



DYNATECH CORPORATION

**PHYSIO-CHEMICAL STUDY OF THE FLAMMABILITY OF
FIRE-RESISTANT MATERIALS FOR AIRCRAFT CABINS**

**- TECHNICAL PROPOSAL -
PCH-190**

**Submitted to:
NATIONAL AVIATION FACILITIES EXPERIMENTAL CENTER
Atlantic City, New Jersey**

**In Response to:
Request for Proposal NAOO-9-12**

June 25, 1969

Progress through Research

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PERSONS AUTHORIZED
TO CONDUCT NEGOTIATIONS

Kenneth D. Roberts, Vice President and Treasurer

John B. Eliot, Controller

F. Robert Johnson, Manager, Government Marketing Department

Section 1

INTRODUCTION

An important field of aircraft safety engineering is the development and utilization of fire resistant and low smoke producing materials of construction. Interior materials of aircraft such as seats, cushions, curtains, paneling, and carpeting have received increasing attention because of a series of crash survivable accidents in late 1965 and early 1966 that revealed the contribution of such materials to loss of life through fire, smoke, and gases. In recent years fire-control engineers concerned with both the construction and transportation fields have learned that smoke from smoldering or burning materials poses as great a threat to life as flames or heat generated in a fire (References 1 and 2). During the past 2 years articles appearing in British and American fire journals suggested that, except for clothing fires, more than half the deaths due to fire are caused by smoke rather than by heat or flames. Accordingly, the National Aviation Facilities Experimental Center, Atlantic City, New Jersey, has issued Request for Proposal NAOO-9-12 outlining the need for a physio-chemical study of the factors affecting the emission of smoke during pyrolysis and the need for screening and developing high temperature stable polymers which emit minimum quantities of smoke.

Based on the experience of Dynatech engineers in plastic and polymer technology and in the development of flame retardant treatments a program in response to RFP NAOO-9-12 has been prepared. The following sections of this proposal outline a specific program:

1. for determining the physio-chemical nature of materials such as molecular structure, composition, inhibitor additives, degradation kinetics, etc., that cause emission of smoke when materials are thermally decomposed as in a typical fire.
2. for conducting a search to identify high-temperature stable polymers potentially useful as cabin replacement materials which inherently emit minimum quantities of smoke (i.e., D_s less than 16 for 90 seconds fire exposure).

3. for developing new additive polymer formulations of polyurethane foam, acrylonitrile-butadiene-styrene, acrylic resins, and vinyl resins, and testing these samples for flammability (by Test Method 5902), for critical optical density D_g 16 (by a modified NBS smoke chamber), and for toxicity (by colorimetrically monitoring CO, HCN, HCl, and HBr).

Dynatech R/D Company has the staff, the association with polymer and pyrolysis experts at the Massachusetts Institute of Technology, and the facilities for conducting a comprehensive investigation into the mechanisms of smoke emission and for formulating new flame and smoke retardant systems. The basic analytical facilities for a study of the molecular aspects of smoke generation, including thermal gravimetric analysis (TGA) and differential thermal analysis (DTA) instruments, are maintained at Dynatech while gas chromatographic, mass spectrometric, and atomic absorption instruments are available in the immediate vicinity. The staff of the Chemical Engineering Department has had a combined total of over 80 years of experience in formulating polymer systems for specialty and exacting applications. Of particular pertinence is a flameproof coating for flammable polymers that was recently developed by the Chemical Engineering Department for NASA-MSC under contract NAS-9-8179. This coating is notable not only because it enables certain polymers to pass the extremely severe silicone igniter test (conducted in pure oxygen at 6.2 psig), but also because it is formulated from an exceptional class of flame retardants that have proved to produce very little smoke when combustion does occur. The experience and techniques developed in meeting the technical requirements for a flameproofing coating are directly applicable to the objectives of the program requested by the National Aviation Facilities Experimental Center.

In addition to the required technical background and facilities necessary for the conduct of the program, Dynatech can point to a record of substantial accomplishment in research and development project management and financial and accounting capability. Dynatech R/D Company has completed more than a thousand projects for both industrial and government clients, establishing an organization skilled in efficient and effective financial and technical management.

Section 2

TECHNICAL DISCUSSION

A. Background

Many polymers have been developed that have exhibited a functionality, ease of fabrication, and low cost that made them highly desirable in applications such as structural components, adhesives, flexible sealants, foams, paneling, and fibers. However, serious fires involving these materials have created much concern over the possible role plastics may have played in these conflagrations. Consequently, more attention is being given to the amount of smoke and toxic fumes as well as the flames that are evolved during combustion of polymers.

Investigations have led to the general agreement that smokes from smoldering or burning materials present three kinds of hazards:

1. They are irritating to the eyes and respiratory system.
2. If dense, they will impede the escape from burning locations and make difficult the entrance of fire-fighters and rescue teams.
3. They are toxic, either directly--by virtue of the components they contain--or indirectly by reducing the quantity of oxygen in the environment.

B. Determination of Smoke Evolution Mechanisms

In the burning process heat causes a material to decompose to solid fragments, liquid distillate, and a gaseous fraction. These decomposition products are then carried out to the flame front where they undergo further oxidation and cracking until they are emitted and cooled.

Thus, smokes are basically a two phase system--a dispersed phase consisting of minute light-scattering particles and a continuous vapor phase. The particles consist of both solid fragments and liquid drops formed by the condensation of the gaseous products of thermal decomposition. The constituents of the vapor phase vary depending upon the material undergoing decomposition, but the predominating constituents are usually H_2O , CO_2 , and CO .

To determine the mechanism or mechanisms by which smoke is evolved, therefore, it is necessary to study both the polymer phase from which the initial

products are generated and the smoke phase that contains the oxidized and cracked products of the further reactions undergone by the initial decomposition species. The mechanism of thermal degradation of organic polymers, of which smoke evolution is a part, may be described as a function of the following three parameters:

- 1) The change of molecular weight of the polymer as a function of temperature and extent of degradation.
- 2) The qualitative and quantitative composition of the volatile and non-volatile pyrolysis products.
- 3) The rates and activation energies of the degradation process.

The techniques used to monitor these parameters are:

- 1) For molecular weight--The measurement of the rate of change of polymer viscosity as a function of conversion by evaluating the viscosity of the polymer residue in the molten state.
- 2a) For qualitative-quantitative composition of the volatile products--flash pyrolysis, hot-wire pyrolysis, or programmed decomposition followed by an effluent gas analysis by one or more of the following techniques:
 - 1) Vapor-phase Chromatography (VPC)
 - 2) Mass Spectrometry
 - 3) Infrared Spectrophotometry
 - 4) Electron Spin Resonance

Often a combination of techniques has proved valuable. For example, particularly fine measurements may be made by utilizing a 500 ft. capillary column (0.02 in) coated with SF 96 (50) (Applied Science Laboratories, State College, Penn.) in VPC and coupling the output from the chromatograph to a time-of-flight mass spectrometer (Ref. 3).

In programmed decompositions, the temperature is increased at a predetermined rate and the products analyzed as they are evolved. Such analysis studies can be conducted simultaneously with differential thermal of thermogravimetric analysis.

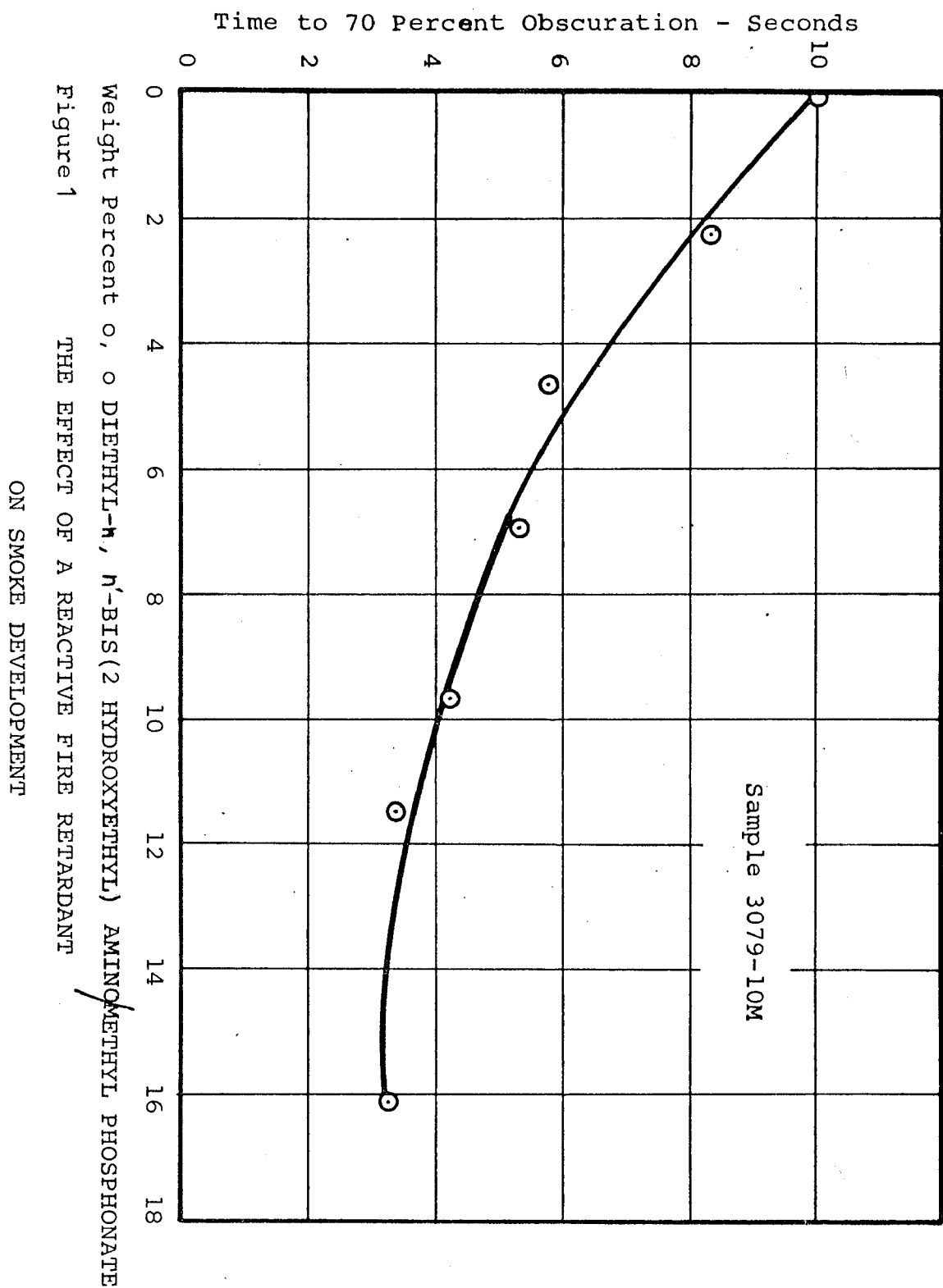
- 2b) For qualitative and quantitative description of nonvolatile products--a method detailed by Hoffman (Ref. 4) for conducting a controlled pyrolysis or combustion of the polymer surface and then successively scraping off thin layers of the surface for analysis. In this manner the decomposition history of the polymer surface may be determined.

Thus, by utilizing the techniques discussed in 2a and b a complete picture of both the solid and gas phase products may be obtained.

- 3) For rates and activation energies--DTA (measures the heat-energy change occurring in a substance as a function of temperature. Typical phenomena that will relate to thermally induced processes include melting, vaporization, polymerization, cross-linking, oxidation, and thermal decomposition) and TGA (monitors the weight loss of a sample as a function of temperature or isothermally as a function of time).

By synthesizing the data on the amount and types of decomposition products formed, the decomposition history of the polymer, the rates of the reactions, and the heat associated with reactions; a description of the thermal decomposition mechanisms may be evolved. From a comparison of the decomposition mechanisms with the smoke generating behavior of flame-retardant and non-flame-retardant polymers, those mechanism associated with smoke emission may be determined.

Most of the fundamental work concerning smokes has been performed on wood and tobacco (Refs. 5, 6, 7) and this work has resulted in the development of some of the analytical tools described above. In the area of polymers Mickelson and Einhorn (Ref. 8) have attempted to correlate the amount of smoke evolved during combustion with the activation energy governing the kinetics of thermal decomposition. They point out that the amount of fire retardant incorporated into a polymer system not only affects the flammability characteristics of the material, but also the dependency of the material to smoke. For example, Figure 1 from their paper shows how increasing the amount of a retardant in the formulation affects the smoking tendency of a urethane. This same behavior has been observed by Gaskill (Ref. 9) for phenolics, polycarbonates, and acrylics. However, no satisfactory smoke emission mechanisms were advanced.



C. Screening of High Temperature Stable Polymers for Smoke Emission

The first standard method for determining smoke evolution was the Steiner Tunnel test (ASTM-E-84). This test was primarily designed for structural materials and so the requirement that test samples be 18" x 25" did not impose a severe requirement. However, for many materials the tunnel test was neither expedient nor applicable, and Rohm and Haas seeking a definitive smoke evolution test, developed the XP-2 smoke chamber. After evaluating the XP-2 chamber, the National Bureau of Standards designed their own apparatus. The Lawrence Radiation Labs then took the NBS design and modified it to suit their own purposes. In Table I is a comparison of the smoke test systems.

Gaskill recently presented the results of his work at LRL in determining smoke density and toxic gas concentrations for various polymer formulations. In the standard nomenclature

$$D_s = \frac{V}{AL} \frac{\log_{10} 100}{T} = \text{specific optical density}$$

T = percent light transmission at any time

$$D = \frac{\log_{10} 100}{T} = \text{optical density}$$

V = chamber volume

A = sample face area

L = light path length

D_m = maximum D_s obtained in a test

$D_s 16$ = time for smoke to reach a critical (i. e., vision obscuring) density.

Some of his results are shown in Table 2.

As can be seen, only cellulose and polysulfone take longer than 90 sec. (1.5 min) to give a critical smoke obscuration value. Since polymers tested had no flame retardants, it can be expected that even shorter times would be necessary to reach critical obscuration with polymers flame retarded with chlorinated and phosphated organics. However, Gaskill does present evidence (see Fig. 2) that a good fire retardant does not have to be a high emitter of smoke. Under nonflaming conditions the inorganic

Table 1
Comparison of Smoke Test Systems for Measuring Smoke Obscuration

	<u>Steiner Tunnel</u>	<u>XP2 Chamber</u>	<u>NBS Chamber</u>	<u>LRL Chamber</u>
Sample size	36 ft ²	1 in ²	6.6 in ²	6.6 in ²
Test duration (min.)	10	4	≥30	≥30
Heat Source	flame	flame	Radiant & optional flame	Same as NBS
Smoke Production mode	Pyrolysis & Combustion	Combustion	Pyrolysis without flame Pyrolysis & Combustion with flame	Same as NBS
Smoke measurement method	Integrated rate	Integrated rate	Accumulation; maximum rate is increased as is obscuration time	Same as NBS
Provisions for smoke sampling and physico-chemical analysis	No	No	No	Yes

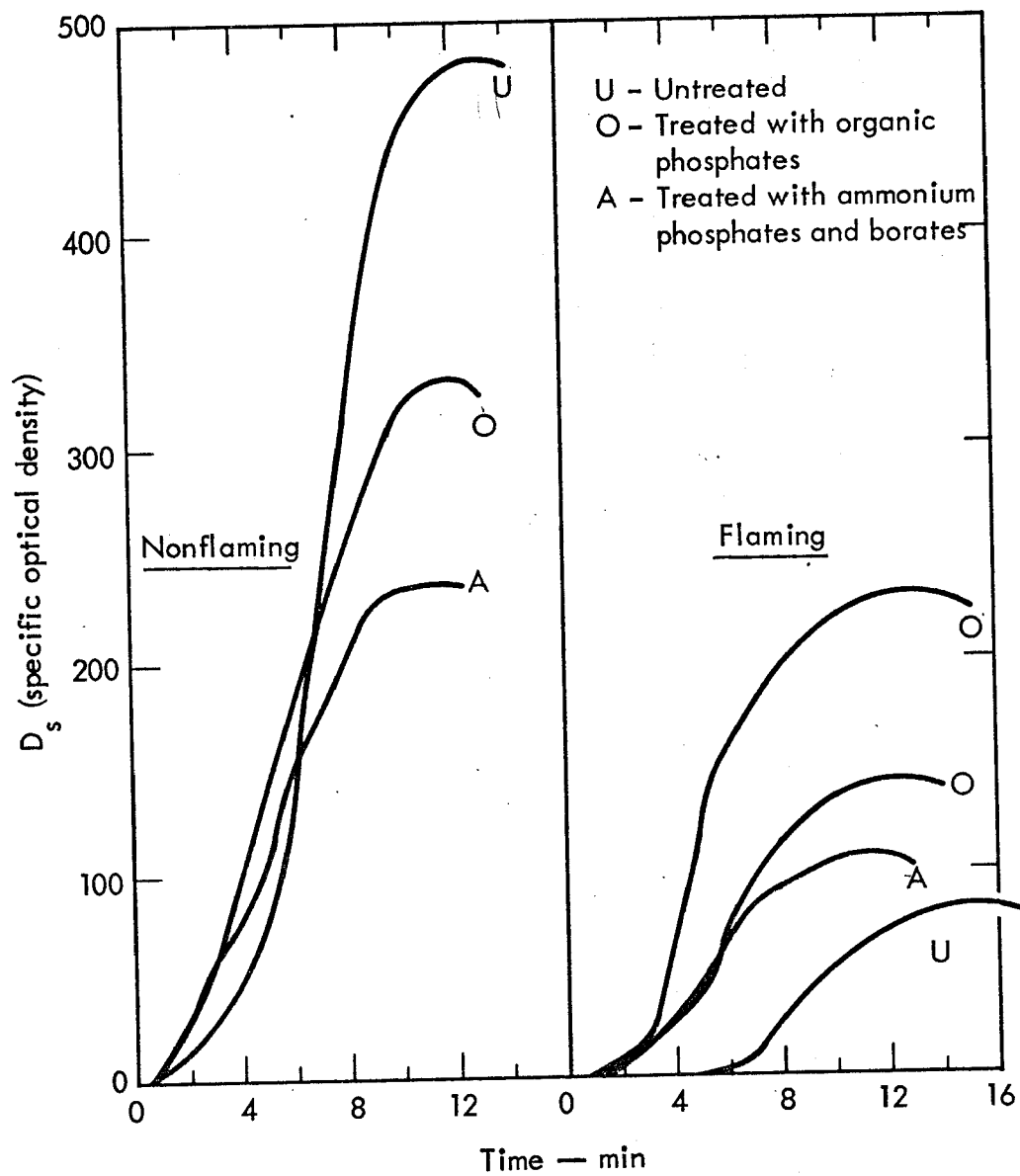


Figure 2

Table 2
Smoke Density for Several Plastics
(Flaming Conditions)

<u>Material</u>	<u>Dm</u>	<u>Ds 16 (min).</u>
Cellulose	18-25	4 - 7.9
Polysulfone	42-100	1.6 - 2.3
Polycarbonate	78-127	0.6 - 1.2
PVC - ABS	309-391	0.33-0.5
Urethane rubber	230	1.5

phosphate and borate treated woods produce the least smoke and in flaming they produce only slightly more smoke than the untreated wood. The only problem with the inorganic salts is that they may be leached out of polymer matrices, but it is possible to minimize losses of the retardant. In fact, the flameproofing coating developed at Dynatech utilizes inorganic phosphates and, yet, can stand immersion in 5% sodium chloride solution for at least one-half hour and still perform its flameproofing function in 100% oxygen environments. It may also be possible to permanently radiation graft inorganic phosphates to polymer chains and, in this way, provide a durably fire-resistant, low smoke producing polymer.

D. Formulation of Low-Smoke-Producing and Fire-Resistant Polymer Systems

While a considerable effort has been expended by researchers in developing flame retardants for polymers (e. g., Delman (Ref.10) lists approximately 130 patents that have been awarded in this area in the last two years alone), very little work has been done in smoke suppression. Einhorn has shown that with urethanes a high degree of aromaticity can be associated with a high degree of smoke development and also that increasing cross-link density also increases smoke production (Ref. 11).

At this point it would be usual to include a discourse on the chemistry of urethanes, vinyls, ABS, etc., and the usual additive, reactive, and coadditive methods for incorporating fire retardants. However, this is singularly useless because all the

commonly used fire-retardants for polymers enhance the emission of smoke in proportion to their effectiveness as fire retarding agents except, apparently, for the inorganic agents. With the addition to cellulose of as little as 5% of ammonium phosphate the tarry decomposition distillates are reduced from 55% to 5% of the weight of the original cotton (Ref. 12). On the other hand, treatments of the antimony oxide-chlorinated compound type, where sublimation of both constituents along with the tars takes place, an apparent tar value of 27% is obtained for the fabric. The higher tar value and the presence of the retardant in the vapor phase where it can inhibit oxidation reactions and promote cracking and polymerization of the hydrocarbons may be one of the reasons for increased emission, but this is at present just speculation. It is necessary to study the gas phase, the solid phase, the heats involved, and the kinetics of the reactions to arrive at a definitive answer.

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Section 3 PROPOSED PROGRAM

A program divided into three phases of activity has been adopted in preparing the program outline. The detailed breakdown of the project tasks is as follows:

	<u>Project Task</u>	<u>Duration, Months From Project Initiation</u>
Phase I	<u>Study of the Mechanisms of Smoke Emission</u>	
1)	Assemble polymer systems of interest. If representative formulations of potentially important retardants are not commercially available (e.g., radiation-grafted allyl phosphates, vanadium pentoxide, nickel plus ceramics containing NH_4I , iron carbonyls, and cyclopentadienyl manganese tricarbonyl), synthesize them.	0 - 3
2)	Gas phase analysis	
	• Develop flash pyrolysis and controlled decomposition techniques	0 - 1
	• Couple pyrolysis (combustion) devices to gas chromatograph-mass spectrometer and examine the representative polymers in N_2 and air.	1/2 - 5 1/2
	• Analyze results and duplicate important experiments	5 - 6
3)	Solid phase analysis	
	• Assemble surface pyrolyzer	0 - 1 1/2
	• Develop standardized techniques for pyrolysis and analysis	1 1/2 - 3
	• Test representative samples in N_2 and air.	3 - 6
	• Analyze results and duplicate important experiments	5 1/2 - 6 1/2

4)	Heat and kinetics analysis	
	• Develop standardized techniques for TGA and DTA analysis	0 - 1
	• Test representative samples in N ₂ and air.	1 - 4
	• Analyze results and duplicate important experiments	4 - 5 1/5
5)	Synthesize the results of the gas solid, heat, kinetic, and smoke chamber (see Phase II) analyses into a coherent view of the mechanisms of smoke emission.	5 - 7
Phase II	<u>Screening of Available High Temperature Stable Polymers</u>	
1)	Search, identify, and obtain high temperature stable polymers.	0 - 3
2)	Construct modified NBS smoke chamber with provision for toxic gas analysis and standardize chamber with that at NBS.	0 - 3
3)	Test polymers for flammability using Test Method 5902, for Ds16 under both smoldering and flaming conditions using smoke chamber, and for toxic gas concentration using colorimetric filters on exhaust from smoke chamber.	3 - 6
4)	Analyze results	5 - 6
Phase III	<u>Formulation of Low Smoke Producing and Fire Resistant Polymers</u>	
1)	Predict from the mechanistic study of Phase I, flame and smoke retardants able to meet the requirements of NAFEC.	6 - 7
2)	Assemble materials	6 - 8
3)	Synthesize prepolymers and retardants. if necessary.	7 - 9

- 4) Formulate and synthesize representative flame and smoke retardant polyurethanes, vinyl resins, ABS plastics, acrylic resins, and other polymers that may be of interest. 7 - 11
- 5) Test formulations with analytical instruments used in Phase I, with Test Method 5902 and with smoke chamber to confirm the predicted flame and smoke retardant behavior. 8 - 12
- 6) Prepare rough draft of Final Report 11 - 12

The program plan has been carefully developed to include provisions for anticipated problems. For example, it is anticipated that modifications of the analytical equipment will be required to permit specialized experiments to be carried out--provisions for the time necessary to make these modifications has been included in Phase I. Also provisions for the duplication of critical experiments is included. The contingency that all the important combinations of polymer and retardants to be examined in Phase I may not be available commercially has been provided for by allocating sufficient staff and materials to formulate and synthesize the otherwise unavailable combinations to insure that a comprehensive survey of emission mechanisms will be obtained.

In Phase II an anticipated problem was that the smoke measurements obtained using the tunnel test and the XP-2 chamber were for particular types of combustion and not necessarily representative of the flame exposure occurring in aircraft. Consequently, a chamber that had provisions to produce both smoldering combustion and flaming combustion, the National Bureau of Standards test chamber, was selected for use. The advantage of this chamber is that smoke production values that bracket all possible combustion conditions can be obtained. In addition, by using the Lawrence Radiation Laboratory's modification of the test chamber, toxic gas concentrations can be obtained colorimetrically under the conditions of the smoking experiments.

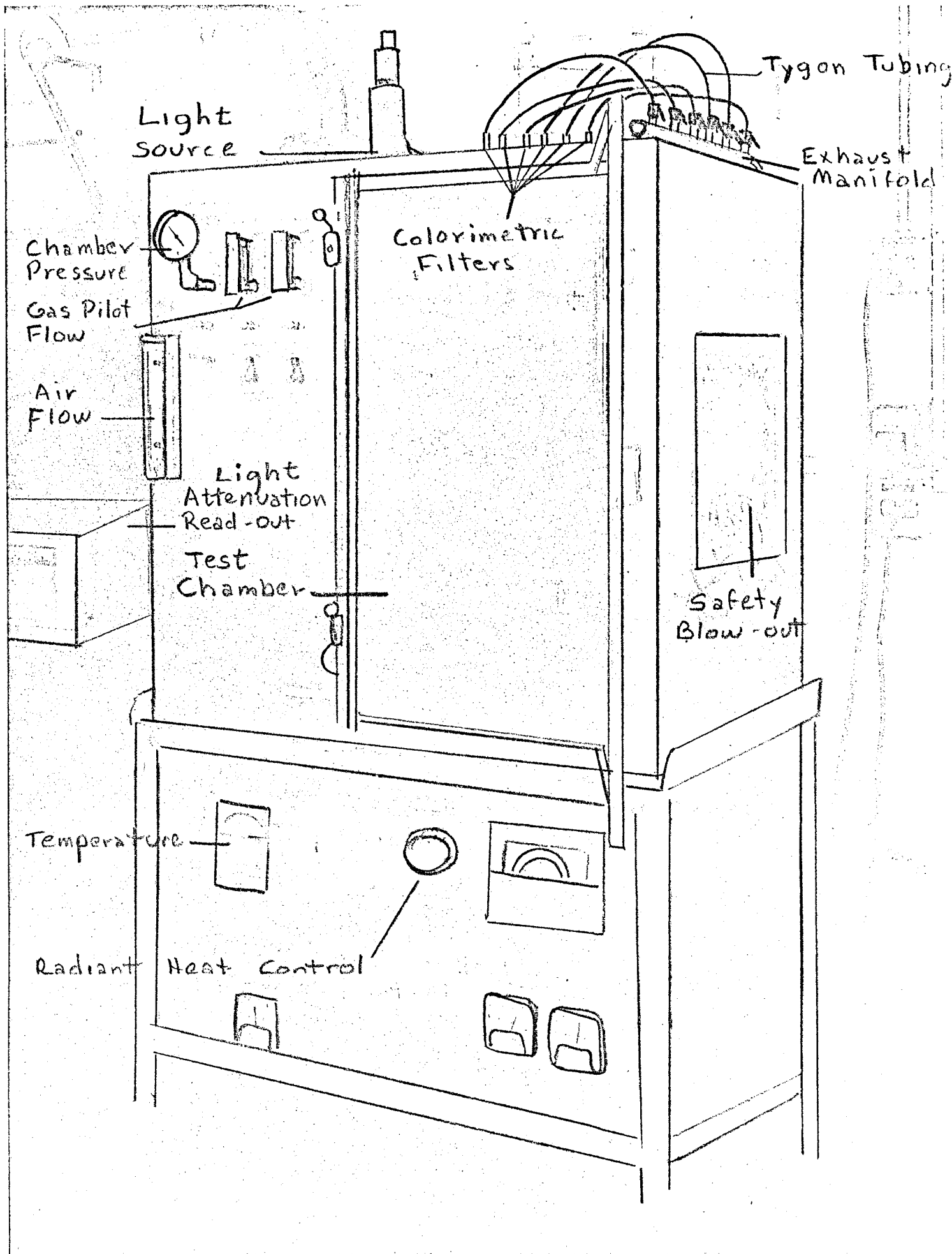


Figure 3 Smoke Chamber

In Phase III the problem that the optimum fire and smoke retardant predicted from the mechanistic study may not be commercially available is anticipated by providing for the actual synthesis of the retardants. Also anticipated is that conventional methods of incorporating retardants into polymers, i.e., additive and reactive methods, may not be the best to provide low smoke emission; and so the provision for an examination of radiation grafting of organic and inorganic groups to the polymers is brought to the program through the inclusion of Prof. Allan S. Hoffman, a polymer and radiation expert, as a consultant to our staff.

Also anticipated in Phase III is that the research & development effort may easily become diffused and ineffective if the approach that every slightly promising lead be fully investigated is taken. Consequently, the major effort in developing low smoke producing and fire-resistant materials will be concentrated on polyurethanes, vinyl resins, ABS, and acrylic resins because these materials have already proved themselves in terms of functionality in aircraft cabins. Of course, other polymer systems that prove promising in the screening experiments of Phase II will also be examined in Phase III if their projected production costs and functionality compare favorably with those of equivalent replacement materials.

Monthly detailed Progress Reports and Financial Management Reports (DD Form 1097) will be submitted for the duration of the contract.

Section 4

PROJECT ORGANIZATION AND MANAGEMENT

4.1 The Project in the Dynatech Organization

The proposed project will be assigned to the Chemical Engineering Department at Dynatech Corporation. This department is one of the five technical operating departments within the Dynatech organization. The relationship of the project to the corporate organizational structure is shown in the accompanying figure.

Within the Chemical Engineering Department at Dynatech R/D Company are two closely cooperating groups. These groups are referred to as the Applied Chemistry Group and the Process Engineering Group.

4.2 Project Management and Contract Administration

Authority for technical and cost control on a project is assigned to the Project Manager, who will be William H. Crandell. Mr. Crandell will maintain direct liaison with the technical manager at National Aviation Facilities Experimental Center. The Project Manager fully controls all project activities, including project personnel activities, experimental direction, and report preparation.

Formal contract liaison with National Aviation Facilities Experimental Center will be maintained by Dynatech's Financial and Administrative Department. This department also monitors project costs and schedules. The accounting and contract administration systems used by Dynatech are specifically designed for research programs and provide for excellent cost control. Direct labor and material costs are forecast on a weekly, monthly, and yearly basis, and actual costs are reported to the Project Manager by the accounting group within two days after the weekly reporting period. This report of status of expenditures for labor and materials on the project is furnished on a weekly control sheet prepared for the Project Manager by the Treasurer from information supplied by the Project Manager. Further fiscal control is exercised through regular monthly meetings of the Project Manager and Treasurer to forecast labor and material budgets.

It is the Project Technical Manager's responsibility to establish procedures for the systematic filing and organization of research and engineering records,

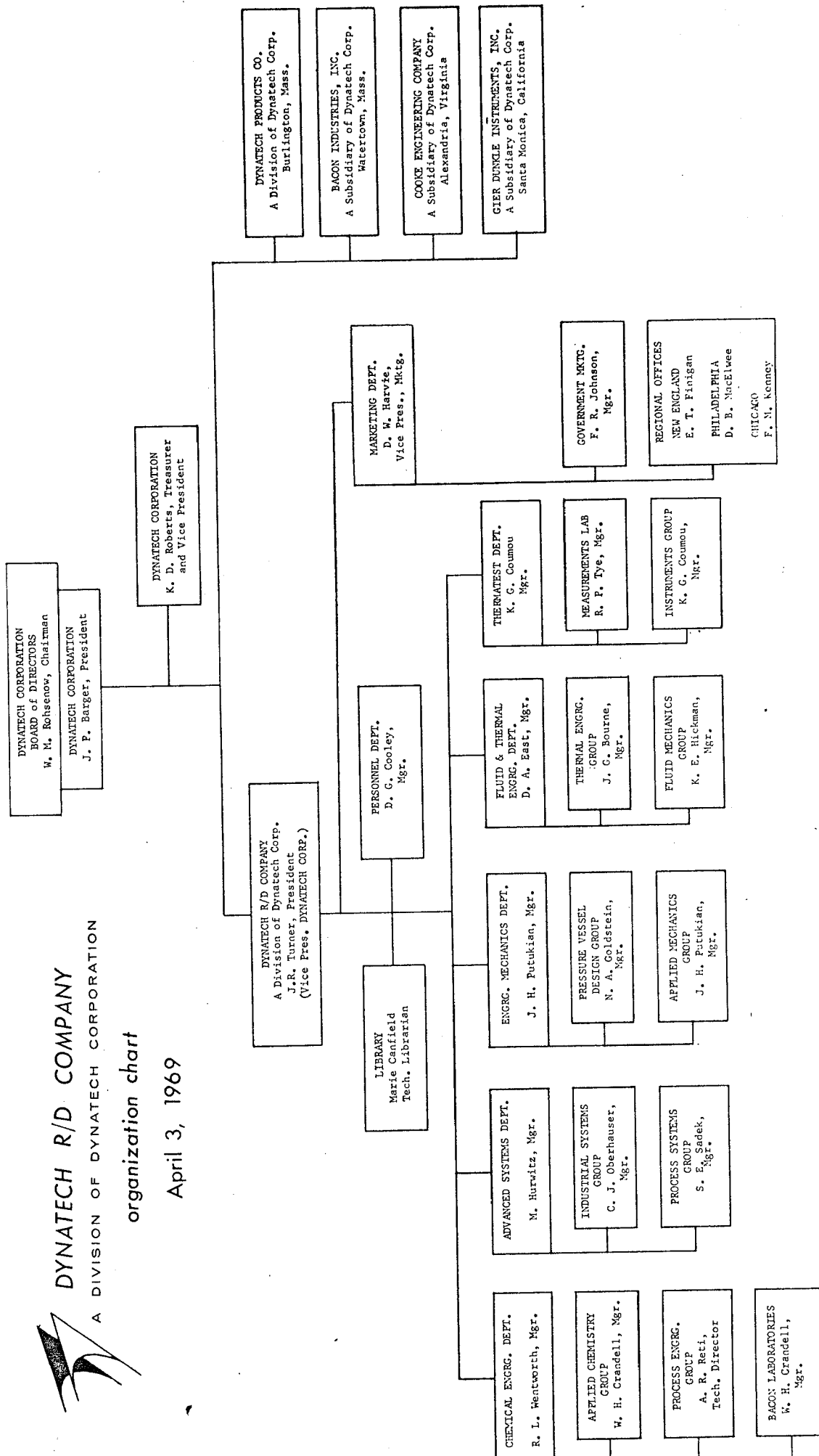


DYNATECH R/D COMPANY

A DIVISION OF DYNATECH CORPORATION

organization chart

April 3, 1969



communications, reports, and literature sources. The preparation of status and final reports to the National Aviation Facilities Experimental Center is the responsibility of the Project Technical Manager. The technical personnel of the project furnish the drafts of these reports and final drafts are prepared by the Project Technical Director.

4.3 Technical and Management Personnel

The program proposed to be carried out in the Chemical Engineering Department at Dynatech R/D Company will be under the direction of William H. Crandell, Manager of the Applied Chemistry Group. He is well versed in the techniques of research and development contract management, having participated in or supervised a large number of Dynatech's projects as well as those at Bacon Laboratories prior to that organization's merger with Dynatech. Included in projects managed by Mr. Crandell have been many involving polymer and resin technology closely allied with the kind which will be utilized in the proposed program. Mr. Crandell's technical experience includes many assignments in materials development and process engineering, including projects to develop flame resistant adhesives, encapsulating compounds, and coating systems for metals and plastics as well as reinforced epoxy and polyester resin systems. Under his direction a host of epoxy resin coatings have been developed, tested, and marketed.

Assisting Mr. Crandell will be Dr. P. L. T. Brian, Technical Director of the Chemical Engineering Department, and Mr. John B. Gregory. Dr. Brian is a noted expert in the field of simultaneous heat and mass transfer with chemical reaction. His incisive analyses have been of telling value on many of Dynatech's projects. John B. Gregory, a specialist in polymer synthesis and formulation, has had extensive experience in formulating urethanes, acrylic resins, and vinyl resins.

Staff engineers on the project will be Glenn K. Armstrong, Kenneth R. Sidman, and Keith J. Sims. Mr. Armstrong and Mr. Sidman have been active in developing unique flame retardant systems that have been utilized by NASA-MSC for protective coatings for flammable polymers. They have had extensive experience in formulating retardant systems to meet exacting physical property requirements. Mr. Sims is a materials specialist who has concentrated on physical properties measurement. He has had two years of experience working with acrylonitrile-butadiene-styrene polymers.

Also available to assist in the formulation phase of the program is Stewart C. Spinney. Mr. Spinney has concentrated on the preparation of polymers for specialty applications.

Skilled chemical technicians will support the engineering activities.

Consultants for the mechanistic study will be Prof. Allan S. Hoffman and Prof. Phillip Issenberg. Prof. Hoffman is a polymer materials specialist (synthesis, structure and properties, and flammability mechanisms). Of particular relevance to the proposed program is his work on thermal stability and flammability mechanisms of polymers, and his background in radiation processing technology. Prof. Issenberg has been concerned with the investigation of the composition of wood smoke. He and his associates have been responsible for developing particularly fine analytical tools for qualitatively and quantitatively analyzing the pyrolysis products of this complex system.

The Treasurer and Controller of Dynatech Corporation, Kenneth D. Roberts, will work with the Project Manager in the fiscal control of the project. Mr. Roberts organized the reporting system utilized at Dynatech for efficiently providing prompt reports of financial status. This system is unusual in providing such information on a weekly basis rather than the commonly-used monthly basis.

4.4 Resumes of Key Personnel

William H. Crandell

John B. Gregory

Glenn K. Armstrong

Kenneth R. Sidman

Keith J. Sims

Stewart C. Spinney

Allan S. Hoffman

Phillip Issenberg

P. L. T. Brian