The conversion of a two-cycle dual fuel power plant engine to a high compression spark ignition gas engine was performed for the Fairbank Morse Company. Experimental work was conducted to generate data necessary to design a combustion chamber. The chamber was designed and the ignition system was modified for use on this engine. The sponsoring company now holds the patents on this combustion chamber and has used modifications of it on other production engines.

Stack Plume Evaluation Industrial Sponsor

A complete qualitative and quantitative analyses was performed on the stack plumes from several large ovens and other air samples for a major manufacturer. The gases involve several irritants, lacrimator and numerous other compounds with temperature and humidity and possible means of scrubbing are being investigated.

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A. Related Project Experience

1. Flammability Characteristics of Vehicle Interior Materials (National Highway Safety Bureau)

The basic objective of this program was to define the flammability characteristics of various categories of vehicle interior materials for the ultimate purpose of defining performance standards.

Statistical information was sought from police, fire and insurance agencies on the incidence of interior fires, and also on the existence of local or state standards pertaining to interior materials.

About 35 currently existing test methods for measuring the flammability of materials were studied, compared, and evaluated for their applicability to vehicle interior materials, and suitable methods were selected for use. More than 200 different interior materials, representing both domestic and some foreign makes of automobiles, were then tested to determine their relative flame spread characteristics.

> 2. <u>Fire Hazard of Combustible Building Materials</u> (Society of the Plastics Industry)

The main goal of this project is to develop a system for evaluating the fire hazard of building finish materials (wall or ceiling covering, plastic light diffusers, etc.). To accomplish this, a number of experiments were conducted to evaluate the effects of these materials on the severity and hazards of actual fires. For this purpose, a three-room structure (two 10 x 15 ft and one 20 x 30 ft room) leading to a 55 ft corridor is used. The experiments involve fires with and without the combustible building finish materials found in normal occupancies and provide guidelines regarding the appropriateness of existing methods and rating systems. This is a continuing program.

3. <u>Development and Evaluation of Test Procedures for</u> Carpeting (American Carpet Institute)

Recent concern over the possible contribution of carpeting to the over-all fire hazard has led to an investigation of potential test methods for discerning differences in the fire behavior of floor-covering materials, and relating these to the observed behavior in real fires. The developed procedure places the sample in its normal orientation and exposes one end to both a radiant source and a piloting flame.

4. Burning Characteristics of Acoustical Material

The program dealt with the determination of the ignition and burning characteristics of solid materials such as acoustical and insulation materials when used as surface treatments in building construction.

5. <u>Fire Spread from Kindling Fuels</u> (Defense Atomic Support Agency)

This investigation was designed to answer the question: What are the requirements of a kindling material to cause sustained combustion of a thick fuel?

Experiments were conducted using both interior and exterior kindling fuels -- the interior fuels mainly from the standpoint of time necessary to produce a "flashover"; the exterior fuels for their ability to ignite nearby structural components.

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CONCLUSION

A thermoanalytical approach has been proposed to investigate the physio-chemical properties of smoke and toxic vapors released from synthetic aircraft cabin materials during combustion. The utilization of thermoanalysis in conjunction with other analytical methods will permit the examination of many more materials and physio-chemical parameters with respect to fire resistance and release of toxic products than would be possible with other approaches.

Correlation of the results with functional groups and the structure of polymers should lead to new formulations which are inherently more flame resistant as well as less smoke and toxic products producing during fire. Based on our extensive experience in combustion research, thermally stable polymers and fire, we are confident of the success of this project.

> Respectfully submitted, IIT RESEARCH INSTITUTE

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Winfried K. Rudloff Research Chemist

Approved by: Eli S. Fréeman

Assistant Director Chemistry Research

WKR:aw

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WINFRIED RUDLOFF Research Chemist, Physical Chemistry Research

B.S., Physical Chemistry, Universitaet Heidelberg, 1957. M.S., Physical Chemistry, Universitaet Heidelberg, 1959. Ph.D., Physical Chemistry, Illinois Institute of Technology, 1965.

Special Interests

Physical chemistry of diffusion and sorption phenomena Physics and chemistry of the solid state Mossbauer spectroscopy Defect structure in correlation to catalysis in condensed phases Physical chemistry of high temperature reactions Thermal analysis Quantum mechanics and computer applications Nuclear physics and related subjects Statistical mechanics Physical chemistry of meteorological phenomena

Experience

Badische Anilin und Soda Fabrik, Ludwigshafen, Germany: chemical technician, 1957.

- Physikalish-Chemisches Institut der Universitaet Heidelberg, Germany: research assistant, 1958-1959.
- Illinois Institute of Technology: teaching assistant, 1959-1960; research assistant, 1960-1963.
- IIT Research Institute: associate chemist, 1964-1967, research chemist, 1967-present.

Professional Affiliations

Gesellschaft Deutscher Chemiker American Chemical Society Phi Lambda Upsilon, Honorary Member American Association for the Advancement of Science

Publications

- "Ueber die Association von Methylamin in Loesungen mit n-Hexan," W. Rudloff, Diplomarbeit, Heidelberg, 1959.
- "Rapid Sorption Processes in Granular Zeolites," W. W Brandt and W. Rudloff, J. Phys. Chem. Solids, <u>25</u>, 167, 1964.

WINFRIED RUDLOFF (cont.)

- 3. "Comprehensive Tables of the Solution of the Diffusion Equation with Changing Boundary Conditions", W. Rudloff and W. W. Brandt, American Documentation Institute, Washington, D.C., No. 7844
- 4. "Gaseous Diffusion in a Natural Zeolite in Relation to Crystalline Disorder", W. W. Brandt and W. Rudloff, Zeitschr. Phys. Chem. (Frankfurt), <u>42</u>, 201, 1964
- 5. "Diffusion of Ethane in Modified Synthetic Zeolites", W. W. Brandt and W. Rudloff, J. Phys. Chem. Solids, <u>26</u>, 741, 1965
- "The Catalytic Effects of Oxides on the Decomposition of Potassium Chlorate", W. Rudloff and E. S. Freeman, Phys. Chem. Section, ACS-Meeting, Atlantic City, Sept. 1965
- 7 "Investigations of the Decomposition-Oxidation Reaction of Cobalt Oxides in Correlation to Electronic Defect Structure," W. Rudloff and E. S. Freeman, Phys. Chem. Section, ACS-Meeting Pittsburgh, March 1966
- 8. "Semiconductivity of Iron Oxides in Correlation to Their Catalytic Activity with Respect to the Decomposition of Potassium Chlorate", W. Rudloff and E. S. Freeman, Phys. Chem. Section, ACS-Meeting New York, Sept. 1966
- 9. "Ethane Diffusion in Ion-Exchanged Synthetic Zeolites of Type A containing Water", W. Rudloff and W. W. Brandt, J. Phys. Chem. <u>71</u>, 3689, 1967
- 10. "The Concentration Dependence of Zeolite Sorption", W. W. Brandt and W. Rudloff, J. Phys. Chem. <u>71</u>, 3948, 1967
- 11. "Thermal Analysis of Inorganic Chlorates and Perchlorates", chapter in "Treatise on Differential Thermal Analysis", E. S. Freeman and W. Rudloff, Acad. Press Inc. (London), Limited Publishers in Press
- 12. "The Catalytic Effect of Metal Oxides on Thermal Decomposition Reaction. I. The Mechanism of the Molten-Phase Thermal Decomposition of Potassium Chlorate in Mixtures with Potassium Chloride and Potassium Perchlorate", W. K. Rudloff and E. S. Freeman, J. Phys. Chem., in Press
- 13. "Thermal Decomposition, Surface Interaction with Gases, and Electrical Properties of Lead Oxides", W. K. Rudloff, E. S. Freeman and M. E. Henry, Phys. Chem. Section, ACS Meeting, Atlantic City, September, 1968.

WINFRIED RUDLOFF (cont.)

- 14. "The Catalytic Effect of Metal Oxides on Thermal Decomposition Reactions. II. The Catalytic Effect of Metal Oxides on the Thermal Decomposition of Potassium Chlorate and Potassium Perchlorate as Detected by Thermal Analysis Methods," W. K. Rudloff and E. S. Freeman, J. Phys. Chem. Submitted for publication.
- "The Reaction of Sulfur Vapor with Potassium Chlorate,"
 W. K. Rudloff, E. S. Freeman and B. Boldt, Phys. Chem. Section, ACS Meeting, Minneapolis, April 1969.
- 16. "Mossbauer Effect Spectroscopy as a Probe for Surface Interactions", 20th Annual Mid-America Symposium on Spectroscopy, Chicago, May 1969; to be published in <u>Developments in Applied Spectroscopy Vol. 8</u>, E. L. Grove, Ed., Plenum Press, New York.
- 17. "Application of the Mossbauer Effect to Studies in Gas Adsorption," W. K. Rudloff, to be published in <u>Proceedings</u> of the First International Conference on Calorimetry and <u>Thermodynamics</u>, Warsaw, 1969.

ELI S. FREEMAN Assistant Director, Chemical Sciences Division

B.S., chemistry, Brooklyn College, 1950. M.A., physical Chemistry, Brooklyn College, 1952. Ph.D., physical Chemistry, Rutgers University, 1960.

Special Interests

Chemical and physical effects of high-energy irradiation Corrosion kinetics Differential thermal analysis Oxidation of reactive metals at high temperatures Physical inorganic chemistry Polymer degradation Preignition reactions Pyrotechnics Radiation effects on inorganic substances Reaction kinetics - theoretical and experimental Thermoanalytical reaction kinetics

<u>Experience</u>

Picatinny Arsenal: Pyrotechnics Laboratory, research chemist, 1952-1955.

Rutgers University: research fellow, 1955-1956; instructor in physics, 1956-1957.

Picatinny Arsenal: Pyrotechnics Laboratory, group leader, 1957-1960; chief, Basic Chemical Research, and specialist in physical chemistry, 1960-1964.

IIT Research Institute: manager of Physical Chemistry Research, 1964-1966; assistant director, Chemical Sciences Division, 1966-present.

Professional Affiliations

American Chemical Society ASTM Sigma Xi

Publications

- "The Behavior of Colloidal Silicate as Revealed by Absorption Indicators," B. Carroll and E. S. Freeman, J. Phys. Chem., 58, 335, 1954.
- "The Kinetics of the Underwater Corrosion of Powdered Mg,"
 E. S. Freeman and S. Gordon, J. Phys. Chem., <u>39</u>, 1009, 1955.
- "The Reaction between Hg and NO₂," E. S. Freeman and S. Gordon, J. Am. Chem. Soc., <u>78</u>, 1913, 1955.

ELI S. FREEMAN (cont.)

- 4. "The Application of the Absolute Rate Theory to the Ignition of Propagatively Reacting Systems. The Thermal Ignition of the Systems Lithium Nitrate-Magnesium, Sodium Nitrate-Magnesium," E. S. Freeman and S. Gordon, J. Phys. Chem., <u>60</u>, 867, 1956.
- "The Kinetics of the Thermal Decomposition of Sodium Nitrate and of the Reaction between Sodium Nitrate and Oxygen,"
 E. S. Freeman, J. Phys. Chem., <u>60</u>, 1487, 1956.
- 6. "The Kinetics of the Thermal Decomposition of Potassium Nitrate and of the Reaction between Potassium Nitrate and Oxygen," E. S. Freeman, J. Phys. Chem., <u>61</u>, 838, 1957.
- 7. "The Application of Thermoanalytical Techniques to Reaction Kinetics. The Thermogravimetric Evaluation of the Kinetics of the Decomposition of $CaC_2O_4H_2O$." E. S. Freeman and B. Carroll, J. Phys. Chem., <u>62</u>, 393, 1958
- B. "The Rouy Method of Photoelectric Polarimetry," B. Carroll,
 B. Tilen, and E. S. Freeman, Anal. Chem., <u>30</u>, 1099, 1958.
- 9. "The Effects of X-Radiation on NH₄ClO₄ as Investigated by Thermogravimetry and Differential Thermal Analysis," E. S. Freeman and D. Anderson, J. Phys. Chem., <u>63</u>, 1344, 1959.
- 10. "A Simple Method for Derivative Differential Thermal Analysis," E. S. Freeman, Anal. Chem., <u>31</u>, 1959,
- 11. "The Kinetics of the Thermal Degradation of the Synthetic Polyester, Laminac 4116," E. S. Freeman, J. Appl. Polymer Sci., 1, 192, 1959.
- 12. "The Characterization of Saturated Polyesters by Differential Thermal Analysis," E. S. Freeman, Anal, Chem., <u>31</u>, 1697, 1959.
- 13. "The Effects of X-Ray and Gamma Ray Irradiation on the Thermal Decomposition of Ammonium Perchlorate in the Solid State," E. S. Freeman and D. S. Anderson, J. Phys. Chem., 64, 1727, 1960.
- 14. "The Kinetics of the Thermal Degradation of Polystyrene and Polyethylene," E. S. Freeman and D. A. Anderson, J. Polymer Sci., <u>54</u>, 253, 1961.
- 15. "Electron Spin Resonance Observations of NH⁴ Formed by X-Ray Irradiation of Ammonium Perchlorate Crystals," J Hyde and E. S. Freeman, J. Phys. Chem., <u>65</u>, 1636, 1961.

ELI S. FREEMAN (cont.)

- 16. "Observation on the Decomposition of X-Ray Irradiated Ammonium Perchlorate," E. S. Freeman and D. A. Anderson, J. Phys. Chem., <u>65</u>, 1662, 1961.
- "The Kinetic Order of the Reaction between Sodium Nitrate and Oxygen," D. A. Anderson and E. S. Freeman, J. Phys. Chem., <u>65</u>, 1648, 1961.
- 18. "The Effects of High Energy Radiation on the Chemical Reactivity of Ammonium Perchlorate," E. S. Freeman, Proceedings of JANAF Solid Propellants Panel, 1962.
- 19. "X-Ray Effects on Ammonium Perchlorate Decomposition," E. S. Freeman, Chem. Eng. News, Oct. 30, 1961.
- 20. "Effects of High Energy Radiation on the Chemical Reactivity of Ammonium Perchlorate," E. S. Freeman, article in "Encyclopedia of X-Rays and Gamma Rays," Reinhold Publishing Co., New York, p. 56, 1963.
- 21. "The Kinetics of the High Temperature Reaction between Magnesium Powder and Oxygen," E. S. Freeman and C. Campbell, Trans. Faraday Soc., <u>59</u>, 165, 1963.
- 22. "Differential Thermal Analysis of Potassium Perchlorate," D. A. Anderson and E. S. Freeman, Nature, <u>195</u>, 1297, 1962.
- "Physical Transitions of the Alkali Metal Nitrates as Revealed by Differential Thermal Analysis," E. S. Freeman and D. A. Anderson, Nature, <u>199</u>, 63, 1963.
- 24. "The Effects of High Energy Radiation on the Chemical Reactivity of Ammonium Perchlorate," E. S. Freeman and D. A. Anderson, Proceedings of the June 1963 ASTM Symposium on the Chemical and Physical Effects of High Energy Radiation on Inorganic Substances, May 1964.
- 25. "Electrical Conductivity of Ammonium Perchlorate," P. Zirkind and E. S. Freeman, Nature, <u>199</u>, 1280, 1963.
- 26. "Thermoanalysis of Some Inorganic Fluorides and Silicofluorides," E. S. Freeman and V. D. Hogan, Anal. Chem., 36, 2337, 1964.
- 27. "The Effect of High Energy Radiation on the Rate of Reaction between Magnesium Powder and Oxygen," C. Campbell and E. S. Freeman, J. Chem. Phys., <u>41</u>, 2565, 1965.

ELI S. FREEMAN (cont.)

38. "Methods of Investigation of Radiation Effects on Polymers", P. Feng and E. S. Freeman, Physical Methods of Macromolecular Chemistry, Vol. 1, Chapter 4, Dekker Press, 1969.

IT RESEARCH INSTITUTE

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GIORI, CLAUDIO Research Chemist, Polymer Research

Ph.D., Polymer Chemistry, University of Milano, Italy, 1962, thesis under Prof. G. Natta.

SPECIAL INTERESTS

Condensation polymers Polymerization kinetics Ring-opening polymerization Degradation mechanisms Polymer stabilization Polymerization catalysis Stereospecific polymerization Phase equilibria Melt viscosity measurements

EXPERIENCE

ENI Group, Polymer Research Laboratories Milano, Italy; Research Chemist: 1963-1965.

Fibers Division, Polymer Research Group Allied Chemical, Petersburg, Virginia; Research Chemist: 1965-1967; Group Leader: 1968.

IIT Research Institute; Research Chemist: 1968-Present

PROFESSIONAL AFFILIATIONS

American Chemical Society

PUBLICATIONS

Giori, C. and Hayes, B.T., "Polymerization of Caprolactam. I. Hydrolysis-Polycondensation Kinetics. II. Vapor-Liquid Equilibria," J. Polymer Sci., to be published.

TALKS

Giori, C. and Hayes, B.T., "Hydrolytic Polymerization of Caprolactam. Some Kinetic Studies," Presented at the Fiber Society Meeting, Princeton, N.J., Sept. 7, 1967.

ALEXANDER GOLDSMITH

Senior Research Engineer, Heat and Mass Transfer

EDUCATION

B.S. in Mechanical Engineering, IIT, 1949 M.S. in Mechanical Engineering, IIT, 1954

SPECIAL INTERESTS

Flammability of materials Thermomedical enginessing Experimental heat transfer Thermophysical properties of materials Heating refrigeration, and a r-conditioning

EXPERIENCE

IITRI, Senior Research Engineer, 1949-present

Except for a short period in privat industry during 1954-1955, Mr. Goldsmith has been with the IIT Research Institute (and its predecessor, the Armour Research Foundation) since 1949. At the present time, he is completing a program on the flammability characteristics of vehicle interior materials for the National Highway Safety Bureau.

His primary experience over the years has been in the field of heat transfer, and he has worked on both analytical and experimental programs. During this period he has contributed toward the successful completion of many projects, whose variety is indicated by the following examples: Analytical and experimental problems of aerodynamic heating dealing with the prediction of remperatures in exposed bodies, and the selection and design of protective systems; design of apparatus, and measurement of thermal properties of materials from -100°F to over 3000°F; prediction of the heat gained by materials exposed to a nuclear explosion; development of Ranque-Hilsch vortex tube as a free-air therm cer

ditions typecal explained and the following, disign of alor iter room to dormance estim of heating a cooling product dispersion of sections and special is lity for evaluations and wall subling daracterist of bound have and subdicy conditions in underground soulders; calibration of

ALEXANDER GOLDSMITH (Continued)

EXPERIENCE

ceiling-type air diffusers, and other projects. Mr. Goldsmith is the principal author of a five-volume Handbook of Thermophysical Properties as identified below.

PROFESSIONAL AFFILIATIONS

Associate Member, American Society of Mechanical Engineers Associate Member, Sigma Xi

PUBLICATIONS

"Handbook of Thermophysical Properties of Solid Materials", Vol. I-V, The Macmillan Co., New York, 1961, (also published as WADC Tech. Rept. 58-476, Revised, 1960).

"Methods and Procedures for the Collection, Evaluation and Consolidation of Thermophysical Properties of Materials", WADD Tech. Rept. 60-812, Jan. 1961.

"Design of Vortex Free-Air Thermometer", WADC Tech. Rept. 57-715, ASTIA Doc. No. AD 142 178, Feb. 1957.

"Investigation of Means and Materials to Combat Thermal Radiation Flash Burns", ASTIA Doc. No. AD 127 857, Jan. 1957.

"Experimental Evaluation of Simulated Heat and Moisture Loads in Shelters", Office of Civil Defense, Work Unit 1211B, Dec. 1965.

"Collation of Power Plant Studies", Maritime Administration, U. S. Dept. of Commerce, Contr. No. MA-3974, Feb. 1967. ERIC AYNSLEY Research Chemical Engineer Fine Particles Research

Ph.D. Chemical Engineering, University of Birmingham, England, 1965.

Special Interests

Air Pollution Inter-particle forces Aerosols Multiphase flow systems

Experience

Consett Iron Company, Ltd., Chemical Engineer, 1962-1966 University of Newcastle-on-Tyne, Senior Research Officer, 1966-1968 IIT Research Institute, Research Chemical Engineer, October 1968 to present

Dr. Aynsley is currently concerned with air pollution monitoring surveying and control techniques. He has had experiences of industrial gas cleaning equipment in the steel and power generation industries. His Ph.D thesis concerned filtration of airborn particules and assessment of particulate properties affecting filtration efficiency and particle agglomerating tendencies. His batchelor's thesis was a survey and appraisal of testing techniques for gas cleaning equipment.

Publications

- "Collection of Airborne Dusts", Part I, Bottrill and Aynsley, British Chemical Engineering, <u>12</u>, 527-530, 1967.
- "Collection of Airbore Dusts", Part II, Bottrill and Aynsley, British Chemical Engineering, <u>12</u>,633-637, 1967.
- 3. "The Electrical Conductivity of Pulverized Fuel Flames", Aynsley, Fells, and Lowes, 4th Int. Con. on M.H.D. Power Generation, Warsaw, Poland, July 1968.
- 4. "Pressure Drop of Two-Phase Air-Water Mixtures in Vertical, Inclined, and Horizontal Pipes", Aynsley.

Part I: Inclination to Vertical: o°, 10°, 20°.

Part II: Inclination to Vertical: 30°, 40°, 50°, 60°

ERIC AYNSLEY (cont)

Part III: Inclination to Vertical: 70°, 80°, 90°.

Int. Symp. on Concurrent Two-Phase Gas-Liquid Flow, University of Waterloo, Waterloo, Ontario Canada, September 18-19, 1968.

- 5. "A Combined Conductivity-Temperature Probe for Use up to 3000°K, J. Meas. and Control, <u>1</u> 285-6, 1968.
- 6. "Hydrodynamics of Droplets in Liquid-Liquid Systems' Aynsley, Liddell and Thornton, Trans. Instn. Chem. Engrs. London, in press.

KURT GUTFREUND Senior Chemist, Polymer Research

B.S., chemistry, University of Buffalo, 1950. M.S., physical chemistry, University of Wisconsin, 1952.

<u>Special Interests</u>

Surface chemistry Rheology Mechanical properties of high polymers Interfacial processes Reaction kinetics Light scattering

Experience

University of Wisconsin: research assistant, 1950-1952. Visking Corporation: assistant chemist, 1952-1954. Dixie Division, American Can Co.: physicist, 1954-1957 IIT Research Institute: associate chemist, 1957-1958; research chemist, 1958-1965; senior chemist, 1965-present.

Professional Affiliations

American Chemical Society Society of Rheology American Society for Testing Materials American Association for the Advancement of Science

Publications

- "Conversion of Fibrinogen to Fibrin: Light Scattering Studies on Bovine Fibrinogen," S. Katz, K. Gutfreund, S. Shulman, and J. D. Ferry, J. Am. Chem. Soc., <u>74</u>, 5706, 1952.
- "Light Scattering Studies on Clotting Systems Inhibited by Hexamethylene Glycol," J. D. Ferry, S. Shulman, K. Gutfreund, and S. Katz, J. Am. Chem. Soc., <u>74</u>, 5709, 1952.
- 3. "Conversion of Fibrinogen to Fibrin: Effect of Calcium on the Formation and Dissociation of Intermediate Polymers," S. Katz. S. Shulman, K. Gutfreund, I. Tinoco, I. H. Billick, and J. D. Ferry, Arch. Biochem. Biophys., <u>47</u>, 165, 1953.

KURT GUTFREUND (cont.)

- 4. "Study of Interface Relationships in Glass-Reinforced Plastics Systems by Sorption Methods," K. Gutfreund, C. Brown, and H. S. Weber, Proceedings, 15th Conf. Reinf. Plastics Div. S.P.I., 10C, 1960.
- "Emulsion Floor Polymer Films," K. Gutfreund, Soap Chem. Specialties, <u>36</u>, 115, 1960.
- 6. "Interaction of Organic Monomers with Fiberglass Surfaces," K. Gutfreund and H. S. Weber, Proceedings, 16th Conf. Reinf. Plastics Div. S.P.I., 8-C, 1961.
- 7. "Interaction of Organic Monomers and Water with Fiberglass," K. Gutfreund and H. S. Weber, SPE J., <u>1</u>, 191, 1961.
- 8. "Instrumental Techniques Applied to Paint Film Deterioration," K. Gutfreund, National Paint, Varnish and Lacquer Association Circular No. 791, pp. 1-60. Feb. 1964.
- 9. "Study of the Deterioration of Paint Films by Measurement of Their Mechanical Properties," K. Gutfreund, National Paint, Varnish and Lacquer Association Circular No. 793, pp. 1-59, Mar. 1965.
- 10. "Interfacial Relationships in Composite Materials." K. Gutfreund, Chapter 6 in "Metallurgy and Materials," Addison-Wesley Publishing Co., Inc., 1967.
- 11. "Critical Evaluation of a Proposed Method for Assessing Paint Quality," K. Gutfraund, National Paint, Varnish and Lacquer Circular No. 794, pp. 1-43, Jun. 1966.
- 12. "Study of Interfacial Relationships in Boron Fiber-Reinforced Plastics by Mechanical and Chemical Methods," J. L. Broutman and K. Gutfreund, Proc. Symposium on Advanced Composite Hardware, Washington, Sept. 21, 1966.
- "Interfacial Investigations of Boron Fiber-Reinforced Plastics," K. Gutfreund, L. J. Broutman, and E. H. Jaffe. Trans. 10th Annual SAMPE Meeting, Nov. 10, 1966.
- 14. "Determination of the Susceptibility of Faint Films to Deterioration." K. Gutfreund, J. Paint Technol., <u>38</u>, Dec. 1966.
- 15. "Investigations of Interfacial Conditions in Boron Fiber-Reinforced Plastics", K. Gutfreund, D. Kutscha and L. J. Broutman, Proceedings 23rd Conf. Reinf. Plastics Div. S. P. I., 16 C, 1968.

KURT GUTFREUND (cont.)

16. "Investigation of Potentially Useful Products Derived from Gluten Hydrolysate", K. Gutfreund, and T. Yamauchi, to be published in J. Agr. Food Chem.

ELI S FREEMAN (cont.)

- "Effect of Radiation on the Electrical Conductivity of Magnesium Oxice," P. Zirkind and E. S. Freeman, J. Chem. Phys., 41, 906, 1964.
- 29. "Effects of Changes in Chemical Reactivity and Decomposition Characteristics of Potassium Perchlorate on the Thermal Ignition of Magnesium-Potassium Perchlorate Mixtures," V D Hogan and E. S. Freeman, Combust. Flame, 9, 19, 1965.
- 30. "Effects of Radiation and Doping on the Catalytic Activity of Magnesium Oxide on the Thermal Decomposition of Potassium Perchlorate," E. S. Freeman and D. A. Anderson, Nature, 206, 378, 1965.
- 31 "The Effects of Doping and of Oxides on the Decomposition Characteristics of Potassium Perchlorate," E. S. Freeman and D. A. Anderson, J. Inorg. Nucl. Chem., 9, 19, 1965.
- 32. "The Effects of Aluminum Fluoride on Combustion of Aluminum in the Presence of Potassium Perchlorate and Oxygen," E S Freeman and D. A. Anderson, Combustion and Flame, 10, 4, 1966
- 33. "Stability of Methyl Radicals Trapped in a Zeolite Matrix." E. S. Freeman, G. A. Noble, R. A. Serway, A. O'Donnell, J. Phys. Chem., <u>71</u>, 4326 (1967).
- 34. "Application of Thermal Analysis Techniques to Criminalistics"
 E. S. Freeman and A. Becker, Law Enforcement Science and Technology, Thompson Book Co./Academic Press, p. 311, 1967.
- 35. "The Thermal Degradation of Nadic Methyl Anhydride Cross-Linked Novolac Epoxy Resin," E. S. Freeman and A. Becker, J. Polymer Science, A-1, 6, 2829 (1968).
- 36 "Thermoanalysis Studies of Inorganic Chlorates and Perchlorates," E. S. Freeman and W. Rudloff, Differential Thermal Analysis, Academic Press, In Press, (1968).
- 37. "The Catalytic Effect of Metal Oxides on Thermal-Decomposition Reactions I. The Mechanism of the Molten-Phase Thermal Decomposition of Potassium Chlorate and of Potassium Chlorate in Mixtures with Potassium Chloride and Potassium Perchlorate", E. S. Freeman and W. Rudloff, Journal of Physical Chemistry, In press (1968).

References

- Salzberg, F., Takata, A. and Labes, W. G., "Prediction of Fire Damage to Installations and Built-up Areas from Nuclear Weapons," Defense Atomic Support Agency, Contract No. DA-49-149-XZ-021, June 1960.
- Graf, S. H., "Ignition Temperature of Various Papers, Woods and Fabrics", Engineering Experiment Station, Oregon State College Bulletin No. 26, Mar. 1949.
- 3. Brown, F. L., "Theories of the Combustion of Wood and Its Control - A Survey of Literature," U.S. Department of Agriculture Report No. 2126, Forest Service, Forest Products Laboratory, Dec. 1958 (10 tables, 140 references, 49 pages).
- Wittich, F. N., "Respiratory Tract Allergic Effects from Chemicals and Air Pollution," Air Pollution Proceedings of U.S. Tech, Cinf. on Air Pollution, McGraw-Hill Co., 1952.
- 5. Information Circular 7718, Ringelmann Smoke Chart", Bureau of Mines. Middleton, W. E. Knowles, "Vision Through the Atmosphere," University of Toronto Press, Canada, 1952.
- "Fire Gas Research Report," NFPA Committee on Fire Gas Research, NFPA Quarterly, Jan. 1952, V. 45, n. 3, p. 280-306.
- Dufour, R. E., "Survey of Available Information on the Toxicity of the Combustion and Thermal Decomposition Products of Certain Building Materials under Fire Conditions", Underwriters Laboratories, Inc., Bulletin of Research No. 53, July 1963.
- 8. Bieberdorf, F. W. and Yuill, A. H., "An Investigation of the Hazards of Combustion Products in Building Fires," Southwest Research Institute, Oct. 1963.
- 9. Marcy, John F. and R. Johnson, "Flaming and Self-extinguishing Characteristics of Aircraft Cabin Interior Materials", Report No. NA-68-30 (DS-68-13), Nat. Aviat. Facilities Experimental Center, Fed. Aviation Agency, July, 1968.
- 10. Interim Federal Standard No. 00136b (COM-NBS) Dec. 26, 1962.
- Rohm and Hass Co. "A Method of Measuring Smoke Density," NFPA Quarterly, Vol. 57, No. 3, Jan. 1964.
- Gross, D. and T. T. Loftus, "A Laboratory Test for Measuring Smoke from Burning Materials". NBS Report 8947, July 29, 1965.