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A PRELIMINARY COMPARISON OF LABORATORY METHODS FOR ASSIGNING A RELATIVE TOXICITY RANKING TO AIRCRAFT INTERIOR MATERIALS

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16. Abstract

Thirteen aircraft interior materials have been tested in order to compare the National Bureau of Standards (NBS) smoke chamber and the microcombustion tube furnace as methods for generating toxic combustion gases. The microcombustion tube furnace ranked nitrogen- and chloride-containing test materials of known composition according to the expected order based upon HCN and HCl concentrations. The nitrogen-containing test materials were not ranked in the expected order by measuring peak gas concentrations in the NBS smoke chamber.

Several methods of sampling combustion gases from the NBS smoke chamber have been investigated in an attempt to maximize sample recovery. These methods include colorimetric detector tubes, bag sampling, impinger sampling, and syringe sampling. In general, syringe sampling resulted in the best sample recoveries of the methods tested in this study.

Animal toxicity tests were conducted for nine aircraft interior materials using Sprague-Dawley rats. A relative toxicity ranking of these predominantly nitrogen-containing materials has been determined by exposing the test animals to the combustion products obtained by decomposing the materials in a combustion tube furnace. A preliminary correlation of animal toxicity data with measured gas concentrations indicates that a possible relationship exists between times-to-incapacitation and hydrogen cyanide concentrations produced by the test materials. The coefficient of correlation is 0.93.

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PREFACE

A determination of the relative toxicities of materials must ultimately involve a study of the physiological effects of combustion products, as well as the analysis of combustion gases. This report is therefore comprised of two parts. The first part is an evaluation of laboratory test methods for generating combustion products from aircraft interior materials.

The more appropriate test method, as determined by the analysis of selected combusiton gases, was then further evaluated in part II by conducting animal tests. The second part of this report describes the correlation of animal toxicity data with toxic gas concentrations, as determined in part I of this study.

Much of the data relating to bag sampling was obtained through the assistance of Mr. Gus Sarkos, who also gave freely of his insights and knowledge in the area of material flammability. The NBS smoke chamber was operated by Mr. Richard Johnson, while Mr. Ray Feher assisted in the operation of the micro-combustion tube furnace.

The animal toxicity tests were conducted at the Civil Aeromedical Institute (CAMI), in Oklahoma City, under the direction of Dr. Paul Smith, Chief, Aviation Toxicology Laboratory, and Dr. Charles Crane, Chief, Biochemistry Research. The author would like to thank Mr. Donald Sanders, Mr. John Abbott, and Mr. Boyd Endecott for their valuable assistance in conducting the animal tests. The test materials were supplied by the National Aviation Facilities Experimental Center (NAFEC), as received from the Aerospace Industries Association.

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INTRODUCTION

PURPOSE.

The purpose of this preliminary study was to select an appropriate laboratory test method for generating and measuring toxic combustion gases released by aircraft interior materials.

BACKGROUND.

The Federal Aviation Administration (FAA) is considering airworthiness regulations that would limit the use of aircraft interior materials in terms of their toxic gas emission characteristics during combustion (reference 1). In this regard, approximately 140 samples of typical cabin interior materials which are in current use were obtained with the cooperation of the Aerospace Industries Association (AIA) and leading seat manufacturers. Seventy-five representative materials were chosen for testing, to be ranked according to their potential toxicity when burned.

The National Bureau of Standards (NBS) smoke chamber and the combustion tube furnace are often employed to generate combustion products. These two test methods have been compared in this study to determine which approach is more appropriate for ranking aircraft interior materials. The unique features of the NBS smoke chamber make it an attractive test method for combustion gas analysis. It offers several qualitative advantages as compared to the combustion tube furnace; such as (1) data on smoke density and toxic gases can be obtained simultaneously, (2) the rates-of-evolution of combustion gases can be measured, (3) it can be used as an animal exposure chamber, and (4) test conditions and decomposition products more closely simulate those encountered in a full-scale fire. However, the ability of a test method to provide data of adequate reliability, as dictated by the objective of a study, is equally important. The NBS smoke chamber and the combustion tube furnace have therefore been compared on the basis of their ability to rank materials of known composition in the expected order.

Since this was an exploratory effort, only 13 of the 75 materials were tested. The study was further expedited by limiting combustion gas analysis to carbon monoxide (CO), hydrogen cyanide (HCN), and hydrogen chloride (HCl).

EXPERIMENTAL SECTION

DESCRIPTION OF TEST MATERIALS.

The materials employed in this study were selected from among the 75 materials that will undergo a full program of testing. A brief description of the 13 test materials is contained in table 1. Materials were chosen which represent the various usage categories found in an aircraft, such as fabrics, foams, carpets, and panels. Since only CO, HCN, and HCl concentrations were measured, material compositions were limited to poly(vinylchlorides) (PVC), urethanes, wools, and Nomex®.

LABORATORY TEST METHODS.

NBS SMOKE CHAMBER. The NBS smoke chamber was a standard commercial design, as supplied by American Instrument Company. This instrument has been fully described in the literature (reference 2). Only minor modifications were made on the chamber, such as painting all uncoated interior surfaces with epoxy paint to reduce corrosion and sample losses. Additional sampling ports were also added. All tests in this series were conducted under standard flaming-exposure conditions (reference 2), with a test time of either 8 minutes (bag sampling) or 10 minutes (direct sampling).

MICROCOMBUSTION TUBE FURNACE. The furnace used in this study was a Thomas Micro Preheater Furnace (5304-BIO), with a heating chamber 110 millimeters (mm) long by 13 mm in diameter. A maximum temperature of 700° Centigrade (C) can be attained when used with a variable transformer. Temperature stability was typically ± 10° C as measured by a chromel-alumel thermocouple. The combustion train is illustrated in figure 1.

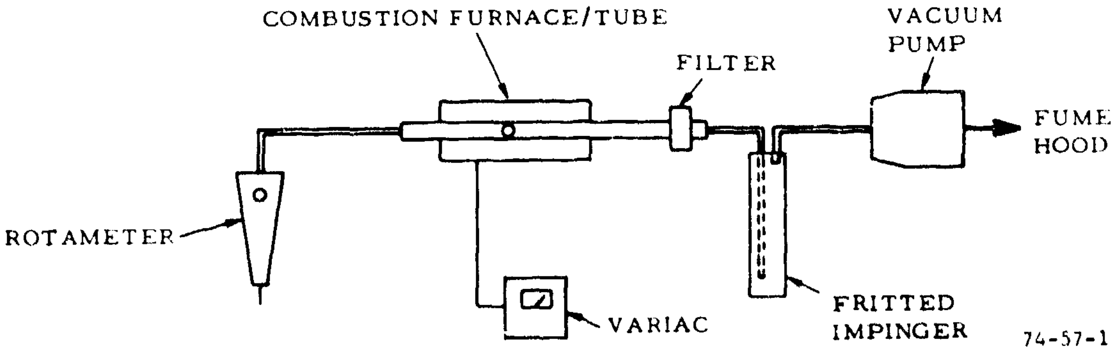

FIGURE 1. SCHEMATIC DIAGRAM OF THE MICROCOMBUSTION TUBE ASSEMBLY

TABLE 1. COMPOSITION OF THE TEST MATERIALS

Material Number	Material Category	Material Composition	Gases Measured
24	Panel	Epoxy-Fiberglas, PVC Core, Epoxy-Fiberglas	CO, HCl
33	Carpet	Wool Pile, Polyester Backing, Latex Coating	CO, HCN
34	Carpet	Wool Pile, Polyester Backing, Latex Coating, Urethane Pad, Nylon Scrim	CO, HCN
56	Panel	PVC, Aluminum, Nomex® Honeycomb, Aluminum	CO, HCl
67	Panel	PVC, Phenolic-Fiberglas, Nomex® Honeycomb, Epoxy-Fiberglas	CO, HCl
78	Fabric	Nomex®	CO, HCN
79	Foam	Polyether Urethane	CO, HCN
81	Fabric	Poly(Vinylchloride)	CO, HCl
82	Fabric	Wool (76 Percent), PVC (24 Percent)	CO, HCl, HCN
86	Foam	Poly(Vinylchloride)	CO, HCl
88	Fabric	Wool	CO, HCN
96	Fabric	Wool (49 Percent), PVC (51 Percent)	CO, HCl, HCN
143a	Foam	Polyester Urethane	CO, HCN

Laboratory air was drawn through the combustion tube at a flow rate of 200 milliliters per minute (ml/min) as measured by a rotameter. The air flow was maintained between tests by the use of an alternate combustion tube in order to stabilize the temperature of the furnace at a nominal value of 600°C. The combustion tube was normally an 8-mm outer diameter (o.d.) by 280-mm Pyrex® tube. A 13-mm o.d. tube was used for testing the panel materials. The filter assembly consisted of a 10-mm polypropylene filter contained in a polyethylene filter holder.

A sample of test material, weighing approximately 10 milligrams (mg), was positioned in the combustion tube so that it would rest at the center of the furnace. This was also the position at which the furnace temperature was measured. The test procedure involved (1) stabilizing the furnace temperature at 600°C, (2) inserting the combustion tube into the furnace and attaching it to the filter assembly, and (3) heating the sample for a period of 10 minutes. In addition to analyzing the contents of the impinger, both the combustion tube and the filter assembly were examined for adsorbed combustion gases.

A modified system was used to measure carbon monoxide concentrations generated using the microcombustion tube furnace. The airflow through the combustion tube was maintained by a tank of purified air. A 12-liter Saran® bag that had been equipped with a stopcock was attached directly to the exit of the combustion tube. Since a flow rate of 200 ml/min passed through the combustion tube for a period of 10 minutes, a total sample volume of 2 liters was collected in the Saran® bag. The CO content of the bag was then measured by infrared spectroscopy.

Impinger collection efficiency was determined for HCN by testing various weights of Nomex® and collecting the combustion products in two fritted impingers which were in series. Sample retention in the first impinger was better than 99.5 percent up to an impinger loading of 60 micrograms per milliliter (μg/ml). Although collection efficiency was less than 98 percent at an impinger loading of 107 μg/ml, experimental concentrations obtained with the various materials never exceeded this value.

The loss of cyanide due to aeration of the sample was also investigated. A known concentration of sodium cyanide (NaCN) in 0.1 molar (M) potassium hydroxide (KOH) was calibrated with the cyanide ion-selective electrode, and a 25-ml aliquot was transferred to a 50-ml fritted impinger. The impinger was attached to a standard combustion train, and 200 ml/min of air was drawn through the impinger for 10 minutes. The cyanide concentration in the aerated sample was then remeasured by the ion-selective electrode. A 10^{-4} M solution exhibited an apparent 6-percent increase in concentration, while duplicate 10^{-5} M solutions exhibited an apparent 12-percent decrease and a 10-percent increase in cyanide concentration. It was concluded that sample losses were less than experimental error.

GAS SAMPLING TECHNIQUES USED WITH THE NBS SMOKE CHAMBER.

BAG SAMPLING (See Reference 7). Eight sample bags were housed in an airtight wooden enclosure, approximately 1 foot by 4 feet by 3 feet, under a vacuum of 8 inches of mercury. The vacuum in the box was chosen so that an evacuated bag would fill with approximately 10 liters of sample in a period of 10 seconds. The bags were made of Saran and had a capacity of 12 liters. The bags were attached to a glass sampling probe in the top of the NBS smoke chamber through a polypropylene transfer line and a polypropylene manifold containing stainless steel three-way valves. The bags were removed from the box after the test had been completed, and the contents were analyzed with colorimetric detector tubes. However, the sample obtained at 8 minutes was reserved for infrared analysis.

DIRECT SAMPLING WITH COLORIMETRIC DETECTOR TUBES.

Direct sampling was used to monitor HCl and hydrogen fluoride (HF), since the concentrations of these gases are unstable and cannot be monitored by bag sampling. Samples were obtained by attaching a detector tube to a 6-mm by 12-inch glass probe in the rear wall of the NBS sample chamber. The contents of the chamber were sampled at approximately 1-minute intervals during the 10-minute test period. The times at which the first pump stroke was initiated and the last pump stroke was completed were noted, and the sample time was recorded as the midpoint of the sampling interval. The actual sampling time varied between 15 and 50 seconds, depending upon the number of strokes and the flow resistance of the detector tube.

SYRINGE SAMPLING. Samples of the combustion gases were taken directly from the NBS smoke chamber with 30-ml, gas-tight syringes. The syringes were constructed of glass, and both the plungers and hubs were made of Teflon®. The sampling probe consisted of a flexible Teflon® needle (Hamilton No. KF20TF), 12 inches in length, encased in a glass tube for rigidity. Samples were taken from the geometric center of the chamber through a sampling port located in the rear wall of the chamber. Samples were taken at 1-minute intervals. Immediately after sampling, 5 ml of the appropriate absorbing solution were added to the gas syringe. The syringe was shaken for 5 minutes, and the absorbing solution was transferred to a 1-ounce polyethlene bottle for later analysis by ion-selective electrode. The syringe was then rinsed with a second 5-ml portion of solution, which was also added to the storage bottle.

IMPINGER SAMPLING. A sampling system employing fritted impingers for sample collection was also used with the NBS smoke chamber. The basic components of the sampling system were a 6-mm o.d. glass sampling probe, a polypropylene transfer line, filter, sampling manifold containing 10 fritted impingers, a rotameter, and a portable vacuum pump. The schematic of this system is presented in figure 2.

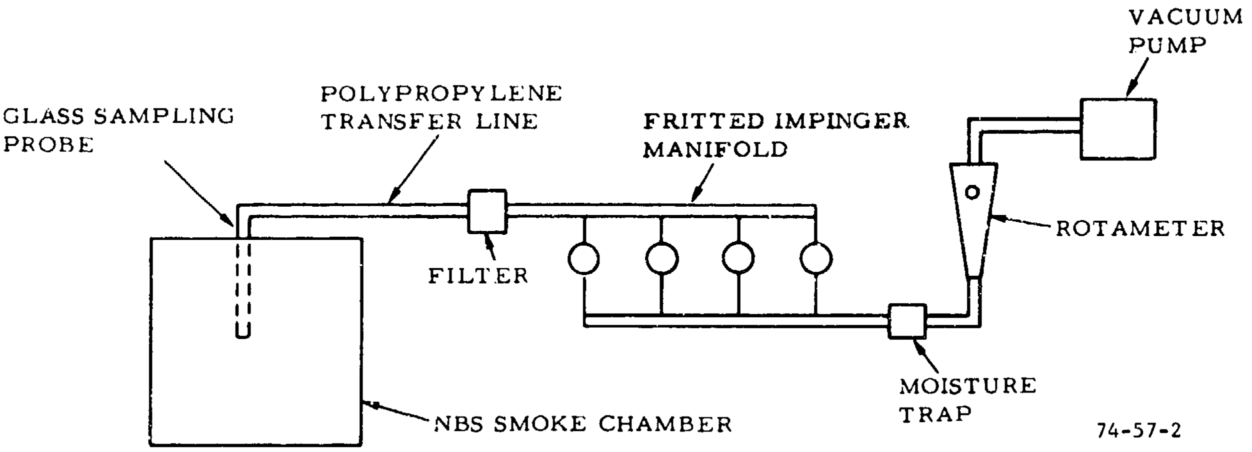

FIGURE 2. SCHEMATIC DIAGRAM OF THE FRITTED IMPINGER SAMPLING SYSTEM

The impingers were used to collect consecutive 1-minute samples during the 10-minute test period. After the completion of a test, each impinger was back-flushed three times with air, and its contents were analyzed by ion-selective electrodes. The filter and filter assembly were also analyzed for the presence of adsorbed sample. Prior to each test, the sampling system was assembled and the flow rate through each impinger was calibrated by placing a wet-test meter upstream of the glass sampling probe. The flow rates for the various impingers generally ranged between 300 and 500 ml/min due to the nonuniform flow resistances of the fritted discs. The value indicated on the rotameter during the flow calibration was recorded, and this value was maintained during the test.

The filter assembly consisted of a Teflon® filter holder machined from 2-inch-diameter Teflon® stock. The filter was a 47-mm polypropylene prefilter with a pore size of 10 microns. The sampling manifold consisted of 10 T-bore glass stopcocks with Teflon®-clad plugs which were connected to the impingers with polypropylene tube fittings. The exhaust manifold was similar in construction, except that Tygon® tubing was used. Each stopcock controlled the flow through a 50-ml fritted impinger equipped with a 9-mm fritted disc. The nominal pore size of the frit was 60 microns. The impingers were filled with 25 ml of the appropriate absorbing solution immediately prior to a test.

INSTRUMENTATION.

COLORIMETRIC DETECTOR TUBES. Draeger colorimetric detector tubes obtined from National Mine Service Co. were used in this study. The only exceptions were the HCl detector tubes, obtained from Mine Safety Appliances, which were used because of their higher range (600 ppm). Both types of detector tube were used in conjunction with the recommended hand-operated pump.

ION-SELECTIVE ELECTRODES. Chloride and cyanide measurements were made with Orion solid state electrodes in conjunction with an Orion double-junction reference electrode. Potentials were measured with an Orion Model 801 digital pH/millivolt (mV) meter. Although the potential of the chloride electrode was stable, the potential of the cyanide electrode would continue to drift for as long as 20 minutes. As a practical matter, the potential of the cyanide electrode was recorded after a period of 10 minutes. This procedure resulted in a reproducible calibration curve.

Both electrodes were calibrated daily when in use. When not in use, the electrodes were stored in distilled water. Combustion gases containing HCN were collected in 0.1 M KOH, while samples containing only HCl were collected in distilled water. All potentials were measured at ambient temperature (22°C) with a constant rate of stirring.

Corrections were not made for sulfide concentrations, which would significantly affect the response of the cyanide electrode. Apparent cyanide concentrations are therefore greater than actual concentrations for materials such as the wools. The chloride electrode was also subject to similar interferences.

CARBON MONOXIDE ANALYZER. A Mine Safety Appliance Model 303 LIRA was used to continuously monitor CO levels in the NBS smoke chamber. The sample gases, at a flow rate of 2 liters per minute, were drawn through a glass sampling probe which extended a distance of 18 inches below the top of the chamber (geometric center). After leaving the probe, the sample gases passed through a glass-fiber filter and a cold trap at -8° C before entering the LIRA. The gases were then exhausted back into the chamber so that a vacuum would not be created. The range of this instrument was 0-2,000 parts per million (ppm) CO.

DISCUSSION OF RESULTS

GAS SAMPLING TECHNIQUES.

Several methods of sampling combustion gases from the NBS smoke chamber have been investigated in an attempt to maximize sample recovery. These techniques included (1) sampling with colorimetric detector tubes directly from the chamber, (2) bag sampling followed by detector tube analysis, (3) sample collection in liquid-filled impingers, and (4) syringe sampling.

COLORIMETRIC DETECTOR TUBES. Peak gas concentrations were felt to be an important parameter; therefore, concentration data for the combustion gases were obtained as a function of time. The simplest approach was to sample the contents of the NBS smoke chamber at 1-minute intervals using Saran® sample bags. The contents of the bags were analyzed with colorimetric detector tubes and the approximated concentration-time curves were constructed from the individual data points.

The decision as to whether or not a particular gas could be collected for qualitative analysis by bag sampling was made by comparing detector tube measurements obtained from bag samples with those obtained by simultaneously sampling directly from the NBS smoke chamber with detector tubes. Any differences in apparent concentrations were attributed to sampling losses. A comparison of maximum concentrations obtained using these techniques appears in table 2. These results indicate that bag sampling losses for the more reactive gases, such as HCl and HF, are approximately 90 percent when compared with direct sampling. Although the apparent concentrations of the other gases show a better agreement between the two sampling techniques, this agreement does not preclude the fact that sampling losses occur for these gases. Such losses may be difficult to detect when colorimetric detector tubes are used as the method of analysis.

TABLE 2. MAXIMUM CONCENTRATIONS OF COMBUSTION GASES AS DETERMINED BY DIRECT AND BAG SAMPLING FROM THE NBS SMOKE CHAMBER*

	Direct Sampling		Bag Sampling		
Combustion Gas	Time (Min)	Concentration (ppm)	Time (Min)	Concentration (ppm)	Ratio** (Percent)
HCl	1.5	2,300	1.0	200	9
HF	1.0	30	0.5	5	17
HCN	4.0	20	4.0	25	125
NO_2	5.0	40	3.0	30	75
SO_2	16.0	600	12.0	600	100

*Analysis by colorimetric detector tubes
**Ratio of maximum concentrations, bag sampling/direct sampling

Nitrogen dioxide concentrations which resulted from burning a wool material in the NBS smoke chamber were measured using both direct sampling with colorimetric detector tubes and the modified Griess-Saltzman procedure (reference 3) in conjunction with syringe sampling. The concentration-time profiles are shown in figure 3. Similar data for HCl are contained in figure 4. Useful qualitative information can be obtained with detector tubes, as illustrated in figure 4. Syringe sampling indicates that the HCl concentration in the NBS smoke chamber reaches a plateau and remains relatively stable; whereas, direct sampling with detector tubes leads to the opposite conclusion. In this case, the HCl concentration reaches a peak and immediately begins to decay. This qualitative difference is probably due to the adsorption of HCl on soot particles (references 4 and 5). The sample gases are filtered by a precleaning layer in the colorimetric detector tubes, but the total sample, including particulates, is collected by syringe sampling.

Colorimetric detector tubes are simple, inexpensive devices which have been found to be useful in many phases of combustion gas analysis. However, the above data suggest that, to varying degrees, colorimetric tubes are not the most appropriate method of analysis for complex mixtures of combustion gases. Although they provide useful qualitative information, quantitative data is difficult to obtain using the sampling procedures described in this report (appendix A, figures A-1 and A-2).

A COMPARISON OF THE FRITTED IMPINGER AND SYRINGE SAMPLING TECHNIQUES. Since the performance of colorimetric detector tubes was found to be questionable, other sampling techniques were investigated. Two techniques, one employing fritted impingers and the other, syringes, appeared to be applicable to the analysis of numerous gases, and still allowed concentration-time curves to be established for the various combustion gases. The approach used to compare these techniques was to measure the concentrations of HCl and HCN produced by materials of known composition. Test materials 81, 96, and 82 contained, respectively, 100-, 51-, and 24- percent PVC fabric. Since PVC is 58.4-percent hydrogen chloride, theoretical yields for the three materials are, in order, 584, 298, and 140 mg of HCl per gram of material (mg/g). Theoretical yields can also be computed on the basis of parts per million (ppm) of HCl produced in the NBS smoke chamber per gram of material. These values are 703, 358, and 169 ppm/g. Theoretical yields of HCN are not available for the nitrogen-containing materials, but absolute values have been compared for the two sampling techniques. Test materials 88, 82, and 96 contain, respectively, 100-, 76-, and 49-percent wool.

Comparisons of the experimental and theoretical yields for HCl, in ppm/g, are contained in table 3. These data indicate that, in general, the best sample recovery is obtained by syringe sampling. Although the best recovery for material 81 was obtained with detector tubes, the tube saturated, and the true value could not be determined. This deficiency in the utility of detector tubes was also evident for other test materials.

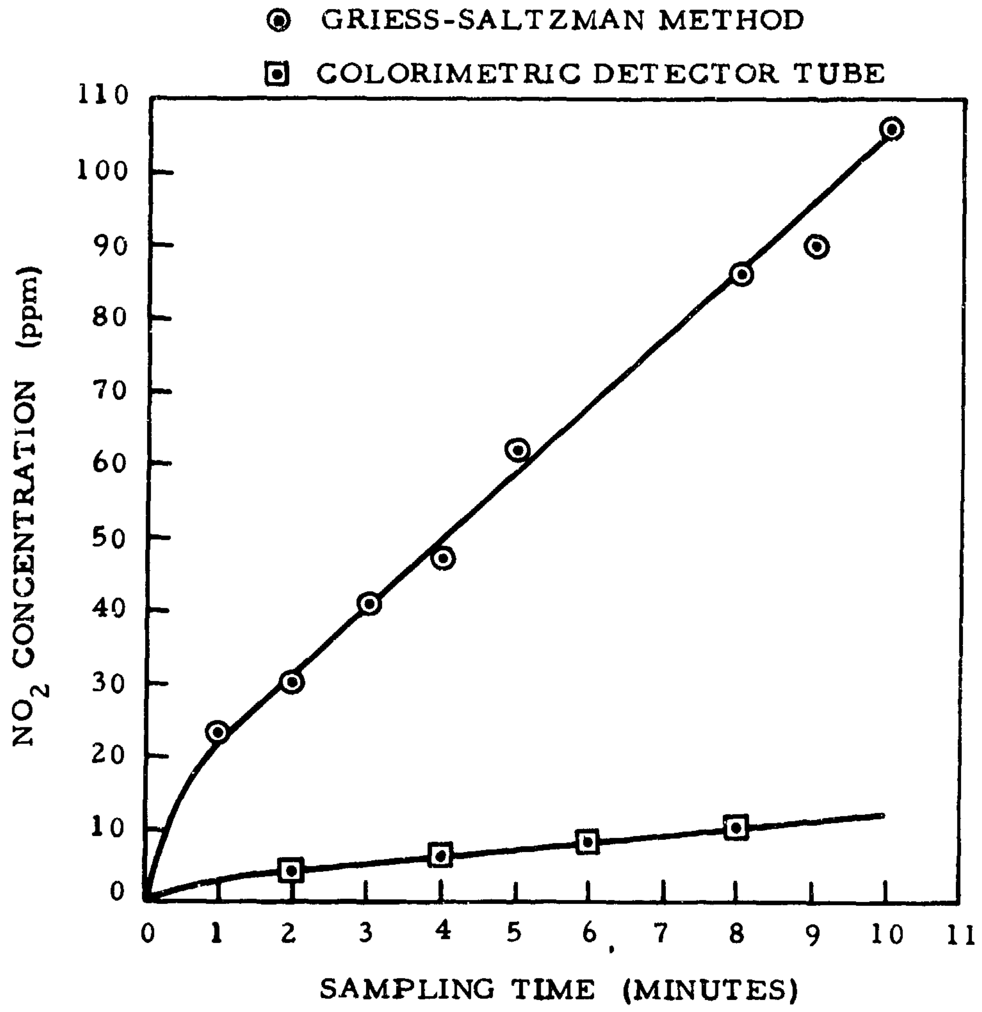

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FIGURE 3. CONCENTRATION-TIME PROFILE OF NO_2 DURING THE FLAMING COMBUSTION OF WOOL CARPET IN THE NBS SMOKE CHAMBER

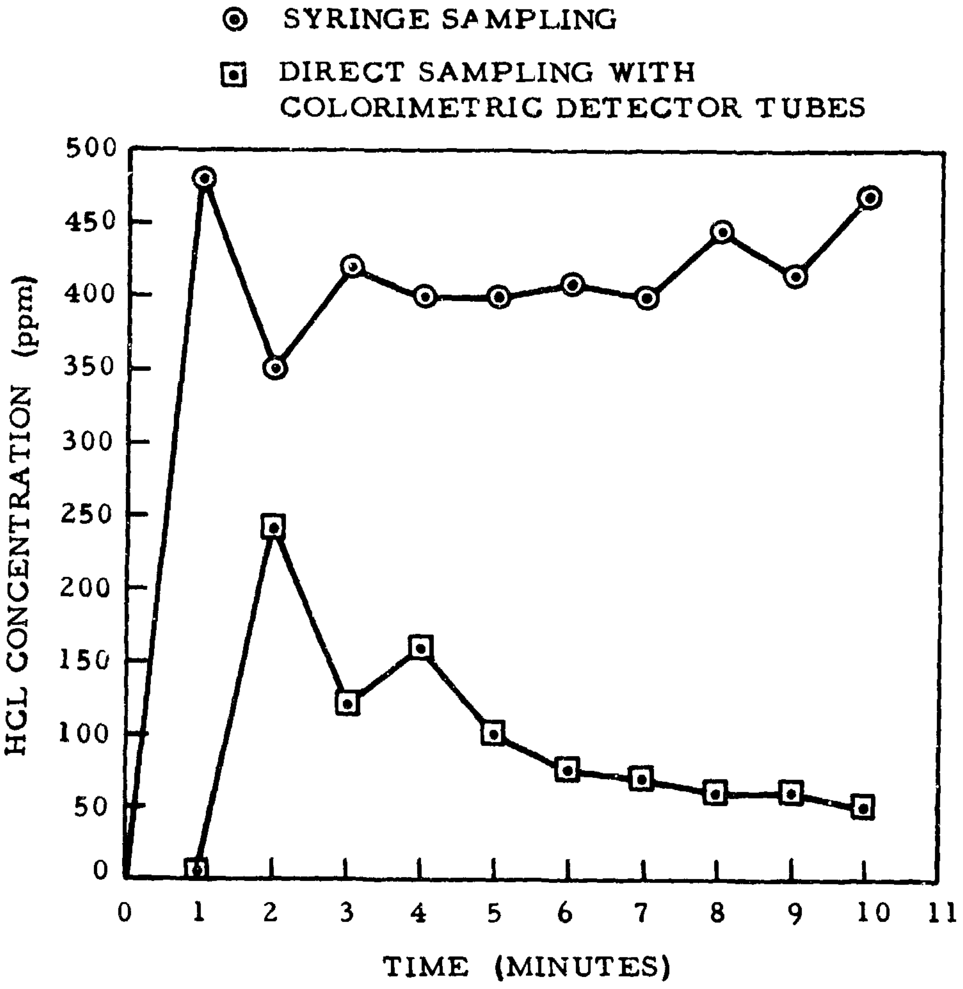

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FIGURE 4. CONCENTRATION-TIME PROFILES OF HCl IN THE NBS SMOKE CHAMBER FOR TEST MATERIAL 82

TABLE 3. EXPERIMENTAL AND THEORETICAL YIELDS OF HYDROGEN CHLORIDE IN THE NBS SMOKE CHAMBER FOR TEST MATERIALS CONTAINING KNOWN AMOUNTS OF CHLORIDE

Material Number	Theoretical Yield (ppm/g)	Detector Tube (ppm/g)	Fritted Impinger (ppm/g)	Syringe Sampling (ppm/g)
81	703	600*	170	435
96	358	170	175	280
82	169	90	65	185

*Saturated detector tube

The use of the fritted-impinger sample collection system presented many practical problems. The time required to assemble the system and calibrate the flow rates was extensive, flow rates were difficult to maintain, and sample recoveries were low. In contrast, syringe sampling was simple, afforded a fast turn-around time, was applicable over a wide concentration range, and provided the best sample recovery of the procedures investigated in this report.

An interesting trend in the data was noticed with syringe sampling. Percent theoretical yields for test materials 81, 96, and 82 were, respectively, 62-, 79-, and 108-percent. The apparent recovery of HCl in the NBS smoke chamber is therefore inversely proportional to concentration. Although the effect could be explained by a concentration-dependent adsorption of HCl by particulate matter, data obtained with the microcombustion tube furnace (table 8) suggest that this trend is the result of interferences. As the wool content of the test material increases, cyanide and sulfide interference becomes more important for the chloride electrode.

The fritted impingers were not used to measure cyanide concentrations in the NBS smoke chamber, both because of their failure to rank the chloride-containing test materials in the expected order and the lengthy time of analysis associated with this technique. As indicated in table 4, neither the colorimetric detector tubes nor syringe sampling, followed by analysis by ion-selective electrode, ranked the wool test materials in the expected order. This may be due to the characteristics of the NBS smoke chamber rather than to the syringe sampling technique itself.

Wall losses in the NBS smoke chamber were investigated by fastening two pieces of Teflon® film, each covering 1 square foot, on the smoke chamber walls. One piece was placed on the wall directly in front of the heat source, and the other was placed on the opposite wall. After testing a sample of PVC, the films were soaked in water, and the solution was analyzed for adsorbed chloride. Wall losses were 0.9 and 1.1 percent for the two locations; indicating that this was not a significant factor in poor sample recovery. This experiment probably provides a reasonable indication of condensation losses, although it may not be a reliable indicator of absorption losses due to the difference in surface characteristics between Teflon® and the chamber wall.

TABLE 4. MAXIMUM EXPERIMENTAL YIELDS OF HYDROGEN CYANIDE IN THE NBS SMOKE CHAMBER FOR TEST MATERIALS CONTAINING A KNOWN PERCENTAGE OF WOOL

Material Number	Wool Content (Percent)	Detector Tube (ppm/g)	Syringe Sampling (ppm/g)
88	100	14	38
82	76	15	18
96	49	52	24

A COMPARISON OF LABORATORY TEST METHODS FOR GENERATING COMBUSTION GASES:

The primary objective of this study was to evaluate the ability of the NBS smoke chamber and the microcombustion tube furnace to rank materials of known composition in the expected order. However, conclusions regarding the characteristics of the two test methods are preliminary in nature, since the analytical procedures upon which they are based were exploratory efforts.

REPRODUCIBILITY. The reproducibilities of the two test methods were compared using test material 81 (PVC) for HCl, material 78 (Nomex®) for HCN, and material 79 (foam) for CO. The results for HCl are contained in table 5. These data, computed as mg/g, indicate that the reproducibilities of both methods are almost identical for HCl.

Table 6 contains data on the reproducibility of the test methods for HCN. The relative standard deviations (RSD) for HCN are significantly different for the NBS smoke chamber (32 percent) and the microcombustion tube furnace (9.2 percent). The production of HCN is dependent upon the burning characteristics of a material, which are especially important for flaming combustion in the NBS smoke chamber. The pattern of combustion is more reproducible in the microcombustion tube furnace, resulting in less variation of the HCN concentrations. As might be expected, the reproducibility of a test method is somewhat dependent upon the composition of the material being tested.

Data on the reproducibility of CO measurements using the two methods are contained in table 7. The RSD's (8.5 and 10 percent) are approximately the same for both methods, indicating no significant difference in reproducibility for CO.

TABLE 5. REPRODUCIBILITY OF HYDROGEN CHLORIDE FOR TEST MATERIAL 81

NBS Smoke Chamber (Syringe Sampling)

Test Number	Sample Weight (g)	Maximum Concentration (ppm)	Yield (mg/g)	Percent Theoretical Yield
1	4.89	2,740	408	69.9
2	5.05	2,970	429	73.4
3	5.01	2,710	395	67.7

Average Yield = 411 mg/g
Average Theoretical Yield = 70 Percent
Relative Standard Deviation = ± 4 percent

Microcombustion Tube Furnace

Test Number	Sample Weight (mg)	Weight Loss (mg)	Yield (mg/g)	Percent Theoretical Yield
1	10.2	9.8	524	89.7
2	10.1	9.6	552	94.5
3	11.0	10.6	554	94.9

Average Yield = 543 mg/g
Average Theoretical Yield = 93 Percent
Relative Standard Deviation = ± 3 percent

TABLE 6. REPRODUCIBILITY OF HYDROGEN CYANIDE FOR TEST MATERIAL 78

NBS Smoke Chamber (Syringe Sampling)

Test Number	Sample Weight (g)	Maximum Concentration (ppm)	Yield (mg/g)
1	2.47	32	37.5
2	2.41	22	23.0
3	2.31	38	45.5

Average Yield = 35.3 mg/g
Relative Standard Deviation = ± 32 percent

Microcombustion Tube Furnace

Test Number	Sample Weight (mg)	Weight Loss (mg)	Yield (mg/g)
1	10.8	9.2	32.7
2	9.8	9.6	33.5
3	10.3	9.5	28.1

Average Yield = 31.4 mg/g
Relative Standard Deviation = ± 9.2 percent

TABLE 7. REPRODUCIBILITY OF CARBON MONOXIDE FOR TEST MATERIAL 79

NBS Smoke Chamber

Test Number	Sample Weight (g)	Maximum Concentration (ppm)	Yield (mg/g)
1	2.59	355	76
2	2.35	360	71
3	2.83	440	87

Average Yield = 78 mg/g
Relative Standard Deviation = ± 10 percent

Microcombustion Tube Furnace

Test Number	Sample Weight (mg)	Weight Loss (mg)	Yield (mg/g)
1	10.4	8.6	34
2	10.2	8.2	40
3	10.3	8.5	38

Average Yield = 37 mg/g
Relative Standard Deviation = ± 8.5 percent

The average RSD's for the data reported in tables 5, 6, and 7 are 15.4 percent for the NBS smoke chamber and 6.3 percent for the microcombustion tube furnace. However, this parameter is dependent upon the composition of the test material as well as the test method.

QUANTITATIVE RECOVERY OF COMBUSTION GASES. Data on the recovery of HCl from the NBS smoke chamber and the microcombustion tube furnace are contained in table 8 (appendix A, figure A-3). These data indicate a significant difference in sample recovery between the two methods. Percent theoretical yield of HCl for test material 81 was 54 percent in the NBS smoke chamber and 95 percent with the microcombustion tube furnace.

As previously mentioned, recovery of HCl in the NBS smoke chamber appeared to be concentration-dependent, but the effect may be due to an increase in the level of interference. This possibility is supported by the data obtained with the microcombustion tube furnace, where percent theoretical recovery increased from 95 percent for material 81 to 144 percent for material 82.

The results obtained for HCN are detailed in table 9 (appendix A, figure A-4). Hydrogen cyanide recoveries are lower in the NBS smoke chamber, even though the materials were tested under flaming conditions. The importance of this difference between the two test methods is illustrated in figure 5, where the quantity of HCN produced by the three test materials is plotted versus percent wool in the material. The data from the microcombustion tube furnace provide a clear distinction between the test materials. In addition, HCN yields are proportional to wool content, and the materials are ranked in the expected order. The data obtained with the NBS smoke chamber do not indicate any significant difference in the recovery of HCN for test materials 82 and 96.

Seventy-five commercially available materials will be tested for the emission of toxic combustion gases in the next phase of this study. Therefore, the demonstrated ability of a test method to rank materials of known composition in the expected order is important. Based upon the data presented in this report, the microcombustion tube furnace seems to be a more suitable test method in this respect than the NBS smoke chamber.

MEASUREMENT OF CARBON MONOXIDE CONCENTRATIONS. An additional comparison of the two test methods was made by measuring CO concentrations for 10 test materials. This was done because the microcombination tube furnace involves smoldering combustion, while the NBS smoke chamber was used with flaming exposure conditions. In addition, only one surface of a test material is exposed to the flames in the NBS smoke chamber, while the entire material is exposed to radiant heat in the microcombustion tube furnace. The NBS smoke chamber is therefore better able to simulate the conditions expected in a full-scale aircraft fire. These differences in the two test methods have been investigated as to their effect on the formation of CO.

Figure 6 is a plot of CO concentrations in the microcombustion tube furnace versus the NBS smoke chamber. Although the data exhibit a great deal of scatter, a qualitative relationship has been indicated for purposes of discussion.

TABLE 8. RECOVERY OF HYDROGEN CHLORIDE

NBS Smoke Chamber (Syringe Sampling)

		Maximum HCl Value			
Test Material	Sample Weight (g)	Time (Min)	Concentration (ppm)	Yield (mg/g)	Percent Theoretical Yield
81	5.15	4	2,230	315	54
96	2.75	2	775	205	69
82	2.63	1	480	133	95

Microcombustion Tube Furnace

Test Material	Sample Weight (mg)	Yield (mg/g)	Percent Theoretical Yield
81	10.1	552	95
96	9.4	319	107
82	9.3	201	144

TABLE 9. RECOVERY OF HYDROGEN CYANIDE

NBS Smoke Chamber (Syringe Sampling)

			Maximum HCN Value		
Test Material	Wool Content (Percent)	Sample Weight (g)	Time (Min)	Concentration (ppm)	Yield (mg/g)
88	100	3.66	10	140	76.5
82	76	2.66	10	50	25.5
96	49	2.80	3	65	30.6

Microcombustion Tube Furnace

Test Material	Sample Weight (mg)	Yield (mg/g)
88	10.1	130
82	10.6	105
96	10.2	67

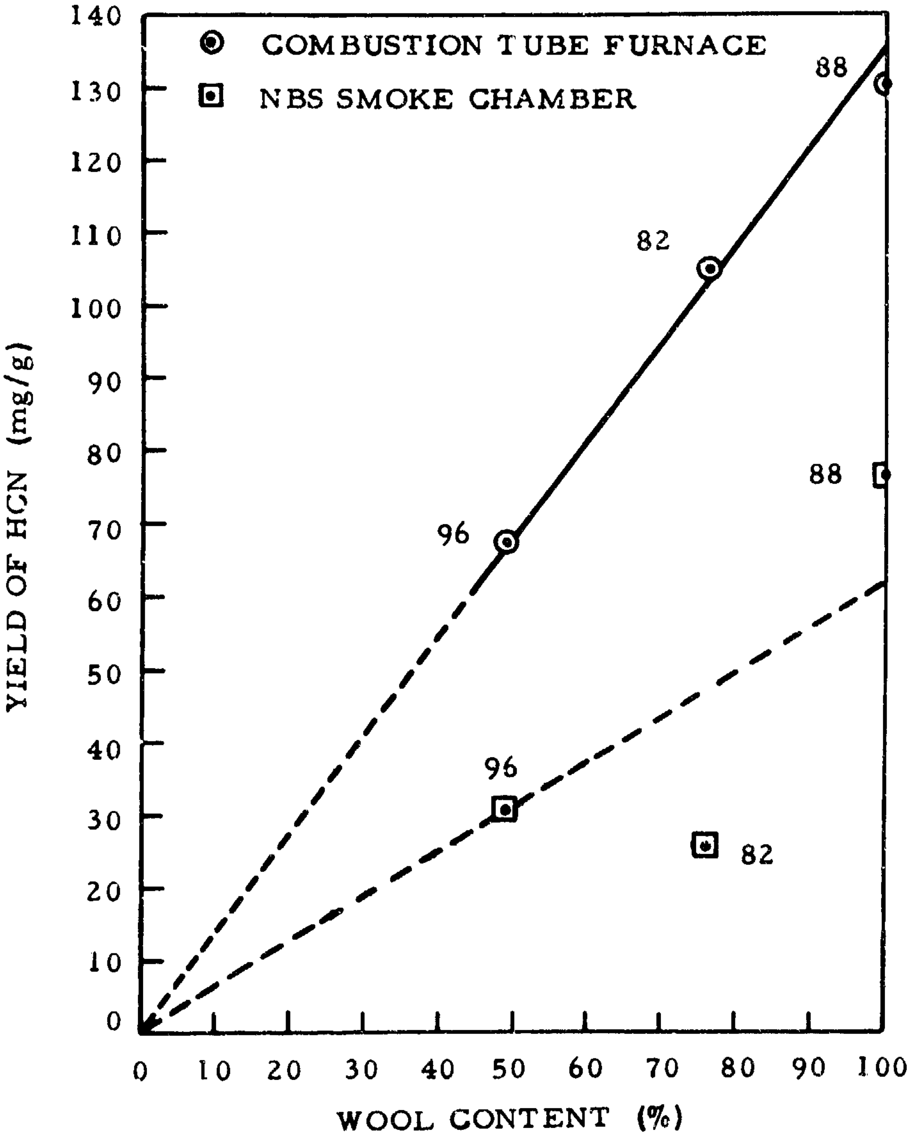

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FIGURE 5. RECOVERY OF HYDROGEN CYANIDE AS A FUNCTION OF WOOL CONTENT IN THE TEST MATERIAL

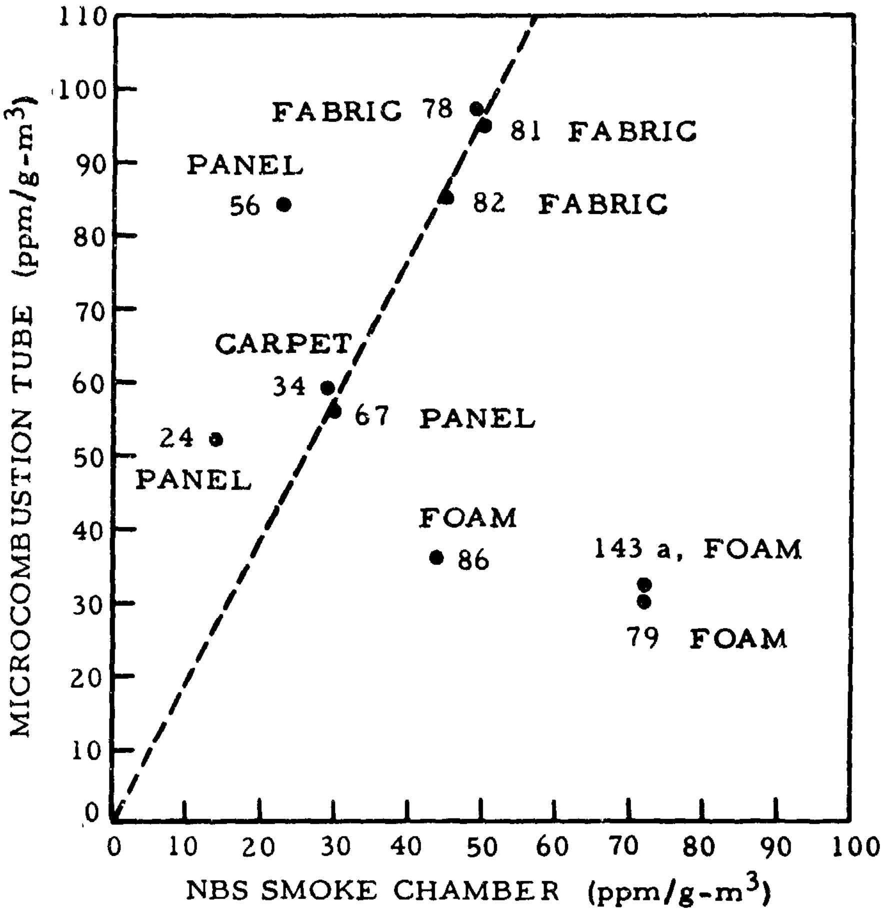

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FIGURE 6. COMPARISON OF CARBON MONOXIDE LEVELS IN THE NBS SMOKE CHAMBER AND THE MICROCOMBUSTION TUBE FURNACE

Five of the test materials deviate significantly from the projected correlation line. Two of the materials, numbers 56 and 24, are panels. These materials produced a much higher level of CO in the microcombustion tube furnace than in the NBS smoke chamber. This can be explained by the fact that only one surface was exposed in the NBS smoke chamber, and the interiors of the panels remained more or less intact. However, in the microcombustion tube furnace all surfaces were exposed to the radiant heat, and both materials were almost completely consumed. The microcombustion tube furnace, therefore, may overestimate the relative hazardousness of some composite materials.

The three foams, materials number 79, 86, and 143a, produced significantly less CO in the microcombustion tube furnace. It is possible that the foams underwent a destructive distillation rather than combustion under the experimental conditions employed in this study. This would account for both the low CO concentrations and the generally heavy condensible fraction associated with the oxidative degradation of these materials. It was concluded that the NBS smoke chamber provides better discrimination between foams with regard to the production of CO.

SUMMARY OF RESULTS

Colorimetric detector tubes, as used in this study, are not applicable for ranking aircraft interior materials, since they are not designed for the analysis of complex mixtures of combustion gases. The HCN and NO_2 detector tubes measured significantly lower gas concentrations than other analytical procedures employed in this study, while the CO detector tubes overestimated CO concentrations as compared to infrared spectroscopy. The HCl detector tubes often became saturated when PVC-containing materials were tested.

The fritted-impinger sampling system did not prove to be satisfactory due to the extensive manifolding required to obtain 10 consecutive 1-minute samples.

Syringe sampling was found to be the most appropriate method for establishing concentration-time curves for the combustion gases measured in the NBS smoke chamber. However, except for the use of colorimetric detector tubes, a large number of samples are generated in gathering enough data points to construct concentration-time curves. If 3 samples of materials are tested per day, 30 samples must be analyzed for each combustion gas measured. Therefore, without the availability of automated instrumentation, establishing concentration-time curves for more than a few gases is not feasible on a routine basis.

Analysis of combustion gases generated in the NBS smoke chamber did not rank the nitrogen-containing test materials in the expected order. The test material containing 49-percent wool produced more HCN than the material containing 76-percent wool. The chloride-containing materials were ranked in the expected order, although the percent theoretical yield was only 54 percent for test material 81. The average RSD of 15.4 percent is also greater than the average RSD of 6.3 percent obtained with the microcombustion tube furnace. It is not possible to determine which ranking order is actually correct, based upon the data presented in this study, since interaction between the wool/PVC blends during flaming combustion could result in a change in the decomposition products. However, the use of the NBS smoke chamber for ranking complex materials does not seem warranted unless the ability to rank materials of known composition can be demonstrated.

The preliminary data contained in this report suggest that neither the NBS smoke chamber nor the microcombustion tube furnace is an entirely satisfactory test method for generating combustion gases. Material exposure conditions in the combustion furnace are not typical of cabin fire conditions, since the material is not actually burned. Decomposition products may therefore differ from those generated under conditions of flaming exposure. In addition, the relative hazardousness of some composite materials, such as panels, may be overestimated due to the total exposure of the sample to the heat source. However, this problem can be resolved by dividing the materials into usage categories prior to testing, so that panels, foams, carpets, etc., are compared to materials in the same usage category.

The microcombustion tube furnace has exhibited the ability to rank materials of known composition in the expected order. The applicability of this test method for ranking aircraft interior materials will therefore be investigated in future studies.

CONCLUSIONS

1. Colorimetric detector tubes are not satisfactory for the quantitative analysis of complex mixtures of combustion gases.

2. Syringe sampling was found to be the most appropriate gas-sampling technique, of those tested, for use with the NBS smoke chamber.

3. Establishing concentration-time curves for combustion gases in the NBS smoke chamber is not feasible on a routine basis due to the time required for sample analysis and data reduction.

4. The microcombustion tube furnace is more suitable for ranking aircraft interior materials of known composition than is the NBS smoke chamber.

PART II: A RELATIVE TOXICITY RANKING OF AIRCRAFT INTERIOR MATERIALS

INTRODUCTION

PURPOSE.

The purpose of the animal toxicity tests was to further investigate the ability of the combustion tube furnace to rank materials in terms of toxic combustion-gas emission characteristics. In addition, the correlation of animal toxicity data with measured toxic gas concentrations was investigated.

BACKGROUND.

The results obtained in part I of this study suggest that the combustion tube furnace is more useful than the NBS smoke chamber for ranking aircraft interior materials in order of the relative toxicity of their thermal decomposition products. However, this conclusion is based upon the analysis of decomposition products for a small number of gases, primarily HCN, HCl, and CO. The existence of a relationship between animal toxicity and the quantity of HCN released by the test materials when decomposed in the microcombustion tube furnace is therefore investigated in part II of this study.

Animal toxicity tests have been conducted at the FAA's Civil Aeromedical Institute (see Preface) for nine of the materials tested in part I of this study. The results of the animal tests have been correlated with the combustion gas analyses performed at NAFEC, as described in part I of this report.

EXPERIMENTAL SECTION

DESCRIPTION OF TEST MATERIALS.

The 9 aircraft interior materials employed in this study were selected from among the 13 materials tested in part I of this study. A brief description of the compositions of the test materials is contained in table 10.

ANIMAL TEST PROCEDURE.

The test animals were male Sprague-Dawley rats obtained from Holtzman Co. Their weights varied from 150 to 190 grams, with an average weight of approximately 170 grams. Two tests were conducted for each material, and two rats (R_1 and R_2) were used for each test. The test animals were marked with either red or green dye for identification purposes. The animals were exposed to the decomposition products of a material for 30 minutes. Those which survived the initial exposure period were observed for 24 hours for any visible signs of disability or delayed death. Times-to-incapacitation (t_i) and times-to-death

TABLE 10. TABULATION OF ANIMAL TOXICITY DATA

Test Material Number	Test Material Description	Sample Weight (mg)	Maximum Furnace Temperature (° C)	Animal Weight (g)		Time-To-Incapacitation (Min)		Time-To-Death (Min)		Average Time-To-Incapacitation [$t_i \pm SD$] (Min)	Peak Gas Concentrations (ppm)	
				R_1	R_2	R_1	R_2	R_1	R_2		HCN	CO
104	Polyester Urethane Foam	502	670	160	170	17.9	17.4	26.0	27.5	21.5 ± 4.4	-	2,552
		506	625	165	180	25.3	25.3	S	S		2	2,290
79	Polyether Urethane Foam	494	645	170	170	N	N	S	S	12.5 ± 0.7	-	2,334
		530	612	190	180	13.0	12.0	22.0	29.5		110	3,628
78	Nomex® Fabric	497	620	148	165	10.0	10.6	22.0	25.0	11.0 ± 0.8	-	2,449
		514	615	150	170	11.8	11.5	25.4	28.3		45	1,975
33	Wool Pile Carpet	509	630	170	155	9.3	8.0	18.6	25.1	8.0 ± 1	-	1,100
		509	618	180	180	7.3	7.2	S	S		60	1,188
34	Wool Pile Carpet with Urethane Pad and Nylon Scrim	502	625	160	175	7.1	7.9	S	S	7.0 ± 0.9	-	926
		529	634	180	170	7.3	5.7	S	S		65	1,345
96	Wool-PVC Fabric (49/51 Percent)	501	620	185	180	4.8	5.1	19.8	8.8	5.7 ± 0.8	-	1,888
		497	606	165	170	6.2	6.5	23.3	*		-	1,487
88	Wool Fabric	504	630	170	170	4.9	4.9	7.4	10.2	4.9 ± 0.2	-	926
		494	615	175	165	5.1	4.6	7.4	15.5		-	926
81	Poly(Vinylchloride) Fabric	504	620	175	175	4.0	4.6	S	S	3.5 ± 1	-	1,928
		530	625	160	160	2.7	2.5	S	S		10	1,975
82	Wool-PVC Fabric (76/24 Percent)	504	620	180	165	2.5	3.0	6.9	7.5	2.7 ± 0.3	-	2,128
		515	610	175	175	2.8	2.5	6.2	7.4		150	1,818

* = Died during the night
N = No incapacitation observed
S = Survived for 24 hours
SD = Standard Deviation (Minutes)

(t_d) were recorded independently by two observers, and the recorded values were averaged for each test animal.

Samples of material with a nominal weight of 500 mg were heated in a Fisher® microcombustion tube furnace using 12-mm by 12-inch Vycor® combustion tubes. The furnace, initially at room temperature, was raised to a maximum temperature of approximately 620°C at 2.5 minutes and then stabilized at roughly 600°C. The temperature profile in the furnace for a typical test is illustrated in figure 7. The furnace was usually turned OFF 10 minutes after the test was initiated. The gas-discharge end of the combustion tube was connected to the gas delivery tube with heat-shrink tubing prior to each test. The other end of the combustion tube was sealed with a rubber septum, through which 100 ml of air was injected into the combustion tube at 3, 5, and 10 minutes after the test was initiated. The air injections, made with a 100-ml glass syringe, were required to force the accumulated combustion gases into the exposure chamber.

The exposure chamber, which was a glass desiccator with an empty volume of 5.6 liters, and associated apparatus are illustrated in figure 8. The top of the chamber was a circular piece of 3/8-inch Plexiglas® which was equipped with a gas delivery tube, a gas-sampling port, and a small, battery-operated fan for mixing the combustion gases. The fan blades were painted with epoxy paint. Gas samples were obtained from the exposure chamber by syringe sampling. Samples for CO analysis were obtained at approximately 3.5, 5.5, and 10.5 minutes, while samples for HCN analysis were taken at 4, 11, and 31 minutes. The contents of the syringes were then analyzed for CO and HCN by gas chromatography.

DISCUSSION OF RESULTS

The animal toxicity data for the nine test materials have been tabulated in table 10. The data include times-to-incapacitation (t_i), times-to-death (t_d), and peak concentrations of hydrogen cyanide (HCN) and carbon monoxide (CO), which were measured in the exposure chamber. Unfortunately, HCN concentrations were not obtained for all of the tests due to experimental difficulties. Since both t_i and t_d were determined visually, the possibility of a subjective bias in the data is difficult to rule out. However, t_i and t_d values for each test animal were recorded independently by two observers and the results were averaged. In addition, the procedure used was to conduct only one test for all nine materials and then repeat the series. The influence of the initial test results on the observers was therefore minimized as much as possible.

Values of t_i and t_d were measured from the times at which the tests were initiated, although the first visable smoke did not enter the exposure chamber until approximately 2 minutes; nor did the test animals appear significantly distressed until that time. Times-to-incapacitation values were found to be more useful for ranking the materials, since under the experimental conditions employed in these tests more than a third of the test animals survived.

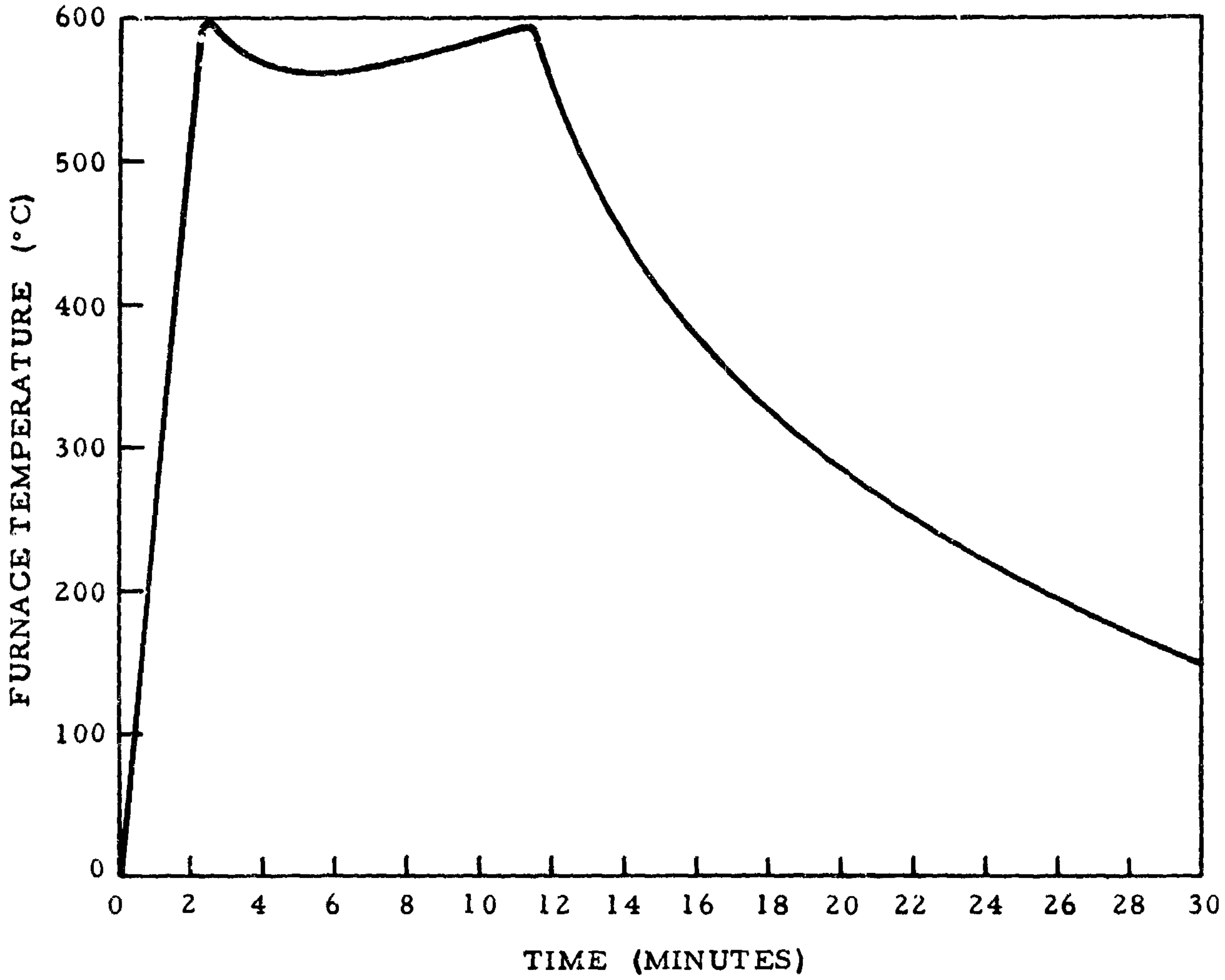

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FIGURE 7. TYPICAL FURNACE TEMPERATURE PROFILE DURING A TEST

In addition, t_i values are considered to be more relevant for judging escape potential as relates to an aircraft cabin fire (reference 5). The test materials in table 10 are therefore listed in order of the average t_i values. Values for the standard deviation (SD) are also included.

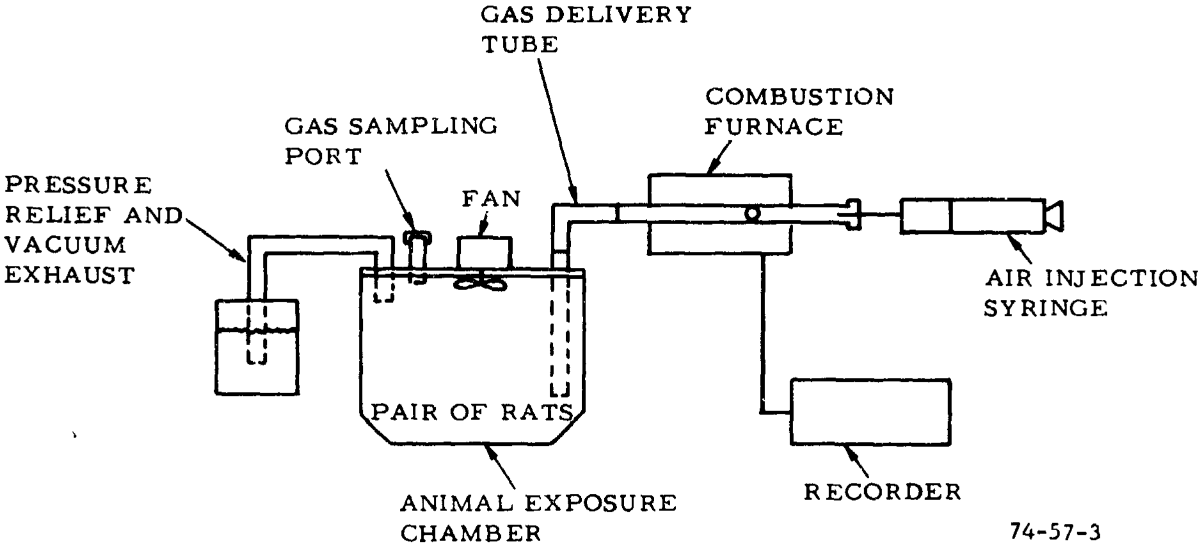

FIGURE 8. ANIMAL TEST APPARATUS

Yields of HCN, HCl, and CO, obtained for the nine test materials using the microcombustion tube furnace described in part I, are listed in table 11. Values of t_i have been included for easier reference. Except for material 81, the test materials are listed according to the quantity of HCN measured during thermal decomposition.

It should be emphasized that the results of the animal tests and the conclusions are of a preliminary nature. Except for the gross chemical composition of these test materials, little information is available concerning the presence of fire retardants or other additives which might influence their toxicity. However, the order of relative toxicity of the test materials is PVC > wool Nomex® > urethanes. The two urethane foams are the least toxic of the test materials, although the polyether urethane is more toxic than the polyester urethane foam.

The data in table 11 suggest that t_i may be related to the HCN yields obtained with the microcombustion tube furnace. This does not mean that the incapacitation of a test animal necessarily resulted from the HCN concentration in the exposure chamber, but it does suggest that the analysis of combustion gases for HCN may provide an indication of animal toxicity with regard to many of the nitrogen-containing test materials. The correlation of t_i with HCN yield is illustrated in figure 9. The coefficient of correlation from linear regression analysis (excluding material 82) is 0.93.

TABLE 11. YIELDS OF COMBUSTION GASES OBTAINED WITH THE MICROCOMBUSTION TUBE FURNACE

Material Number	HCN (mg/g)	HCl (mg/g)	CO (mg/g)	t^*_i
104	2.5	-	-	21.5
79	6.6	-	38	12.5
78	33.5	-	123	11.0
33	34.2	-	-	8.0
34	51.4	-	73	7.0
96	67	320	-	5.7
82	105	200	108	2.7
88	130	-	-	4.9
81	-	550	121	3.5

*Time-To-Incapacitation (Minutes)

The behavior of test materials 82 and 78 deserve special comment. The relatively high toxicity of material 82 (76-percent wool and 24-percent PVC) compared to the other test materials cannot be explained on the basis of HCN concentrations. Although many of the materials produced convulsions in the test animals between 10 and 20 minutes, material 82 caused convulsions as early as 2.5 minutes into the test. The t_i for material 82 is 2.7 minutes, while t_d is 7.0 minutes. In comparison, t_i for material 96 (49-percent wool and 51-percent PVC) is 5.7 minutes, and t_d is greater than 17 minutes. A simple synergistic response due to the combination of PVC and wool is therefore not indicated. The results obtained for test material 82 illustrate one of the major difficulties associated with applying a purely analytical approach to the area of material toxicity.

A possible explanation for the observed toxicity of material 82 is the fire retardant treatment received by this material in order to meet current flammability standards. Material 82 is treated with a zirconium fluoride fire

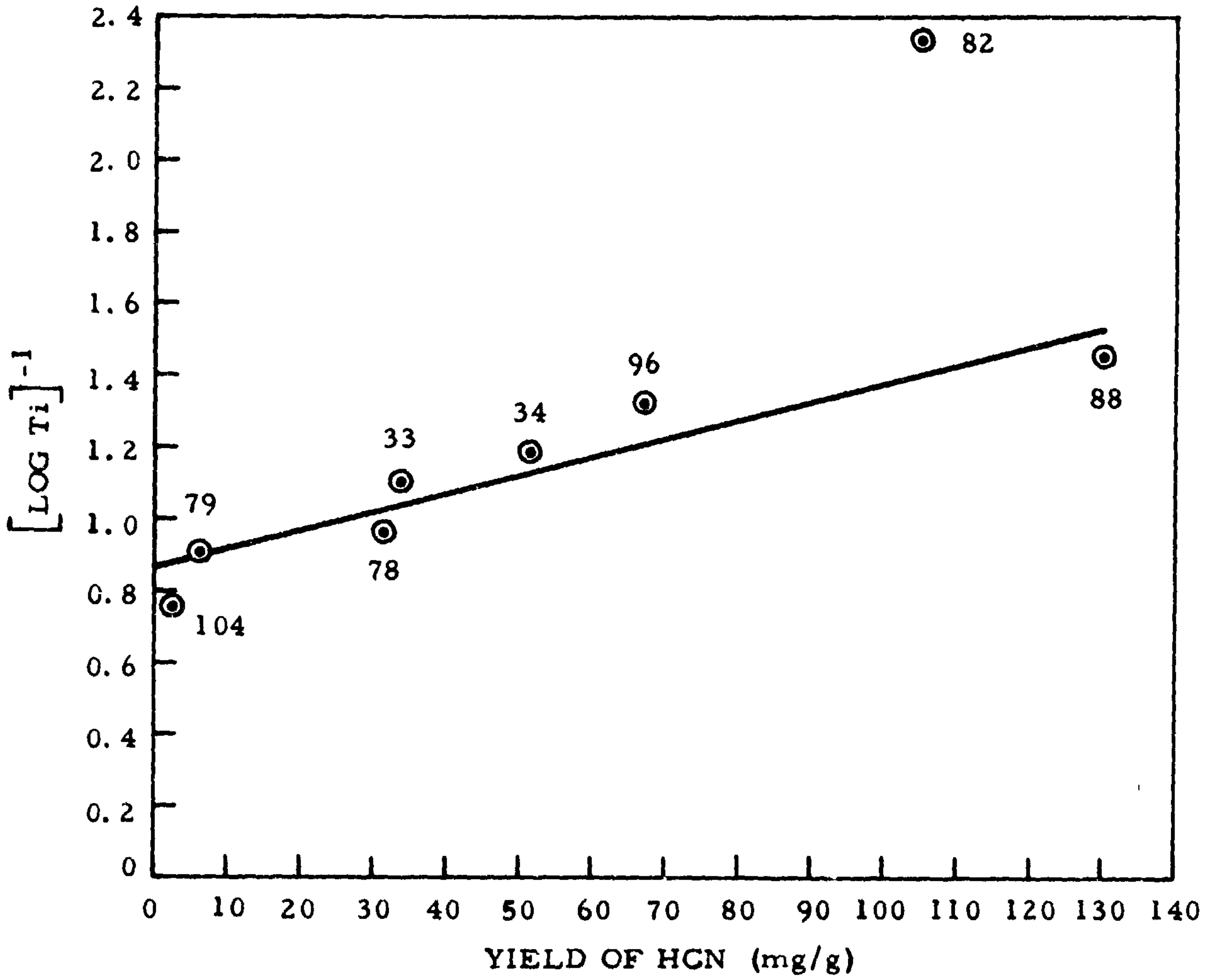

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FIGURE 9. AVERAGE TIMES-TO-INCAPACITATION AS A FUNCTION OF HCN YIELDS OBTAINED WITH THE MICROCOMBUSTION TUBE FURNACE

retardant, while material 96 does not require additional treatment due to the already high chloride content of this material. Thermal gravimetric analysis in air revealed that material 96 is completely decomposed at 850°C, while a 12-percent residue remains for material 82.

The effect of test material 78 (Nomex®) on the test animals was also anomalous. The test animals did not exhibit any signs of undue distress at 10 minutes, immediately prior to the last flushing of the combustion tube with air. However, approximately 1 minute after the final combustion gases entered the exposure chamber, the test animals were incapacitated. Nomex® is thermally stable and undergoes a relatively slow decomposition (appendix A, figure A-4). But once decompositon has occurred, the resulting products appear to be highly toxic.

Test material 81 (PVC) is not included in the previous discussion since it is not a nitrogen-containing material. This material exhibits the greatest relative toxicity, except for material 82, with a t_i of 3.5 minutes. Although the test animals were incapacitated rather quickly by the PVC fabric, they were generally active 9-10 minutes later and appeared to be functioning normally. The conditions of the test animals were normal after 24 hours, with no visible signs of dysfunction. Material 81 is one of the more hazardous materials if the comparison is made on the basis of t_i values, while it is one of the least hazardous if t_d values are considered.

An attempt was made to correlate t_i values with the peak concentrations of HCN measured in the exposure chamber, even though data is not available for all the materials. This relationship is illustrated in figure 10. If test material 82 is not included in the calculation, the coefficient of correlation is 0.34, with the correlation line indicated in the figure. The relationship between the two parameters is worse than one might have expected from figure 9. However, the same difficulty is encountered in sampling gases from the exposure chamber as was found to occur when sampling gases from the NBS smoke chamber as described in part I. Only a small portion of the total sample was analyzed, and the assumption was made that the combustion gases in the chamber were homogeneous mixtures. This assumption is not always valid. In addition, sample transfer losses for gaseous HCN have been shown by CAMI scientists to be a significant factor in the analytical procedure.

SUMMARY OF RESULTS

In general, the order of relative toxicity of the materials tested in this study is PVC>wool>Nomex®>urethane foam. This ranking is based on t_i values, although it is interesting to note that PVC is one of the least hazardous if the materials are ranked according to t_d values. Except for material 82, the test materials were ranked in the same order of relative toxicity by both the animal tests and combustion gas analysis using the microcombustion tube furnace. The coefficient of correlation between t_i values and HCN yields

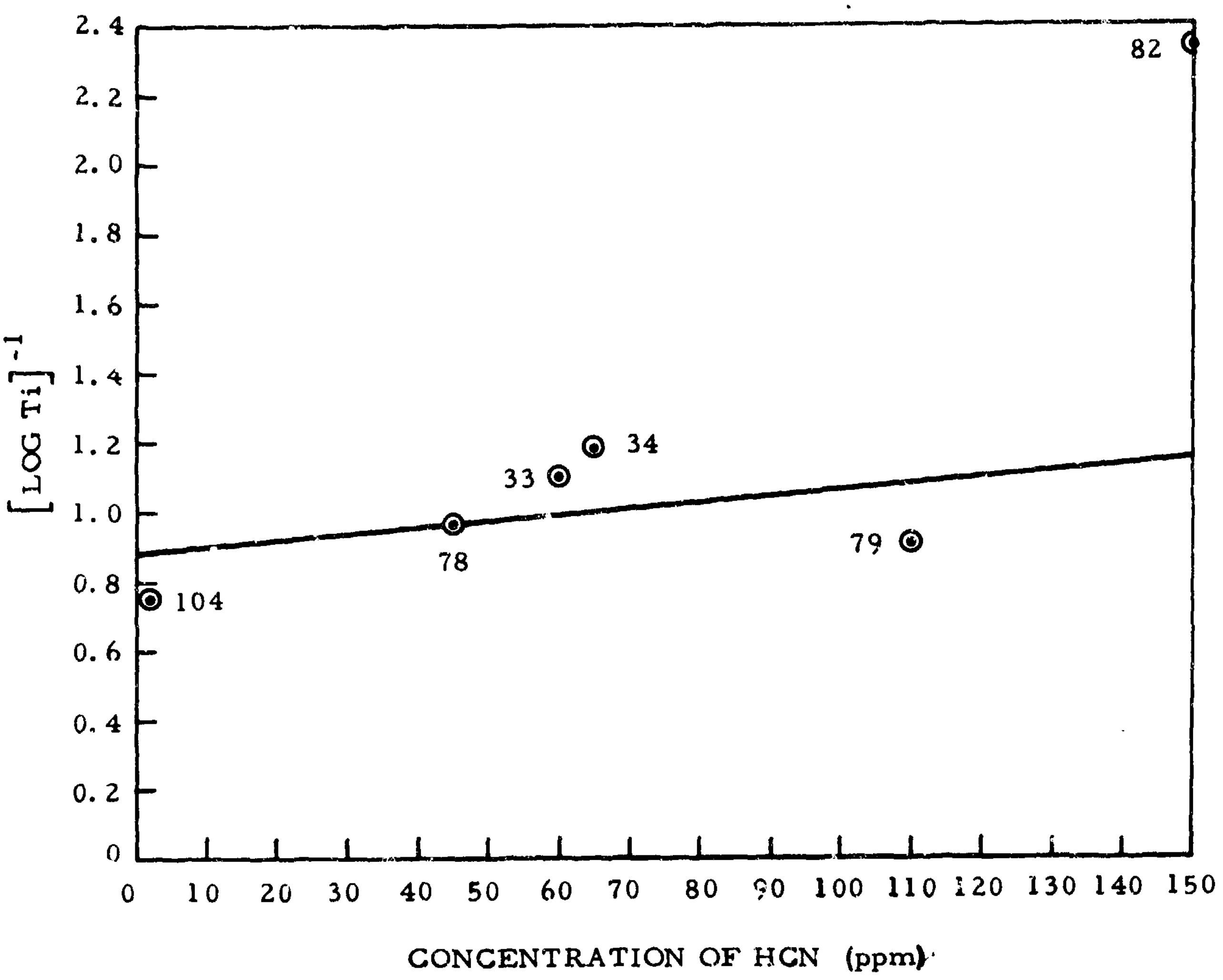

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FIGURE 10. AVERAGE TIMES-TO-INCAPACITATION AS A FUNCTION OF PEAK HCN CONCENTRATIONS MEASURED IN THE ANIMAL EXPOSURE CHAMBER

obtained with the microcombustion tube furnace is 0.93. However, the correlation coefficient for peak HCN concentrations measured in the exposure chamber is only 0.34.

Test material 82 (76-percent wool and 24-percent PVC) is uniquely toxic. This behavior could be due to the fire-retardant treatment received by this material. The unexpected toxicity of material 82 illustrates the need for animal tests in assigning relative toxicities to aircraft interior materials.

CONCLUSIONS

1. Time-to-incapacitation (t_i) is a useful parameter for ranking aircraft interior materials according to animal toxicity.

2. It may be possible to establish a relationship between concentrations of specific combustion gases and observed animal toxicity. This would require a more sophisticated effort involving both combustion gas analysis and animal toxicity tests for a broad selection of materials, including materials of known composition.

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APPENDIX A

REPRESENTATIVE CONCENTRATION-TIME CURVES FOR SELECTED GASES IN THE NBS SMOKE CHAMBER

Various concentration-time curves have been included in the appendix to support statements made in the text of this report.

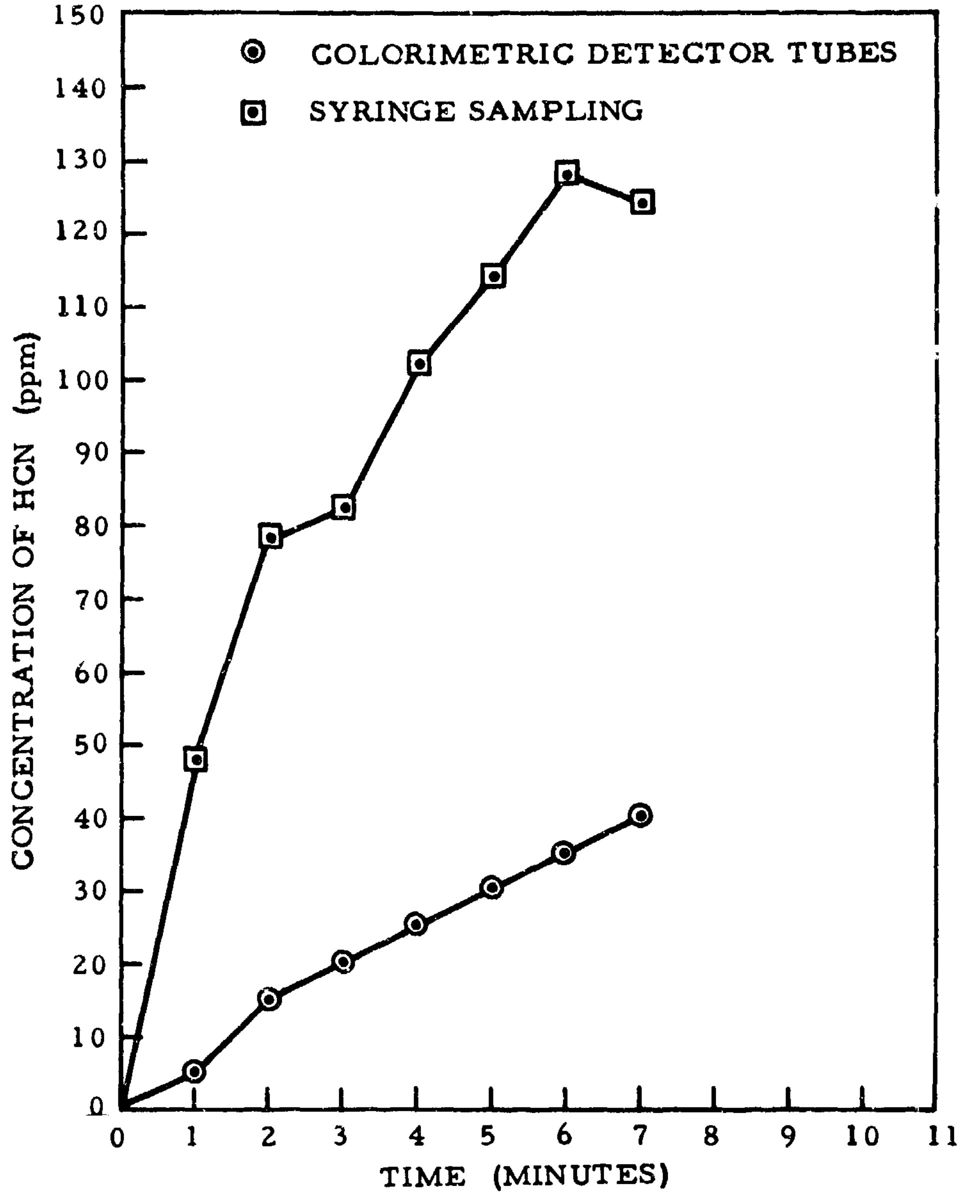

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FIGURE A-1. ANALYSIS OF HCN IN THE NBS SMOKE CHAMBER BY COLORIMETRIC DETECTOR TUBES AND SYRINGE SAMPLING FOR TEST MATERIAL 88

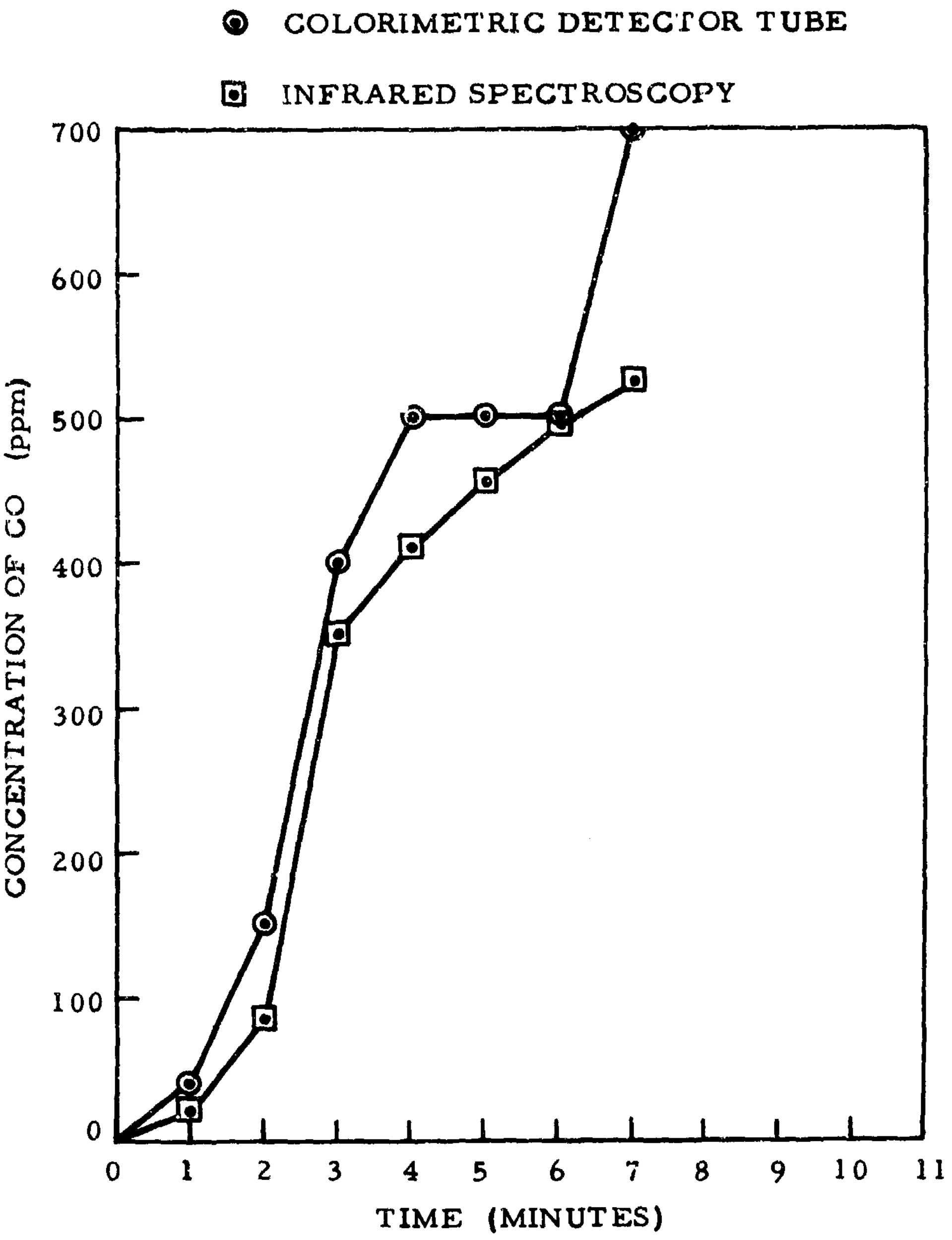

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FIGURE A-2. MEASUREMENT OF CO IN THE NBS SMOKE CHAMBER BY COLORIMETRIC DETECTOR TUBE AND INFRARED SPECTROSCOPY FOR TEST MATERIAL 34

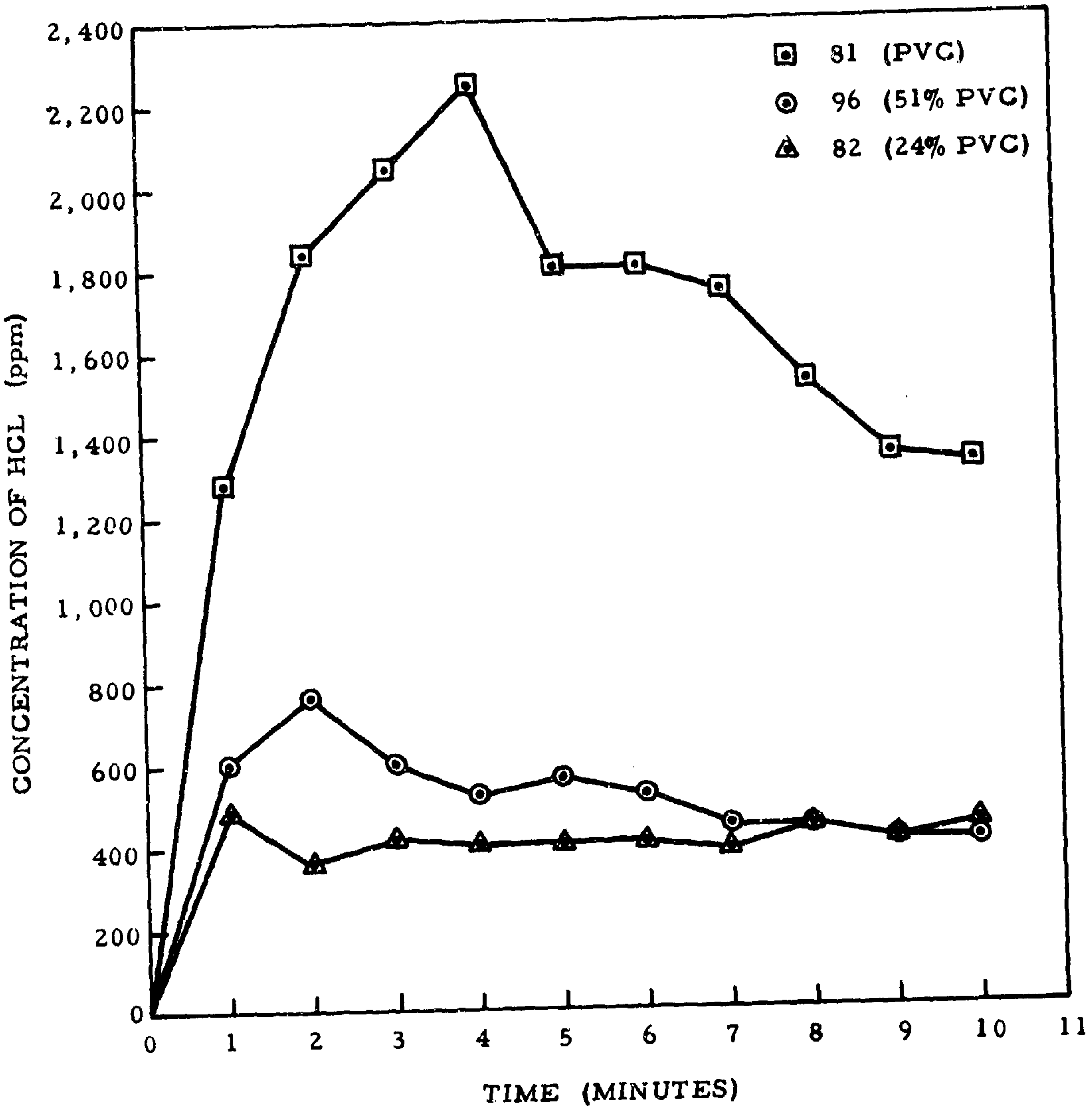

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FIGURE A-3. SYRINGE SAMPLING OF HCl CONCENTRATIONS IN THE NBS SMOKE CHAMBER

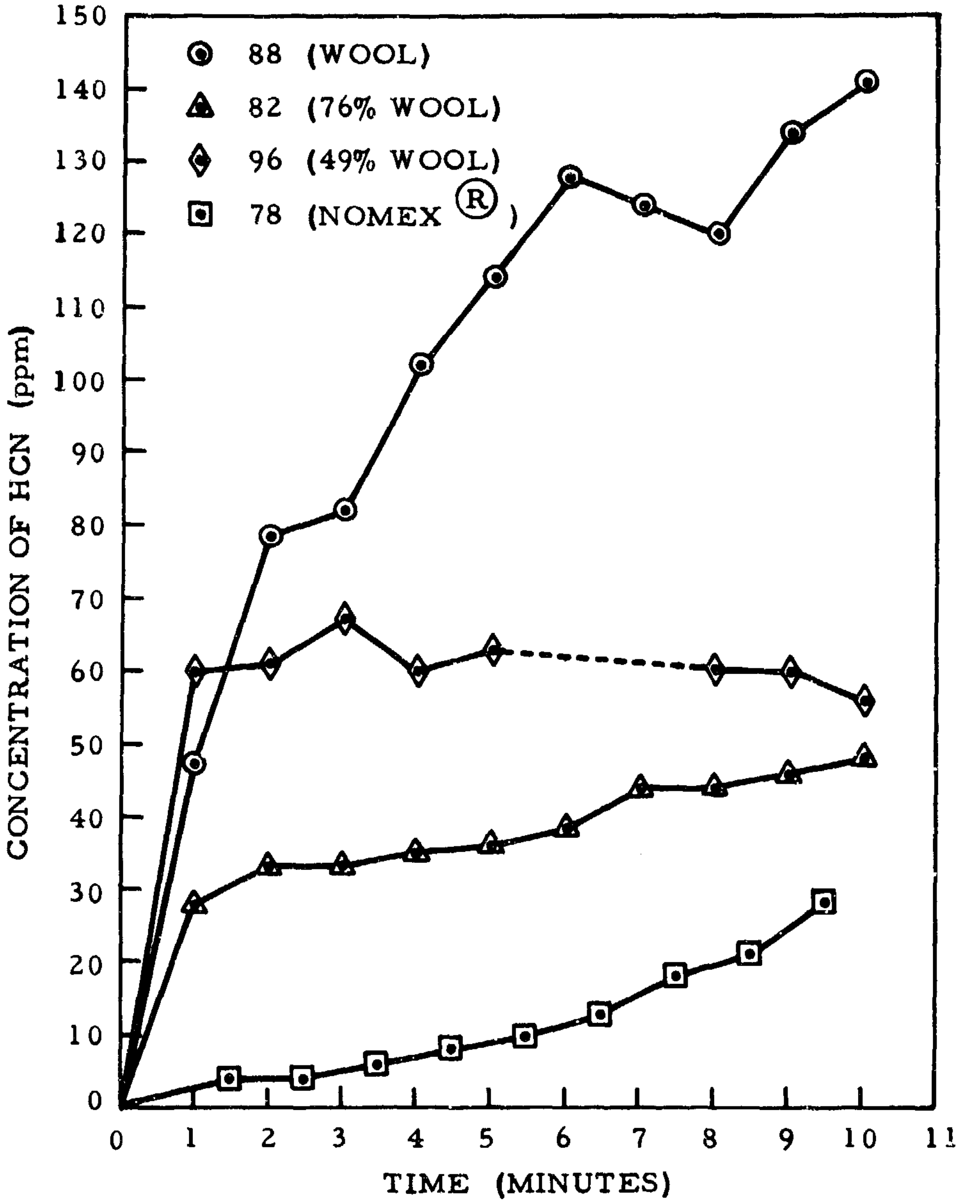

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FIGURE A-4. SYRINGE SAMPLING OF HCN CONCENTRATIONS IN THE NBS SMOKE CHAMBER