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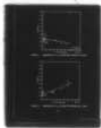
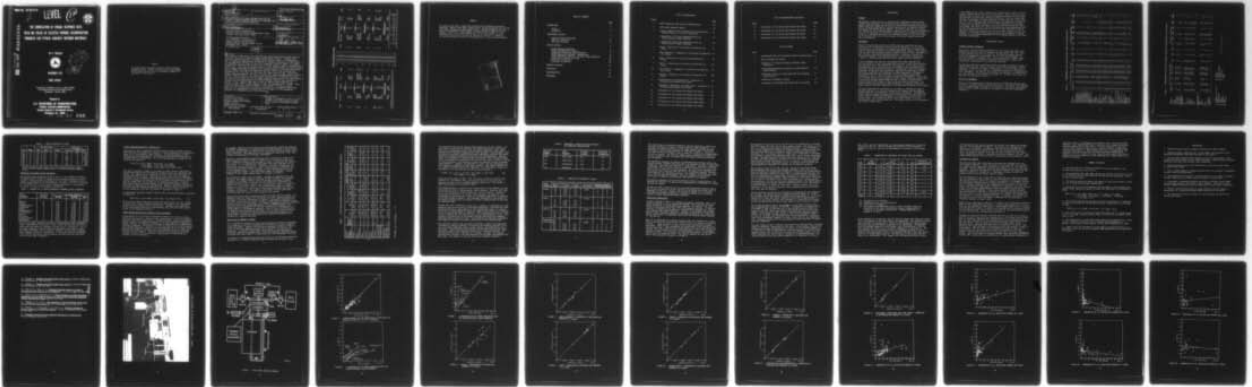
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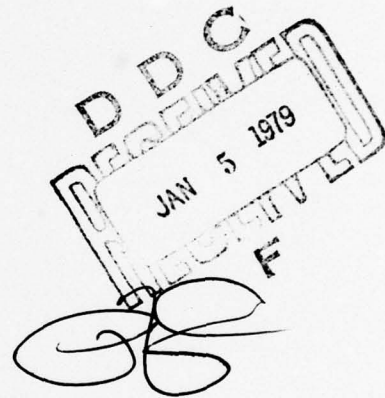
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**THE CORRELATION OF ANIMAL RESPONSE DATA  
WITH THE YIELDS OF SELECTED THERMAL DECOMPOSITION  
PRODUCTS FOR TYPICAL AIRCRAFT INTERIOR MATERIALS**

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Joe C. Spurgeon



NOVEMBER 1978

FINAL REPORT

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Prepared for

**U.S. DEPARTMENT OF TRANSPORTATION  
FEDERAL AVIATION ADMINISTRATION  
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<p>16. Abstract The purpose of this report is to describe the correlations between animal response data and the yields of selected thermal decomposition products. Seventy-five aircraft interior materials, including panels, fabrics, foams, and thermoplastics were thermally decomposed under conditions of oxidative pyrolysis in a tube furnace. In one experiment performed at the Civil Aeromedical Institute (CAMI), the thermal decomposition products were directed into an animal exposure chamber containing male albino rats. Both times-to-incapacitation (<math>t_i</math>) and times-to-death (<math>t_d</math>) were recorded. In a separate experiment performed at the National Aviation Facilities Experimental Center (NAFEC), the thermal decomposition products (TDP's) were collected and analyzed for CO, HCN, H<sub>2</sub>S, HCl, HBr, NO<sub>2</sub>, SO<sub>2</sub>, HCHO, and HF yields. Multivariate linear regression analysis is used to correlate the times-to-incapacitation with the yields of the nine TDP's. The coefficients of correlation (R) between TDP yields and <math>1/t_i</math> values exceed 0.95 for all usage categories except panels, for which R=0.80. In addition, the coefficients of correlation between observed and predicted <math>1/t_i</math> values for four "unknown" materials in each of several usage categories are: panels = 0.393, foams = 0.948, fabrics = 0.968, and thermoplastics = 0.988. Times-to incapacitation are related primarily to the yields of systemic toxicants (HCN, CO, etc.) rather than the yields of irritant TDP. In general, HCN is more important for describing <math>1/t_i</math> values than CO, although CO is the predominant toxicant for thermoplastics and coated fabrics.</p>			
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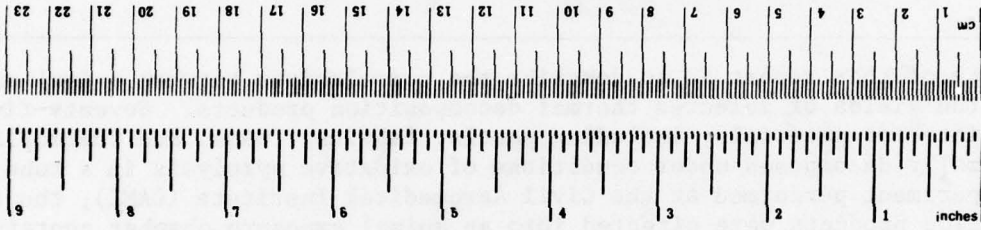
## METRIC CONVERSION FACTORS

### Approximate Conversions to Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
<b>LENGTH</b>				
in	inches	2.5	centimeters	cm
ft	feet	30	centimeters	cm
yd	yards	0.9	meters	m
mi	miles	1.6	kilometers	km
<b>AREA</b>				
in <sup>2</sup>	square inches	6.5	square centimeters	cm <sup>2</sup>
ft <sup>2</sup>	square feet	0.09	square meters	m <sup>2</sup>
yd <sup>2</sup>	square yards	0.8	square meters	m <sup>2</sup>
mi <sup>2</sup>	square miles	2.6	square kilometers	km <sup>2</sup>
	acres	0.4	hectares	ha
<b>MASS (weight)</b>				
oz	ounces	28	grams	g
lb	pounds	0.45	kilograms	kg
	short tons (2000 lb)	0.9	tonnes	t
<b>VOLUME</b>				
tsp	teaspoons	5	milliliters	ml
Tbsp	tablespoons	15	milliliters	ml
fl oz	fluid ounces	30	milliliters	ml
c	cups	0.24	liters	l
pt	pints	0.47	liters	l
qt	quarts	0.95	liters	l
gal	gallons	3.8	liters	l
ft <sup>3</sup>	cubic feet	0.03	cubic meters	m <sup>3</sup>
yd <sup>3</sup>	cubic yards	0.76	cubic meters	m <sup>3</sup>
<b>TEMPERATURE (exact)</b>				
°F	Fahrenheit temperature	5/9 (after subtracting 32)	Celsius temperature	°C

### Approximate Conversions from Metric Measures

When You Know	Multiply by	To Find	Symbol
<b>LENGTH</b>			
millimeters	0.04	inches	in
centimeters	0.4	inches	in
meters	3.3	feet	ft
kilometers	1.1	yards	yd
	0.6	miles	mi
<b>AREA</b>			
square centimeters	0.16	square inches	in <sup>2</sup>
square meters	1.2	square yards	yd <sup>2</sup>
square kilometers	0.4	square miles	mi <sup>2</sup>
hectares (10,000 m <sup>2</sup> )	2.5	acres	ac
<b>MASS (weight)</b>			
grams	0.035	ounces	oz
kilograms	2.2	pounds	lb
tonnes (1000 kg)	1.1	short tons	st
<b>VOLUME</b>			
milliliters	0.03	fluid ounces	fl oz
liters	2.1	pints	pt
liters	1.06	quarts	qt
liters	0.26	gallons	gal
cubic meters	35	cubic feet	ft <sup>3</sup>
cubic meters	1.3	cubic yards	yd <sup>3</sup>
<b>TEMPERATURE (exact)</b>			
Celsius temperature	9/5 (then add 32)	Fahrenheit temperature	°F



\*1 in = 2.54 (exactly). For other exact conversions and more detailed tables, see NBS Misc. Publ. 286, Units of Weights and Measures, Price \$2.25, SD Catalog No. C13.10.286.

PREFACE

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## INTRODUCTION

### PURPOSE.

The purpose of this report is to describe the correlations between animal response data obtained on 75 aircraft interior materials at the Civil Aero-medical Institute (CAMI) and the yields of nine thermal decomposition products (TDP's) which were measured for the same materials at the National Aviation Facilities Experimental Center (NAFEC). The primary objective is to determine whether or not an observed animal response, such as time-to-incapacitation ( $t_i$ ), can be described by a statistical model based on the measured yields of TDP's. A secondary objective is to determine whether or not such a model can be employed when only a limited number of the more commonly monitored TDP's are measured.

### BACKGROUND.

Several recent studies conducted at NAFEC have been concerned with the analysis of TDP's released by aircraft interior materials. In an initial investigation, the materials were thermally decomposed under flaming conditions in the National Bureau of Standards (NBS) smoke chamber. The yields of selected TDP's were estimated using colorimetric detector tubes (reference 1). Later studies, employing instrumental methods of analysis, involved the oxidative pyrolysis of the same materials in a tube furnace (references 2, 3, and 4). The TDP yields utilized in this report are from reference 2.

It was recognized very early in the program that animal response data would be required to rank these materials according to their relative potential toxicities in a fire environment (reference 5). The yields of TDP's, by themselves, are not sufficient for this purpose since the significance of these data cannot be determined without a toxicological point of reference. Scientists at CAMI have had considerable experience in experimental animal toxicology, including the observation of animal responses in relation to pure gas exposures of carbon monoxide (CO) and hydrogen cyanide (HCN) (references 6 and 7), and the testing of materials (references 5 and 8). The FAA's Systems Research and Development Service (SRDS), therefore, established a cooperative program between NAFEC and CAMI in order to obtain animal response data and TDP yields on typical aircraft interior materials. The animal response data obtained at CAMI (reference 9) have been utilized to assign relative potential toxicities to the materials discussed in this report.

The effort described in this report is, in effect, a feasibility study to determine whether or not further research by the Federal Aviation Administration (FAA) into bio-analytical correlations is warranted. Correlations between observed animal responses and the concentrations of toxic TDP's commonly present in combustion atmospheres, if successful, could be useful in several areas. One is quality control tests, while another is initial screening tests for aircraft materials. This might reduce, but not eliminate, the need for animal tests.

A more immediate use could be made of such correlations to assist in interpreting proposed FAA full-scale fire test results. Animal test protocols commonly used to compare the relative combustion hazards of materials have been developed for use under laboratory conditions. However, current FAA programs are designed to compare materials in full-scale fire tests (reference 10). Test durations will be restricted to approximately 5 minutes or less in order to simulate realistic fire scenarios and evacuation times. In addition, the sample loading will be low in comparison with most laboratory animal test protocols. Short test durations, high temperatures, and low TDP concentrations may limit the utility of most presently available laboratory animal test protocols for use in the FAA's full-scale fire test program. If a suitable animal test protocol is not available, it may be necessary to base material comparisons on the chemical analysis of the full-scale fire environment.

## EXPERIMENTAL SECTION

### AIRCRAFT INTERIOR MATERIALS.

The aircraft interior materials discussed in this report were chosen from among those which were used in wide-body aircraft during 1972-1973. Many of these materials are still in service. The materials comply with the latest FAA flammability requirements, and are "self extinguishing" in a vertical orientation (reference 10). They were supplied to NAFEC through the cooperation of the Aerospace Industries Association of America and leading seat manufacturers for the aircraft industry.

The materials have been classified according to usage categories in order to compare materials with similar functions. They include 13 panels, 9 panel components, 12 fabrics, 9 foams, 8 thermoplastics, 6 flooring materials, 5 cargo bay liners, 4 coated fabrics, 4 insulation materials, 3 transparencies, and 2 elastomers. The chemical and physical characteristics of these materials are described in more detail in reference 1. However, their basic compositions are described in table 1.

### NAFEC TEST PROCEDURE.

Details of the experimental procedures employed at NAFEC and CAMI have been described in previous reports (references 2 and 9). Therefore, only summaries of the two experimental approaches are presented in this report. The material samples tested at NAFEC and CAMI are not exposed to identical experimental conditions.





This is due to limiting factors imposed by the different objectives of the two laboratories. The NAFEC tests are designed to obtain TDP yields, while the CAMI tests are designed to measure animal responses. However, the two approaches are similar, and parametric studies (reference 2 and unpublished NAFEC data) indicate that the thermal decomposition products generated in both laboratories are probably comparable for many, but not all, of the 75 materials.

A 250-milligram (mg) sample of material is exposed to 600 degrees Celsius ( $^{\circ}\text{C}$ ) in a tube furnace. The material is heated in a Vycor<sup>®</sup> tube for 5 minutes, while air is drawn through the tube at a flow rate of 2 liters per minute ( $\ell/\text{m}$ ). Eight of the nine thermal decomposition products are sampled using four liquid-filled bubblers, each of which contains an appropriate absorbing solution. The tube furnace and associated apparatus are depicted in figure 1.

The contents of the bubblers are analyzed for hydrogen cyanide (HCN), hydrogen sulfide ( $\text{H}_2\text{S}$ ), hydrogen chloride ( $\text{HCl}$ ), hydrogen bromide (HBr), and formaldehyde (HCHO) by differential pulse polarography. Nitrogen dioxide ( $\text{NO}_2$ ) and sulfur dioxide ( $\text{SO}_2$ ) are analyzed by spectrophotometric procedures, while hydrogen fluoride (HF) is measured by ion selective electrode. Carbon monoxide (CO) is collected by replacing the bubblers with a Saran<sup>®</sup> sample bag and passing the required airflow through the combustion tube from a cylinder of high purity air. The CO is analyzed by nondispersive infrared spectroscopy. Three replicate tests are made on each material, and the reported yields are an average of the three results. An additional series of three replicate tests is conducted for the CO analysis.

#### CAMI TEST PROCEDURE.

A 750-mg sample of material is exposed to  $600^{\circ}\text{C}$  in a tube furnace. The material is heated in a Vycor tube for 10 minutes. During this time, an airflow rate of 4  $\ell/\text{m}$  is maintained through the Vycor tube. The thermal decomposition products enter an animal exposure chamber constructed of Plexiglas and are then recirculated through the Vycor tube. The total volume of the exposure system is 12.6 liters. The recirculation fan is turned off after the first 10 minutes of a test. However, the animals are left in the exposure chamber until the last animal dies, or for a maximum of 30 minutes.

Male Sprague-Dawley rats weighing between 150 and 300 grams (g) are exposed to the thermal decomposition products in groups of three. The animals are housed in individual Plexiglas wheels rotating at 6 revolutions per minute (r/min). Both the temperature and the oxygen ( $\text{O}_2$ ) concentration in the exposure chamber are monitored throughout a test. The temperature is not allowed to exceed  $35^{\circ}\text{C}$ , while the partial pressure of  $\text{O}_2$  is not allowed to fall below 95 percent of ambient. The CAMI test chamber (reference 9) is illustrated in figure 2.

The reported times-to-incapacitation ( $t_i$ ) and times-to-death ( $t_d$ ) are the average of at least three tests (nine animals) for all the materials except the panel components. Only two tests (six animals) were made on these materials. These data have been normalized on the basis of a 200-g test animal and a 1-g material sample weight (reference 9).

## RESULTS SECTION

### ANIMAL RESPONSE PARAMETERS.

The rotating wheel developed at CAMI provides two toxicological endpoints,  $t_i$  and  $t_d$ . Values of  $t_i$ ,  $t_d$ , and  $1/t_i$  for the 75 materials, along with the yields of the nine TDP's, are contained in table 1. The inverses of  $t_i$  values are used throughout this report in order to linearize the data. A least-squares linear regression analysis of  $1/t_i$  and  $1/t_d$  values results in a coefficient of correlation (R) of 0.914 for the 56 materials that produced a  $t_d$ . Therefore, either endpoint could be used to rank the materials according to their relative potential toxicities. However,  $t_i$  values have been used to rank materials for two reasons.

1. Incapacitation is a more relevant response when one is concerned with emergency evacuations from potentially hazardous environments, such as post-crash cabin fires. Pure gas studies (reference 6) utilizing albino rats have shown that 30 percent of the lethal CO dose will produce incapacitation, while only 16 percent of the lethal HCN dose is required to produce incapacitation. Therefore, material rankings that are based on death rather than incapacitation as an endpoint may be misleading for some applications.

2. A more pragmatic reason for using  $t_i$  as the endpoint is that 71 of the 75 materials produced incapacitation, while only 56 of the materials produced death. For the experimental conditions employed in these tests,  $t_i$  values allow a greater percentage of the materials to be ranked. This parameter is, therefore, of more value with respect to the present data.

### THERMAL DECOMPOSITION PRODUCTS.

A brief analysis of the TDP yields is contained in table 2. The TDP's are listed in order of the frequency with which they were detected. Carbon monoxide, NO<sub>2</sub>, HCN, HCHO, and HCl are released by 60 percent or more of the materials, while SO<sub>2</sub>, HBr, HF, and H<sub>2</sub>S are released by a third or less of the materials tested. In addition to the minimum and maximum yields, table 2 contains the mean and median yields for each of the nine TDP's. Due to a small number of comparatively large yields, the median yield is more representative of the "average" yield than the mean.

The last two columns in table 2 are a comparison of TDP yields in milligrams per gram (mg/g) and equivalent concentrations in parts per million (p/m) recorded in the animal exposure chamber. For example, if a material produces 100 mg/g of CO in the NAFEC test apparatus, 750 mg of the material should produce 5,287 p/m of CO in a volume of 12.6 liters. The equivalent concentrations in p/m are provided as a readily available basis of comparison for other data banks. Also, yields in mg/g can sometimes be misleading. For example, the median yields for HBr and HF are approximately equal, but the equivalent concentrations in p/m indicate a substantial difference in exposure levels.

TABLE 2. MEAN AND MEDIAN TDP YIELDS

TDP	Number	TDP Yields (mg/g)				Equivalent Concentration*	
		Mean	Median	Minimum	Maximum	mg/g	p/m
CO	74	113	96	9	406	100	5,287
NO <sub>2</sub>	56	0.3	0.1	0.01	2	0.1	3.2
HCN	52	9.2	5.7	0.6	62.4	5	275
HCHO	48	4.9	2.2	0.4	63.4	2	98.7
HCl	45	92.8	34.4	4.2	536	30	1,216
SO <sub>2</sub>	25	3.1	1.9	0.3	16.6	2	46.3
HBr	23	12.2	7.1	1.7	47	10	183
HF	17	23.1	8.3	4.1	152	10	740
H <sub>2</sub> S	13	5.7	5.3	0.2	14.2	5	218

\*The TDP concentration (p/m) equivalent to the indicated yield (mg/g) for a sample weight of 750 mg, an exposure chamber volume of 12.6 liters, and a chamber temperature of 30 °C.

COMPARISON OF MATERIAL USAGE CATEGORIES .

The material usage categories listed in table 3 are arranged according to the magnitudes of the mean  $t_i$  values of the individual materials in each category. Incapacitation was obtained for 71 of the 75 materials, with  $t_i$  values ranging from 1.15 to 14.96 minutes. The mean  $t_i$  for the 71 materials is 5.79 minutes ( $1/t_i = 0.173$ ) with a standard deviation of 3.05 minutes. The median  $t_i$  value is 5.06 minutes ( $1/t_i = 0.198$ ).

TABLE 3. ANALYSIS OF  $T_i$  VALUES ACCORDING TO MATERIAL USAGE CATEGORIES

Usage Category	Materials Included	$t_i$ Values (minutes)		
		Minimum	Maximum	Mean
Fabrics	12	1.15	7.57	2.58
Panels	13	2.36	5.85	3.48
Transparencies	2	3.80	7.56	5.06
Components	9	3.22	13.02	5.29
Foams	9	4.29	9.58	5.53
Thermoplastics	8	3.70	10.79	5.56
Insulations	3	3.70	12.26	5.95
Liners	3	3.99	10.33	6.28
Flooring	6	4.94	10.90	6.67
Coated Fabrics	4	6.97	13.71	9.00
Elastomers	2	9.16	14.96	10.36

Seven of the usage categories in table 3: transparencies, panel components, foams, thermoplastics, insulations, cargo liners, and flooring have mean  $t_i$  values that are similar to the mean  $t_i$  value for all 71 materials. Two of the usage categories: coated fabrics and elastomers, have mean  $t_i$  values that exceed the mean  $t_i$  of 5.8 minutes. However, the two usage categories which together account for the majority of the surface area in an aircraft cabin, fabrics and panels, exhibit greater relative potential toxicities than the other usage categories.

#### LINEAR REGRESSION ANALYSIS: GROUPED DATA.

Least-squares linear regression equations relating animal response data and TDP yields can be calculated for either: (1) the 71 materials as a generalized group, or (2) each material usage category. The appropriateness of each approach has been investigated. The regression equation describing the calculated times-to-incapacitation ( $t_c$ ) for the grouped data (71 materials) is contained in equation 1.

$$\begin{aligned} 1000 (1/t_c) = & 16.7 (\text{HCN}) + 0.615 (\text{CO}) - 11.1 (\text{H}_2\text{S}) \\ & + 41.2 (\text{NO}_2) + 6.49 (\text{SO}_2) - 0.103 (\text{HCl}) \\ & - 1.36 (\text{HCHO}) - 0.661 (\text{HBr}) - 0.121 (\text{HF}) + 84.3 \end{aligned} \quad (1)$$

The terms in equation 1 appear in the order of their relative importance in minimizing residuals, and the equation is based on both zero and nonzero TPD yields. The coefficient of correlation is 0.915, while the coefficient of determination ( $R^2$ ) is 0.837. Since the  $R^2$  value is the ratio of explained variation to total variation, this regression equation can account for approximately 84 percent of the variation in  $1/t_i$  values, even though only nine of the more common TDP's are included in the regression equation. Although HCHO and HF yields have been included in the regression equation, only one of these TDP was measured for any one material. However, deleting both HCHO and HF yields from the regression equation results in an R value of 0.913. A scatter diagram for  $1/t_i$  and  $1/t_c$  values is contained in figure 3.

An abbreviated regression equation based on HCN, CO, and  $\text{H}_2\text{S}$  yields is contained in equation 2.

$$1000 (1/t_c) = 12.7 (\text{HCN}) + 0.627 (\text{CO}) - 12.7 (\text{H}_2\text{S}) + 81.6 \quad (2)$$

The R value for this equation is 0.891, while the  $R^2$  value is 0.793. Yields of these three systemic toxicants account for 95 percent of the explainable variation in  $1/t_i$  values (0.793 versus 0.837). Therefore, the correlation is not significantly improved by including  $\text{NO}_2$ ,  $\text{SO}_2$ , HCl, HBr, HCHO, and HF yields in the regression equation.

#### LINEAR REGRESSION ANALYSIS: MATERIAL USAGE CATEGORIES .

An examination of the residuals (differences between  $1/t_i$  and  $1/t_c$  values) resulting from equation 1 indicates that, statistically, it may not be appropriate to treat the 71 materials as a collective group. Positive and negative residuals should be randomly distributed if the data represent a homogeneous sample. However, the algebraic distribution of residuals is not random within the usage categories. For example, 9 of the 13 panels and 9 of the 12 fabrics have positive residuals. Seven of the nine foams and six of the eight thermoplastics have negative residuals, while the residuals for the six flooring materials are all negative. These data indicate that equation 1 describes a family of curves, and is, therefore, inappropriate for describing the behaviors of the 71 materials.

For example, figures 4 and 5 illustrate the least-squares regression lines for CO and HCN, respectively, for various material usage categories. These figures illustrate the diversity of responses that can be obtained for different groupings of materials. The result is a set of nonparallel regression lines, each of which is influenced by a different environment.

Therefore, linear regression equations have been calculated for each of the usage categories listed in table 4. Values of  $1/t_i$  calculated using equations 3 through 10 (table 4) are included in table 1, along with the observed  $1/t_i$  values. It is a mathematical requirement that  $(n+1)$  materials can be described by no more than  $(n)$  TDP. Therefore, elastomers, insulations, and cargo liners have been combined in order to form a group of sufficient size to treat mathematically. It is necessary to work with grouped data if the usage categories contain a relatively small number of materials. Scatter diagrams for each usage category appear in figures 6 through 13.

The equations in table 4 are not unique. A number of equations can be generated for each usage category by varying the TDP upon which the equations are based. For example, the  $R^2$  value for equation 6 actually increases somewhat when CO yields are deleted from the regression calculations and replaced with  $SO_2$  yields. However, all the equations in table 4, except for equation 3, satisfy two criteria. They contain enough terms to achieve a minimum  $R^2$  value of 0.95 and as many of the terms as possible have positive regression coefficients. The negative coefficients for  $H_2S$  result from including 0-mg/g  $H_2S$  yields in the regression calculations.

Panels provide the worst correlation between TDP yields and  $1/t_i$  values. The maximum  $R^2$  value for panels is a relatively low 0.636. Several factors may contribute to the poor correlation for panels. One possible explanation is that TDP's other than those listed in table 1 significantly contribute to the relative toxicities of the panels. For example, organonitriles may be important for panels due to the thermal degradation of the Nomex<sup>®</sup> honeycomb cores. Another possible explanation for the poor correlation is sampling error. A 250-mg sample of a heterogenous composite, often an inch in thickness, may not always be representative of the panel, since a large excess of adhesive can substantially alter the weight fraction of each panel component in a 250-mg sample. Therefore, more sophisticated sampling techniques may be required for composite materials.

#### PREDICTION OF UNKNOWN $T_i$ VALUES.

Once a descriptive equation has been generated using one sample of materials, it is tempting to use such an equation as a predictive tool for a sample of similar materials. However, the descriptive equations contained in table 4 could not have been generated without first obtaining  $t_i$  values for the materials. In addition, these equations only apply to the experimental conditions employed to generate them. Different regression equations (based on animal data) would have to be established for each experimental condition.

One approach to evaluating the predictive powers of the calculated regression equations is to calculate predicted times-to-incapacitation ( $t_p$ ) for the

TABLE 4. LEAST-SQUARES REGRESSION EQUATIONS FOR EACH MATERIAL USAGE CATEGORY

Material Usage Category	Equation Number	R <sup>2</sup>	k*	Regression Coefficients									
				CO	HCN	H <sub>2</sub> S	HCL	HBr	NO <sub>2</sub>	SO <sub>2</sub>	HCHO	HF	
Panels	3	0.636	80.2	0.132	30.8	-182	0	1.20	0	-36.5	0	7.09	
Panel Components	4	0.960	-56.3	1.62	5.88	0	0	3.24	0	0	0	0	
Foams	5	0.958	98.6	0.456	2.79	226	0	0	0	-29.6	7.56	0	
Fabrics	6	0.948	296	0.051	10.2	-17.9	-0.317	0	0	0	0	0	
Coated Fabrics	7	0.996	6.32	1.22	0	0	0	0	0	0	0	0	
Flooring	8	0.963	4.06	1.48	7.52	0	0	0	0	0	0	0	
Thermoplastics Transparencies	9	0.973	83.6	0.480	0	0	0	0	0	0	0	0	
Elastomers Insulations Cargo Liners	10	0.956	46.1	0.492	14.8	0	0.448	0	0	0	1.03	0	

\*(1,000) 1/t<sub>c</sub> = a<sub>1</sub>x<sub>1</sub>+a<sub>2</sub>x<sub>2</sub>+ . . . + k

four materials that did not produce incapacitation in the test animals. One would like to know how such materials are ranked analytically, even though this is not an appropriate use of the descriptive equations. Since these materials do not produce incapacitation, they represent a biased sample. They should not be evaluated using the calculated regression equations since these equations are based on materials which do produce incapacitation. However, since it is impossible to determine this fact without first performing animal tests, the appropriate regression equations have been used to predict  $t_i$  values for these materials. The predicted  $t_i$  value for material 109 was calculated using equation 9. However, the equations in table 4 are not necessarily the best predictive equations for each usage category. They simply represent one of a number of possible descriptive equations. The predicted  $t_i$  values for materials 25, 66, and 118A were calculated using equation 11

$$(1,000) 1/t_i = 18.2 + 1.30 (\text{CO}) + 20.3 (\text{HCN}) + 0.224 (\text{HCl}) - 141.5 (\text{NO}_2) + 1.35 (\text{HCHO}) \quad (11)$$

which has an  $R^2$  value of 0.983. This is a better predictive equation than equation 10. For example, the  $t_p$  for material 66 is 6.0 minutes using equation 10, while it is 7.1 minutes based on equation 11. The results of these calculations are contained in table 5.

The maximum observed  $t_i$  value for the 71 materials is 14.96 minutes, and only eight materials have  $t_i$  values greater than 10 minutes. Materials 109, 118A, and 25 have predicted  $t_i$  values between 10.7 and 20.2 minutes. Assuming a log-normal distribution of  $t_i$  values, these materials are predicted to be less hazardous than 90 percent or more of the sample population, as indicated in the last column of table 5. The log-normal percentile is the fraction of the sample population that is expected to produce incapacitation in less than the indicated time.

Material 66, with a predicted  $t_i$  value of 7.1 minutes, is predicted to be less hazardous than only 60 percent of the sample population. The relatively high predicted toxicity is due primarily to the reported HCN yield of 7.3 mg/g, a level that is indeed capable of producing incapacitation within 4 to 8 minutes (based on similar HCN yields in table 1). Since incapacitation did not occur, one must conclude that the reported HCN yield is significantly in error. This material illustrates one of the primary problems associated with comparing materials based on analytical data. The complexity of combustion atmospheres often makes it difficult to obtain reliable data on TDP concentrations.

The predictive powers of the regression equations in table 4 have also been evaluated by a second technique. One material was selected from each of the four main usage categories: panels, foams, fabrics, and thermoplastics by a blind draw. These materials were deleted from the data bank, and new linear regression equations were generated based on the remaining materials. The new equations were used to predict the "unknown"  $t_i$  values. The deleted materials were returned to the data bank and this procedure was repeated three more times. The results of the four trials are contained in table 6.

TABLE 5. PREDICTED  $t_i$  VALUES FOR THOSE MATERIALS NOT PRODUCING INCAPACITATION

<u>Material Number</u>	<u>Usage Category</u>	<u>Predicted <math>t_p</math> (min)</u>	<u>Log-Normal Percentiles</u>
118A	Cargo Liner	20.2	96
25	Cargo Liner	17.3	95
109	Transparency	10.7	90
66	Insulation	7.1	60

TABLE 6. PREDICTION OF UNKNOWN  $t_i$  VALUES

<u>Usage Category</u>	<u>Material Number</u>	<u>Observed <math>t_i</math> (min)</u>	<u>Predicted <math>t_p</math> (min)</u>	<u>Correlation Coefficient</u>	<u>Relative Ranking</u>	
					<u>Observed</u>	<u>Predicted</u>
Panels	20	2.36	3.3	0.393	1	2
	14	2.38	4.3		2	4
	2	3.07	2.9		3	1
	37	3.90	3.4		4	3
Foams	73	4.29	4.8	0.948	1	1
	79	4.80	5.2		2	3
	74	5.04	4.9		3	2
	80	7.55	15.1		4	4
Fabrics	127	1.15	1.0	0.968	1	1
	78	2.23	2.5		2	2
	82	3.47	2.9		3	3
	96	4.64	4.0		4	4
Thermoplastics Transparencies	113	4.04	4.0	0.988	1	1
	117	5.19	5.7		2	2
	99	6.01	6.5		3	3
	100	9.41	9.1		4	4

The coefficients of correlation between  $t_1$  and the predicted  $t_1$  values ( $t_p$ ) have been calculated for each of the four material usage categories. In addition, the deleted materials in each usage category have been compared according to their relative ranking, as indicated in table 6. There is a very good correlation between  $t_1$  and  $t_p$  values for fabrics and thermoplastics, but not for panels. One would not expect a good correlation for panels due to their relatively poor correlation with TDP yields, which has previously been discussed.

The regression equations for fabrics and thermoplastics probably contain the primary systemic toxicants associated with the materials in each of these usage categories. These TDP's are HCN and CO, respectively. Therefore, regression coefficients are not greatly affected by material deletions, with the result that  $t_1$  and  $t_p$  values are in good agreement. The more erratic agreement for foams may be due to the fact that not all of the dominant systemic toxicants are included in the regression equation. For example, isocyanates are highly toxic, but they were not measured. This may contribute to the greater fluctuation in regression coefficients when materials are deleted.

The relative rankings of the deleted materials follow the same pattern. The rankings are identical for fabrics and thermoplastics, intermediate for foams, and poor for panels.

The data in table 6 indicate that the calculated regression equations should not be used as predictive tools, especially considering the present state of knowledge in this area. However, the results do suggest that a combined analytical/toxicological approach is potentially useful if it can be sufficiently refined through additional research.

#### SINGLE-GAS CORRELATIONS.

Literally hundreds of TDP's are released by polymeric materials. When a test animal is exposed to such a complex environment, each of the TDP will make some contribution to an observed animal response. In addition, the interaction of the various TDP's will alter their apparent toxicity. It would be difficult to quantitate the degree of contribution associated with a single toxic species under these conditions. However, it is possible, using least-squares linear regression analysis, to determine whether or not a particular TDP is associated to a discernable degree with the observed animal response, in this case  $t_1$ .

When the 71 materials that produced incapacitation are considered as a group, eight of the nine TDP's individually correlate with  $1/t_1$  values, as indicated in table 7. These correlations only include those materials with nonzero TDP yields. Seven of the correlations are significant at the 1-percent probability level ( $P_1$ ). These are the correlations for CO, NO<sub>2</sub>, HCN, SO<sub>2</sub>, HBr, HF, and H<sub>2</sub>S. The correlation for HCl is also significant, but at the 5-percent probability level. The correlation for HCHO yields, however, is not significant at even the 10-percent level. The scatter diagrams for the data in table 7 are contained in figures 14 through 22.

The regression lines for three of the TDP's that are significantly correlated with  $1/t_1$  values, HCl, HBr, and HF, have negative slopes. The yields of these TDP's are inversely proportional to observed toxicities. This is probably due to their properties as respiratory irritants, since pure-gas studies indicate that such TDP's tend to reduce an animal's respiratory minute volume (reference 11). This would result in a lower rate of inhalation of the available systemic toxicants than otherwise would occur, and  $t_1$  values subsequently increase. Although these TDP's prolong observed  $t_1$  values in rats, this does not imply that the presence of irritants in a fire environment will increase human escape potential (references 12, 13, 14, and 15). A more appropriate conclusion may be that additional research is required before materials producing significant quantities of irritants can be properly evaluated.

The regression lines for CO, HCN, H<sub>2</sub>S, NO<sub>2</sub>, and SO<sub>2</sub> have positive slopes. Carbon monoxide, HCN, and H<sub>2</sub>S are systemic toxicants. Since they are capable of producing an incapacitation response at relatively low concentrations, positive slopes are expected for these TDP's. Nitrogen dioxide and SO<sub>2</sub>, however, are considered to be respiratory irritants (for laboratory animals) at the low levels and short exposure times encountered in these tests (references 16, 17, and 18). The median yield of NO<sub>2</sub> corresponds to an average exposure concentration of 3.2 p/m in a 12.6 liter exposure chamber. The median yield of SO<sub>2</sub> corresponds to an average exposure concentration of 46 p/m. The positive correlation with  $1/t_1$  values for these TDP's, therefore, may be due to the colinearity of NO<sub>2</sub> and SO<sub>2</sub> yields with toxic nitrogen-and-sulfur-containing TDP's that were not measured.

Another possible contribution to the positive correlations is the variability of the data. The average relative standard deviations (ARSD) for the NO<sub>2</sub>, SO<sub>2</sub>, and HCHO yields are between 53 and 60 percent (reference 2). Such a wide variability in these TDP yields, combined with an ARSD of approximately 15 percent for the animal response data (reference 9), could affect the slopes of the correlation lines if only a small number of data points are involved.

The R value for HCN in table 7 corresponds to an R<sup>2</sup> value of 0.607, while the corresponding R<sup>2</sup> value for CO is 0.089. In addition, the standard error of estimate is less for HCN than for CO, even though there are fewer data points for the HCN. These data suggest that HCN rather than CO is the predominant toxic species for many of the 71 materials that produced incapacitation. On the basis of usage categories, HCN is the predominant toxicant for fabrics, flooring, and elastomers/cargo liners/insulations. Carbon monoxide is the predominant toxicant for foams, coated fabrics, and thermoplastics. Hydrogen cyanide and CO are approximately equally important for panels and panel components.

The last column in table 7 contains concentrations of selected TDP's calculated using the regression coefficients in that table. Statistically, these concentrations will produce incapacitation in a 200 g rat in 5 minutes (appendix D, of reference 9) under the experimental conditions employed in the CAMI animal exposure system. Due to the nature of these calculations, however, the predicted concentrations should only be evaluated on a qualitative basis. Calculations based on pure gas studies (appendix D, of reference 9) indicate

that a 200 g rat will incapacitate in 5 minutes when exposed to a CO concentration of 4,981 p/m. The CO concentration predicted by the regression equation in table 7, 4,516 p/m, is approximately 91 percent of the pure-gas value.

TABLE 7. CORRELATION OF INDIVIDUAL TDP YIELDS WITH  $1/t_1$  VALUES

TDP	Times Detected	R	$S_e$ (1)	P $\lambda$ (2) (%)	a (3)	b (3)	Predicted Concentration (4)
H <sub>2</sub> S	13	0.962	0.059	1	20.6	120	170
HCN	51	0.779	0.091	1	9.14	168	193
HBr	21	-0.774	0.096	1	- 1.40	295	-
HF	17	-0.626	0.077	1	- 1.59	289	-
SO <sub>2</sub>	25	0.601	0.187	1	5.7	198	-
NO <sub>2</sub>	53	0.579	0.117	1	139	181	-
HCl	44	-0.307	0.127	5	- 0.30	219	-
CO	70	0.298	0.125	1	0.48	159	4,516
HCHO	45	-0.084	-	-	-	-	-

- (1) Standard Error of Estimate
- (2) Probability Level of the Correlation
- (3)  $1000 (1/t_1) = a (\text{mg/g}) + b$
- (4) The predicted concentration (p/m) of a TDP, calculated using the equations in this table, which should produce incapacitation for a 200g rat in 5 minutes (reference 9). Chamber temperature is assumed to be 30°C.

Carbon monoxide is a stable gas, and one would not expect wall losses nor losses due to the presence of the test animal to be significant. The calculated results seem to support this expectation. Unlike CO, HCN and H<sub>2</sub>S are reactive gases, and losses from the gas phase are expected to be significant. Calculations based on pure-gas exposures for HCN predict that a concentration of 107 p/m will incapacitate a 200 g rat in 5 minutes (appendix D, of reference 9). The concentration of 193 p/m calculated from the regression equation in table 7 indicates that approximately half of the generated HCN may be lost from the gas phase in the animal exposure system. The magnitude of this result does not appear to be unreasonable. This suggests that the median HCN yield to which

the test animals were actually exposed in the CAMI test chamber was probably half the 275 p/m reported in table 2. This would be in agreement with the median  $t_i$  of 5.8 minutes. In addition, both HCN and H<sub>2</sub>S are expected to have similar toxicities (reference 19). The 170 p/m obtained for H<sub>2</sub>S in table 7 is within 12 percent of the 193 p/m value obtained for HCN, indicating an equivalent toxicity if gas-phase losses are assumed to be equal. The median animal exposure level for H<sub>2</sub>S is also probably overestimated in table 2.

#### DISCUSSION OF RESULTS.

The data upon which these correlations are based were obtained under less than optimum conditions. Since the majority of the nine TDP's are reactive, it is expected that significant quantities of these TDP's are lost from the gas phase. This is especially true when test animals are used for whole-body exposures. Therefore, the degree of correlation between  $1/t_i$  values and TDP yields is expected to increase for an experiment in which the TDP's are sampled directly from the animal exposure chamber.

The concentrations of the various TDP's to which the test animals are exposed vary as a function of time. There are also differences in the concentration-time profiles between the TDP's produced by a material. For example, material 88 (wool fabric) releases H<sub>2</sub>S as a single peak within the first minute of the test. Hydrogen cyanide and CO are released during the majority of the test period, and appear as double peaks (reference 2). Therefore, the total yield of a TDP is only a simple approximation of the environment to which the test animals are actually exposed. A more precise approach involves measuring the concentration of each TDP as a function of time and integrating the area under the curve up to the time-to-incapacitation. However, this approach is too complex for an initial feasibility study. In addition, the results suggest that the use of total TDP yields is adequate for the experimental conditions employed in these studies.

Another factor affecting the degree of correlation is the difference between the NAFEC and CAMI experimental procedures. A 250-mg sample weight and 2 l/m airflow rate are employed at NAFEC, while a 750-mg sample weight and 4 l/m airflow rate are employed at CAMI. In addition, the heating time is 5 minutes at NAFEC and 10 minutes at CAMI. These factors can result in a noticeable difference in at least CO yields for some materials (reference 2).

Although only the three systemic toxicants (HCN, CO, and H<sub>2</sub>S) are included in equation 2, the  $R^2$  value for this correlation is 0.793, versus 0.837 for equation 1. Therefore, including such irritant TDP's (in low concentrations) as NO<sub>2</sub>, SO<sub>2</sub>, HCl, HCHO and HF in the calculations does not significantly improve the correlation. There are several factors that might account for the apparent lack of influence these TDP's have on calculated  $1/t_c$  values. One factor is that their gas-phase concentrations tend to be unstable in reactive environments. This may result in a substantial reduction in their concentration before they can significantly affect the responses of the test animals. However, HCN and H<sub>2</sub>S are also reactive TDP's, and their influence is evident. This factor cannot be evaluated without first performing experiments in which TDP concentrations are monitored in the animal exposure chamber as a

function of time. An additional factor might be that the test procedures employed in the combined NAFEC/CAMI studies may tend to emphasize systemic toxicants rather than irritant TDP's. Irritants represent an important class of TDP's because of their possible negative effects on human escape potential (reference 12). These effects can occur at concentrations substantially below those required to produce short-term incapacitation. Therefore, the use of rats as the test animal and/or incapacitation as the endpoint may not be adequate for those materials that produce significant yields of irritants.

#### SUMMARY OF RESULTS

1. The coefficient of correlation between  $1/t_i$  and  $1/t_d$  values for the 56 materials producing a  $t_d$  is 0.914.
2. Carbon monoxide,  $\text{NO}_2$ , HCN, HCHO, and  $\text{HCl}$  are released by 60 percent or more of the materials tested, while  $\text{SO}_2$ , HBr, HF, and  $\text{H}_2\text{S}$  are released by a third or less of the materials.
3. The group  $t_i$  values for fabrics and panels are less than the mean  $t_i$  value for the 71 materials producing incapacitation.
4. Values of  $1/t_i$  for the 71 materials are described by the following least-squares linear regression equation in which the coefficient of correlation is 0.915.

$$1000 (1/t_c) = 16.7 (\text{HCN}) + 0.615 (\text{CO}) - 11.1 (\text{H}_2\text{S}) + 41.2 (\text{NO}_2) \\ + 6.49 (\text{SO}_2) - 0.103 (\text{HCl}) - 1.36 (\text{HCHO}) - 0.661 (\text{HBr}) \\ - 0.121 (\text{HF}) + 84.3$$

5. The following abbreviated regression equation describing the 71 materials is based on only HCN, CO, and  $\text{H}_2\text{S}$  yields and has a coefficient of correlation of 0.891.

$$1000(1/t_c) = 12.7 (\text{HCN}) + 0.627 (\text{CO}) - 12.7 (\text{H}_2\text{S}) + 81.6$$

6. The coefficients of correlation between TDP yields and  $1/t_i$  values exceed 0.95 for all usage categories except panels, for which the correlation coefficient is 0.80.
7. The coefficients of correlation between observed and predicted  $1/t_i$  values for four "unknown" materials in each of several usage categories are: panels = 0.393, foams = 0.948, fabrics = 0.968, and thermoplastics = 0.988.
8. Eight of the nine TDP yields are individually correlated with  $1/t_i$  at a probability level of 5 percent or greater; HCHO yields are not correlated with  $1/t_i$  values.

## CONCLUSIONS

1. Times-to-incapacitation are highly correlated with times-to-death.
2. Hydrogen cyanide, rather than CO, is the primary toxic species for the materials and exposure conditions discussed in this report.
3. The two usage categories which together account for the majority of the surface area in an aircraft cabin, fabrics and panels, exhibit greater relative potential toxicities than the other usage categories.
4. Times-to-incapacitation can be described by a statistical model based on selected TDP yields.
5. Only a limited number of commonly monitored TDP's are required to adequately describe times-to-incapacitation.
6. The incapacitation response is related primarily to the yields of systemic toxicants (HCN, CO, etc.) rather than the yields of irritant TDP's.
7. An analysis of residuals indicates that similar materials, those within the same usage category, often tend to group together. Therefore, a least-squares regression equation should be calculated for each family of materials.
8. Material classifications based on usage categories may not always be appropriate for correlations between animal responses and TDP yields.
9. The potential toxicities of panels are not adequately described by the measured TDP yields.

## RECOMMENDATIONS

1. The relative importance of HCN and CO as primary toxicants in enclosure fires involving largely synthetic materials should be investigated.
2. The effect of exposure conditions (flaming combustion versus oxidative pyrolysis) on the results contained in this report should be investigated.
3. Additional TDP's contributing to the incapacitation response should be identified and included in the regression equations.
4. The adequacy of present animal test protocols for comparing materials which release significant quantities of irritant TDP should be investigated.
5. The applicability of a combined analytical/toxicological approach should be investigated through additional research.
6. The predictive powers of the regression equations for each usage category should be investigated by evaluating untested materials.

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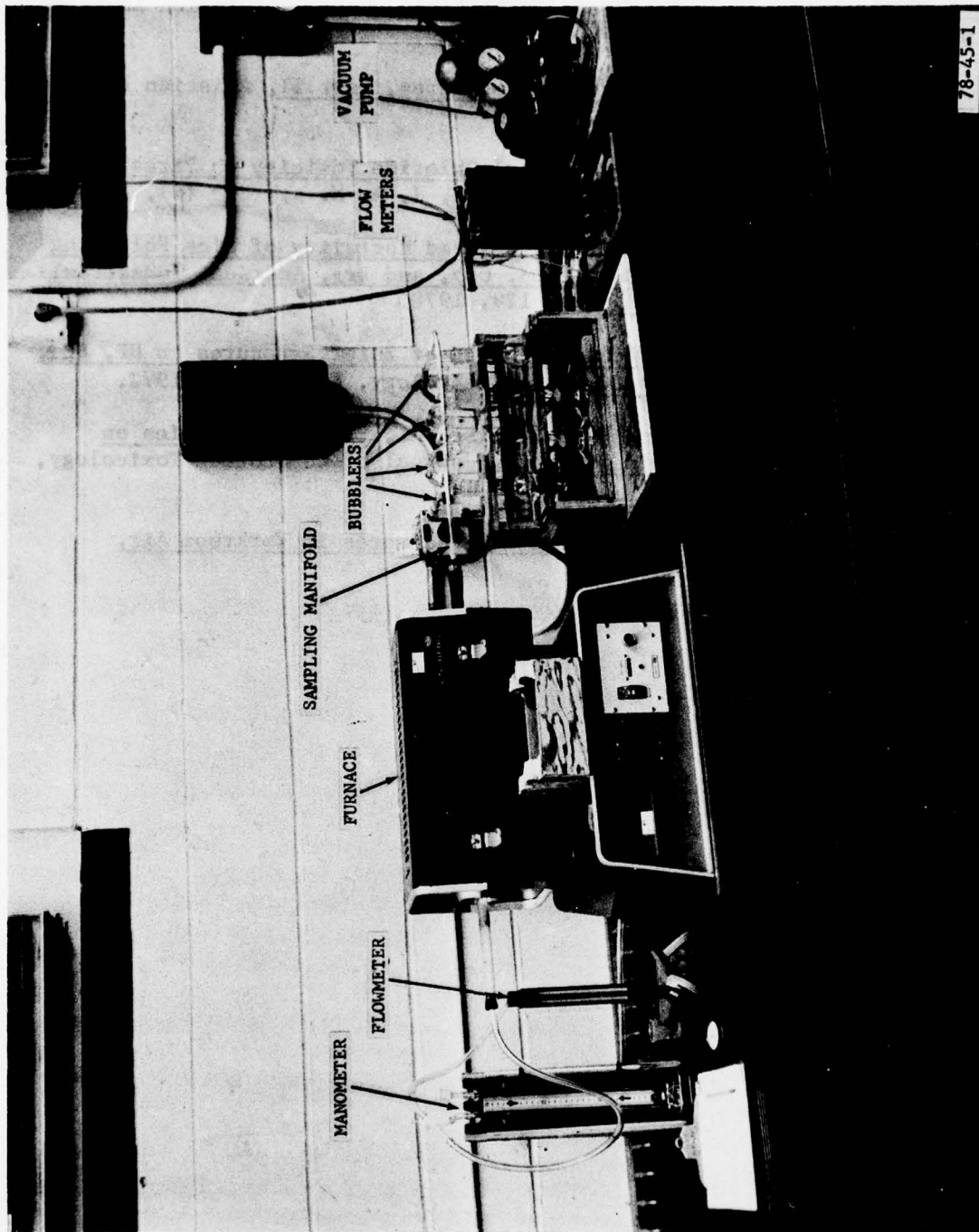
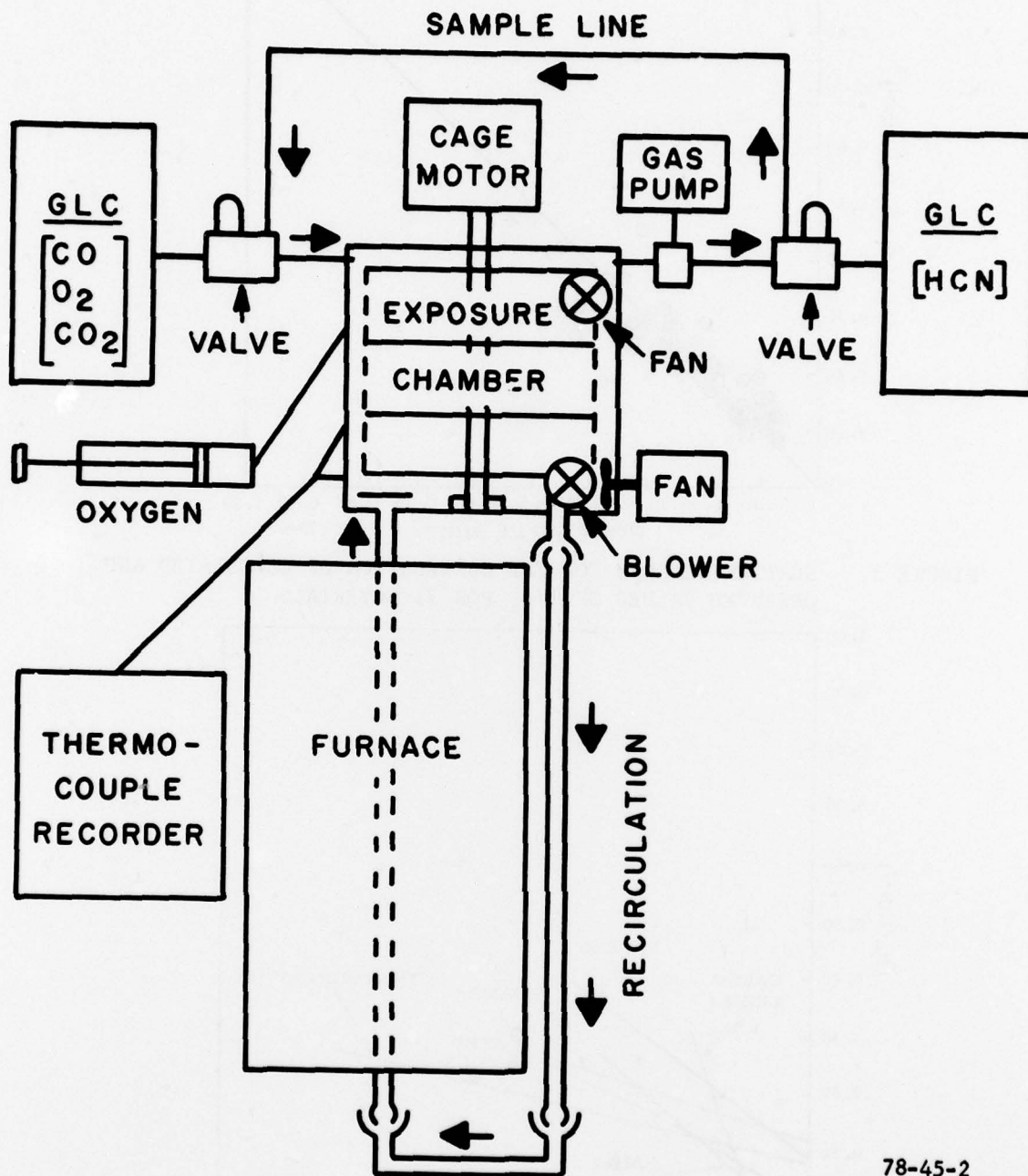


FIGURE 1. NAFEC COMBUSTION TUBE AND TDP SAMPLING SYSTEM



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FIGURE 2. CAMI ANIMAL EXPOSURE CHAMBER

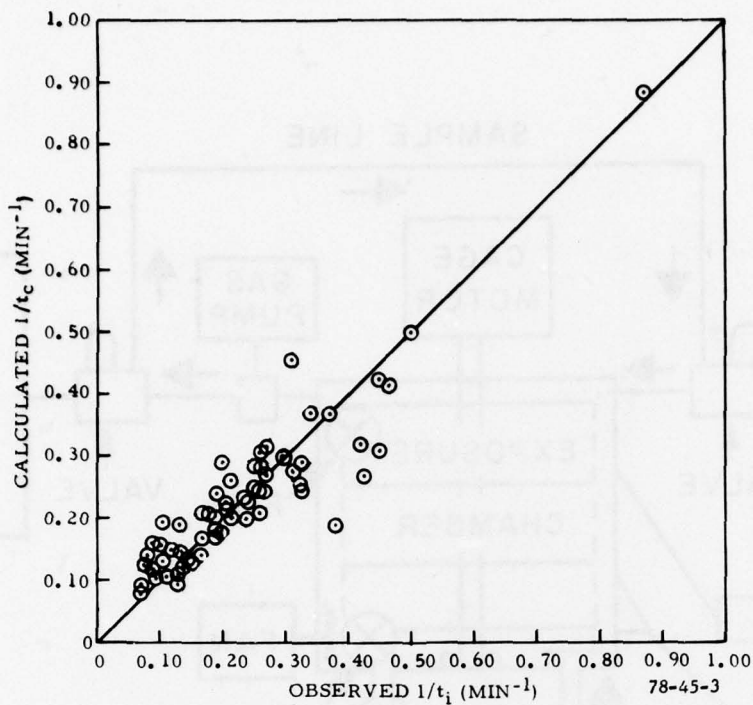


FIGURE 3. SCATTER DIAGRAM FOR THE CORRELATION OF CALCULATED AND OBSERVED VALUES OF  $1/t_i$  FOR 71 MATERIALS

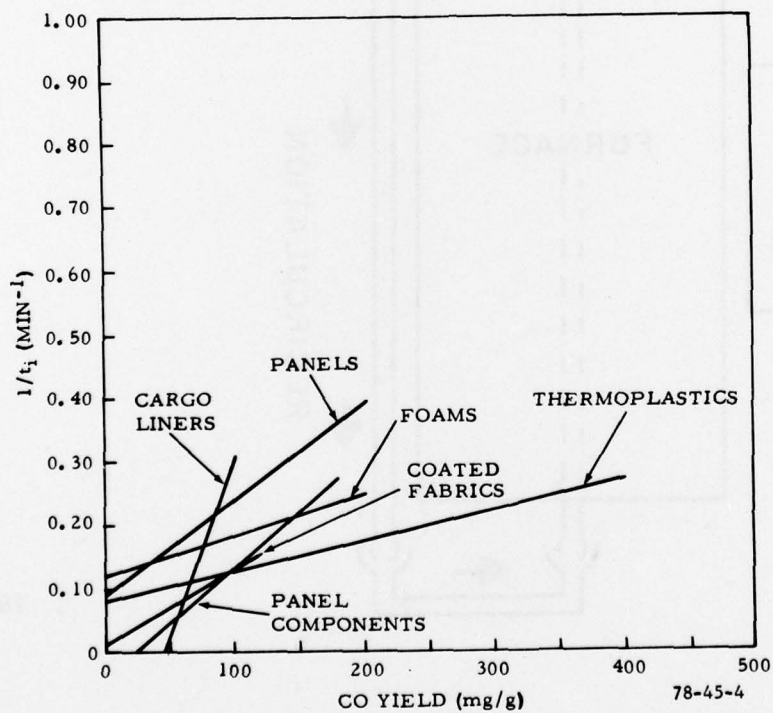


FIGURE 4. A COMPARISON OF CO LINEAR REGRESSION PLOTS FOR VARIOUS MATERIAL USAGE CATEGORIES

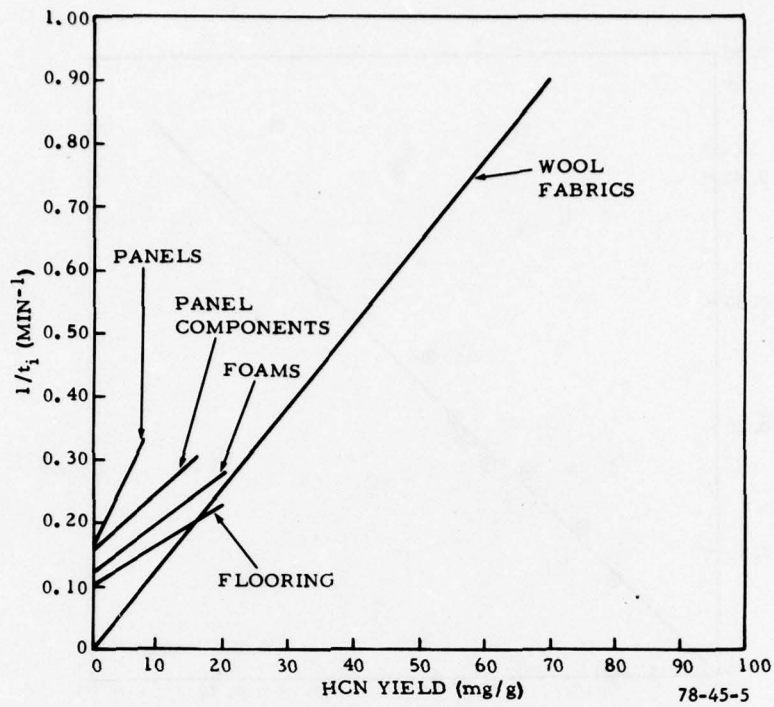


FIGURE 5. A COMPARISON OF HCN LINEAR REGRESSION PLOTS FOR VARIOUS MATERIAL USAGE CATEGORIES

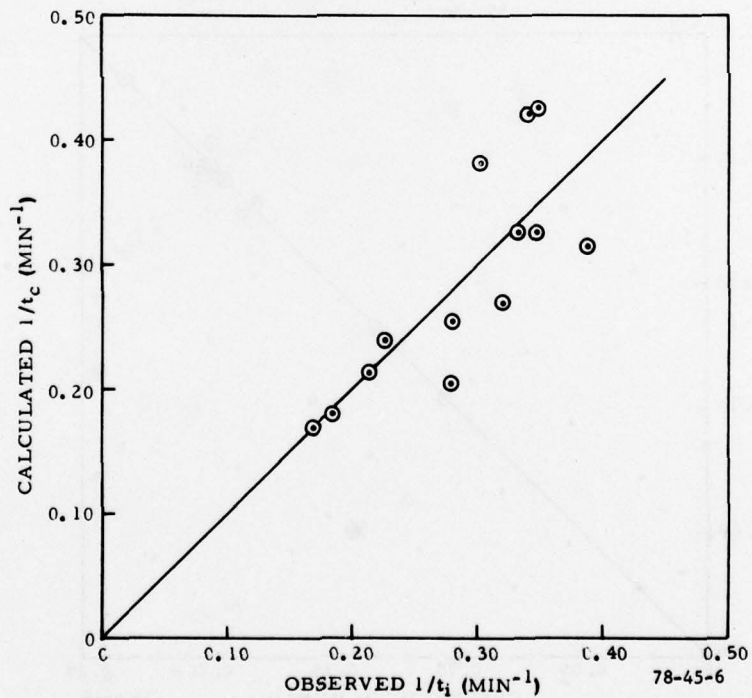


FIGURE 6. PANELS - COMPARISON OF CALCULATED AND OBSERVED  $1/t_1$  VALUES

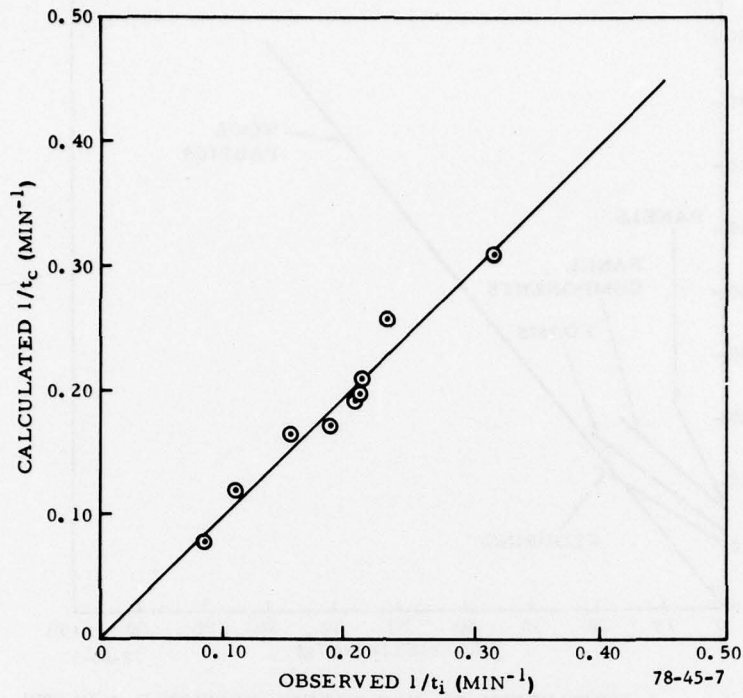


FIGURE 7. PANEL COMPONENTS - COMPARISON OF CALCULATED AND OBSERVED  $1/t_i$  VALUES

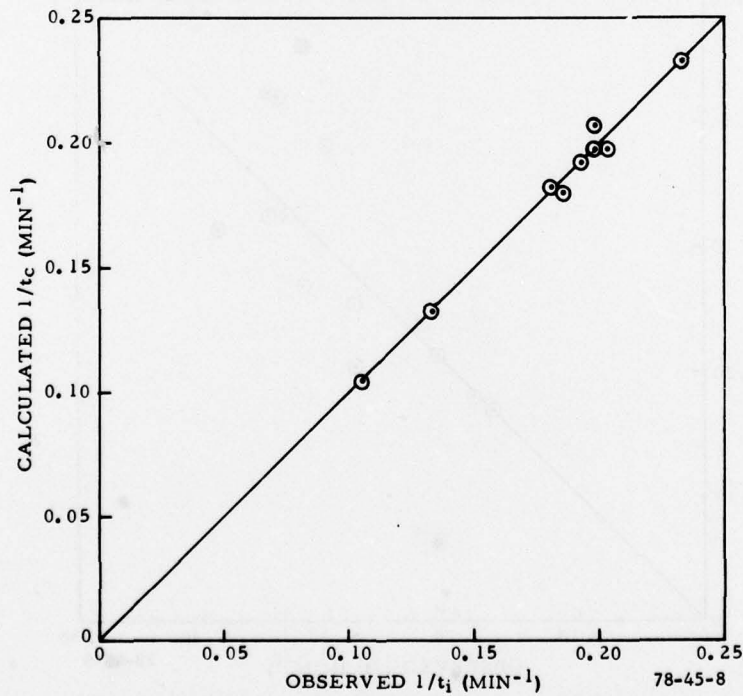


FIGURE 8. FOAMS - COMPARISON OF CALCULATED AND OBSERVED  $1/t_i$  VALUES

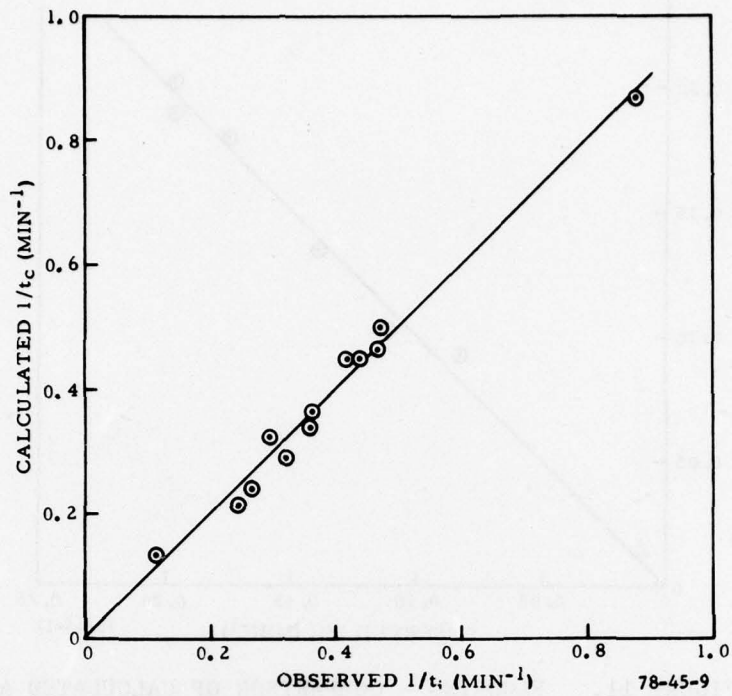


FIGURE 9. FABRICS - COMPARISON OF CALCULATED AND OBSERVED  $1/t_i$  VALUES

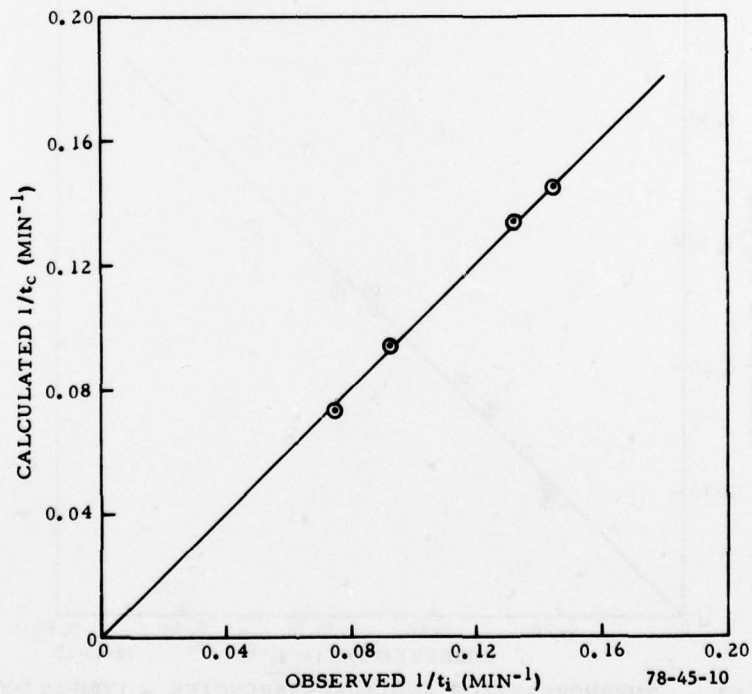


FIGURE 10. COATED FABRICS - COMPARISON OF CALCULATED AND OBSERVED  $1/t_i$  VALUES

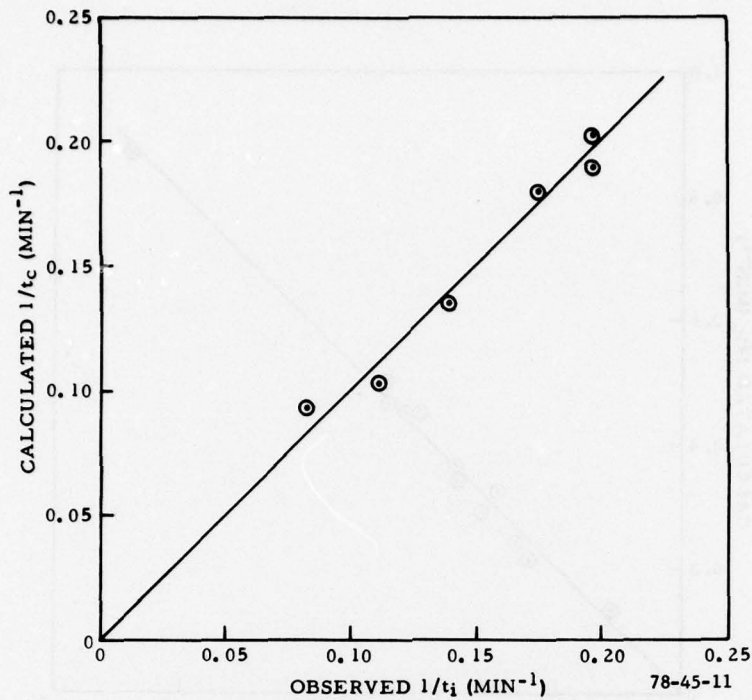


FIGURE 11. FLOORING - COMPARISON OF CALCULATED AND OBSERVED  $1/t_i$  VALUES

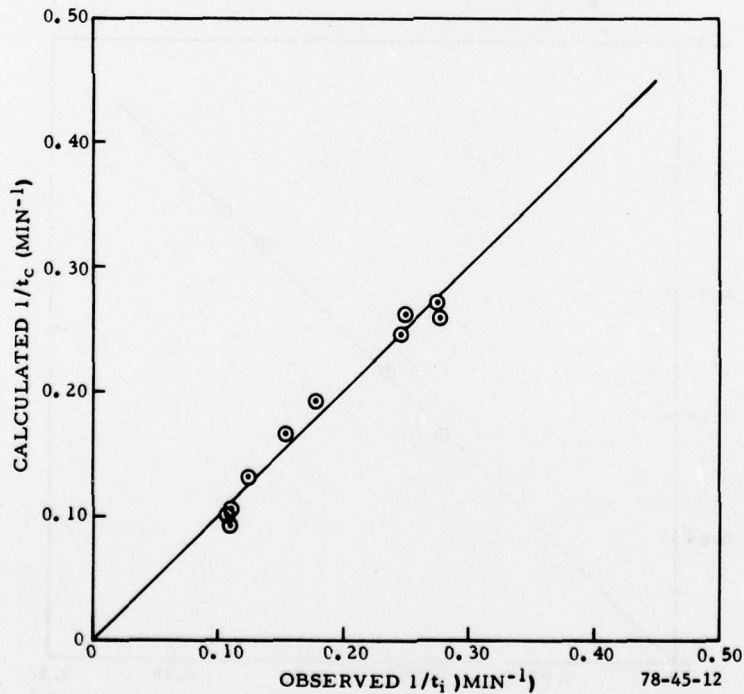


FIGURE 12. THERMOPLASTICS AND TRANSPARENCIES - COMPARISON OF CALCULATED AND OBSERVED  $1/t_i$  VALUES

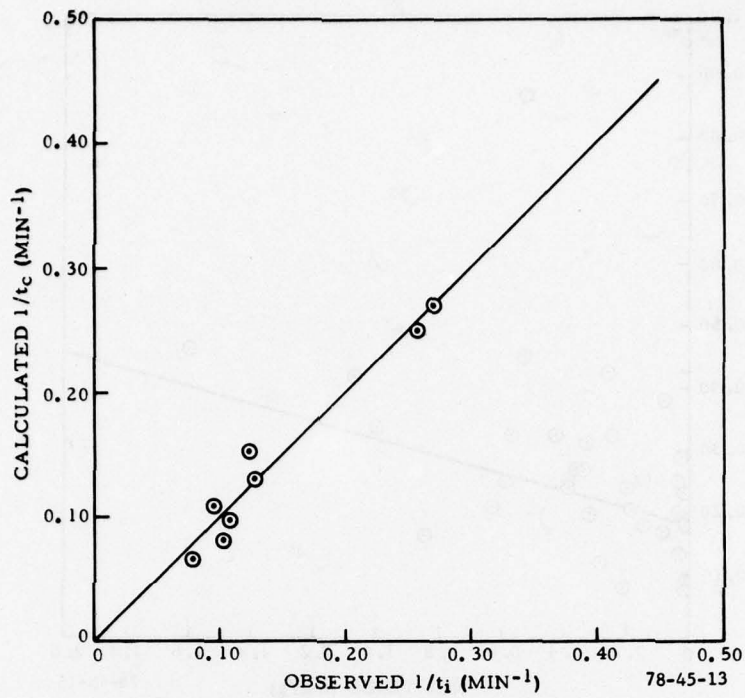


FIGURE 13. ELASTOMERS, INSULATIONS, AND CARGO LINERS - COMPARISON OF CALCULATED AND OBSERVED  $1/t_1$  VALUES

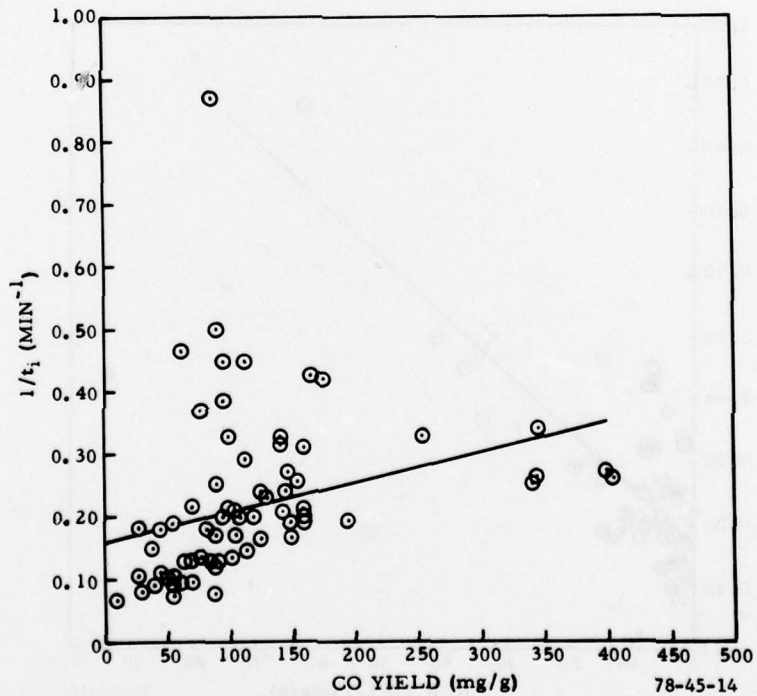


FIGURE 14. CORRELATION OF  $1/t_1$  VALUES WITH NONZERO CO YIELDS

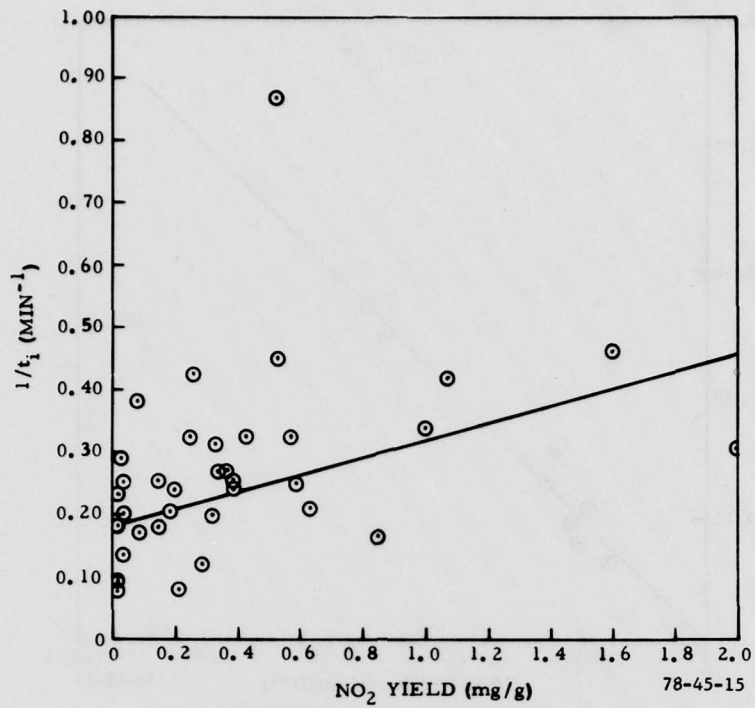


FIGURE 15. CORRELATION OF  $1/t_i$  VALUES WITH NONZERO  $\text{NO}_2$  YIELDS

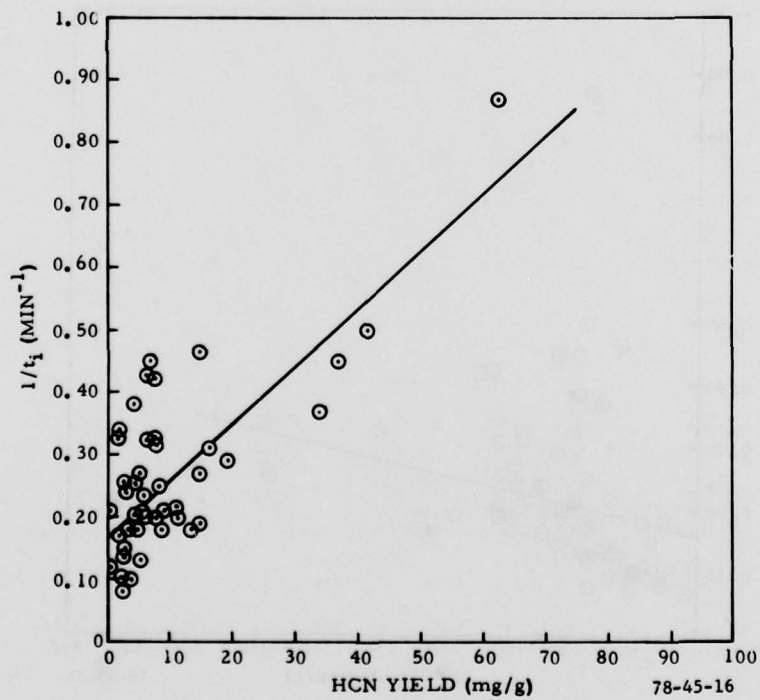


FIGURE 16. CORRELATION OF  $1/t_i$  VALUES WITH NONZERO HCN YIELDS

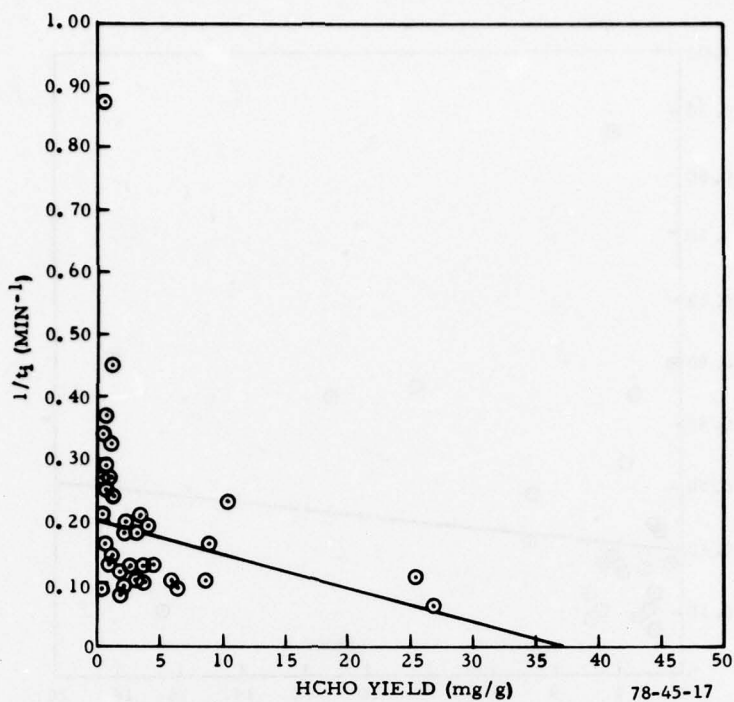


FIGURE 17. CORRELATION OF  $1/t_1$  VALUES WITH NONZERO HCHO YIELDS

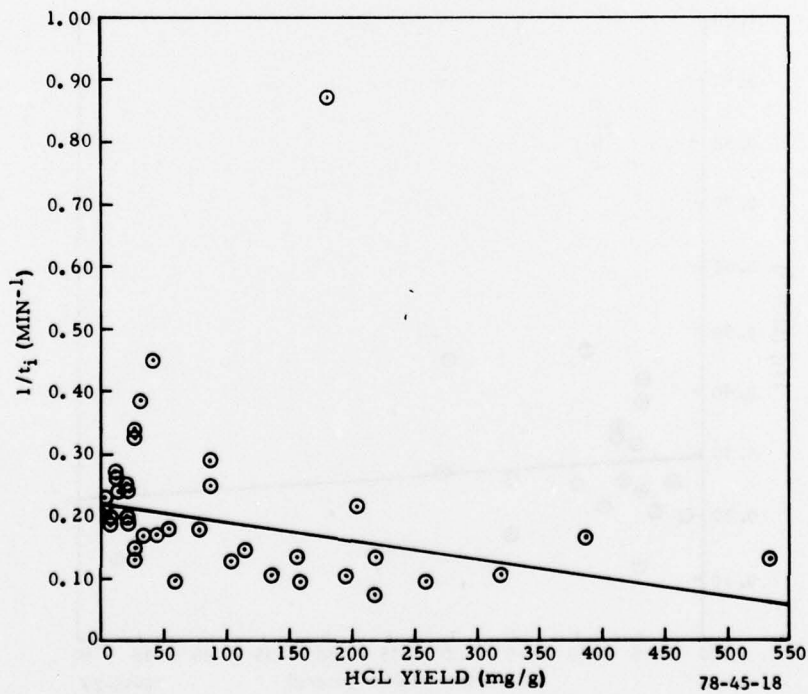


FIGURE 18. CORRELATION OF  $1/t_1$  VALUES WITH NONZERO HCL YIELDS

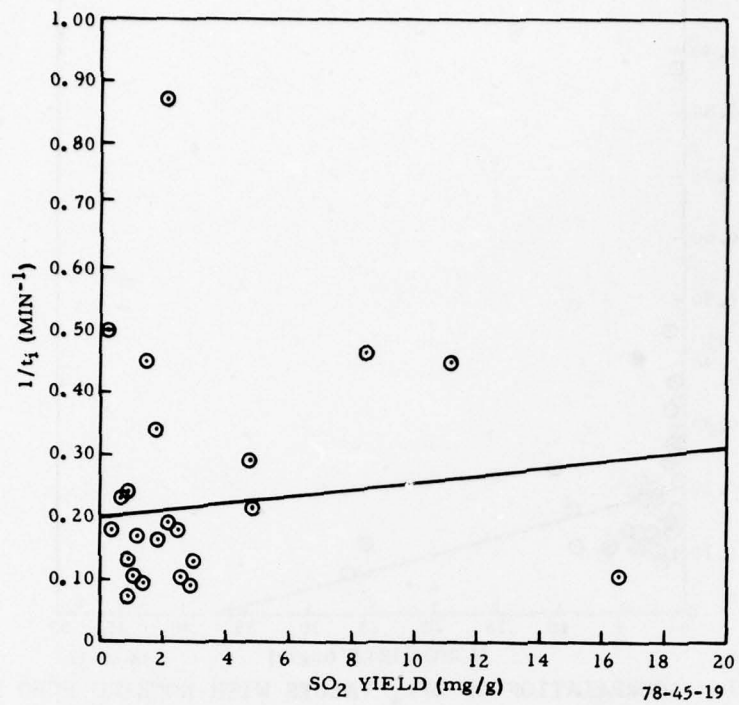


FIGURE 19. CORRELATION OF  $1/t_1$  VALUES WITH NONZERO SO<sub>2</sub> YIELDS

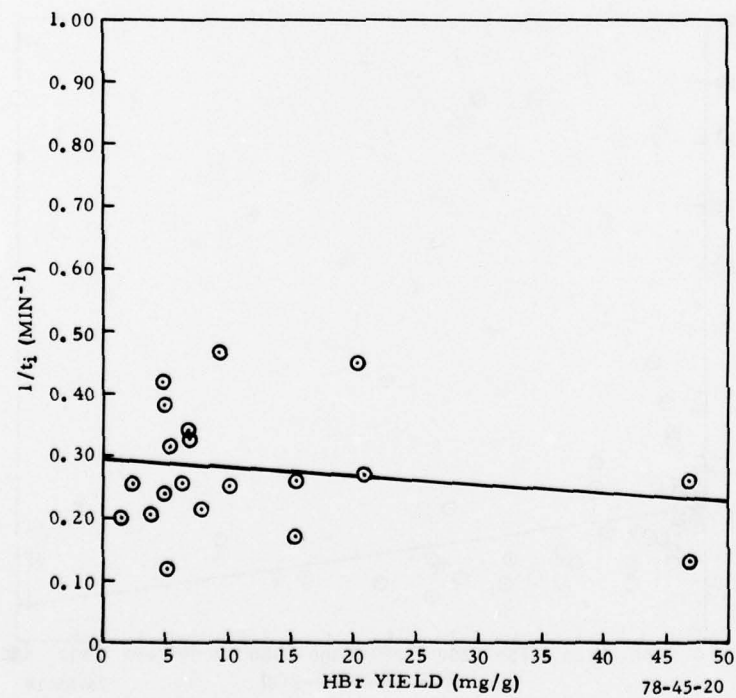


FIGURE 20. CORRELATION OF  $1/t_1$  VALUES WITH NONZERO HBr YIELDS

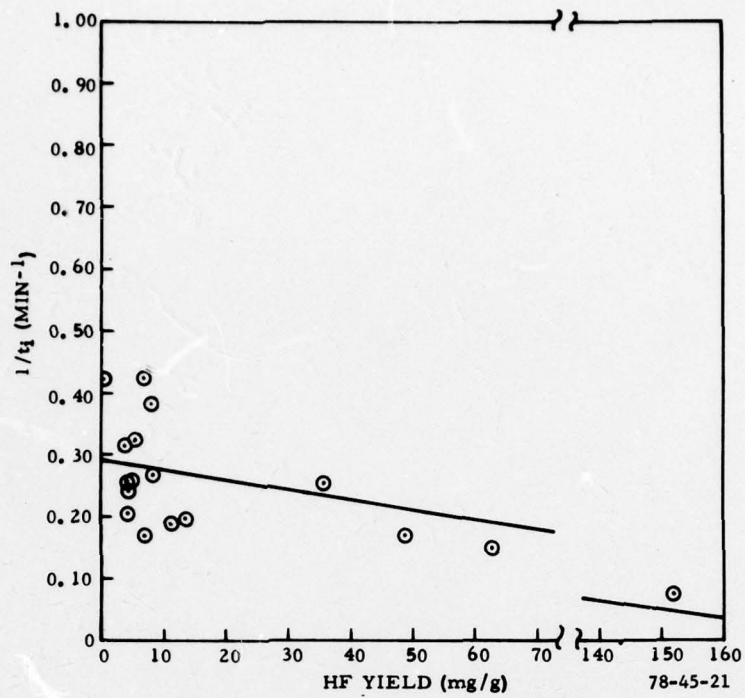


FIGURE 21. CORRELATION OF  $1/t_1$  VALUES WITH NONZERO HF YIELDS

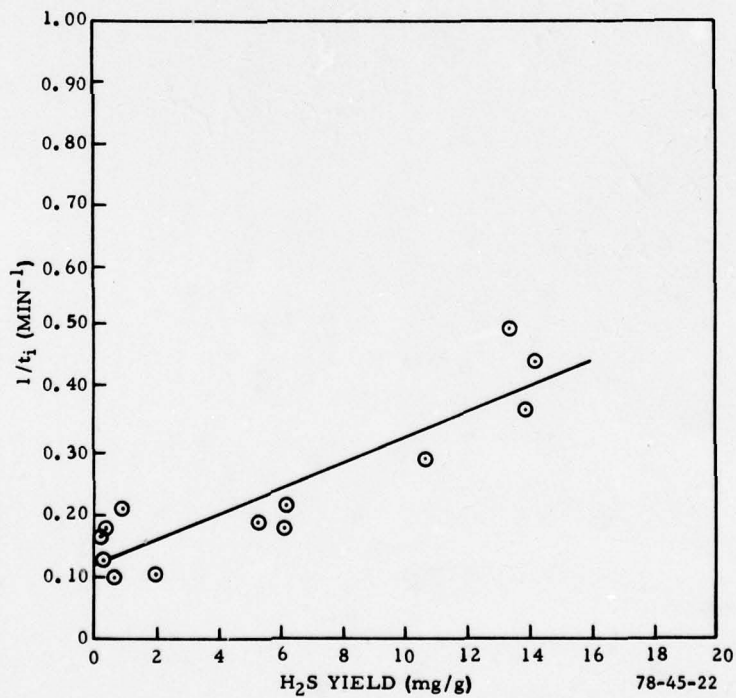


FIGURE 22. CORRELATION OF  $1/t_1$  VALUES WITH NONZERO H<sub>2</sub>S YIELDS