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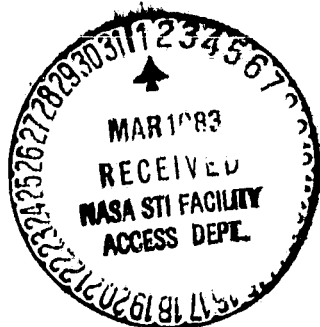
NASA CONTRACTOR REPORT 166432

Development of Aircraft Lavatory Compartments With
 Improved Fire Resistance Characteristics - Phase IV

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Development of Aircraft Lavatory Compartments With
Improved Fire Resistance Characteristics - Phase IV

Boeing Commercial Airplane Company
Seattle, Washington 98124

Prepared for
Ames Research Center
Under Contract NAS2-9864

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National Aeronautics and
Space Administration

Ames Research Center
Moffett Field, California 94035

CONTENTS

CONTENTS (Concluded)

TABLES

TABLES (Concluded)

FIGURES

DEVELOPMENT OF AIRCRAFT LAVATORY COMPARTMENTS WITH IMPROVED FIRE RESISTANCE CHARACTERISTICS – PHASE IV

SANDWICH PANEL DECORATIVE INK DEVELOPMENT

Arumugham Jayarajan, Gerald A. Johnson, Gailerd L. Korver, and Roy A. Anderson

Boeing Commercial Airplane Company

1.0 SUMMARY

Five chemically different resin systems with improved fire resistance properties were studied for a possible screenprinting ink application. Fire resistance is hereby defined as the cured ink possessing improvements in flammability, smoke emission, and thermal stability. The developed ink shall be suitable for application to polyvinyl fluoride film. Only clear inks without pigments were considered.

Preliminary evaluation and compatibility studies of the five formulations were made and compared with KC4900 clear acrylic ink, which was used as a baseline. The tests used in the screening evaluation included viscosity, smoke and toxic gas emission, Limiting Oxygen Index (LOI), and polyvinyl fluoride film (PVF) printability. Based on the results of this evaluation, Firestone Plastics Company, No. 461 chlorofluorocarbon resin (FPC461) was selected for optimization studies.

Two ink formulating companies were identified for optimization of FPC461 chlorofluorocarbon resin-based screenprinting ink: Midwest Coatings of Kansas City, Missouri and Seri-Print of Canada, Ltd. The parameters for optimization included screenprinting process performance, quality of coating, and flammability of screenprinted 0.051-mm (0.002-in.) white Tedlar. The quality of the screenprinted coating on Tedlar is dependent on viscosity, curing time, adhesion to polyvinyl fluoride film, drying time (both in-screen and as an applied film), and silk screen mesh material and porosity. During a series of trials, one formulation from each of the participating companies was developed to yield a good quality coating, satisfactory performance during the screenprinting operation, and a high LOI.

Midwest P2028 was the only optimized formulation that became available in time for the performance evaluation tests. KC4900 clear was used as the baseline in evaluating the performance of Midwest P2028 clear. The two inks were tested as: (1) cured ink systems for evaluating thermal stability and weight loss in air and nitrogen atmospheres, (2) cured ink systems laminated between 0.051-mm (0.002-in.) white and 0.025-mm (0.001-in.) clear PVF films to evaluate LOI, 12- and 60-s vertical flammability, and peel strength, and (3) item (2) laminates bonded to a cured, three-ply laminate of phenolic-fiberglass prepreg.

Chlorofluorocarbon resin-based Midwest P2028 ink showed consistently better performance in thermal stability, flammability, and smoke emission testing than KC4900. Data on heat release and toxic gas evolution indicated no significant differences between the two systems.

2.0 INTRODUCTION

The Federal Aviation Administration (FAA) has investigated new regulations to control the smoke and toxic gas emission characteristics of commercial aircraft interior materials (refs. 1 and 2). Large-scale tests conducted by NASA-JSC have demonstrated that it may be possible to obtain improvements in these characteristics through the use of newly developed materials (refs. 3 and 4). Research on new materials under laboratory conditions is a cost effective way to determine the extent of improvement that could be expected. Related studies in the areas of flammability and thermochemical characterization of aircraft interior materials can be found in References 5 through 15.

This report describes the results of a NASA-ARC contract with the Boeing Commercial Airplane Company (BCAC) (ref. 16) to develop a fire-resistant screenprinting ink. The program was funded by NASA-ARC as part of its continuing flammability studies on aircraft interior materials and is Phase IV of an overall effort to improve the fire resistance of interior sandwich panels. Reports covering efforts in Phases I, II, and III can be found in References 13, 14, and 15, respectively.

Sidewall panels (sandwich as well as crushed core) used in the interior of commercial aircraft are decorated with the respective airline's colors and pattern designs. The medium or material that carries these color pigments and designs is the screenprinted ink resin. The ink, which is mixed in solvents to obtain fluidity, is screenprinted on the decorative Tedlar film. The solvent in the ink is then evaporated by passing the printed film through a drying oven. This report describes the identification, development, and testing of an ink resin containing no pigments to gain improvements in fire resistance without sacrificing screenprinting process performance characteristics.

Since the amount of ink material is small, at most 0.0203 mm (0.0008 in.) for two coats, the study is concerned with the inherent thermal stability of ink, its effect on fire initiation and propagation, and smoke and toxic gas evolution of the whole panel.

V. C. Chin (Boeing – Manufacturing Research and Development (MR&D)) fabricated screenprints and panels. W. C. Crowe, C. A. Boydston, C. A. Hatlestad, and C. N. Lutz (Boeing – Boeing Materials Technology (BMT)) performed fire and flammability tests. Thermogravimetric Analysis/Differential Thermal Analysis (TGA/DTA) testing was done by K. M. Tanino (Boeing – BMT). F. L. Doty, E. D. Mott, S. W. Rawllins, and T. J. Jaenicke (Boeing – BMT) prepared test specimens, made viscosity measurements, and performed a number of other laboratory-type tasks.

3.0 TEST PROGRAM

The test program section has been divided into two separate parts:

(1)   Test methods

(2)   Test materials

3.1 TEST METHODS

A broad range of tests, including flammability, thermochemical, and mechanical, was conducted to fully characterize the candidate materials and select the appropriate test methods to be used in future programs. The extensive laboratory testing in the flammability area was necessary because the implications of laboratory-scale test results are not fully understood. That is, the correlation between small-scale and large-scale tests has not been established.

3.1.1 FLAMMABILITY TESTS

It was desirable to measure five basic properties of the materials: (1) propensity to burn, (2) smoke emission, (3) heat release, (4) toxic gas emission, and (5) flame propagation. In some cases, more than one test apparatus was used to measure the same property, thus giving a comparison of test methods.

3.1.1.1 Propensity to Burn

The propensity to burn was measured using standard Bunsen burner exposure tests and the Limiting Oxygen Index (LOI) test.

The 12- and 60-s vertical ignition Bunsen burner tests were chosen because they are the standard flammability tests required by the Federal Aviation Administration (FAA) for wide-cabin interior materials (ref. 17). These tests measure burn length and the extinguishing time after the igniting flame is removed. The procedures are described in Appendix A (sec. A.1), and typical test setups are shown in Figures 1 and 2.

The LOI test was run to determine the propensity of the materials to burn. This test exposes the specimen to an open flame in a controlled nitrogen/oxygen atmosphere. The ratio of N_2 to O_2 is regulated; thus, concentrations up to 100 percent O_2 can be obtained (ref. 18). A higher amount of O_2 necessary to sustain burning indicates a greater resistance to burning; an index rating of 100 indicates that the material would only burn in an atmosphere of 100 percent O_2. The LOI gives a ranking index that may be used to compare materials. The procedure is described in Appendix A (sec. A.2), and Figures 3 and 4 show the apparatus and test setup.

3.1.1.2 Smoke Emission

The smoke emission characteristics of the candidate materials were determined using two techniques: (1) smoke accumulation in an enclosure was measured using the National Bureau of Standards (NBS) smoke chamber, and (2) smoke emission in an exposed air stream was measured using the Ohio State University (OSU) release rate apparatus. The OSU apparatus was operated at 5.0 W/cm² (264.3 Btu/ft²/min); while the NBS chamber was operated at 2.5 W/cm² (132.2 Btu/ft²/min) and 5.0 W/cm² (264.3 Btu/ft²/min) to determine the responses of materials to various fire environments.

The NBS chamber was selected in the FAA's proposed smoke regulation (ref. 1) because it is a laboratory simulation thought to represent a cabin fire. The chamber is sealed during the test; thus, oxygen depletion takes place and smoke builds up during the exposure. Specimens are exposed to a radiant heat source and pilot flame. The smoke obscuration is measured by passing

3

a light beam through the cabinet and measuring light transmission loss. The procedure is described in Appendix A (sec. A.3), and Figures 5 and 6 show the apparatus.

The OSU release rate apparatus exposes specimens to a radiant heat source in a chamber through which air is ducted. Smoke emission is measured by recording the light transmission across the exhaust stack. The procedure is described in Appendix A (sec. A.4), and Figures 7 and 8 show the apparatus.

3.1.1.3 Heat Release

Heat release characteristics of the candidate materials were determined using the technique of heat release in an exposed air stream measured using the OSU release rate apparatus. The apparatus was operated at an incident heat flux of 5.0 W/cm^2 (264.3 $Btu/ft^2/min$) to determine the response of materials to this fire environment.

The OSU release rate apparatus exposes specimens to a radiant heat source in a chamber through which air is ducted. Heat release is calculated from recorded inlet and exhaust air temperatures during a test. The procedure is described in Appendix A (sec. A.4), and Figures 7 and 8 show the apparatus.

3.1.1.4 Toxic Gas Emission

Toxic gas emissions were measured by gas accumulation in the NBS chamber. The NBS chamber exposure represents an open-fire condition where only partial (or surface) burning takes place.

Samples were taken using colorimetric tubes (for HCN, SO_2, and NO_x), NaOH absorber solutions (for halide gases), and online gas detectors (for CO and CO_2). The NaOH solutions were analyzed using specific ion electrodes. This technique involves gases from the NBS chamber being taken as a grab sample and results expressed as a concentration (ppm) of the gas in the accumulation chamber.

The NBS smoke chamber procedure is described in Appendix A (sec. A.3), and Figures 5 and 6 show the apparatus.

3.1.1.5 Flame Propagation

The flame propagation properties were measured on the American Society for Testing and Materials (ASTM) E162 flame spread apparatus. This device measures the surface flammability characteristics of materials. This method of measuring material surface flammability uses a radiant heat source in front of which an inclined specimen of the material is placed. The orientation of the specimen is such that ignition is forced near its upper edge, and the flame front progresses downward. A factor derived from the rate of progress of the flame front (ignition properties) and another relating to the rate of heat liberation by the material under test are combined to provide a flame spread index, I_s.

The procedure for determining flame spread is described in Appendix A (sec. A.5), and Figures 9 and 10 show the apparatus.

3.1.2 THERMOCHEMICAL TESTS

Both Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) tests were run to determine the decomposition rates of the materials. These tests were used to determine the exothermic (or endothermic) rate of the materials as they were decomposing as well as their weight loss. The materials with high exothermic rates were considered undesirable because of their contribution to a fire scenario. Materials with a high weight loss at temperatures below 260°C (500°F) were undesirable because the gases given off at these low temperatures would contribute ignitable fuel to a fire.

The DTA/TGA test procedures are described in Appendix A (sec. A.6).

4

3.1.3 MECHANICAL TEST

A mechanical property test was run on the candidate materials to determine their bond strength. This bond strength was measured by determining the peel strength between the 0.025-mm (0.001-in.) clear Tedlar (top film) and the ink/0.051-mm (0.002-in.) white Tedlar (substrate film). Details of the peel strength test procedure are described in Appendix A (sec. A.7), and a schematic of the specimen configuration and test setup is shown in Figure 11.

3.1.4 ADDITIONAL TESTS

Tests were run on the candidate ink systems to determine their viscosity, coating uniformity, and printability. All of these tests relate to the usability of the ink systems in a manufacturing environment on production materials and processes. Viscosity was measured with a Brookfield Viscosimeter at $25 \pm 1°C$ ($77 \pm 1.8°F$). Coating uniformity was determined by measuring the variation in ink film thickness after applying two coats over 0.051-mm (0.002-in.) white Tedlar. Printability was determined by screenprinting two coats of the candidate ink systems on 0.051-mm (0.002-in.) white Tedlar while observing the effects of dry time (both in-screen and as an applied film), the cohesion between successive coats of ink, and the print quality using the standard production screen material and mesh size. The following list shows the location of the detailed test procedures in this report (i.e., sections of appendix A):

- Viscosity – Section A.8
- Uniformity of Coating – Section A.9
- Printability – Section A.10

3.2 TEST MATERIALS

New materials were selected to determine reduction in fire hazard through burning more slowly, emitting less smoke, emitting less toxic gas, and/or retarding flame spread during a fire exposure. The candidate ink systems tested included epoxy, polyester, chlorofluorocarbon, silicone, and urethane. None of these inks were color pigmented. Cure of the urethane system was initiated by ultraviolet (UV) while all others were heat activated. All candidate ink systems were tested as potential replacements for the acrylic-type ink used by aircraft manufacturers today for decorative coating of commercial airplane interior sandwich panels.

During the evaluation testing, inks were considered: (1) individually, (2) coated on 0.051-mm (0.002-in.) white Tedlar, (3) sandwiched between 0.025-mm (0.001-in.) clear Tedlar and 0.051-mm (0.002-in.) white Tedlar, and (4) sandwiched as in item (3) above and bonded to a three-ply phenolic-fiberglass composite.

A complete list of candidate materials appears in Table 1 along with the baseline acrylic system.

3.2.1 BASELINE SYSTEM

Two basic types of decorative sandwich panels are currently used by aircraft companies as shown in Figure 12. The first system consists of a precured blank panel onto which is bonded a decorative polyvinyl chloride (PVC) outer layer. The materials used in the sandwich skins are normally flame-retarded (FR) epoxy-fiberglass (e.g., halogenated). The honeycomb core, before 1970, was phenolic/Kraft paper but has subsequently been changed to phenolic/polyamide paper. The second type of decorative sandwich consists of an integral decorative skin that replaces the polyvinyl chloride as well as forming the structural member of the sandwich panel. The decorative layer in this type of construction is printed or silk screened polyvinyl fluoride. The face sheets are FR epoxy-fiberglass, and the honeycomb core is phenolic-polyamide. It was this second type of panel that was considered in this program. The epoxy-fiberglass face sheets and phenolic-polyamide honeycomb core portions of the sandwich panel were addressed in Phase II (ref. 14), resulting in the identification of phenolic-fiberglass as an improved material from the

standpoints of flammability, smoke, and toxicity. The decorative film was considered in Phase III (ref. 15) and demonstrated that Tedlar possessed superior qualities when compared to all the other candidate materials. In Phase IV (this effort), as in Phase III, only the decorative layer was considered – with particular emphasis on the silk screen ink.

3.2.2 TEST SYSTEMS

Five types of resins were considered: (1) silicone, (2) chlorofluorocarbon, (3) urethane, (4) brominated polyester, and (5) phosphorylated epoxy. Each polymer possessed its own advantages. Silicone and phosphorylated epoxy offered generic thermal stability without additives; chlorofluorocarbon and brominated polyester used halogens for fire resistance; and urethane (UV initiated) presented a solventless ink system. See Table 1 for a list of candidate resin systems.

4.0 RESULTS AND DISCUSSION

A survey of the industry, which was conducted to select possible resins for development into a screenprinting ink with improved fire resistance over the current ink system (KC4900 series), yielded five candidates (sec. 4.1). In the screening evaluation phase (secs. 4.2 and 4.3), the objective was to select the most viable ink vehicle material among these candidates to fulfill the objectives of this contract.

In the optimization phase (secs. 4.4 and 4.5), two companies that specialize in the compounding of inks from basic resin materials were selected. With the help of these companies, one formulation from each source was developed by conducting tests on the compounded ink and the screenprinted coating on Tedlar. A trial and error optimization approach was used in arriving at the maximum· Limiting Oxygen Index (LOI) for the ink coated on 0.051-mm (0.002-in.) white Tedlar without diminishing the screenprinting and curing performance.

A large quantity of the optimized formulation from each company was ordered. Only the formulation from Midwest Coatings was received in time to compare the performance of the optimized ink with the baseline acrylic ink. These two inks were tested as: (1) cured ink systems, (2) cured ink systems laminated between 0.051-mm (0.002-in.) white and 0.025-mm (0.001-in.) clear polyvinyl fluoride (PVF) films, and (3) item (2) laminates bonded to a cured, three-ply laminate of phenolic-fiberglass prepreg. Details of this performance evaluation testing are discussed in Section 4.6.

4.1 CANDIDATE MATERIALS

An extensive industry survey was made to identify candidate ink resins for consideration in this program as discussed in Section 3.2. Consequently, five new resin systems of different generic chemical types were selected as candidates for screening evaluation. These are listed in Table 1 along with the respective manufacturers, product designations, and methods of curing. They were chosen as potential ink vehicles without laboratory testing. The candidate ink vehicles fall into the following chemical types: (1) chlorofluorocarbon, (2) silicone, (3) polyurethane, (4) brominated polyester, (5) phosphorylated epoxy, and (6) acrylic (baseline).

4.2 SCREENING EVALUATION

Among the materials selected for the screening evaluation, only KC4900 clear (baseline) was available in a readily usable form. Midwest Coatings was contacted and agreed to supply screenprinted samples of ultraviolet- (UV-) curable polyurethane for testing at Boeing. The rest of the candidates (viz., chlorofluorocarbon, silicone, and brominated polyester) were available only as resins. Hence, preparation of workable inks from these resins became the first task. This

6

meant identifying the right solvents and solution techniques. Solution viscosity measured as described in Section 3.1.4 was used as the criterion to establish amounts of resin and solvent. Dissolving silicone resin was the most problematic and time consuming part since different solvents and techniques had to be explored and experimented with. Eventually, solutions of resins in appropriate solvents were successfully prepared with viscosity levels appropriate for the screenprinting process.

During the ink preparation stage, phosphorylated epoxy and brominated polyester resins were eliminated from further consideration. Phosphorylated epoxy was not available to Boeing in sufficient quantities for testing. Reasons for eliminating brominated polyester will be explained later in Section 4.2.1.

The ink solutions of silicone, chlorofluorocarbon, and KC4900 clear were spray coated on 0.051-mm (0.002-in.) white Tedlar and ovendried to prepare workable coatings (sec. 4.2.2).

LOI, smoke emission, and toxic gas emission characteristics of three new ink systems coated on 0.051-mm (0.002-in.) white Tedlar were determined and compared to test results on KC4900 clear ink coated on 0.051-mm (0.002-in.) white Tedlar. These efforts are described in Sections 4.2.3, 4.2.4, and 4.2.5, respectively.

4.2.1 SOLUTION PREPARATION

The solution preparation is discussed by treating each resin individually.

(1)   Chlorofluorocarbon

The selected solvent had to fulfill the screenprinting process requirements. Fast and easy drying of the screenprinted ink became the criterion. KC4900-30 thinner is a mixture of low boiling solvents (table 2) and is used at Boeing to dilute the inks to obtain the required viscosity for the screenprinting line. Hence, KC4900-30 thinner was first tried as the solvent for all the resins, including chlorofluorocarbon.

A sample of FPC461 chlorofluorocarbon resin was received in the powder form. This powder was successfully dissolved in KC4900-30 thinner, resulting in a solution viscosity of about 15 000 cp when determined according to Appendix A (sec. A.8). After ordering and receiving a larger quantity of resin, a 1.9-l (0.5-gal.) solution of chlorofluorocarbon in KC4900-30 was prepared, containing 37.2 parts of chlorofluorocarbon by weight to 100 parts of solution to yield a viscosity of 14 500 cp.

(2)   Brominated Polyester

Brominated polyester is a liquid resin. It was impossible to mix it with solvents and obtain a solution with a usable viscosity. The highest viscosity value achieved was 250 cp with 43.6 parts by weight resin to 56.4 parts KC4900-30 thinner. The resin was neither clear nor colorless, which was unacceptable. Based on the supplier's information, it could not be coated on Tedlar. In view of the above, brominated polyester was removed from further evaluation.

(3)   Silicone

Silicone resin (DC6-2230) is a powder. However, sufficient quantities could not be dissolved in KC4900-30. The maximum viscosity level reached was 375 cp with 59.8 parts by weight of silicone resin to 40.2 parts of KC4900-30.

Dow Corning was contacted regarding the selection of solvent for DC6-2230. Xylene was suggested, but no definite solution preparation technique was available. After a series of trials, a solution was prepared with 16 300 cp viscosity. Final mix ratio was six parts

silicone resin to one part xylene. Slight heating was performed to facilitate the absorption of xylene into the silicone matrix. Final viscosity is sensitive to temperature. The total amount of silicone-xylene solution prepared was 1525g (3.36 lb).

4.2.2 COATING PREPARATION

The ink solutions of silicone-xylene, chlorofluorocarbon/KC4900-30 thinner, KC4900 clear, and a roll of 0.051-mm (0.002-in.) white Tedlar were shipped to NASA-ARC. There, equipment was available to spray coat Tedlar without using a screen and to produce a uniform coating after ovendrying. A description of this coating method is included as Appendix B.

Samples of silicone, chlorofluorocarbon, and the baseline KC4900 inks coated on 0.051-mm (0.002-in.) white Tedlar were received from NASA-ARC. There was an appreciable variation in thickness from ink to ink. Table 3 lists the average thickness of each coating received, including UV-cured polyurethane ink coated on 0.051-mm (0.002-in.) white Tedlar from Midwest Coatings.

The silicone coating was brittle and cracked and flaked off during handling, exposing the substrate Tedlar. This cracked coating was expected to have an adverse influence on the flame spread by channeling the flame through the cracks and increasing the total surface area exposed to ignition conditions, thereby facilitating the combustion reaction.

4.2.3 LIMITING OXYGEN INDEX

Table 4 lists the measured LOI test (sec. 3.1.1.1) results for KC4900, chlorofluorocarbon, silicone, and UV-cured polyurethane inks coated on 0.051-mm (0.002-in.) white Tedlar. The LOI of plain 0.051-mm (0.002-in.) Tedlar is also included in Table 4 for comparison purposes.

Silicone- and polyurethane-coated Tedlar both exhibit a lower LOI than plain Tedlar. On the other hand, KC4900 acrylic- and FPC461 chlorofluorocarbon-coated Tedlar show an improvement in LOI when compared to plain Tedlar.

UV-cured polyurethane ink from Midwest Coatings exhibits the lowest LOI, which is lower than the mole percent of oxygen in the atmosphere. Oxygen concentration in the atmosphere is more than sufficient to feed the combustion reaction of polyurethane ink-coated Tedlar film. In other words, the inherent resistance of Tedlar to continuing combustion in normal atmospheric conditions is lost by coating it with polyurethane ink.

Silicone is ranked third in LOI behind FPC461 chlorofluorocarbon and KC4900 acrylic coatings. Its LOI is slightly less than that for plain Tedlar. No improvement in flame resistance is obtained by using silicone on Tedlar. The low LOI obtained is mainly due to the nature of the coating as described in Section 4.2.2 and should not be attributed to the resin itself.

KC4900 acrylic ink coating yields a better LOI than plain Tedlar. Hence, Tedlar receives additional protection from this ink. The LOI of KC4900, which is the baseline against which fire resistance properties of other resins were compared, is the second best. The best LOI value obtained was for FPC461 chlorofluorocarbon resin-based ink coated on Tedlar. Its LOI of 30.3 shows inherently high resistance to combustion in air.

Figure 13 visually highlights the differences in LOI values of these inks.

4.2.4 SMOKE EMISSION

National Bureau of Standards (NBS) chamber (sec. 3.1.1.2) smoke evolution test results at 2.5 W/cm^2 (132.2 Btu/ft^2/min) and 5.0 W/cm^2 (264.3 Btu/ft^2/min) heat fluxes for KC4900 acrylic, FPC461 chlorofluorocarbon, DC6-2230 silicone, and Midwest UV-cured polyurethane are reported and compared in this section.

8

Table 5 lists the specific optical density, D_s, values measured at 2.5 W/cm² (132.2 Btu/ft²/min) heat flux for each ink coating at 1.5 min, 4.0 min, and the time at which the maximum D_s value is reached. Normalization of the thickness of coating is essential for proper comparison since the amount of smoke emitted is directly proportional to the amount or thickness of ink coated on 0.051-mm (0.002-in.) Tedlar. Table 6 lists D_s values per mil of coating obtained by dividing D_s values in Table 5 by the thickness of the coating. Figures 14 and 17 have been plotted using the values in Tables 5 and 6, respectively, with no measurable smoke (i.e., $D_s = 0$) at zero time.

Similarly, measured D_s values at 5.0 W/cm² (264.3 Btu/ft²/min) heat flux are shown in Table 7 and Figure 15. D_s values at 5.0 W/cm² (264.3 Btu/ft²/min) normalized for the thickness of coating are shown in Table 8 and Figure 18.

Figure 16 shows measured D_s values at both 2.5 W/cm² (132.2 Btu/ft²/min) and 5.0 W/cm² (264.3 Btu/ft²/min) heat fluxes. As expected, D_s values at 5.0 W/cm² (264.3 Btu/ft²/min) heat flux are consistently higher than those at 2.5 W/cm² (132.2 Btu/ft²/min). Also, the time required to reach a given D_s value is shorter at the higher heat flux.

Based on the data plotted in Figures 17 and 18, UV-cured polyurethane evolves the largest amount of smoke per mil of coating, and silicone evolves the least at both heat fluxes. At 2.5 W/cm² (132.2 Btu/ft²/min) heat flux, chlorofluorocarbon resin is better than baseline KC4900 acrylic and almost as good as silicone, which is the best. At 5.0 W/cm² (264.3 Btu/ft²/min) heat flux, the smoke release of chlorofluorocarbon is comparable to the baseline acrylic when the data up to 4 min are considered.

Hence, the ranking based on smoke emission data from zero time up to 4 min is as follows (1 = Best):

(1)   DC6-2230 silicone

(2)   FPC461 chlorofluorocarbon

(3)   KC4900 acrylic

(4)   Midwest UV-cured polyurethane

4.2. TOXIC GAS EMISSION

Tables 9 and 10 list the toxicant concentrations measured in the NBS chamber at 2.5 W/cm² (132.2 Btu/ft²/min) and 5.0 W/cm² (264.3 Btu/ft²/min), respectively. Tables 11 and 12 show the toxicant concentrations normalized for the coating thickness. The toxicants considered were HCN, NO_x, CO, CO_2, HCl, HF, and SO_2.

It is more reasonable to compare the values listed in Tables 11 and 12 because the toxicant concentrations, as well as the amount of smoke generated, are properties that depend on the amount of material burned, which is proportional to the thickness of coating.

HF concentrations at 5.0 W/cm² (264.3 Btu/ft²/min) (table 10) show lower values when compared with those at 2.5 W/cm² (132.2 Btu/ft²/min) (table 9). The HF concentrations should have increased or at least maintained the same level when the heat flux was doubled. The data on other toxicants exhibit increased concentration with increased heat flux, and only HF data are contrary to this general trend. Hence, caution must be exercised when conclusions are drawn based on the NBS chamber data for HF evolution. At best, these data may be used only for comparisons among the materials under constant heat flux conditions.

No abnormal toxicant levels were noted. Since biological response tests have not been conducted, no conclusions can be drawn regarding the comparative toxicity of the ink resins. However, based on the concentration levels of the toxicants considered, the candidate resins should be toxicologically equivalent or better than KC4900.

4.3 SELECTION OF ONE CANDIDATE INK VEHICLE FOR OPTIMIZATION

The objective of the screening evaluation task described in Section 4.2 was to select one candidate ink vehicle from the three considered. KC4900 acrylic was used as the baseline for the evaluation. Performance of candidate ink vehicles in the screening evaluation is summarized in Table 13.

UV-cured polyurethane performed the least acceptably. Its LOI was lower than atmospheric oxygen content. It evolved the largest amount of smoke compared to all the inks considered. It was eliminated from consideration for optimization.

Based on the smoke and toxic gas emission data, DC6-2230 silicone would be the superior system. Its low LOI is related to the poor quality of coating obtained on Tedlar. Since silicone is more chemically stable, its solubility is very low in solvents normally used for making screenprinting inks. Also, it exhibits poor adhesion to Tedlar. The silicone resin was dissolved in xylene with difficulty. Hence, even though silicone has potentially the best fire resistance properties, its poor solubility in standard screenprinting ink solvents (solvents used in screenprinting inks dictate curing time, drying in the screen, and flow characteristics) and poor ability to coat Tedlar eliminate silicone from further consideration for optimization.

Thus, by a process of elimination, FPC461 chlorofluorocarbon resin was selected for optimization from the candidate ink systems studied. FPC461 exhibits a superior LOI, significantly lower smoke evolution, and more or less equivalent toxicity compared to the present KC4900 ink system. The solutions having the appropriate viscosity in the screenprinting range can be easily made by dissolving chlorofluorocarbon resin in the normally available ink thinner solvents. Its Tedlar coating ability and adhesion were satisfactory. A peel strength test (sec. 3.1.3) was conducted to evaluate the adhesive properties and will be discussed later.

4.4 SELECTION OF COMPANIES FOR OPTIMUM INK FORMULATION

FPC461 chlorofluorocarbon resin was selected for further optimization. Information on this resin, which is available from Hooker Chemical, Plastics Division, Firestone Blvd., P.O. Box 699, Pottstown, Pennsylvania 19464, is included as Appendix C. Two ink manufacturing companies were contacted for assistance in compounding a screenprintable ink out of the basic resin without diminishing its fire resistance characteristics as discussed in the foregoing Section 4.3. This condition required solvents, plasticizers, and other additives that would not adversely affect the fire resistance capabilities of the resin.

The participants were: (1) Midwest Coatings, Kansas City, Missouri and (2) Seri-Print of Canada Ltd., Scarborough, Ontario, Canada. It was agreed between Boeing and the participants that their formulations would remain as their proprietary information and would be identified and tested with their respective batch numbers.

4.5 OPTIMIZATION STUDIES

The ink formulations received from the participating companies were screenprinted on 0.051-mm (0.002-in.) white Tedlar to obtain a 0.6- x 0.6-m (2- x 2-ft) print on the handprinter (discussed in sec. A.10 of appendix A). They were then dried at 71.1°C (160°F) for 90s. LOI tests were run on these prints. The prints were also laminated with a 0.025-mm (0.001-in.) clear Tedlar on top. Some of these screenprinted specimens were embossed to prepare a decorative laminate (DEC-LAM) (clear Tedlar/two coats of ink/white Tedlar/fiberglass-epoxy, one-ply). Since only clear inks were tested, "cut-through" and "wash-out" properties of the ink could not be checked. However, these trials were run to ensure that no major problems would be encountered. The printability was checked as explained in Section 3.1.4.

Each formulation was thus evaluated for its screenprintability and flammability properties. The results were fed back to the respective ink formulators to adjust the subsequent formulation in an attempt to improve the printability and flammability performance. Thus, a trial and error approach was used in progressively arriving at the maximum LOI without diminishing the screenprinting and curing performance. Optimization of a formulation from each participating company is described in the following sections.

4.5.1 FORMULATIONS OF MIDWEST COATINGS

Table 14 summarizes the optimization effort expended on formulations received from Midwest Coatings. The initial formulation (P1116) coated on 0.051-mm (0.002-in.) Tedlar exhibited an undesirable thickness variation, viz., 0.0203 mm to 0.0356 mm (0.0008 in. to 0.0014 in.). It dried too rapidly on the screen during the application of the second coating. It also showed a slight yellowing when embossed during preparation of the DEC-LAM.

The LOI of P1116 ink coated on 0.051-mm (0.002-in.) white Tedlar was measured as 28.8. During the screening evaluation phase, an LOI of 30.3 was obtained for chlorofluorocarbon resin (table 4). This value was treated as the target value in the optimization phase. Although the LOI of 28.8 for P1116 was lower than the target value, it was believed that it could be improved.

Based on the above observations by Boeing, which were supplied to Midwest Coatings, a new formulation (P2028) was submitted for evaluation. P2028 performed satisfactorily during the screenprinting and embossing stages despite a very slight yellowing observed after cure. Its LOI when measured as coated on 0.051-mm (0.002-in.) white Tedlar was 30.0. Even though this value is less than the target value of 30.3, it was deemed to be sufficient improvement. Hence, P2028 was the optimized ink formulation from Midwest Coatings. A larger order was placed for this formulation with Midwest Coatings for use in the next phase of performance evaluation and comparison to KC4900 ink.

4.5.2 FORMULATIONS OF SERI-PRINT OF CANADA, LTD.

Table 15 gives a summary of the formulations evaluated during optimization of chlorofluorocarbon resin-based ink from Seri-Print of Canada, Ltd. Seri-Print D1062 ink coating was more or less uniform in thickness, 0.0152 mm to 0.0203 mm (0.0006 in. to 0.0008 in.). It dried too rapidly on the screen during the application of the second coating and also showed a slight yellowing when embossed during preparation of the DEC-LAM.

The LOI of D1062 was measured as 28.8. This is lower than the target value of 30.3 measured for chlorofluorocarbon resin in Table 4. The results of this evaluation were reported to Seri-Print, and an improved formulation was requested.

Seri-Print submitted D1073 ink as the improved formulation for further evaluation. However, the viscosity of D1073 was too low for screenprinting purposes. Hence, Boeing was not able to screenprint Tedlar using this batch of ink. Upon learning this, Seri-Print resubmitted a similar formulation with a more desirable viscosity.

Seri-Print batch No. D1089 performed satisfactorily during the screenprinting operation and yielded a coating with uniform thickness. The LOI was measured as 30.2 for this ink coated on 0.051-mm (0.002-in.) Tedlar. The LOI for D1089 was the closest to the target value of 30.3. Even though this formulation showed slightly fast drying in-screen during the second coat, its performance was sufficiently optimized with respect to flammability to proceed with the next task of performance evaluation. A larger quantity of D1089 was ordered from Seri-Print to complete the performance evaluation.

4.6 PERFORMANCE EVALUATION

Among the two formulations identified in the optimization phase, only Midwest Coatings could deliver the large order of P2028 ink formulation in time for the performance evaluation studies. Seri-Print of Canada, Ltd. experienced delays in raw material delivery and could not meet the time constraints of the contract. Batch D1089, which is one of the optimized formulations, was delivered to Boeing only a month and a half ahead of the expiration date of the contract. There was insufficient time to include it in the performance evaluation studies.

A performance evaluation was conducted on the optimized formulation of P2028 from Midwest Coatings to determine the improvements in fire response characteristics that had been achieved. KC4900 clear acrylic ink was used as the baseline. Specimens used in the performance evaluation and tests conducted on the respective specimens are indicated in the following:

(1) Cured ink system

    a. Thermogravimetric Analysis (TGA) (in nitrogen and in air)

    b. Differential Thermal Analysis (DTA) (in nitrogen and in air)

(2) Cured ink system laminated between 0.051-mm (0.002-in.) white and 0.025-mm (0.001-in.) clear polyvinyl fluoride films

    a. LOI

    b. Peel strength

    c. Federal Aviation Regulation (FAR) 25-32 (12- and 60-s vertical ignition)

(3) Item (2) laminate bonded to a three-ply laminate of Fiberite MXB6070-7781 with a 0.025-mm (0.001-in.) silicone film adhesive (Densil from Dennison Manufacturing Company)

    a. NBS chamber – 5.0 W/cm^2 (264.3 Btu/ft^2/min) – flaming mode – smoke and toxic gases

    b. Ohio State University (OSU) chamber – 5.0 W/cm^2 (264.3 Btu/ft^2/min) – flaming mode – vertical orientation – smoke and heat release characteristics

    c. Flame spread index, American Society for Testing and Materials (ASTM) E162

4.6.1 TEST SPECIMEN PREPARATION

The cured inks were prepared by evaporating the solvent at 71.1°C (160°F) for a minimum of 10 min. This procedure yields a film of residue, which includes either chlorofluorocarbon for P2028 or acrylic for KC4900. TGA and DTA were run using this cured ink.

The test inks were screenprinted in the production screenprinter and dryer (fig. 19). This yielded a cured-ink film of about 0.025-mm (0.001-in.) thickness for two coats on 0.051-mm (0.002-in.) white Tedlar. This screenprinted Tedlar was laminated with a 0.025-mm (0.001-in.) thick clear Tedlar on top. Specimens were cut out of these rolls for LOI, peel strength, and FAR 25-32 (12- and 60-s vertical ignition) tests.

The laminate of 0.025-mm (0.001-in.) clear Tedlar/ink/0.051-mm (0.002-in.) white Tedlar was cut to the required sizes and bonded to a three-ply laminate of Fiberite MXB6070-7781 with a 0.025-mm (0.001-in.) silicone film adhesive. The three-ply laminate of Fiberite MXB6070-7781 was press-cured at 160°C (320°F) and 6.9 x 10^5 N/m^2 (100 psi) for 10 min. Layup for this fabrication is diagrammed in Figure 20.

4.6.2 CURED INK SYSTEM

TGA data were generated for Midwest P2028 and KC4900 inks in nitrogen and air. Data obtained were percent weight loss versus temperature and percent residue remaining, if any. Simultaneously, DTA data were generated during the weight loss run. DTA data resulted in heat evolved (exothermic) or absorbed (endothermic) during the weight loss transformation.

Data obtained in air are closer to reality than in nitrogen. Since air contains nitrogen as well as oxygen, data obtained in a nitrogen atmosphere act as a datum in understanding the thermal stability of materials in the burning reaction. (Only oxygen is a reactant; nitrogen is an inert participant.) Hence, the data in nitrogen represent mainly heating and thermal decomposition effects; whereas the data in air include effects of oxygen reacting with the material (oxidation or burning) in addition to the above. Enthalpy and weight loss are the main effects relevant to this study. These are discussed in the following sections.

4.6.2.1 Thermogravimetric Analysis

TGA data obtained in nitrogen and in air are listed in Tables 16 and 17, respectively, for Midwest P2028 and KC4900 screenprinting inks. Data were plotted in Figures 21, 22, 23, and 24 individually for the two inks. Combined TGA data for the inks can be found in Figures 25 and 26 for easy comparison.

As shown in Figure 26, nitrogen atmosphere plot, the major weight loss (up to 80 percent) temperature for KC4900 is about 250°C (482°F); whereas that for Midwest P2028 is about 400°C (752°F). Hence, P2028 chlorofluorocarbon resin is more stable than KC4900 acrylic in heating, and its inherent thermal stability is greater. The amount of residual material is not significantly different for KC4900 and Midwest P2028 resins.

When the temperatures corresponding to 70 percent weight loss are compared, Figure 25 shows that Midwest P2028 decomposes around 350°C (662°F) and KC4900 around 240°C (404°F) in air. At all temperatures up to 750°C (1382°), the percent weight remaining is higher for Midwest P2028 than for KC4900. Hence, even when heating and burning are combined, Midwest P2028 performs better than KC4900, and fewer smoke particles are expected from P2028 than from KC4900 based on these results.

4.6.2.2 Differential Thermal Analysis

DTA data obtained in nitrogen and in air are listed in Tables 18 and 19, respectively, for Midwest P2028 and KC4900 screenprinting inks. Data are schematically plotted in Figures 21, 22, 23, and 24, showing the relationship to the weight loss curve.

Considering the heat effects up to 80 percent weight loss in nitrogen (figs. 21 and 22), KC4900 has a net endotherm of 32 cal/g (58 Btu/lb) with 300°C (572°F) as the end of peak; whereas Midwest P2028 has a net exotherm of -8.3 cal/g (-14.9 Btu/lb) with 460°C (860°F) as the end of peak. There are no further heat effects above 80 percent weight loss due to gas phase reactions since nitrogen atmosphere is maintained.

As seen in Figures 23 and 24, heat contributions from gas phase reactions show up after major weight loss has occurred because reactive oxygen is now available from the air environment. Considering weight loss up to 100 percent, KC4900 releases a net heat of 261.3 cal/g (470.3 Btu/lb); whereas Midwest P2028 releases only 89.9 cal/g (161.8 Btu/lb). Hence, Midwest P2028 ink resin would not add as much fuel and heat energy to a fire as KC4900 during decomposition.

4.6.3 DECORATIVE LAMINATE

Tests for evaluation of the performance of decorative laminates prepared with KC4900 and Midwest P2028 inks included LOI, peel strength, and FAR 25-32 (12- and 60-s vertical ignition).

13

Test specimens were 0.025-mm (0.001-in.) clear Tedlar/ink/0.051-mm (0.002-in.) white Tedlar. The following sections contain results and discussion of these tests.

4.6.3.1 Limiting Oxygen Index

Table 20 compares LOIs of KC4900 clear and Midwest P2028 clear inks laminated between 0.051-mm (0.002-in.) white and 0.025-mm (0.001-in.) clear polyvinyl fluoride films. Midwest P2028 exhibits a higher value than KC4900 laminate. Hence, Midwest P2028 ink-coated laminate has a lesser propensity to burn than KC4900.

4.6.3.2 Peel Strength

A peel strength test indicated that clear Tedlar film could not be peeled from the cured ink coated on 0.051-mm (0.002-in.) white Tedlar. Clear Tedlar film broke even before the peeling could start. Breaking load for KC4900 clear was 0.65 kg (1.43 lbs) and that for Midwest P2028 was 0.76 kg (1.68 lbs). Adhesive forces are stronger than the breaking load for clear Tedlar in the respective ink laminates.

4.6.3.3 FAR 25-32 (12- and 60-s Vertical Ignition)

FAR 25-32 results are summarized in Table 21. Midwest P2028 ink-coated laminate gives significantly lower burn lengths in both 60-s vertical and 12-s vertical tests. The only difference in the laminate construction is the inks; viz., Midwest P2028 and KC4900. It is reasonable to conclude that Midwest P2028 retards flame propagation better than KC4900. The amount of ink material involved in a typical laminate, similar to the one used in this test, is very small compared to the amount of Tedlar film. These vertical flammability results are noteworthy since they seem to bring out the differences in the role played by the ink material in the combustion of such laminates.

4.6.4 DECORATIVE LAMINATE BONDED TO THREE-PLY LAMINATE OF PHENOLIC-FIBERGLASS PREPREG

NBS chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – flaming mode – smoke and toxic gases, OSU chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – flaming mode – vertical orientation – smoke and heat release characteristics, and flame spread index (ASTM E162) tests were run on decorative laminates of KC4900 and Midwest P2028 inks between 0.051-mm (0.002-in.) white and 0.025-mm (0.001-in.) clear polyvinyl fluoride films bonded to three-ply laminates of phenolic-fiberglass prepreg. A 0.025-mm (0.001-in.) transfer tape adhesive made by the Dennison Company was used for bonding. The following sections contain the results and discussion on these tests.

4.6.4.1 Smoke Emission

NBS chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – flaming mode – smoke test results are shown in Table 22 and Figure 27. The panel containing Midwest P2028 ink evolved significantly less smoke than the panel containing KC4900 ink.

4.6.4.2 Toxicity Characteristics

NBS Chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – flaming mode – toxicant concentrations for KC4900 and Midwest P2028 panels are compared in Table 23. The concentration values for HF are too low and unrealistic. However. both KC4900 and Midwest P2028 are similarly affected. The toxicant concentrations for the other gases also show no major differences between the panels containing KC4900 and Midwest P2028 inks.

4.6.4.3 Smoke Release Rate

OSU chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – flaming mode – vertical orientation – smoke release results for KC4900 and Midwest P2028 are shown in Tables 24 and 25 and plotted in Figures 28 and 29.

Table 24 shows the rate of release of smoke particles, S, per unit area of specimen surface and rate of change of specific optical density, D_{s}, for Midwest P2028. Table 25 shows the same for KC4900. The smoke particles release data versus time, t, are plotted in Figure 28 and specific optical density, D_{s}, data versus time are plotted in Figure 29.

From Figures 28 and 29, it can be seen that Midwest P2028 performs far better than KC4900. The rate of release of smoke particles from Midwest P2028 is always less than KC4900 except in the 8- to 15-s interval. It is evident that even though the amount of ink material involved is small, it makes a difference in smoke evolution from the panels tested.

4.6.4.4 Heat Release Rate

OSU chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – flaming mode – vertical orientation – heat release results for KC4900 and Midwest P2028 are tabulated in Tables 26 and 27, respectively. For easy comparison, Figure 30 shows the plots of these data for both KC4900 and Midwest P2028. As seen from this figure, heat release rates for the two ink systems are more or less equivalent even though Midwest P2028 has a slightly smaller total heat release.

Heat contribution from ink material is not big enough to make a significant difference in the overall heat evolution when the panel is tested in the OSU chamber under the conditions of 5.0 W/cm² (264.3 Btu/ft²/min) radiant heat and flaming mode.

4.6.4.5 Flame Spread

No useful results could be gained from the ASTM E162 test on the panels for studying the contribution of inks to the flame spread phenomenon. As soon as the flame started at the top, both Tedlar films, 0.025-mm (0.001-in.) clear and 0.051-mm (0.002-in.) white, burned and shrank away within a few seconds. Silicone film adhesive, used in bonding the decorative laminate and the glass-phenolic panel together, contributed to the destruction of the decorative laminate within a few seconds by starting an extremely non-uniform flame front. It was difficult to transfer the silicone film adhesive, 0.025-mm (0.001-in.) thick, to the panel to give a uniform coating. Usually patches and islands of adhesive were unavoidable.

5.0 CONCLUSIONS

FPC461 chlorofluorocarbon resin has been identified as a workable screenprinting ink vehicle. It has satisfactory printability, curing properties, and adhesion to Tedlar.

The propensity to burn and the smoke emission of panels can be significantly lowered by utilizing a screenprinting ink (like Midwest P2028) formulated with this resin instead of KC4900 acrylic. Even though chlorofluorocarbon as a basic resin was more thermally stable and contributed only about one-third the heat energy for burning in air when compared with KC4900 acrylic, the actual amount of ink used to decorate the panels is negligible, resulting in only a minor contribution to the total heat energy release during a fire. Toxic gas emissions of FPC461 chlorofluorocarbon resin are comparable to KC4900 acrylic.

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16

6.0 FUTURE WORK

Chlorofluorocarbon resin-based screenprinting ink was tested as clear, that is, without containing any pigments. In order to evaluate the ink vehicle for "cut-through" and "wash-out" properties during the texturing stage, color pigments have to be included in future studies. In addition, the impact on the fire resistance, smoke, and toxicity properties of the resins that contain pigments has to be evaluated.

Chlorofluorocarbon resin is expensive for screenprinting ink application when compared with KC4900 acrylic in the quantities used in this study. Cost impact studies that consider production-scale requirements for chlorofluorocarbon resin have to be performed.

APPENDIX A
DETAILS OF TEST PROCEDURES

A.1 FLAMMABILITY, 12- AND 60-s VERTICAL

FAR 25-32 flammability tests are required by the FAA for flight hardware certification. In accordance with FAR 25-32 (ref. 17), the 12- and 60-s vertical ignition tests were conducted. The procedure described in the following paragraphs is applicable to both tests. Typical test setups are shown in Figures 1 and 2.

The Bunsen burner was operated on commercially pure methane gas supplied from a storage tank at a flow rate of 2212 ±10 cm³/min (135 ±0.6 in.³/min). The flame was adjusted to give a minimum temperature of 843 ±10°C (1550 ±18°F) with a flame height of 3.8 cm (1.5 in.) total and a blue cone height of 1.9 cm (0.75 in.). Flame temperature was measured using a Leeds & Northrop Model 8659 bridge-type potentiometer and chromel-alumel thermocouple that was mounted to the specimen holder frame for accurate positioning during measurement.

The specimens were mounted vertically as shown in Figure 2. Three specimens of each material were tested at these conditions. The time during which the burner flame was applied to the specimen and the time of specimen burning following removal of the burner flame were measured using an electric timer accurate to within 0.1s. Burned length was determined by measurement with a steel scale graduated in 0.25-mm (0.01-in.) increments. The test specimens were 7.6-cm (3-in.) wide x 30.5-cm (12-in.) long and were conditioned prior to testing for a minimum of 24 hrs at 21.1 ±2.8°C (70 ±5°F) and 50 ±5% relative humidity.

A.2 LIMITING OXYGEN INDEX

LOI tests were performed in the oxygen-nitrogen test apparatus shown in Figures 3 and 4. The tests were conducted in accordance with ASTM D2863 (ref.18).

The initial concentration of oxygen and nitrogen chosen was based on past experience with similar materials. The gases were allowed to flow for 30s to purge the system. The specimen was ignited so that the entire tip was burning. The relative flammability was determined by adjusting the concentration of gases rising past the specimen to a point at which the oxygen concentration was the minimum that would allow the specimen to burn; i.e., the specimen burns 3 min or longer or burns 5 cm (3 in.). Volumetric flow of the oxygen and nitrogen gases was measured by calibrated glass flowmeters. The LOI was calculated using the following formula:

$$LOI = \frac{(100)\,(V_{f1})}{V_{f1} + V_{f2}}$$

where V_{f1} and V_{f2} are the volumetric flow rates in cm³/s of O_2 and N_2, respectively.

The length and width of the specimens were as specified in ASTM D2863. However, specimen thickness varied.

A.3 NBS SMOKE CHAMBER

Smoke and toxic gas generation was determined in an accumulating chamber of the design used by the NBS and described in NBS Technical Note 708 (ref. 19). The test equipment and operation are described in the following paragraphs.

The test chamber is a sealed metal box, 0.91-m (3-ft) wide x 0.61-m (2-ft) long x 0.91-m (3-ft) high, with a total capacity of 0.51 m³ (18 ft³). The test chamber contains a furnace, specimen

holder, and photometer system and has provision for the attachment of a gas burner. The chamber is shown in Figures 5 and 6.

The photometric system consists of a high-intensity light source and photocell. The light path is vertical within the chamber to reduce errors arising from smoke stratification. A sensitive amplifier with large meter scales for accurate readings is supplied as the readout system and, by this means, values of light transmittance are obtained. A recorder is connected to the meter so that a continuous plot of transmittance is obtained.

The percentage change in the light transmission is converted to an optical density value by means of the following equation:

$$D_s = \frac{V}{AL} \log_{10} \frac{100}{Plt}$$

where:

D_s = specific optical density

V = chamber volume, 0.51 m^3 (18 ft^3)

L = light path length, 0.91m (3 ft)

A = exposed test specimen surface area, 42.35 cm^2 (6.56 in.^{2})

Plt = percent light transmission

The test specimen size was approximately 7.6 x 7.6 cm (3 x 3 in.). The back, edges, and unexposed front surfaces of the specimen were covered by a single sheet of aluminum foil. The foil-protected specimens then were backed by a 7.6- x 7.6- x 1.3-cm (3- x 3- 0.5-in.) sheet of asbestos millboard. The use of asbestos sheet minimizes the heat loss through the rear of the specimen. The microjet gas burner was placed in front of the radiant furnace so that the jets impinged on the bottom surface of the specimen. The air/propane mixture was adjusted to the correct ratio and flow rate by adjustment of two independent flowmeters. The specimen then was slid across into the heat path of the furnace and in front of the gas jets, and burning commenced. After completion of each test, the cabinet was vented and the photocell cleaned. At least three specimens were tested at each radiant heat flux; viz., 2.5 W/cm^2 (132.2 Btu/ft^2/min) and 5.0 W/cm^2 (264.3 Btu/ft^2/min).

The specimen under test is irradiated by means of an electrically heated radiant energy source mounted within an insulated ceramic tube, which is positioned so that the desired irradiance level averages over the central 3.8-cm (1.5-in.) diameter area of the vertically mounted specimen. The irradiance level is determined by the applied voltage to the furnace, which is controlled by a rheostat.

The gas burner has six flamelets, two of which are directed horizontally at right angles to the sample surface, two of which are canted downward 45 deg to the sample surface, and two of which are canted downward approximately 60 deg, impinging on the sample holder reservoir.

The specimen holders, fabricated from stainless steel, are designed to expose a 6.5-cm (2.562-in.) square specimen area to the radiant heat of the furnace. The gas jets emerge along the bottom edge of the specimen. The specimen, supported as previously described, is located vertically, 3.8 cm (1.5 in.) in front of the furnace opening. A 7.6-cm (3-in.) square of asbestos millboard is used to back the specimen, and the whole assembly is retained by a bent spring of phosphor bronze and a steel retaining rod.

Toxic gas generation was determined quantitatively using colorimetric (Dräger) tubes, NaOH absorber solutions, and online gas detectors. Each Dräger tube was designed by the manufacturer

to measure a specific type of gaseous product. The NaOH solutions were analyzed using specific ion electrodes.

A.4 OSU RELEASE RATE APPARATUS

Heat release characteristics were determined using the OSU release rate apparatus. The test equipment (figs. 7 and 8) and operation are described in the following paragraphs.

The temperature difference between the air entering the environmental chamber and that leaving was monitored by a thermopile for heat release calculations. An electrically heated panel was used as the radiant heat source.

A pilot flame was used as the ignition source for the specimens tested. The flame was positioned 10 mm (0.4 in.) from and perpendicular to the exposed surfaces of the vertical specimens. The centerline at the outlet of the pilot burner tube intersected the vertical centerline of the vertical specimens 5 mm (0.2 in.) above the lower edge.

The specimens measured 15.2 x 15.2 cm (6 x 6 in.). The specimens were conditioned for 24 hrs in an oven at 60°C (140°F) and then placed in a cabinet at 21.1 ±2.8°C (70 ±5°F) and 50 ±5% relative humidity for a minimum of 24 hrs before testing. Three specimens from each panel were tested at 5.0 W/cm² (264.3 Btu/ft²/min).

The pilot flame was ignited and positioned. The radiant panel was set for the desired heat flux. The air flow to the equipment was set at 2.38 ±0.11 m³/min (84 ±4 ft³/min) for atmospheric pressure and 23.3°C (74°F) temperature conditions. Steady-state conditions, such that the radiant heat flux did not change more than 0.511 kW/m² (0.045 Btu/ft²/s) over a 10-min period, were maintained before the specimen was injected.

The specimen was placed in the hold chamber with the radiation shield doors closed. The airtight outer door was secured, recording devices started, and output of the thermopile set to "zero" on the recorder. The specimen was retained in the hold chamber 60 ±5s before ignition.

Test duration was 5 min. A blank run (baseline test) was performed during which the specimen holder, with a piece of asbestos in place of a specimen, was injected and heat release versus time data taken.

The total heat release is calculated by integrating the temperature rise over the length of the run.

$$\text{Total heat evolved} = H_t = \frac{C_k}{A} \int_0^t T_0 dt$$

where:

t   = time

C_k = constant (function of V, T_i, and heat flux)

A   = area of sample, 232.3 cm² (0.25 ft²)

T_i = inlet temperature, °C (°F)

T_0 = outlet temperature, °C (°F)

V   = volume of air, 2.4 m³/min (85 ft³/min)

A.5 FLAME SPREAD

The flame spread index was determined using the radiant panel apparatus shown in Figures 9 and 10. Tests were conducted in accordance with ASTM E162 (ref. 20). The test equipment and procedure are described in the following paragraphs.

The apparatus uses a vertically mounted, porous refractory panel that has a radiating surface of 30.5 x 45.8 cm (12 x 18 in.) and can operate up to 816°C (1500°F). The panel has a venturi-type aspirator for mixing propane and air that is supplied by a blower at approximately atmospheric pressure. Mounted in front of the panel is a framework that supports the specimen holder in the middle of the panel at an angle of 30 deg from the vertical (see fig. 11). A pilot burner igniter is also mounted in front of the panel and is in such a position that the flame impinges on the specimen 13 mm (0.5 in.) from the top edge of the specimen and 6.35 mm (0.25 in.) from the nearer side of the specimen holder opening. Located above the panel and framework is a hood with a stack mounted within it, situated directly over the panel and specimen location. Mounted within the stack is a thermopile consisting of eight chromel-alumel thermocouples connected in parallel.

Additional equipment includes a potentiometer recorder for recording temperature variations of the stack thermocouples. A radiation pyrometer is used to standardize the thermal output of the radiant panel. The final piece of equipment is a timer that is calibrated to read to 0.01 min for recording the time of events during the test.

Specimens tested measured 15.2 x 45.8 cm (6 x 18 in.). Conditioning of the specimens consisted of a 24-hr exposure to 60°C (140°F) in an oven followed by an equilibration cycle at an ambient temperature of 23 ±3°C (73 ±5°F) and a relative humidity of 50 ±5%. Normally, 24 hrs is sufficient for specimens to attain equilibrium. Four specimens of each material were tested.

To conduct a test, combustion products are first removed from the thermocouples in the stack. The propane-air mixture running through the radiant panel is ignited and adjusted to provide a blackbody operating temperature of 670 ±4°C (1238 ±7°F) as determined by the radiation pyrometer. The pilot flame is ignited and adjusted to provide a 5.1- to 7.6-cm (2- to 3-in.) long flame. The stack temperature is recorded. The specimen, mounted in a holder, is placed on the supporting framework and the timer simultaneously started. The times are recorded for the formation of a flame front on the surface of the specimen and for the arrival of the flame front at each of the 7.6-cm (3-in.) indicator marks on the specimen holder. The test run is completed when the flame front has progressed to the 38.1-cm (15-in.) mark or after an exposure time of 15 min, whichever occurs first, provided the maximum temperature at the stack thermocouples has been reached.

The flame spread index is the product of the flame spread factor, F_s, and the heat evolution factor, Z, as follows:

$$I_s = F_s Z$$

where:

I_s = flame spread index

F_s = flame spread factor, which is calculated using the times taken for the flame front to reach the 7.6-cm (3-in.) event marks on the specimen holder

Z = $\dfrac{CI}{B}$

C = a constant

I   = the compensated increase in stack temperature

B   = the calibration constant for the test apparatus

A.6 THERMAL ANALYSES

Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) tests were performed simultaneously on the same sample of material. The Mettler Thermoanalyzer was employed in both air and nitrogen environments with a heating rate of 10°C/min (18°F/min). Analytical results were obtained in the form of specimen weight remaining in milligrams versus temperature in °C (TGA) and total amount of energy given off during specimen decomposition in calories/gram (DTA).

A.7 PEEL STRENGTH

The peel strength was determined by peeling the 0.025-mm (0.001-in.) clear Tedlar (top film) from the ink/0.051-mm (0.002-in.) white Tedlar (substrate film). An Instron tensile testing machine that has a constant rate of crosshead movement was used. The grip separation rate was 5.1 cm/min (2.0 in./min). Eight specimens of each material were tested. Figure 11 shows the specimen configuration and test setup.

A.8 VISCOSITY

The ink was thoroughly mixed in a one-quart container and allowed to condition for 2 hrs without agitation at 25 ±1°C (77 ±1.8°F). The viscosity was determined at 25 ±1°C (77 ±1.8°F) using a Brookfield Viscosimeter, Model RVF. A reading was taken after equilibrating 3 min using a number six spindle at 20 rpm.

A.9 UNIFORMITY OF COATING

The uniformity of the coating was determined by measuring the coating thickness at several randomly selected locations. A micrometer that measured down to 0.025 mm (0.0001 in.) was used.

A.10 PRINTABILITY

Printability was determined by screenprinting two coats of the candidate ink system onto 0.051-mm (0.002-in.) white Tedlar, utilizing the apparatus shown in Figures 31, 32, and 33 and evaluating the following parameters:

● Curing time – condition of the ink film following cure for 90s at 71°C (160°F)

● Adhesion to polyvinyl fluoride film – determined by measuring the peel strength per Appendix A (sec. A.7)

● Dry time (both in-screen and as applied film) – determined by observing the apparent adhesion between successive ink coats and the condition of the second and final coat

● Silk screen mesh material and porosity – ease of screenprinting with production screen (viz., 156 strands/in. and polyester fabric)

● Uniformity of coating – determined by measuring the coating thickness variation per Appendix A (sec. A.9)

● Color shift – determined by observing yellowing, if any, of the coating following cure

In addition, viscosity of the ink system prior to screenprinting was determined per Appendix A (sec. A.8).

APPENDIX B
PROCEDURE FOR SPRAY COATING TEDLAR

B.1 EQUIPMENT

Spray gun .... Devilbiss

Model .... Type MBC

Air cap no. 30

Material nozzle .... AV 15 EX

B.2 PROCEDURE

The panels were sprayed at 45 psi in a slanted position. The hand spray technique was such that each pass overlapped the previous one by about 50 percent. Each pass deposited a coating that was about 0.005-in. thick. After coating, the panels were dried in an air-circulating oven for 1 hr at 32.2°C (90°F).

APPENDIX C
FPC461 (MANUFACTURER'S INFORMATION)

C.1 GENERAL

FPC461 is a copolymer of trifluorochloroethylene and vinyl chloride with excellent heat and light stability. In addition, FPC461 is soluble in conventional lacquer solvents, allowing it to be used as an air dry coating. It can also be used as a dry processing material; i.e., molding, extrusion, and calendaring.

C.2 TYPICAL POLYMER PROPERTIES

Typical polymer properties are as follows:

- Form   White powder

- Specific gravity   1.70

- Solution bulking   0.070 gal./solid lb

- Relative viscosity (1% in cyclohexanone)   1.50

C.3 FILM PROPERTIES

Film properties are as follows:

- Transparent   • Heat and light stable

- Air drying – thermoplastic   • Weather resistant

- Resistant to corrosive chemicals   • Flame resistant

C.4 TYPICAL SOLUTION CHARACTERISTICS

Toluene or xylene (25%)

Chlorinated hydrocarbons (25%)

Ketones (30%)

Esters (30%)

Aliphatic hydrocarbon tolerance – limited

Alcohol tolerance – limited

FPC461 is soluble in toluene or xylene as well as ketone and ester solvents. It is easily dissolved by sifting the resin into the solvent under agitation. A small portion of the solvent can be held for addition after "cutting" to ensure the development of high viscosity necessary for high shear during solvation. Mild heating and/or high shear agitation will reduce the mixing time. Agglomeration of resin particles must be prevented for minimum solvation time, and agitation must be continued until solvation is complete. "Lacquer"-type resins always show solubility and viscosity differences with variation in mixing (i.e., shear rate, time, temperature, etc.). The solvent system will determine the viscosities, processing characteristics, and drying schedules.

24

The time necessary for complete solution of the resin is usually diminshed by utilizing a high shearing action mixer and/or gentle heating of the solution. The aforementioned technique is desirable in the higher percent solid ranges.

C.5 MOISTURE VAPOR TRANSMISSION

Figure 34 is a graph showing moisture vapor transmission versus time for 25 percent FPC461 in MEK.

C.6 TYPICAL SOLUBILITY

The typical solubility data presented in Figures 35 and 36 and below were obtained from solutions prepared with minimum shear at room temperature to obtain maximum viscosities. The viscosities were measured using the Brookfield Viscometer, Model LVF.

Solution	Ratio (Resin/Solvent)	Viscosity, cp (6/60 rpm)
FPC461/MEK	45/55	5600/7380
FPC461/Acetone	45/55	4000/5340
FPC461/Acetone/Cyclohexanone	45/25/30	13 400/14 600
FPC461/Acetone/Toluene	45/45/10	5200/6320
FPC461/Acetone/Toluene/Methanol	45/25/25/5	7000/8120
FPC461/MEK/Toluene	45/45/10	7400/8940

Figure 37 shows the effect of heating a high solids FPC461 solution with respect to solution viscosity. The lowering of the solution viscosity with heat should enable the system to be "hot sprayed" or "curtain coated".

C.7 FPC461 COMPOUNDING STUDIES

C.7.1 PLASTICIZERS AND OTHER MODIFIERS

Figure 38 shows the effect of Paraplex (Rohm and Haas Company, Washington Square, Philadelphia, Pennsylvania) G-62, Saniticizer (Monsanto Chemical Company, Organic Chemicals Division, P.O. Box 478, St. Louis, Missouri) 409, and DOP upon an FPC461 solution based on 20 parts of FPC461 (by weight), 25 parts of xylene, 45 parts of MIBK, and 10 parts of cyclohexanone with respect to solution viscosity. Figure 39 shows the effect of the Acryloid (Rohm and Haas Company, Washington Square, Philadelphia, Pennsylvania) A-101 upon solution viscosity. Figure 40 shows the effect of various Elvacite (E.I. duPont DeNemours & Company, Inc., Electrochemicals Dept., Wilmington, Delaware) resins upon solution viscosity.

C.7.2 SOLID KETONE RESIN AND METHANOL

Table 28 illustrates the effects of various solid ketone (Mohawk Industries Inc., P.O. Box 187, 44 Station Road, Sparta, New Jersey) and methanol combinations upon an FPC461 system.

C.7.3 COLOR PIGMENTS

The effects of various pigments upon the rheological properties of various FPC461 solutions are shown in Table 29. All of the systems shown in this table are based on 20 percent total solids.

The chemical resistance of the pigments shown in Table 29 in FPC461 solutions and the viscosity stability of these systems have not been investigated. The hiding power of these formulations, as indicated by Morest Cards, appears to be satisfactory except for the white. Probably a slight increase in pigmentation would be required to obtain complete hiding. The cyclohexanone was incorporated into the formulation in order to achieve a slow drying film. Spraying and casting indicated that the quantities of cyclohexanone could be appreciably reduced and still obtain a long drying time.

C.8 SALT SPRAY -- WEATHER-O-METER EXPOSURE

Salt spray and Weather-O-Meter exposures have been completed for a series of coatings based upon FPC461-pigmented systems for the coil coating industry. The aging period was 1000 hrs for both tests.

Panels placed in the salt spray were given a 90 deg bend in the center with an X scribe on the lower half. After 1000 hrs, no significant change was noted. There was no undercutting of the film at the scribe marks. The Aluminum Association allows 1/16-in. maximum creep at the score line. (Color and Quality Standards, January 1965).

The Weather-O-Meter was more severe. Generally, all displayed a good appearance except for the usual water stains. No chalking was evident on any of the samples. All panels, except two, lost some gloss. Except for three panels, this loss in gloss was quite small. Some of the colors, however, showed appreciable color change. The blue, green, and yellow were the poorest. All changed to some extent either by fading or darkening. The Aluminum Association allows only slight chalking or color change in addition to the normal water staining.

Coatings were applied to solvent-cleaned, Bonderite 721 HS aluminum panels at a dry film thickness of 1.0 ± 0.1 mil. After a short air dry, they were baked in a forced-draft oven for 4 min at 460°F. The time taken to reach 460°F was 3 min; so the coating was actually subjected to the temperature for 1 min.

Formulations and test results are shown in Table 30.

C.9 PHYSICAL PROCESSING

FPC461 can be milled at approximately 250°F roll temperature. A stearate lubricant improves the processing characteristics.

FPC461 can be injection molded at 225°F with 10 000 psi. Compression moldings can be made at 180°F with 2000 psi.

Since FPC461 contains fluorine, caution should be exercised during heat processing to avoid inhalation of fumes.

C.10 FILM FORMATION

FPC461, by virtue of its solubility and solvent selection, can easily be formulated as a room temperature film former. Slightly elevated temperatures (i.e., 140°F) can be used to accelerate solvent evaporation. Relatively low baking temperatures are required to obtain unprimed metal adhesion.

26

C.11 ADHESION TO METALS

Air dried films of FPC461 do not have metal adhesion. However, successful air dry primers have been made for air dry FPC461 top coats as follows:

		Parts by Weight
Primer:	FPC461	30
	Neolyn 23 (Hercules Powder Co., Wilmington, Del.)	6
	Toluene	30
	MEK	17
	MIBK	17

<div align="center">or</div>

		Parts by Weight
Primer:	FPC461	30
	MR-74	5
	Xylene	25
	MIBK	45
	Cyclohexanone	10
Typical Topcoat:	FPC461	30
	MIBK	25
	Toluene	45

Acrylics such as Acryloid B-44 or B-72 (Rohm and Haas Company, Washington Square, Philadelphia, Pennsylvania) or Elvacite 2013 (E.I. duPont DeNemours & Company, Inc., Electrochemicals Dept., Wilmington, Delaware) when blended with FPC461 at about 3.5/1 (FPC461/acrylic ratio) will promote adhesion to metals.

Other additives may be blended with FPC461 to promote adhesion. Several examples are cited in Table 31. It was noted that the adhesion improves with time; i.e., early testing of samples may result in premature failure.

FPC461 coatings when baked at elevated temperatures do develop metal adhesion. The following study shows the effect of baking temperature upon FPC461 metal adhesion.

Formulation:	Parts by Weight
FPC461	30
MIBK	53
EAK	5
Xylene	39.5
Propylene Oxide	0.5
Paraplex G-62	5
Zopaque 88S	36
Dicalite L-5	12
Troy Anti-Float	1.2

PVC	39.5%
Total Solids	45.8%
60 deg Gloss	10

Baking time: 4 min (time for panel to reach 460°F was 3 min; therefore film was at 460°F for only 1 min).

Film thickness: 1 mil

Substrate: 721 HS bonderized aluminum

Bake Temperature	Adhesion No. 600 Scotch Tape over 100 squares (1/16 in.)	Knife Test
150°F	Excellent	Poor
175°F	Excellent	Poor-Fair
200°F	Excellent	Fair
225°F	Excellent	Fair
250°F	Excellent	Fair-Good
275°F	Excellent	Good
300°F	Excellent	Good-Excellent
325°F	Excellent	Excellent
350°F	Excellent	Excellent
375°F	Excellent	Excellent
400°F	Excellent	Excellent
460°F	Excellent	Excellent

The above data indicate that baking temperatures as low as 325°F will produce films with excellent adhesion.

C.12 RESISTANCE

FPC461 is extremely resistant to weather, heat, and light and will not yellow or decompose under continuous exposure to temperatures of 150°C.

C.13 ADDITIONAL DATA

Additional data on FPC461 are shown in tables 32, 33, 34 and as follows:

o   Oxygen permeability – similar to butyl rubber
o   Water absorption–after 24 hrs – 0.05% at 23°C

28

APPENDIX D
SYMBOLS AND ABBREVIATIONS

A	specimen surface area
AA	Aluminum Association
Aminco	American Instrument Company
ANPRM	advance notice of proposed rule making
ARC	Ames Research Center
ASTM	American Society for Testing and Materials
Ave.	avenue
B	calibration constant
BCAC	Boeing Commercial Airplane Company
Blvd.	boulevard
BMT	Boeing Materials Technology
Btu/ft^2/min	British thermal units per square foot per minute
Btu/ft^2/s	British thermal units per square foot per second
Btu/lb	British thermal units per pound
C	a constant
C_k	a constant
^{o}C	degrees Celsius
^{o}C/min	degrees Celsius per minute
cal/g	calories per gram
cfm	cubic feet per minute
cm	centimeter
cm^2	square centimeter
cm/min	centimeters per minute
cm^3/min	cubic centimeters per minute
cm^2/s	square centimeters per second
cm^3/s	cubic centimeters per second
Co.	company

CO	carbon monoxide
CO_2	carbon dioxide
conc	concentrated
Corp.	corporation
cp	centipoise
CR	contract report
D_m	maximum specific optical density
D_m/mil	maximum specific optical density per 0.001 inch
D_s	specific optical density
D_s/mil	specific optical density per 0.001 inch
$d(D_s)/dt$	derivative of D_s with respect to time
DEC-LAM	decorative laminate
deg	degree (not temperature)
Del.	Delaware
Dept.	department
dia	diameter
dil	dilute
dQ/dt	derivative of heat release with respect to time
dS/dt	derivative of smoke release with respect to time
dt	derivative of time
DTA	Differential Thermal Analysis
EAK	ethyl amyl ketone
e.g.	for example
etc.	and so forth
°F	degrees Fahrenheit
F_s	flame spread factor
FAA	Federal Aviation Administration
FAR	Federal Aviation Regulation
FEP	fluorinated ethylene propylene

fig.	figure
°F/min	degrees Fahrenheit per minute
FR	flame-retarded
ft	foot
ft^3	cubic foot
ft-lb	foot pound
ft^3/min	cubic feet per minute
gal.	gallon
gal./solid lb	gallons per solid pound
ΔH	enthalpy
H_t	total heat evolved
HCl	hydrogen chloride
HCN	hydrogen cyanide
HF	hydrogen fluoride
HNO_3	nitric acid
H_2O_2	hydrogen peroxide
hr	hour
HS	high strength
H_2SO_4	sulfuric acid
I	compensated increase in stack temperature
I_s	flame spread index
i.e.	that is
in.	inch
in.^{2}	square inch
Inc.	incorporated
in./min	inches per minute
in.^{3}/min	cubic inches per minute
J/cm^2	Joules per square centimeter
J/cm^2/s	Joules per square centimeter per second

JSC	Johnson Space Center
kg	kilogram
K.C.	Kansas City
kW/m²	kilowatts per square meter
l	liter
L	length
LOI	Limiting Oxygen Index
Ltd.	limited
m	meter
m³	cubic meter
MEK	methyl ethyl ketone
MeOH	methyl alcohol
MIAK	methyl isoamyl ketone
MIBK	methyl isobutyl ketone
mil	0.001 inch
min	minute
mm	millimeter
m³/min	cubic meters per minute
MR&D	Manufacturing Research and Development
MVT	moisture vapor transmission
N_2	nitrogen
NaOH	sodium hydroxide
NASA	National Aeronautics and Space Administration
NBS	National Bureau of Standards
NH_4OH	ammonium hydroxide
N/m²	Newtons per square meter
no.	number
NO_3	nitrate
NO_x	oxides of nitrogen

NPRM	notice of proposed rule making
O_2	oxygen
ohm/cm	ohms per centimeter
OSU	Ohio State University
oz	ounce
PA	Pennsylvania
phr	parts per hundred of resin by weight
Plt	percent light transmission
P.O.	Post Office
pp.	pages
ppm	parts per million
prepreg	preimpregnated with resin
psi	pounds per square inch
PVF	polyvinyl fluoride
PVC	polyvinyl chloride
Q	total heat release
ref.	reference
rpm	revolutions per minute
R.T.	room temperature
s	second
s^{-1}	per second
S	smoke
sec.	section
SO_2	sulfur dioxide
St.	Saint
STAR	Scientific and Technical Aerospace Reports
t	time
T_i	inlet temperature
T_m	time of maximum smoke

T_o	outlet temperature
temp	temperature
TGA	Thermogravimetric Analysis
THF	tetrahydrofuran
U.S.	United States
UV	ultraviolet
V	volume
V_{f1}	volumetric flow rate of O_2
V_{f2}	volumetric flow rate of N_2
viz.	namely
Vol.	volume
volts/mil	volts per 0.001 inch
W/cm^2	Watts per square centimeter
wt	weight
yr	year
Z	heat evolution factor
%	percent
$\pm$	plus or minus
&	and
=	equal
x	times or by
*	asterisk
o	degrees (temperature)
$\int_0^t$	integral from zero to time t

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Table 1.–Candidate Ink Vehicle Materials

No.	Method of Curing	Generic Chemical Type	Product Designation	Manufacturer
1	Thermal	Chlorofluorocarbon	FPC461	Hooker Chemicals
2	Thermal	Silicone	DC6-2230	Dow Corning
3	UV	Polyurethane	–	Midwest Coatings
4	Thermal	Brominated Polyester	USS 17046A	US Steel
5	Thermal	Phosphorylated Epoxy	–	Hughes Aircraft
6	Thermal	Acrylic[a]	KC4900	KC Coatings

[a]Baseline

Table 2.–Solvents in KC4900-30 Thinner

SC 150: a petroleum distillate
Cyclohexanone
Ethyl Ether Ethylene Glycol
Butyrolactone

Table 3.–Average Thickness of Coatings Included in Screening Evaluation

No.	Ink Coated on 0.051-mm (0.002-in.) Tedlar	Total Thickness		Thickness of Coating	
		mm	(in.)	mm	(in.)
1	KC4900 Acrylic	0.1727	(0.0068)	0.1219	(0.0048)
2	FPC461 Chlorofluorocarbon	0.1245	(0.0049)	0.0737	(0.0029)
3	DC6-2230 Silicone	0.1676	(0.0066)	0.1168	(0.0046)
4	Midwest UV Polyurethane	0.1016	(0.0040)	0.0508	(0.0020)

Table 4.–LOI of Screening Evaluation Candidate Inks Coated on Tedlar

No.	Ink Coated on Tedlar	LOI	Ranking (1 = Best)	Remarks
1	KC4900 Acrylic	28.0	2	Baseline, improvement over plain Tedlar
2	FPC461 Chlorofluorocarbon	30.3	1	Only ink better than baseline and plain Tedlar
3	DC6-2230 Silicone	24.3	3	Worse than baseline and plain Tedlar
4	Midwest UV Polyurethane	19.0	4	Grounds for elimination since less than standard atmospheric condition
5	Plain Tedlar	25.9	–	–

Table 5.–NBS Smoke Chamber – 2.5 W/cm² (132.2 Btu/ft²/min)

No.	Ink Coated on Tedlar	D_s (1.5 min)	D_s (4 min)	D_m	T_m (min)
1	KC4900 Acrylic	16.77	20.87	23.43	7.83
2	FPC461 Chlorofluorocarbon	1.53	4.23	11.43	15.81
3	DC6-2230 Silicone	3.8	6.13	9.0	13.47
4	Midwest UV Polyurethane	14.0	15.13	16.2	3.28

Table 6.–NBS Smoke Chamber – 2.5 W/cm² (132.2 Btu/ft²/min) – D_s Values After Dividing by Coating Thickness

No.	Ink Coated on Tedlar	D_s/mil (1.5 min)	D_s/mil (4 min)	D_m/mil	T_m (min)
1	KC4900 Acrylic	3.49	4.35	4.88	7.83
2	FPC461 Chlorofluorocarbon	0.53	1.46	3.94	15.81
3	DC6-2230 Silicone	0.83	1.33	1.96	13.47
4	Midwest UV Polyurethane	7.0	7.57	8.1	3.28

Table 7.–NBS Smoke Chamber – 5.0 W/cm² (264.3 Btu/ft²/min)

No.	Ink Coated on Tedlar	D_s (1.5 min)	D_s (4 min)	D_m	T_m (min)
1	KC4900 Acrylic	36.9	43.3	43.6	5.2
2	FPC461 Chlorofluorocarbon	19.4	27.4	36.7	8.8
3	DC6-2230 Silicone	20.1	27.6	35.7	9.4
4	Midwest UV Polyurethane	19.3	21.6	22.3	5.1

Table 8.–NBS Smoke Chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – D_s Values After Dividing by Coating Thickness

No.	Ink Coated on Tedlar	D_s/mil (1.5 min)	D_s/mil (4 min)	D_m/mil	T_m (min)
1	KC4900 Acrylic	7.7	9.0	9.1	5.2
2	FPC461 Chlorofluorocarbon	6.7	9.4	12.7	8.8
3	DC6-2230 Silicone	4.4	6.0	7.7	9.4
4	Midwest UV Polyurethane	9.7	10.8	11.2	5.1

Table 9.–NBS Smoke Chamber – 2.5 W/cm² (132.2 Btu/ft²/min) –
Toxicant Concentration (ppm) at 4 Minutes

No.	Ink Coated on Tedlar	HCN (ppm)	NOₓ (ppm)	CO (ppm)	CO₂ (ppm)	HCl (ppm)	HF (ppm)
1	KC4900 Acrylic	1	3	80	800	80	149
2	FPC461 Chlorofluorocarbon	0	1	67	767	50	116
3	DC6-2230 Silicone	0	1	80	767	25	143
4	Midwest UV Polyurethane	1	5	80	667	23	72

Table 10.–NBS Smoke Chamber – 5.0 W/cm² (264.3 Btu/ft²/min) –
Toxicant Concentration (ppm) at 4 Minutes

No.	Ink Coated on Tedlar	HCN (ppm)	NOₓ (ppm)	CO (ppm)	CO₂ (ppm)	HCl (ppm)	HF (ppm)
1	KC4900 Acrylic	1	5	113	833	200	79
2	FPC461 Chlorofluorocarbon	0	2	133	900	75	76
3	DC6-2230 Silicone	0	2	107	867	45	79
4	Midwest UV Polyurethane	0	6	100	800	35	69

*Table 11.–NBS Smoke Chamber – 2.5 W/cm² (132.2 Btu/ft²/min) –
Toxicant Concentration (ppm/mil of Coating) at 4 Minutes*

No.	Ink Coated on Tedlar	HCN (ppm)	NO$_x$ (ppm)	CO (ppm)	CO$_2$ (ppm)	HCl (ppm)	HF (ppm)	SO$_2$ (ppm)
1	KC4900 Acrylic	0.2	0.6	16.7	167.0	16.7	31.0	0
2	FPC461 Chlorofluorocarbon	0	0.3	23.1	264.5	17.2	40.0	0
3	DC6-2230 Silicone	0	0.2	17.4	166.7	5.4	31.1	0
4	Midwest UV Polyurethane	0.5	2.5	40.0	333.6	11.5	36.0	0

*Table 12.–NBS Smoke Chamber – 5.0 W/cm² (264.3 Btu/ft²/min) –
Toxicant Concentration (ppm/mil of Coating) at 4 Minutes*

No.	Ink Coated on Tedlar	HCN (ppm)	NO$_x$ (ppm)	CO (ppm)	CO$_2$ (ppm)	HCl (ppm)	HF (ppm)
1	KC4900 Acrylic	0.2	1.0	23.5	173.5	41.7	16.5
2	FPC461 Chlorofluorocarbon	0	0.7	45.9	310.3	25.9	26.2
3	DC6-2230 Silicone	0	0.4	23.3	188.5	9.8	17.2
4	Midwest UV Polyurethane	0	3.0	50.0	400.0	17.5	34.5

Table 13.–Performance Summary of Candidate Ink Vehicles in Screening Evaluation (1 = Best)

Ink Vehicle	LOI	Smoke	Toxicity	Coating Characteristics
FPC461 Chlorofluorocarbon	1	2	Acceptable	Good
Silicone	2[a]	1	Acceptable	Poor[a]
UV-cured Polyurethane	3[a]	3[a]	Acceptable	Good

[a]Performance worse than baseline KC4900 Acrylic

Table 14.–Optimization of Midwest Coatings' Chlorofluorocarbon Resin-Based Ink Formulation

No.	Formulation	LOI	Printability	Remarks
1	P1116	28.8	● Non-uniform coating thickness = 0.8 to 1.4 mil ● Fast drying on the screen ● Yellowing when embossed	Adjustments needed
2	P2028	30.0	● Uniform coating thickness = 0.8 to 1.0 mil	Optimization completed

Table 15.–Optimization of Seri-Print of Canada, Ltd., Chlorofluorocarbon Resin-Based Ink Formulation

No.	Formulation	LOI	Printability	Remarks
1	D1062	28.9	● Coating thickness = 0.6 to 0.8 mil ● Fast drying on the screen ● Yellowing when embossed	Adjustments needed
2	D1073	–	● Very low viscosity ● Could not be screenprinted	Requested new submission
3	D1089	30.2	● Uniform coating thickness = 0.7 mil	Optimization completed

Table 16.–TGA in Nitrogen of Screenprinting Inks: Midwest P2028 and KC4900

Heat Rate: 10°C/min (18°F/min)

Ink	Temp Range, °C (°F)	Wt Loss (%)
Midwest P2028	25-140 (77- 284)	3.5
	140-200 (284- 392)	12.8
	200-460 (392- 860)	56.8
	460-860 (860-1580)	19.6
	Total =	92.7
	Remaining char =	7.3
KC4900	25- 80 (77- 176)	0.8
	80-300 (176- 572)	67.7
	300-420 (572- 788)	5.8
	420-520 (788- 968)	11.7
	520-920 (968-1688)	6.1
	Total =	92.1
	Remaining char =	7.9

Table 17.–TGA in Air of Screenprinting Inks: Midwest P2028 and KC4900

Heat Rate: 10°C/min (18°F/min)

Ink	Temp Range, °C (°F)	Wt Loss (%)
Midwest P2028	25-170 (77- 338)	2.2
	170-250 (338- 482)	16.0
	250-300 (482- 572)	1.1
	300-460 (572- 860)	56.0
	460-760 (860-1400)	24.7
	Total =	100.0
KC4900	25-170 (77- 338)	5.4
	170-300 (338- 572)	63.8
	300-430 (572- 806)	4.7
	430-730 (806-1346)	26.1
	Total =	100.0

Table 18.–DTA in Nitrogen of Screenprinting Inks: Midwest
P2028 and KC4900

Heat Rate: 10°C/min (18°F/min)

Ink	Peak Starts °C(°F)	Peak Ends °C(°F)	Enthalpy, ΔH cal/g (Btu/lb)
Midwest P2028	140 (284) 300 (572)	230 (446) 460 (860)	4.3 (7.7) [Endothermic] -12.6 (-22.7) [Exothermic]
KC4900	120 (248)	300 (572)	32.0 (57.6) [Endothermic]

Table 19.–DTA in Air of Screenprinting Inks: Midwest
P2028 and KC4900

Heat Rate: 10°C/min (18°F/min)

Ink	Peak Starts °C(°F)	Peak Ends °C(°F)	Enthalpy, ΔH cal/g (Btu/lb)
Midwest P2028	170 (338) 320 (608) 450 (842) 560 (1040)	250 (482) 450 (842) 560 (1040) 760 (1400)	5.9 (10.6) [Endothermic] -14.3 (-25.7) [Exothermic] -28.3 (-50.9) [Exothermic] -53.2 (-95.8) [Exothermic]
KC4900	170 (338) 440 (824)	300 (572) 700 (1292)	30.7 (55.3) [Endothermic] -292.0 (-525.6) [Exothermic]

Table 20.–LOI of KC4900 Clear and Midwest P2028 Clear Inks Laminated Between
0.051-mm (0.002-in.) White and 0.025-mm (0.001-in.) Clear Polyvinyl Fluoride Films

Ink	LOI
KC4900 Clear	25.4
Midwest P2028	26.2

Table 21.–FAR 25-32 (12- and 60-s Vertical Ignition) Results for KC4900 Clear and Midwest P2028 Clear Inks Laminated Between 0.051-mm (0.002-in.) White Tedlar and 0.025-mm (0.001-in.) Clear Tedlar Films

Ink	Test Method	Extinguishing Time (s)	Burned Length cm (in.)	Drip Extinguishing Time (s)
KC4900	60-s vertical 12-s vertical	0.0 0.0	11.9 (4.7) 11.2 (4.4)	None None
Midwest P2028	60-s vertical 12-s vertical	0.0 0.0	7.6 (3.0) 8.1 (3.2)	None None

Table 22.–NBS Chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – Flaming Mode – Specific Optical Densities for KC4900 Clear and Midwest P2028 Clear Inks Laminated Between 0.051-mm (0.002-in.) White and 0.025-mm (0.001-in.) Clear Polyvinyl Fluoride Films

Ink	D_s (1.5 min)	D_s (4 min)	D_m	T_m (min)
KC4900 Clear	55.1	91.1	95.0	6.36
Midwest P2028 Clear	41.5	66.5	71.7	6.89

Table 23.–NBS Chamber – 5.0 W/cm² (264.3 Btu/ft²/min) – Flaming Mode – Toxicant Concentrations for KC4900 Clear and Midwest P2028 Clear Inks Laminated Between 0.051-mm (0.002-in.) White Tedlar and 0.025-mm (0.001-in.) Clear Tedlar Films

Ink	HCN (ppm)	NO_x (ppm)	CO (ppm)	HCl (ppm)	CO_2 (ppm)	HF (ppm)	SO_2 (ppm)
KC4900	2	4	260	100	2800	5[a]	0
Midwest P2028	2	3	280	105	2867	6[a]	0

[a]Too low, unrealistic

Table 24.–OSU Chamber Smoke Release Data – 5.0 W/cm² (264.3 Btu/ft²/min) for the Panel Containing Midwest P2028 Clear Ink (Average of Three Specimens)

Average Time, t (s)	Average dS/dt (particles/cm²/s)	Average d(D_s)/dt (s^{-1})	Average D_s	Average S (particles/cm²)
5	0.00	0.04	0.15	0.00
10	0.01	0.29	0.63	0.01
15	0.03	1.23	5.58	0.13
20	0.04	1.61	11.44	0.27
25	0.05	2.04	19.10	0.45
30	0.06	2.47	30.28	0.71
35	0.03	1.30	39.22	0.92
40	0.01	0.49	43.46	1.02
45	0.00	0.16	44.84	1.05
50	0.00	0.08	45.44	1.07
55	0.00	0.05	45.73	1.08
60	0.00	0.03	45.93	1.08
65	0.00	0.04	46.10	1.08
70	0.00	0.03	46.25	1.09
75	0.00	0.02	46.41	1.09

Table 25.–OSU Chamber Smoke Release Data – 5.0 W/cm² (264.3 Btu/ft²/min) for the Panel Containing KC4900 Clear Ink (Average of Three Specimens)

Average Time, t (s)	Average dS/dt (particles/cm²/s)	Average d(D_s)/dt (s^{-1})	Average D_s	Average S (particles/cm²)
5	0.00	0.08	0.37	0.01
10	0.00	0.07	0.76	0.02
15	0.02	1.06	3.35	0.08
20	0.05	2.24	12.91	0.30
25	0.06	2.45	24.50	0.58
30	0.07	3.12	37.95	0.89
35	0.05	2.03	51.13	1.20
40	0.02	0.86	58.07	1.37
45	0.01	0.32	60.76	1.43
50	0.00	0.11	61.68	1.45
55	0.00	0.06	62.09	1.46
60	0.00	0.08	62.43	1.47
65	0.00	0.08	62.84	1.48
70	0.00	0.08	63.24	1.49
75	0.00	0.03	63.61	1.50

Table 26.—OSU Chamber Heat Release Data – 5.0 W/cm² (264.3 Btu/ft²/min) for the Panel Containing KC4900 Clear Ink (Average of Three Specimens)

Average Time, t (s)	Average dQ/dt (J/cm²/s)	Average Q (J/cm²)
5	-0.09	- 1.91
10	0.13	- 3.02
15	1.99	2.04
20	4.24	18.02
25	5.63	43.85
30	7.82	77.04
35	7.69	117.21
40	6.09	152.84
45	4.92	179.46
50	3.26	199.87
55	2.57	214.50
60	2.17	225.85
65	1.89	234.69
70	0.97	241.13
75	0.17	246.78

Table 27.—OSU Chamber Heat Release Data – 5.0 W/cm² (264.3 Btu/ft²/min) for the Panel Containing Midwest P2028 Ink (Average of Three Specimens)

Average Time, t (s)	Average dQ/dt (J/cm²/s)	Average Q (J/cm²)
5	-0.13	- 1.36
10	0.68	- 1.09
15	2.74	9.18
20	4.40	26.10
25	6.72	55.15
30	7.93	92.46
35	6.79	130.47
40	4.78	159.81
45	3.69	180.20
50	2.26	194.93
55	1.95	205.89
60	1.85	214.48
65	1.34	221.66
70	1.08	227.94
75	0.16	234.29

Table 28.–Effects of Various Solid Ketone and Methanol Combinations Upon an FPC461 System

	Parts by Weight				
					Control
FPC461	20	20	20	20	20
MR-74	5		5		
MR-85		5		5	
Xylene	25	25	15	15	25
MIBK	45	45	45	45	45
Cyclohexanone	10	10	10	10	10
MeOH			10	10	
Viscosity, cp at 23°C Initial Brookfield 6/60 rpm	150/150	160/139	120/114	110/113	150/144
Solution Clarity	Clear	Clear[a]	Clear	Clear[a]	Clear

[a]Clear solutions contain fine suspended material, indicating the possibility of the solubility of MR-85 being exceeded. The MeOH appears to be compatible in the above formulations.

Table 29.–Effects of Various Pigments Upon the Rheological Properties of Various FPC461 Solutions

	Parts by Weight				
FPC461	20	20	20	20	20
Alcoa 209		16			
Titanox RA-50			20		13
Philblack A (Phillips Petroleum Co.)				5	
Fastolux Blue 48T-13 (Ansbacher-Siegle)					5
Xylol	25	43	50	31	47
MIBK	30	53	59	37	56
Cyclohexanone	25	46	51	32	48
Viscosity, cp at 23°C Initial Brookfield 6/60 rpm	120/116	96/84	64/65	160/165	1240/382

Table 30.–Accelerated Agings – TS-1190

TS-1190 No. / AA Color No.		41 (White) AA11003	41B (White) AA11003	41D (White) AA11003	41E (White) AA11003	64 (White) AA11003	65 (Blue) AA61003	66 (Green) AA51002	67 (Red) AA71003	68 (Pink) AA71002	69 (Gray) AA21002	70 (Yellow) AA31002	71 (Beige) AA41002
FPC461		30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0
MIBK		38.0	38.0	38.0	38.0	53.0	53.0	38.0	38.0	38.0	38.0	38.0	38.0
EAK		20.0	20.0	20.0	20.0	5.0	5.0	20.0	20.0	20.0	20.0	20.0	20.0
Xylene		39.5	39.5	39.5	39.5	39.5	39.5	39.5	39.5	39.5	39.5	39.5	39.5
Propylene Oxide		0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Paraplex G-62	Parts by Weight					5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
TiPure R-610		18.5											
TiPure R-992													
TiPure R-902			32.0	32.0									
Zopaque 88S					32.0	36.0	36.0	27.0	10.0	43.0	28.0	28.0	14.0
Dicalite L-5						12.0	9.0	8.0	4.0	4.0	1.0	6.5	10.0
Troy Anti-Float						1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Shephard Blue No. 9							6.5	0.42					
Shephard Black No. 1							0.28	14.0	1.1	0.8	3.0	0.3	0.3
Shephard Green No. 5								1.74					
Shephard Green No. 4								5.2					
Shephard Yellow No. 22											3.0	22.8	
C. K. Williams R.O. 8097									26.4				
Mapico Red 567									15.5		0.3		
Shephard Red No. 63										4.1			
Shephard Yellow No. 28										12.5			5.0
Shephard Gray No. 19											29.7		
Shephard Golden Yellow No.55												3.04	
Shephard Brown No. 8													16.0
Weather-O-Meter (1000 hrs)													
Appearance		Excellent	Heavy yellow stain	Excellent	Excellent	Yellow salt spots	Faded	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
Chalking		None	None	None	None	None	None	None	None	None	None	None	None
Change in 60 deg gloss		-33	+4	-3	-34	-2	-2	-1	-1	+1	-2	-13	-5
Color change				Very slight yellow color				Darker	Slightly darker	Slightly darker	More yellow	Much lighter	Slightly darker
Salt Spray (5% and 1000 hrs)													
Appearance		Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
Adhesion		Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
Blisters		None	None	None	None	None	None	None	None	None	None	None	None

Note: These coatings are in excellent condition except for some color fade after three years' exposure at 45 deg south at Pottstown, Pennsylvania.

49

Table 31.–Metal Adhesion of FPC461 Blended With Various Additives

FPC461		25	25	25	25
Lubrizol CA-21[a]		2	2		
Silane Z-6040[b]		5			
Silane A-1100[b]	Parts			2.5	1.0
ERL 4221[b]	by			2.5	1.0
DER-331[c]	Weight				
MEK		55	55	55	55
Toluene		70	70	70	70
Antifoam Q[c]		0.03	0.03	0.03	0.03
Knife Adhesion (Air dry)		Good to excellent	Good to excellent	Excellent	Good
Film Color		Slightly blue	Slightly yellow	No color	No color

[a]The Lubrizol Corp., Box 3057, Cleveland, Ohio
[b]Union Carbide Corp., 270 Park Avenue, New York, New York
[c]Dow Corning Co., Midland, Michigan

Table 32.–Chemical Exposures (Molded Samples) – 240 Hours at Room Temperature

Material	Effect
NH$_4$OH (conc & dil)	No effect
NaOH (20% and 40%)	No effect
H$_2$O$_2$ (90%)	Gas penetration – no degradation
Red Fuming HNO$_3$	
20% excess NO$_3$	Slight softening
7% excess NO$_7$	No effect
Chlorosulfonic Acid	Slight discoloration
Moist bleaching powder	No effect
Chromic Acid (70% H$_2$SO$_4$)	No effect
HCl (conc & dil)	Gas penetration – no degradation
Hydrofluoric Acid (48%)	No effect
Aqua Regia	No effect
Acetic Acid (glacial)	Partial solubility
Sulfuric Acid (battery acid)	No effect
Sulfuric Acid (conc)	No effect
Water	No effect
Aniline	Softened
Carbitol	No effect
Ethyl Alcohol (95%)	No effect
Methyl Alcohol	No effect
Ethylene Glycol	No effect
Mesityl Oxide	Soluble
Heptane	Softened
V.M.P. Naphtha	Soluble
Hi-Flash Naphtha	Partial solubility
Butyl Mercaptan	Soluble
Fuel Oil	Slight softening
Gasoline	Soluble
Hydraulic Fluid	No effect

Table 33.– Properties of Molded Material

Tensile Strength	4500 to 5500 psi		
Elongation	10% to 50%		
Flexural Modulus	200 000 to 300 000 psi at R.T.		
Impact Strength (Izod)	0.5 to 1.0 ft-lbs		
Rockwell Hardness	R75 to R85		
Shore Hardness	D69 to D75		
Folding Endurance	170 to 180 flexes (0.016 in. thick)		
	60 Cycles	1000 Cycles	1 000 000 Cycles
Dielectric Constant (24°C)	2.84	2.82	2.4
Power Factor (24°C)	0.02	0.01	0.01
Loss Factor (24°C)	0.05	0.03	0.02
DC Resistivity (70°C)	5 x 10^{14} ohm/cm		
Dielectric Strength (24°C)	860 volts/mil		

Table 34.– Outdoor Studies – FPC461

Parts by Weight

TS-667 No.	2	3	4	6	107	103	109
FPC461	20	20	20	20	20	20	20
MIBK	53	59	52	56	30	42.5	42.5
Cyclohexanone	46	51	17	49	20	20	20
Xylene	45	50	31	47	29	41.5	41.5
Propylene Oxide					1	1	1
Titanox RA-50		20		13			
Titanox RANC					20	10	10
Philblack A			5				
Alcoa 209 Paste	16						
Phthalo Blue (Holland-Suco F-4500)				5		10	
Phthalo Green (Cyanamid 15-3100)							10
Thermolite 31					0.2	0.2	0.2
Exposed 45 deg south Pottstown, PA (yrs)	6	6	6	6	5	5	5
Appearance	Excellent, slightly darker	Excellent, bleached	Excellent, no change	Excellent, faded	Excellent, slightly dirty	Excellent, some fading	Excellent, some darkening
Chalking (ASTM D659-44)	9	2	8	8	9	8	8
Peeling or Flaking	None	None	None	None	None	None	None

Parts by Weight

TS-667 No.	110	111	112	113	114	116
FPC461	20	20	20	20	20	20
MIBK	30	30	30	30	30	30
Cyclohexanone	20	20	20	20	20	20
Xylene	29	29	29	29	29	29
Propylene Oxide	1	1	1	1	1	1
Alcoa Al. 109	20					
Philblack A		0.8			0.43	
Titanox RANC			15	15	13	20
Phthalo Blue (Holland-Suco F-4500)			5			
Phthalo Green (Cyanamid 15-3100)				5		
Mohawk MR-74						5
Thermolite 31	0.2	0.2	0.2	0.2	0.2	0.2
Exposed 45 deg south Pottstown, PA (yrs)	5	5	5	5	5	5
Appearance	Excellent, slightly darker	Excellent, no change	Excellent, darker	Excellent, darker	Excellent, darker	Excellent, slightly dirty
Chalking (ASTM D659-44)	9	9	9	8	9	9
Peeling or Flaking	None	None	None	None	None	None

Table 34.–(Concluded)

Parts by Weight

TS-667 No.	117	118	119	121	122	123
FPC461	20	20	20	20	20	20
MIBK	42.5	42.5	42.5	42.5	42.5	42.5
Cyclohexanone	20	20	20	20	20	20
Xylene	41.5	41.5	41.5	41.5	41.5	41.5
Propylene Oxide	1	1	1	1	1	1
Mohawk MR-74	5	5	5	5	5	5
Titanox RANC	10	10		15	15	13
Alcoa Al. 209			20			
Phthalo Blue (Holland-Suco F-4500)	10			5		
Phthalo Green (Cyanamid 15-3100)		10			5	
Philblack A						0.43
Thermolite 31	0.2	0.2	0.2	0.2	0.2	0.2
Exposed 45 deg south Pottstown, PA (yrs)	5	5	5	5	5	5
Appearance	Excellent, darker	Excellent, darker	Excellent, darker	Excellent, darker	Excellent, darker	Excellent, darker
Chalking (ASTM D659-41)	8	8	9	8	8	9
Peeling or Flaking	None	None	None	None	None	Slight

Note: Formulations TS-667-2 to 6 sprayed on aluminum Q panels. TS-667-107 to 123 sprayed on aluminum and steel Q panels. All were exposed at 45 deg south at Pottstown, Pennsylvania. Exposures are still in progress. Panels were air dried at room temperature.

53
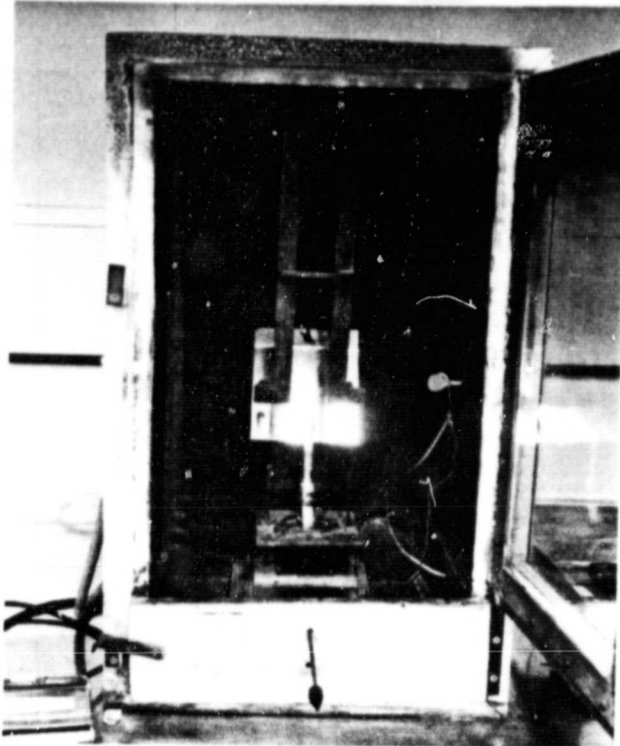
Figure 1.–Vertical Burn Test Chamber, FAR 25-32 Type
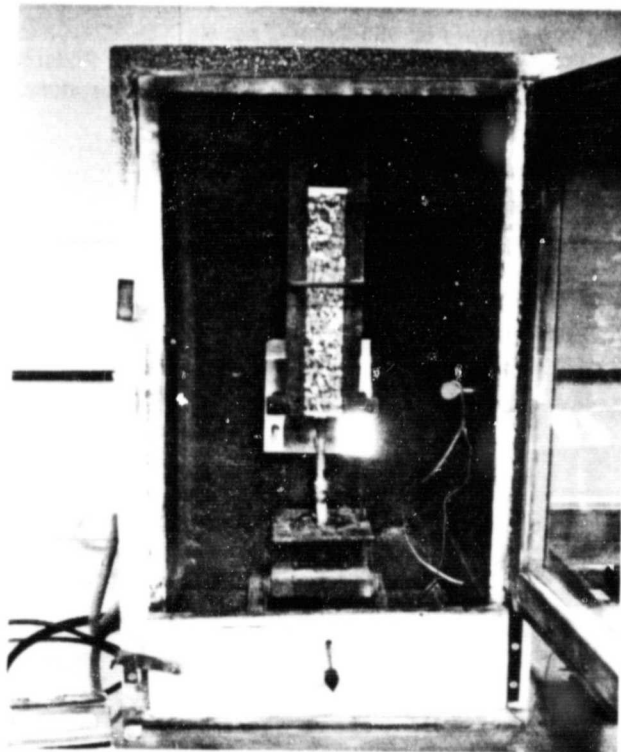
Figure 2.–Vertical Burn Test Chamber Showing Specimen and Burner Flame Positioning
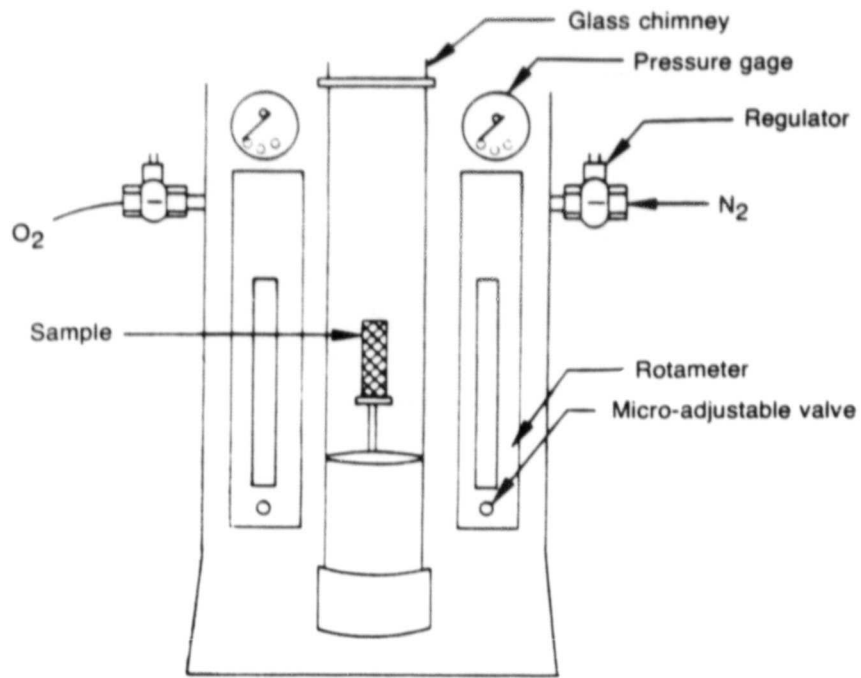
Figure 3.–Schematic of LOI Test Apparatus
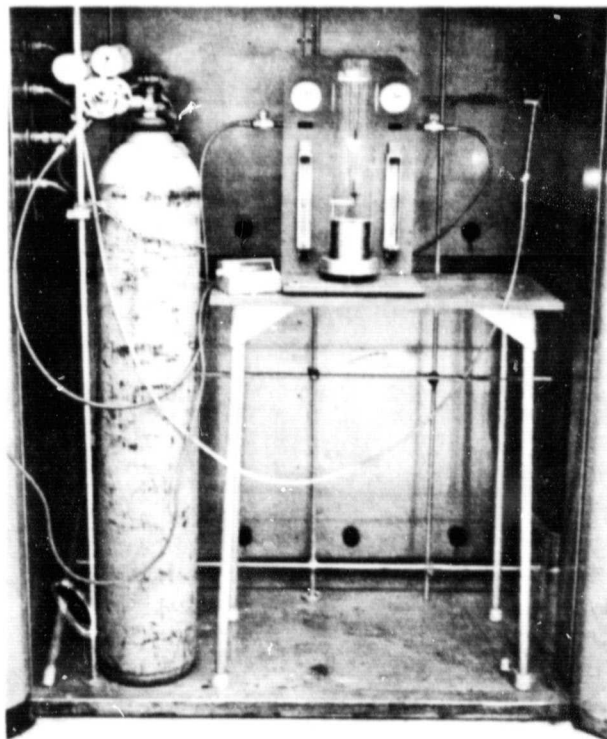
Figure 4.–LOI Test Apparatus
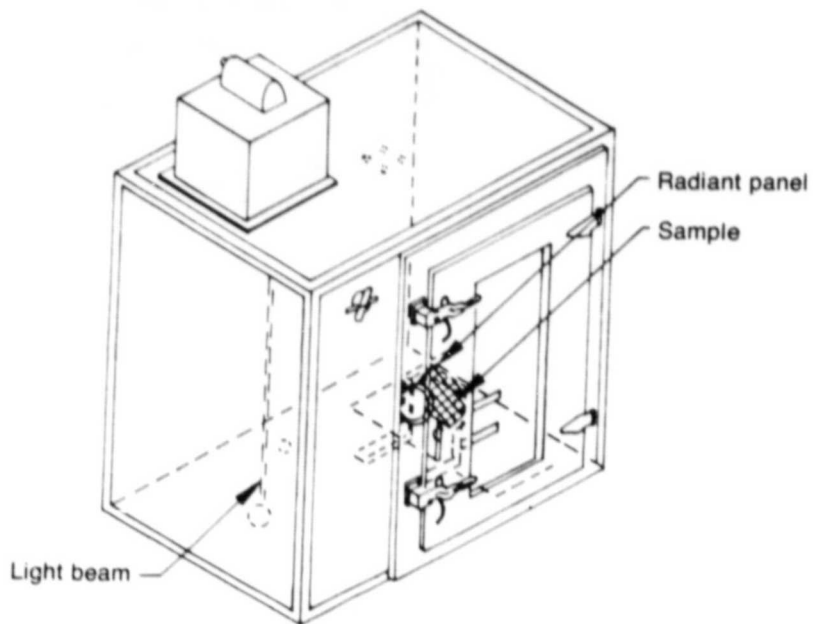
Figure 5.–Schematic of NBS Smoke Chamber
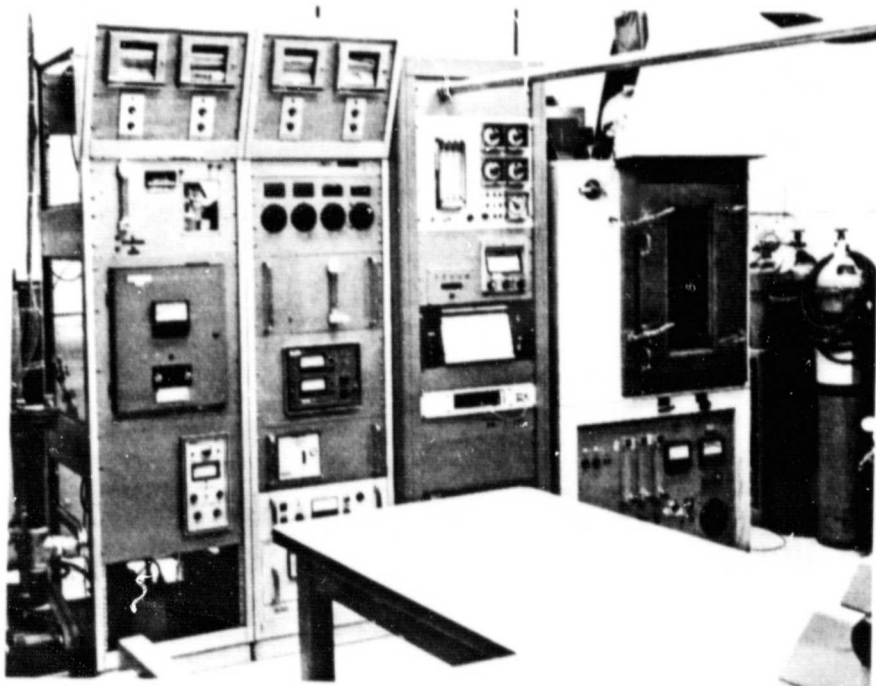
Figure 6.–Aminco–NBS Smoke Chamber
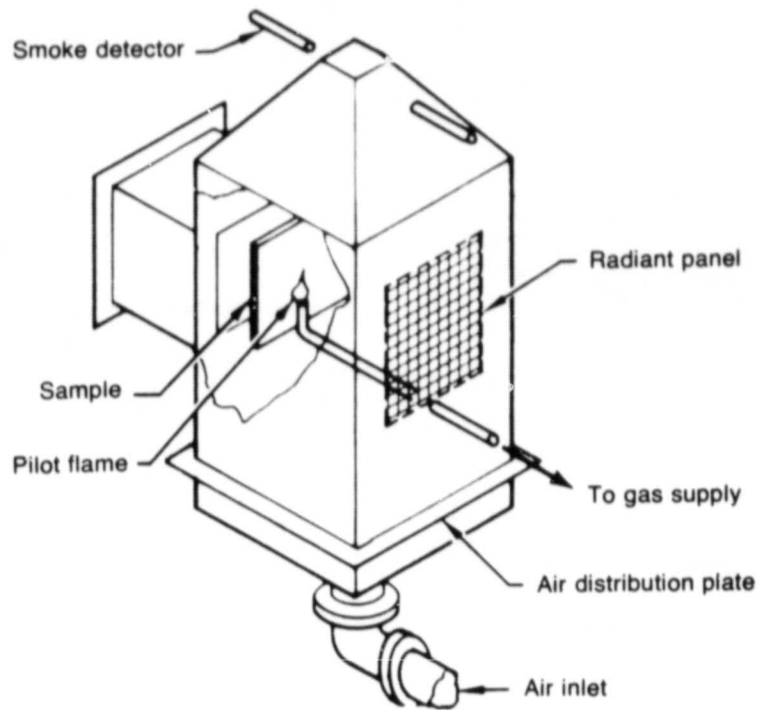
Figure 7.–Schematic of OSU Release Rate Apparatus
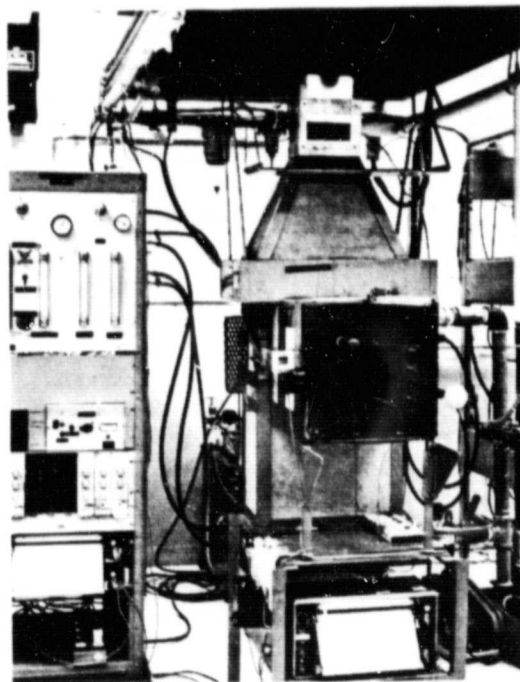
Figure 8.–OSU Release Rate Apparatus

57
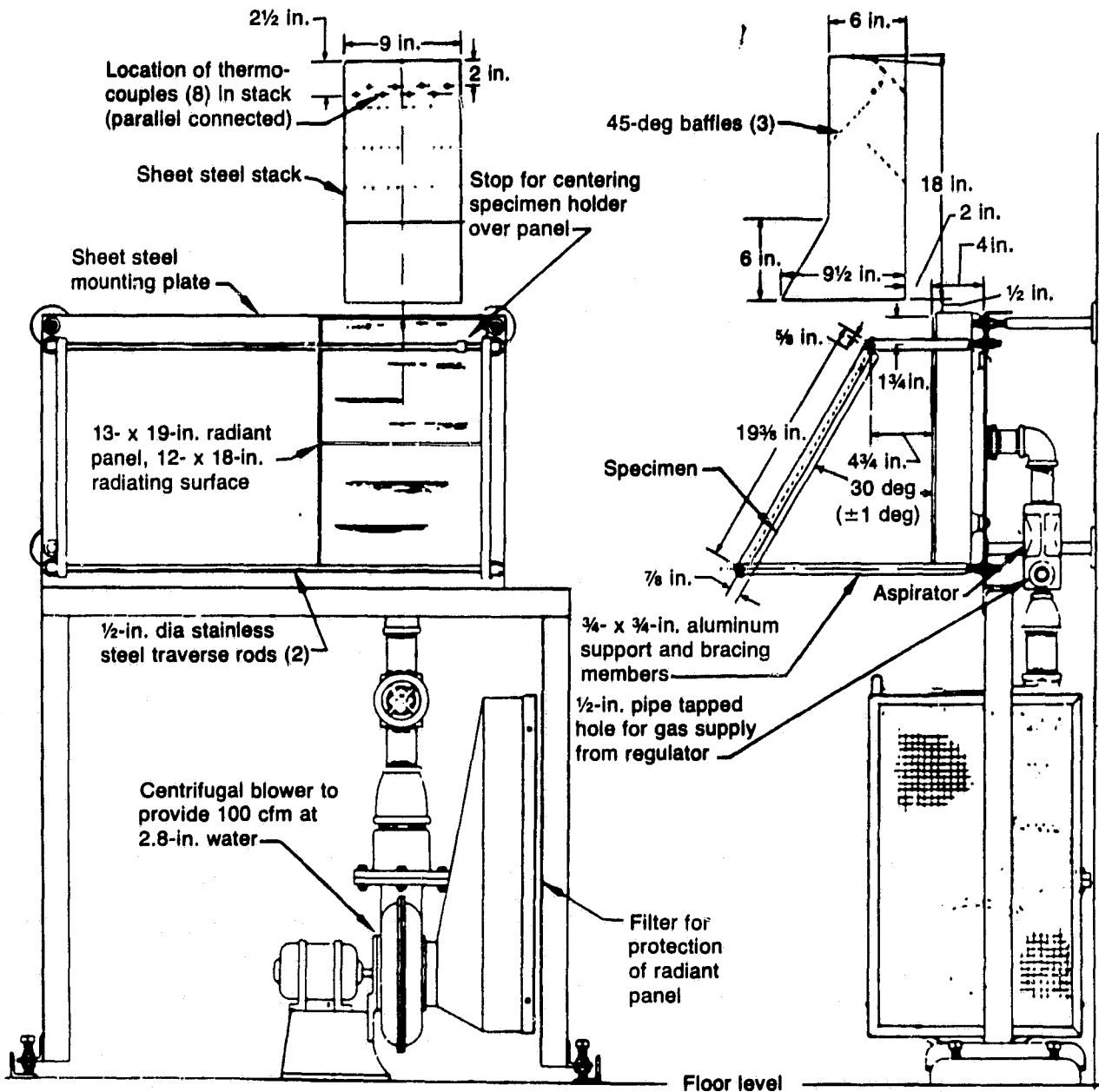
Figure 9.–Schematic of Flame Spread Apparatus

Figure 10.–Flame Spread Apparatus
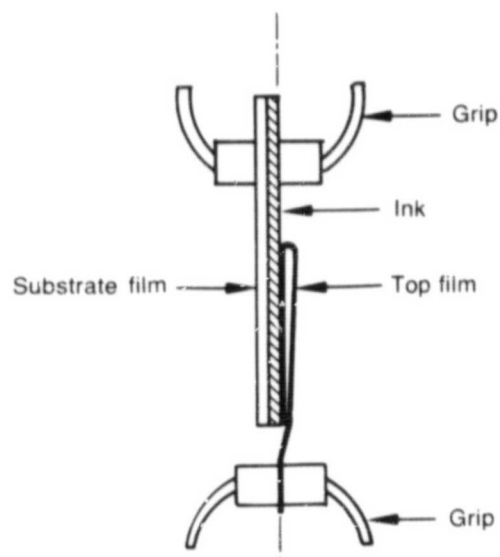
Figure 11.–Peel Strength Specimen Configuration and Test Setup

**POLYVINYL CHLORIDE (PVC) –
EPOXY SANDWICH PANEL**

0.025-mm (0.001-in.) clear PVF

0.254-mm (0.010-in.) printed PVC

0.381-mm (0.015-in.)
epoxy-fiberglass skins

Phenolic-polyamide
honeycomb core

Precured sandwich
panel blank

Secondary bonding
(PVF-PVC) to sandwich

Prelaminated and textured PVF-PVC

POLYVINYL FLUORIDE (PVF) – EPOXY SANDWICH

0.025-mm (0.001-in.) clear PVF

0.051-mm (0.002-in.) printed white PVF

0.254-mm (0.010-in.) epoxy-fiberglass skin

0.127-mm (0.005-in.) epoxy-
fiberglass bond ply

Phenolic-polyamide
honeycomb core

0.127-mm
(0.005-in.)
epoxy-fiberglass
skin – 2 plies

Secondary sandwich bond

Prelaminated and textured

Figure 12.–Baseline Sandwich Panels
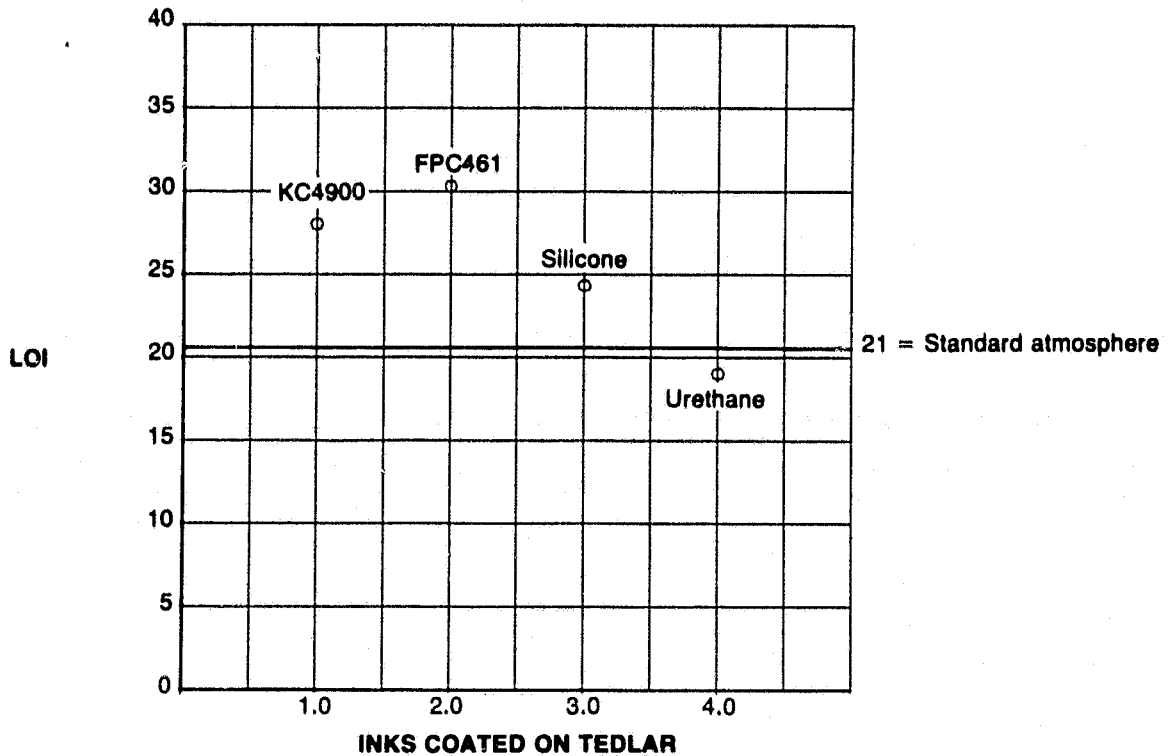
Figure 13.–LOI of KC4900, FPC461, Silicone, and Urethane Inks Coated on Tedlar
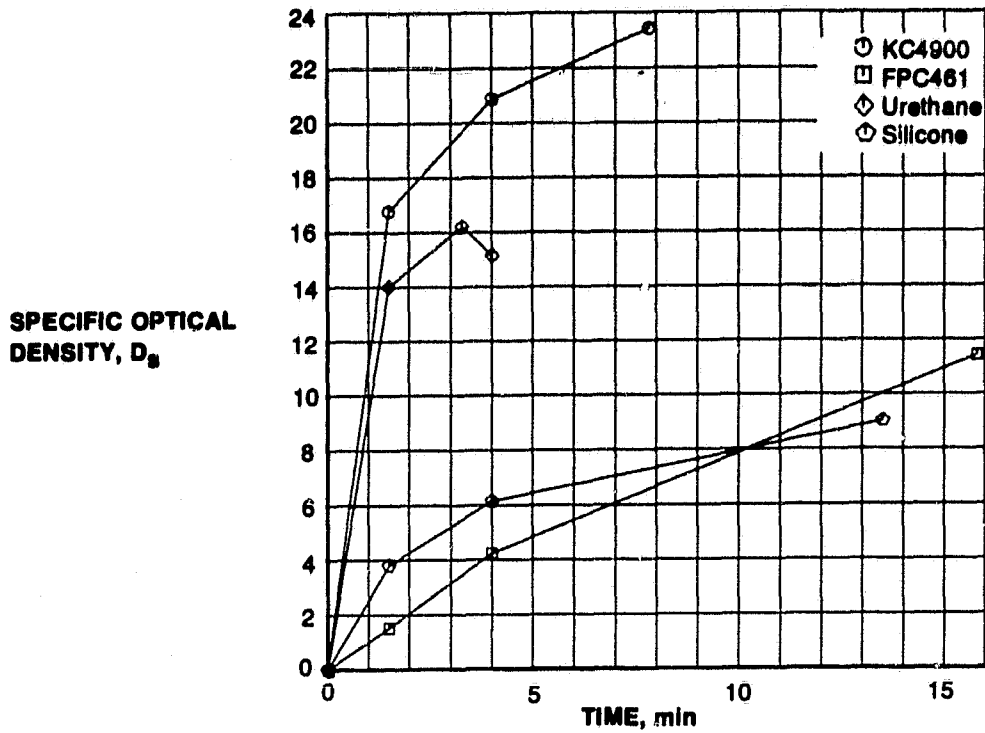
Figure 14.–Specific Optical Density of KC4900, FPC461, Silicone, and UV Urethane – 2.5 W/cm² (132.2 Btu/ft²/min)
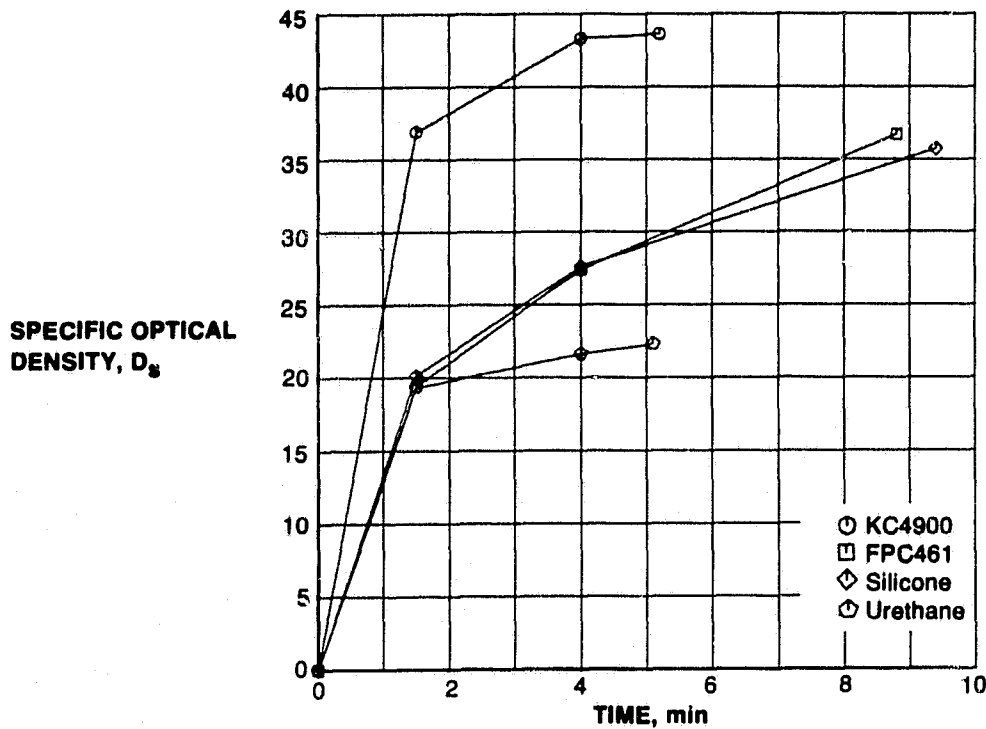
Figure 15.–Specific Optical Density of KC4900, FPC461, Silicone, and UV Urethane – 5.0 W/cm² (264.3 Btu/ft²/min)

61
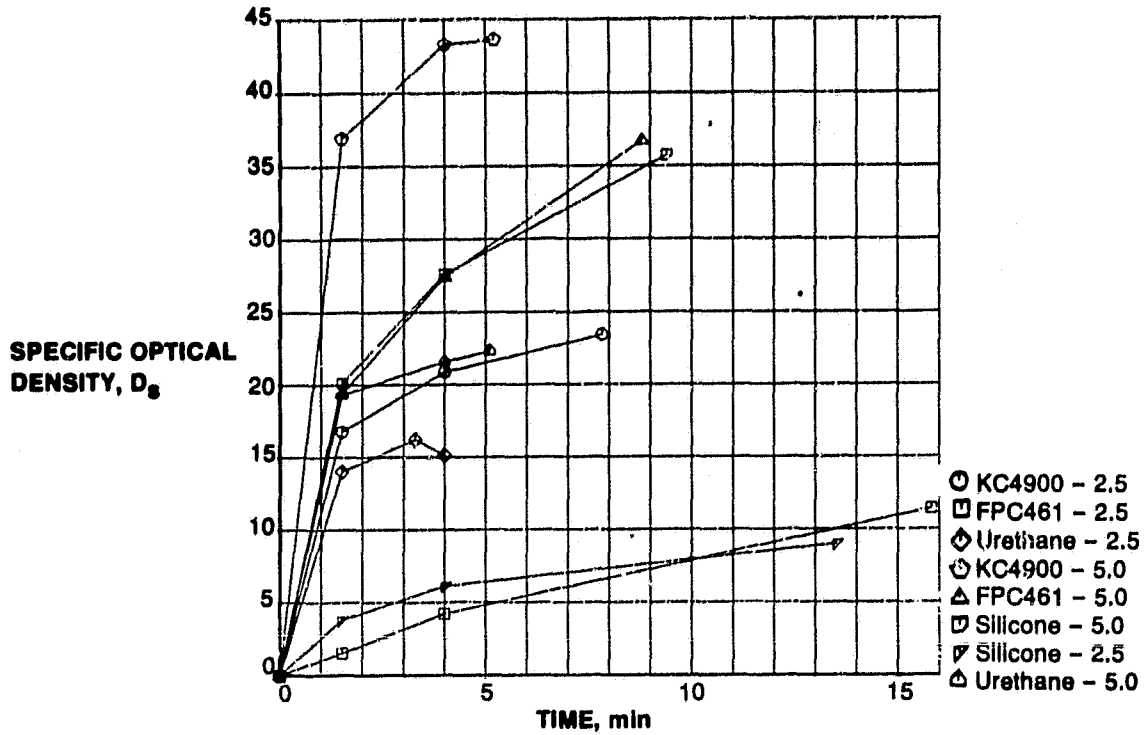
Figure 16.–Specific Optical Density of KC4900, FPC461, Silicone, and UV Urethane – 2.5 W/cm² (132.2 Btu/ft²/min) and 5.0 W/cm² (264.3 Btu/ft²/min)
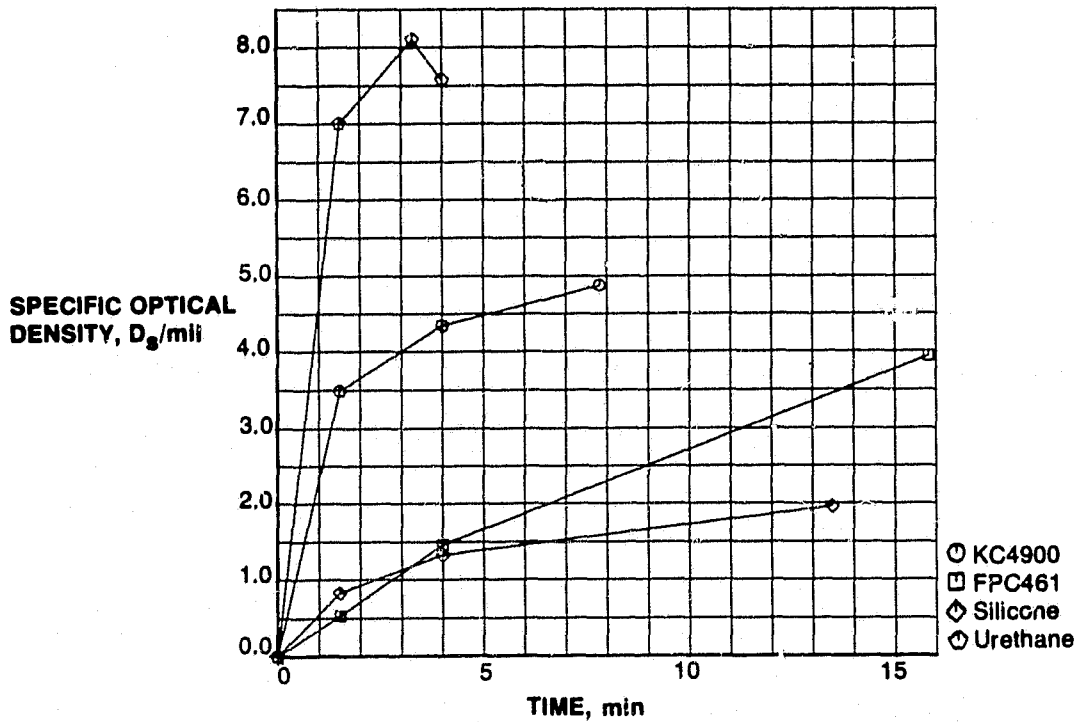
Figure 17.–NBS Smoke – 2.5 W/cm² (132.2 Btu/ft²/min)–D_S per mil of Coating

62
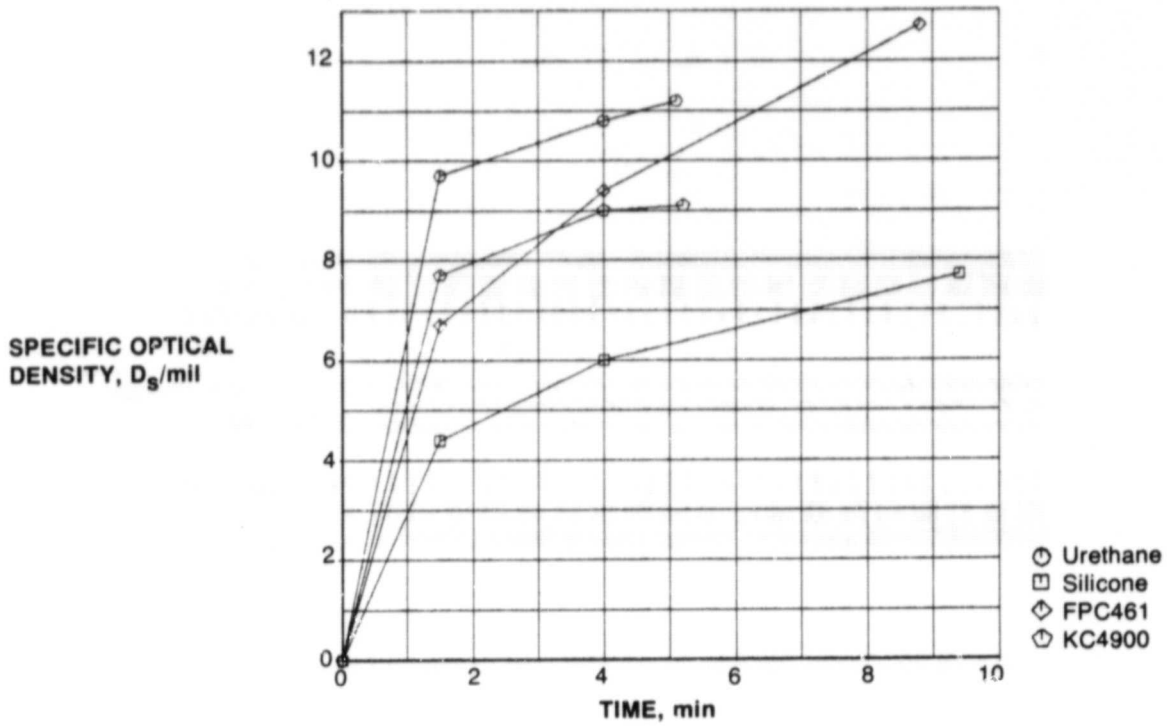
Figure 18.–NBS Smoke – 5.0 W/cm² (264.3 Btu/ft²/min)–D_S per mil of Coating
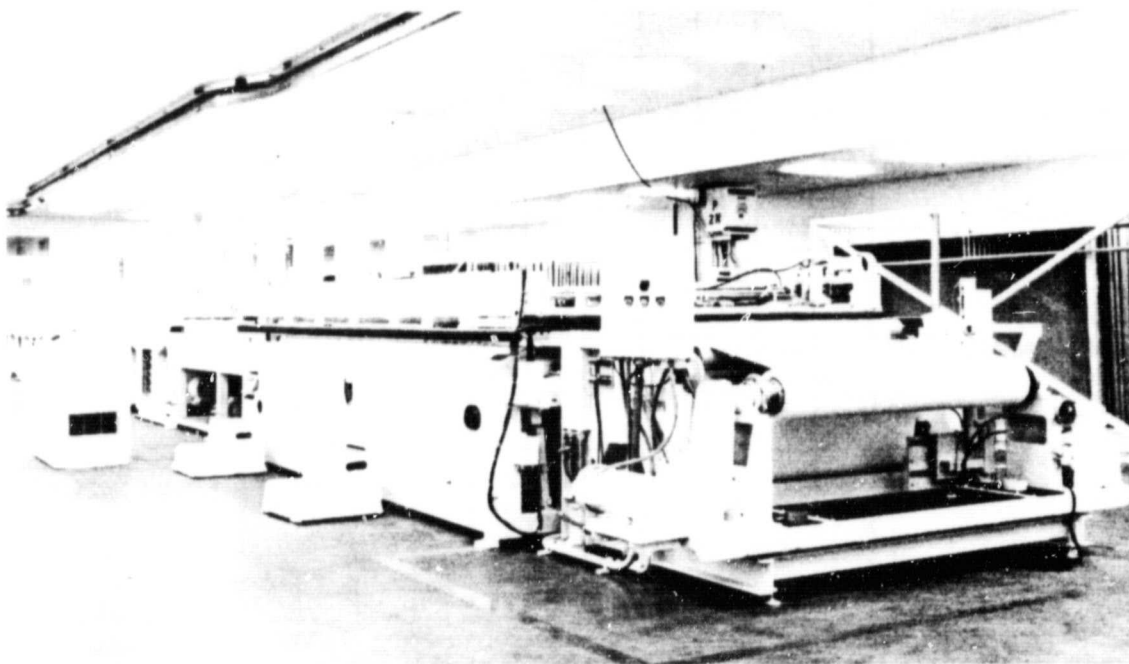
Figure 19.–Production Screenprinter and Dryer

63
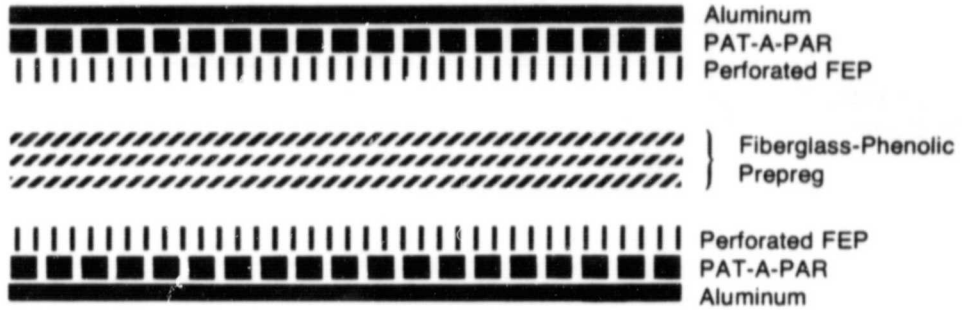
Figure 20.–Three-Ply Fiberglass-Phenolic Laminate Layup
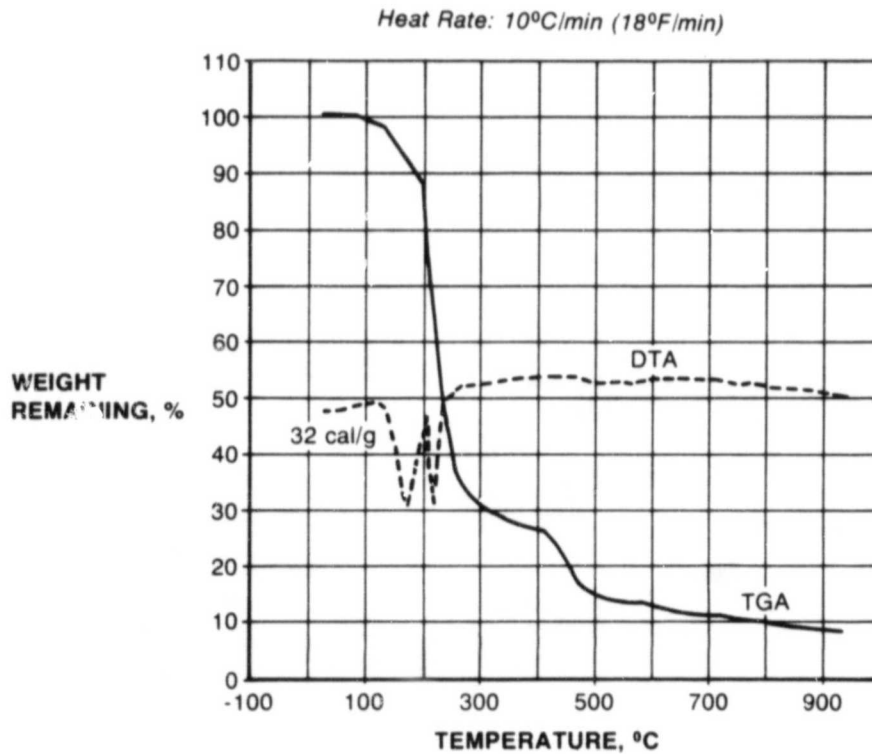
Figure 21.–TGA/DTA of KC4900 in Nitrogen

Heat Rate: 10°C/min (18°F/min)
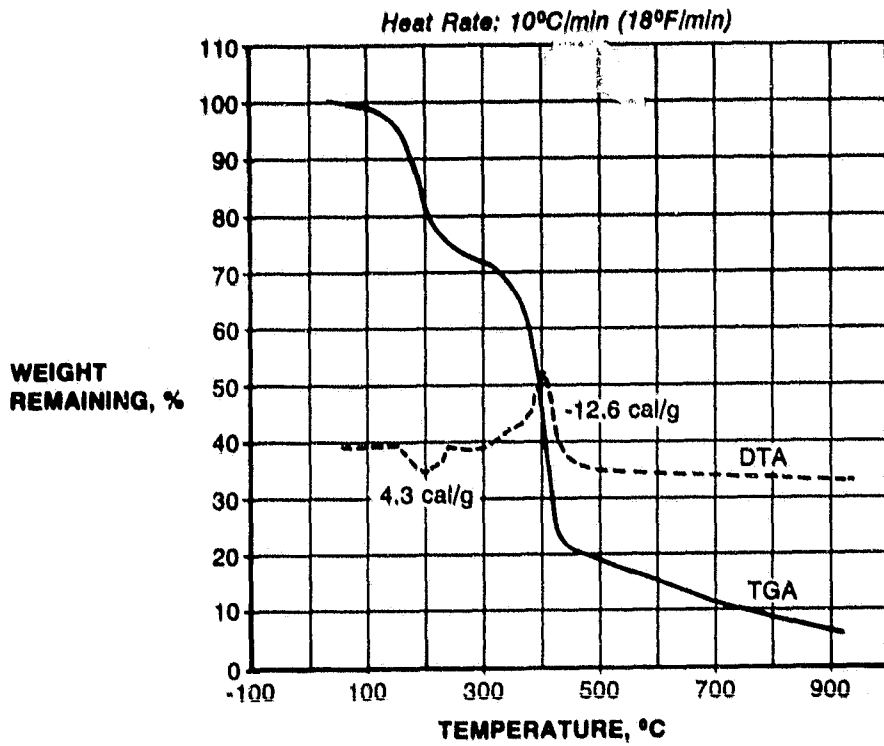
Figure 22.–TGA/DTA of Midwest P2028 in Nitrogen

Heat Rate: 10°C/min (18°F/min)
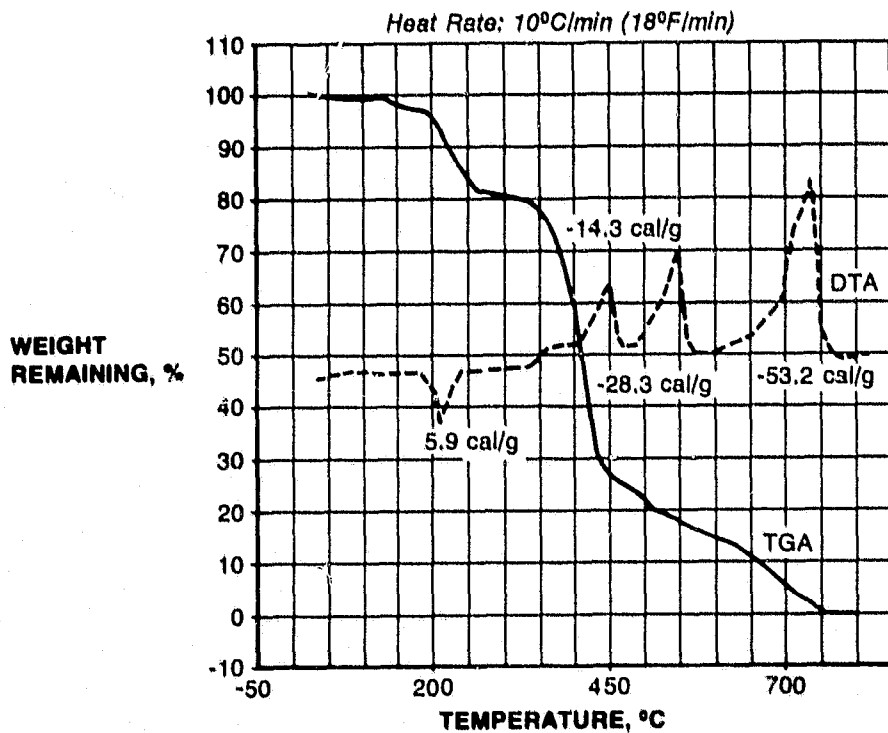
Figure 23.–TGA/DTA of Midwest P2028 in Air
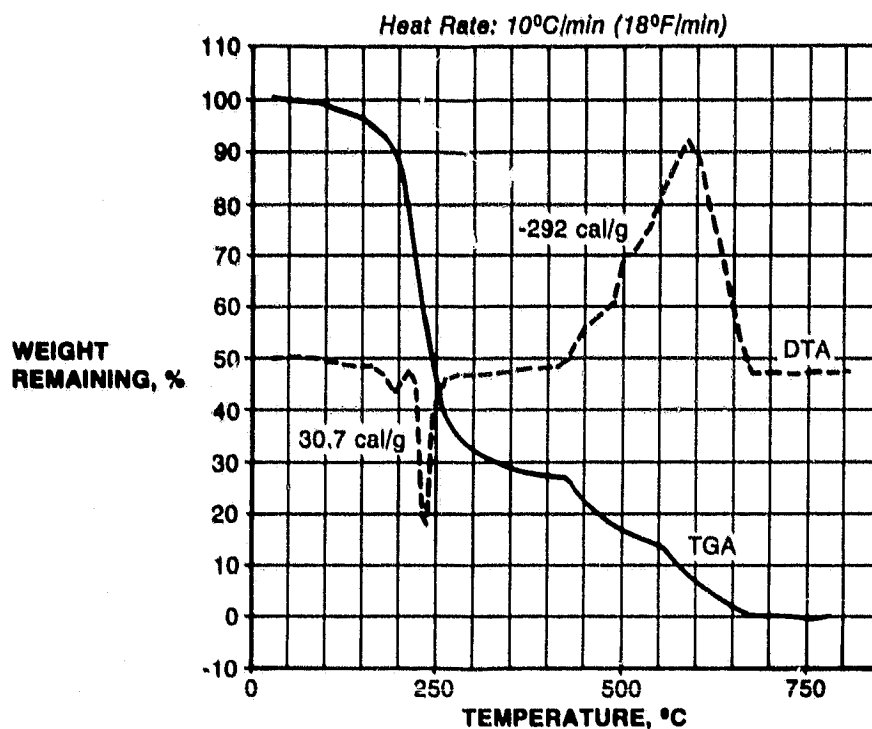
Figure 24.–TGA/DTA of KC4900 in Air
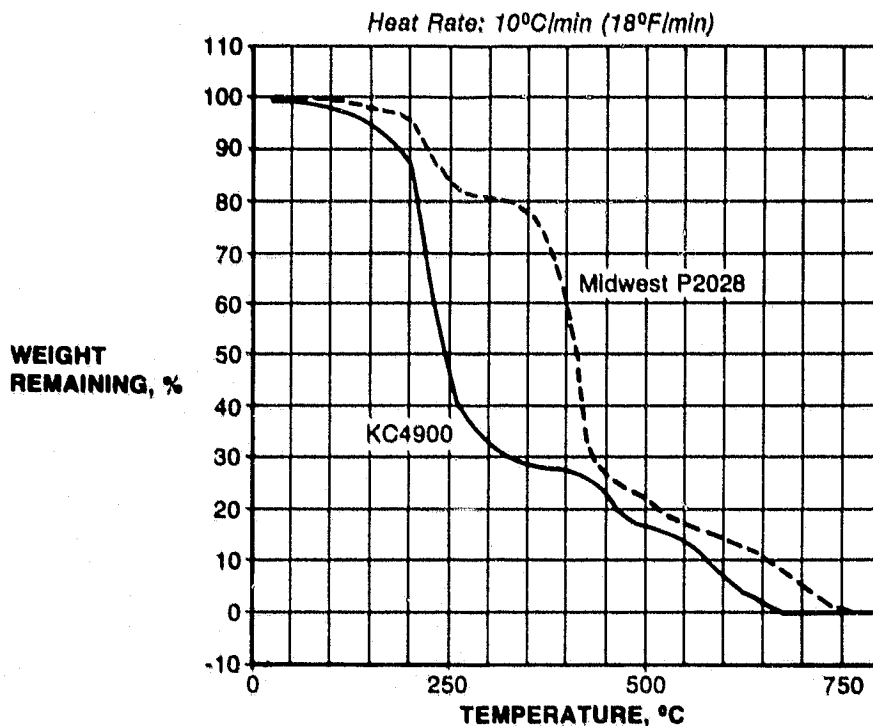
Figure 25.–TGA Comparison of Midwest P2028 and KC4900 in Air

66
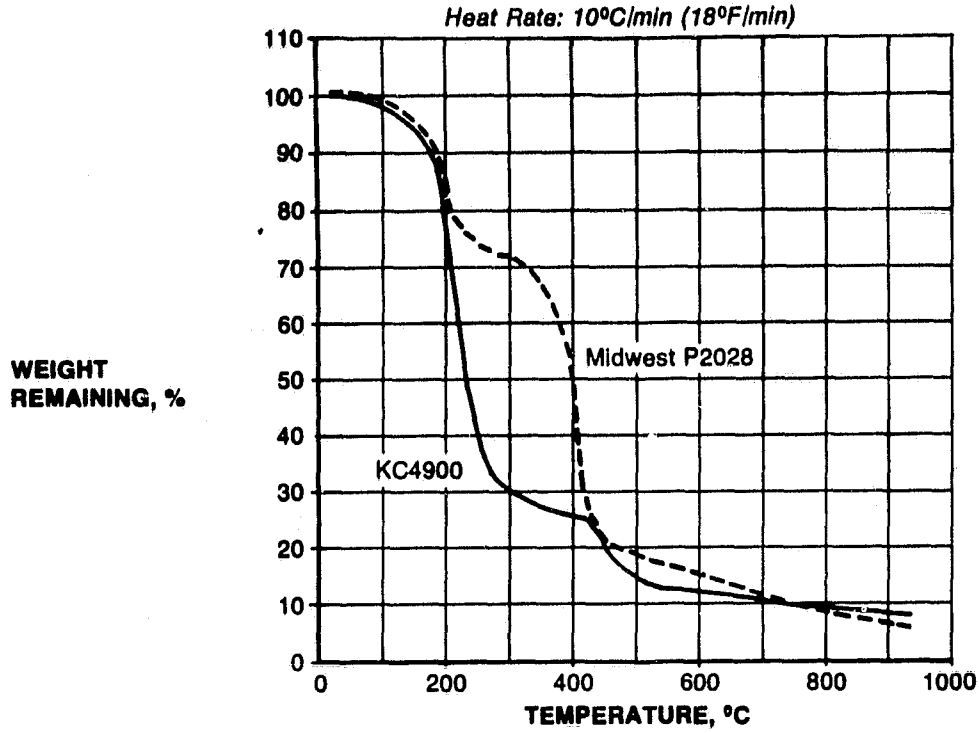
Figure 26.–TGA Comparison of Midwest P2028 and KC4900 in Nitrogen
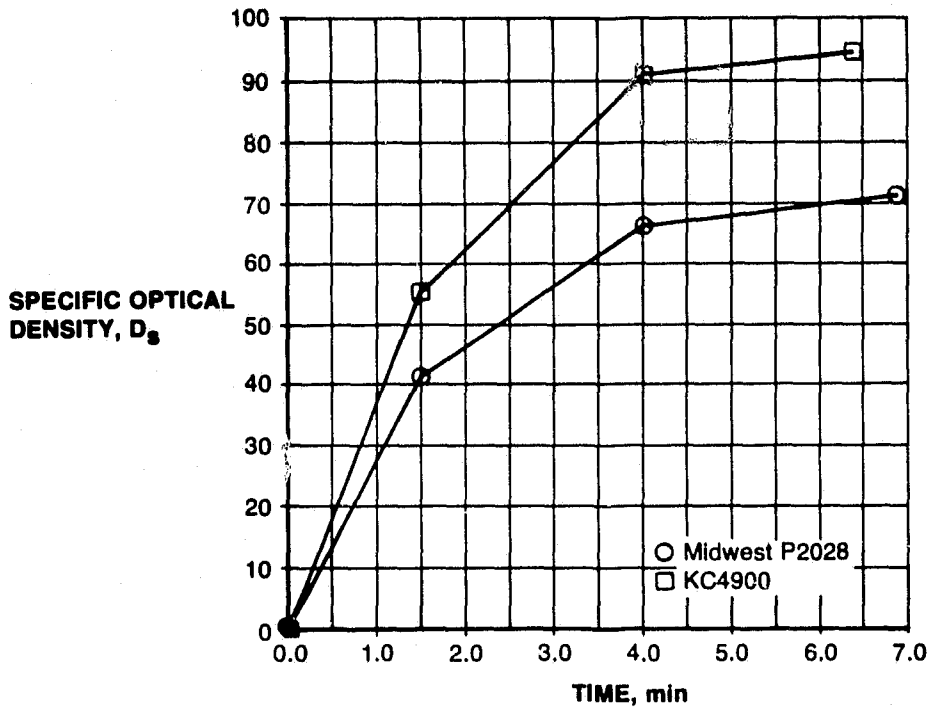
Figure 27.–NBS Chamber–5.0 W/cm² (264.3 Btu/ft²/min)–Flaming Mode–Smoke Evolution
From Test Panels Containing Midwest P2028 and KC4900
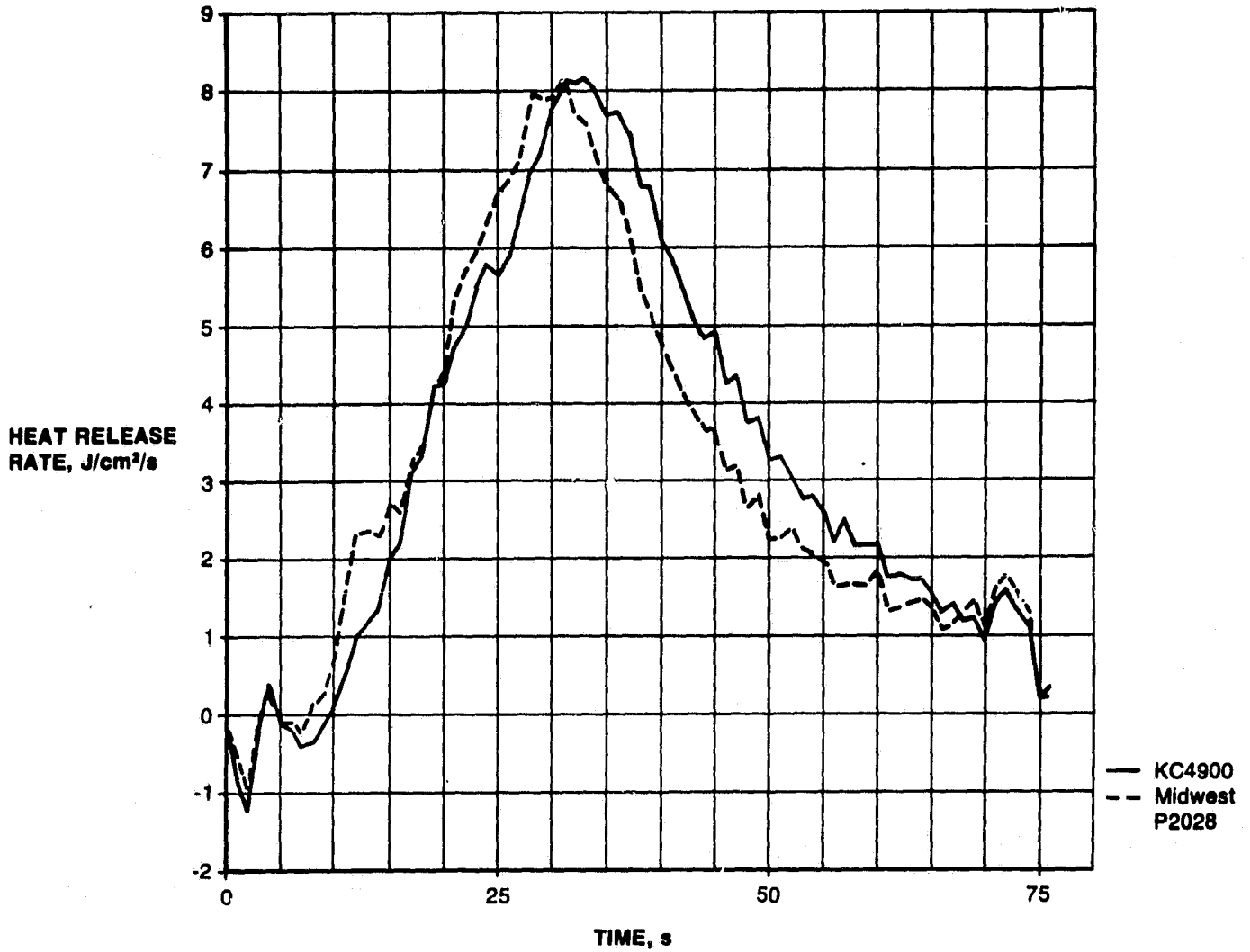
67

SMOKE RELEASE
RATE,
PARTICLES/ft²/s
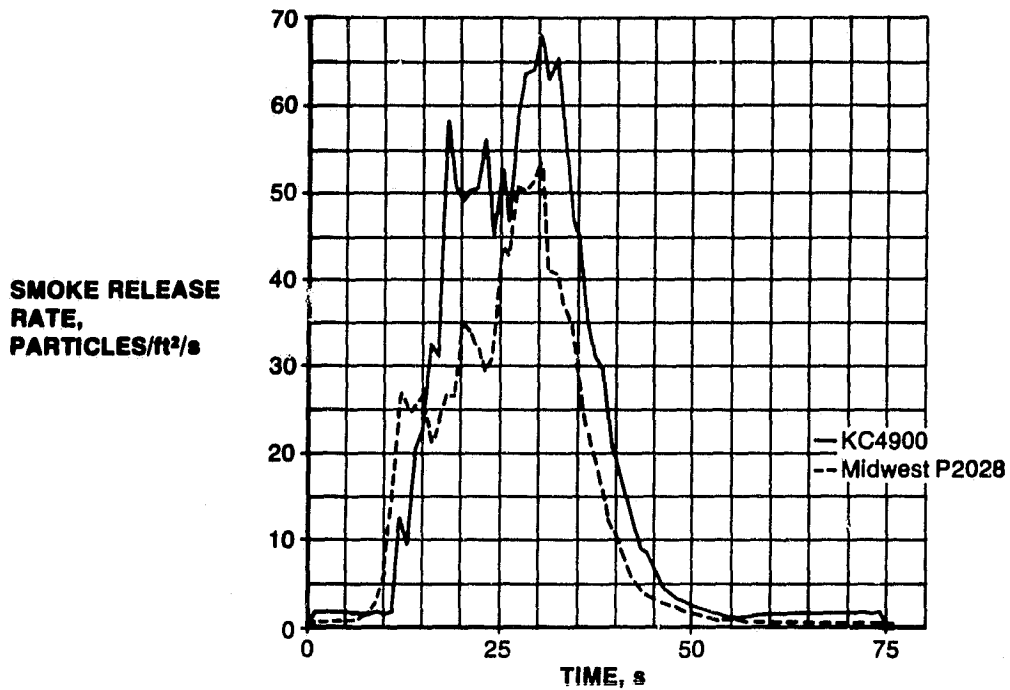
*Figure 28.-OSU Apparatus-5.0 W/cm² (264.3 Btu/ft²/min)-Flaming Mode-Smoke Release
Rate From Test Panels Containing Midwest P2028 and KC4900*

RATE OF
SPECIFIC OPTICAL
DENSITY, d(D$_s$)/dt
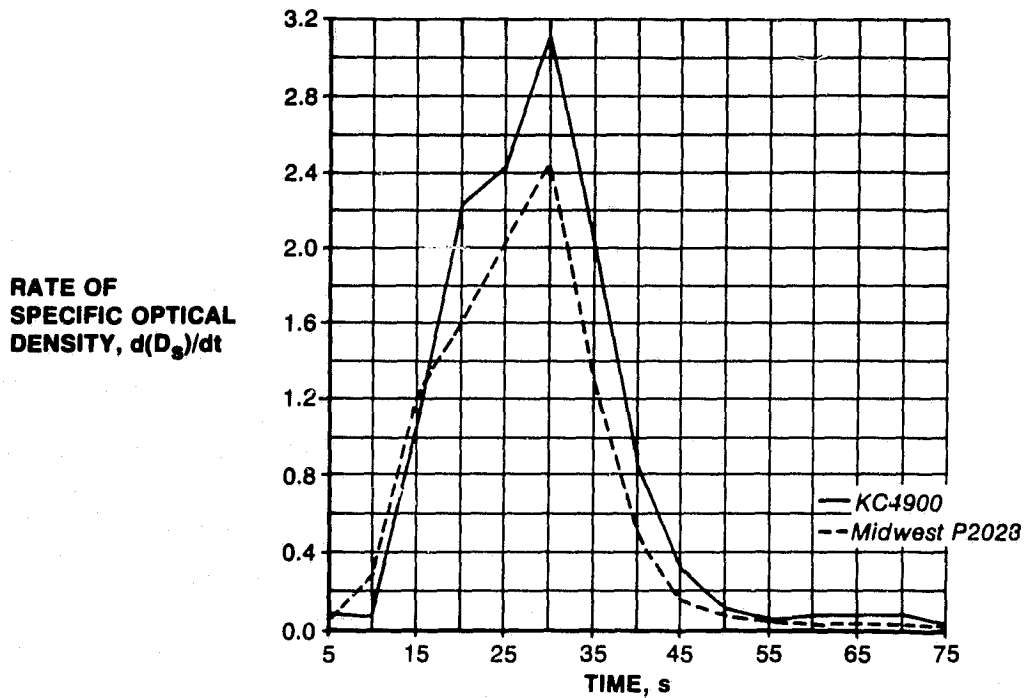
*Figure 29.-OSU Apparatus-5.0 W/cm² (264.3 Btu/ft²/min)-Flaming Mode-Specific Optical
Density Change for Test Panels Containing Midwest P2028 and KC4900*

68

Figure 30.–OSU Apparatus–5.0 W/cm² (264.3 Btu/ft²/min)–Flaming Mode–Heat Release
Rate From Test Panels Containing Midwest P2028 and KC4900

69

Figure 31.–Laboratory Screenprint Apparatus
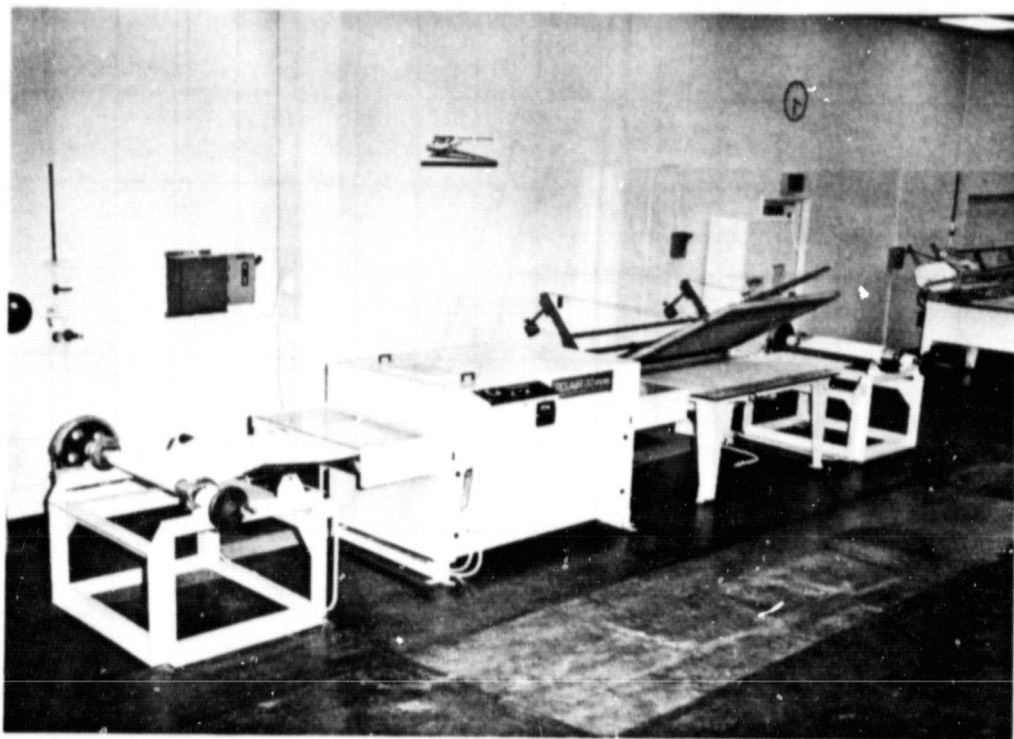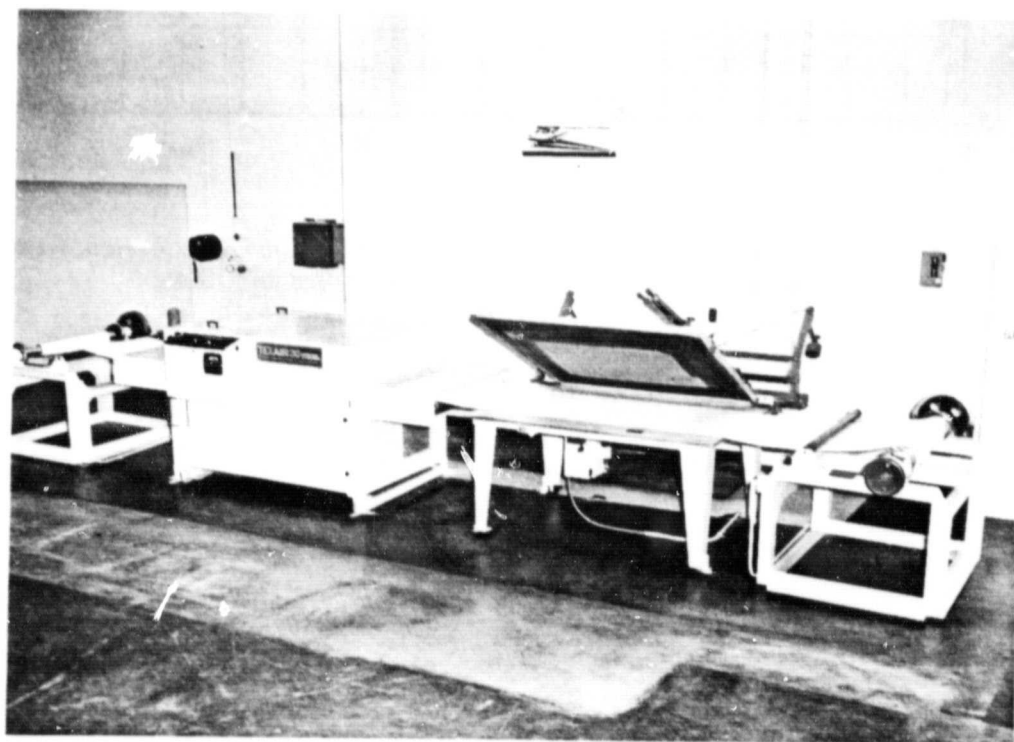

Figure 32.–Laboratory Screenprint Apparatus

70
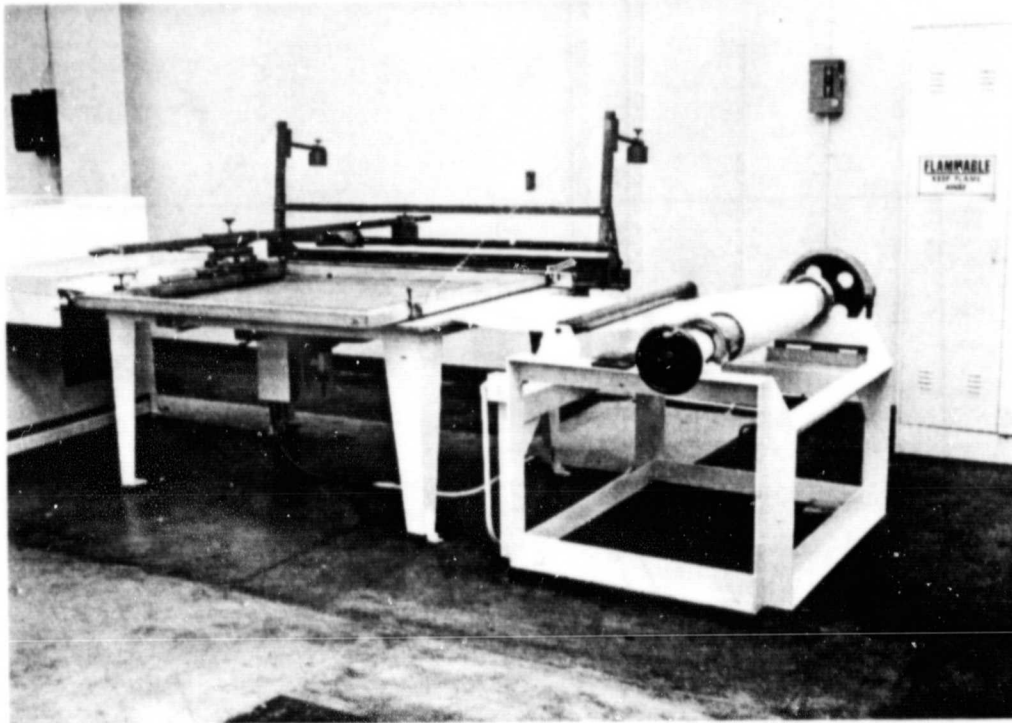
Figure 33.–Laboratory Screenprint Apparatus

25% FPC461 IN MEK

(Weight Basis)

Film thickness–1.8 mil
Temperature–100° F
Relative humidity–95%
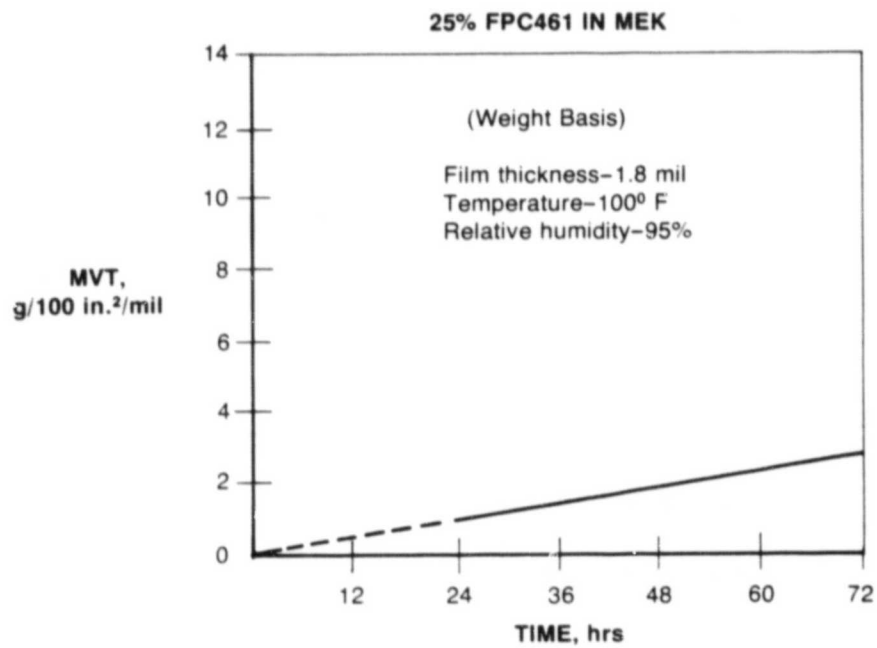
MVT,
g/100 in.²/mil

TIME, hrs

Figure 34.–Moisture Vapor Transmission Versus Time
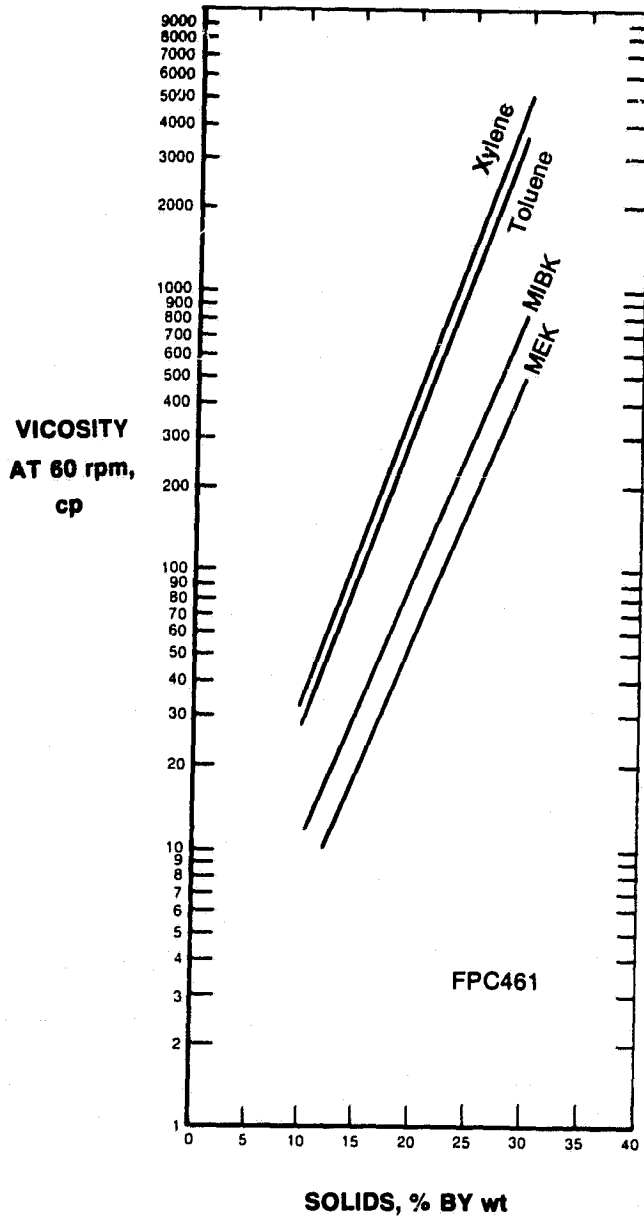
Figure 35.–Viscosity Versus Solids Content
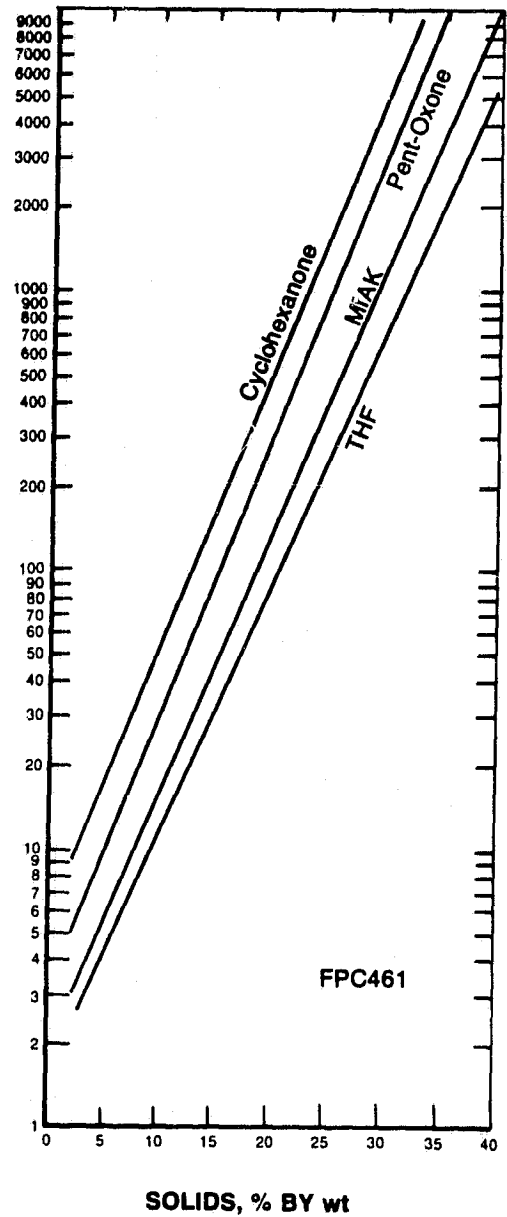
Figure 36.–Viscosity Versus Solids Content
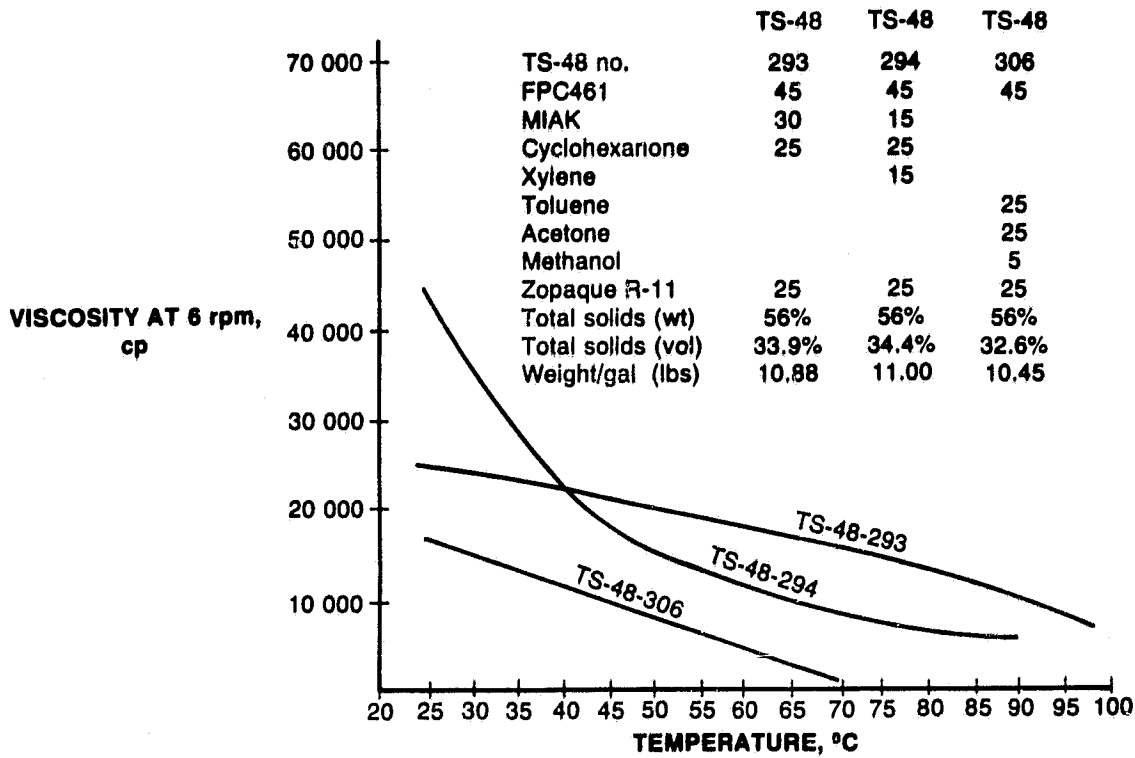
72

VISCOSITY AT 6 rpm, cp

	TS-48	TS-48	TS-48
TS-48 no.	293	294	306
FPC461	45	45	45
MIAK	30	15	
Cyclohexanone	25	25	
Xylene		15	
Toluene			25
Acetone			25
Methanol			5
Zopaque R-11	25	25	25
Total solids (wt)	56%	56%	56%
Total solids (vol)	33.9%	34.4%	32.6%
Weight/gal (lbs)	10.88	11.00	10.45

Figure 37.–Low Shear Rate Solution Characteristics of FPC461 Formulations

VISCOSITY AT 60 rpm, cp

Test Procedure		Parts by Weight		
1. Liquid materials weighed into 4-oz jar	FPC461	20	20	20
	Xylene	25	25	25
2. Spatula-stirred after addition of resin	MIBK	45	45	45
	Cyclohexanone	10	10	10
3. Rolled 24 hrs	Plasticizer	1	2	3
4. Viscosity run at 23°C				
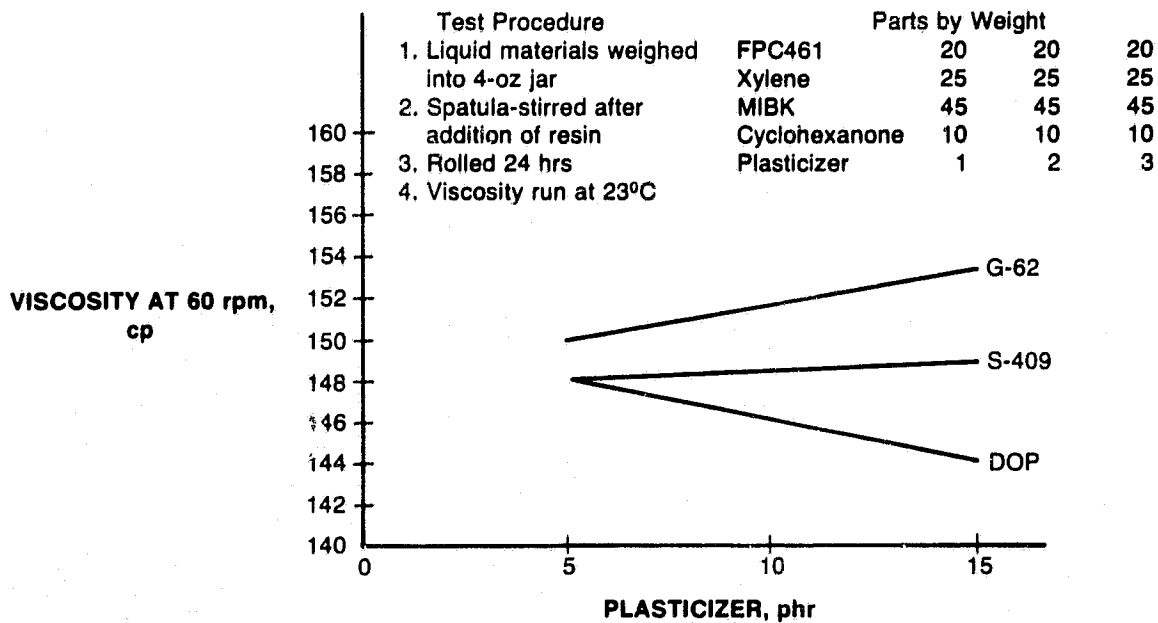
Figure 38.–Viscosity Versus Plasticizer

73

Test Procedure		Parts by Weight		
1. Liquid materials weighed	FPC461	20	20	20
into 4-oz jar	Xylene	25	25	25
2. Spatula-stirred after	MIBK	45	45	45
addition of resin	Cyclohexanone	10	10	10
3. Rolled 24 hrs	Acryloid A-101	1	2	3
4. Viscosity run at 23°C				

VISCOSITY AT 60 rpm, cp
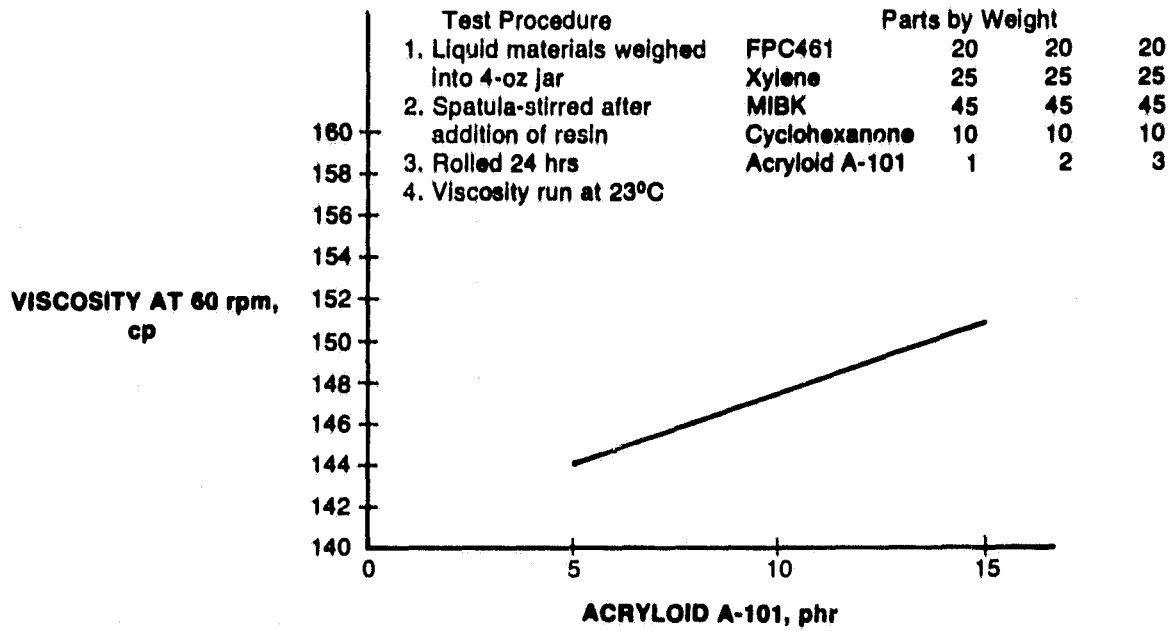
ACRYLOID A-101, phr

Figure 39.–Viscosity Versus Acryloid A-101

FPC461/Elvacite resin   15% by weight
MEK   85% by weight

VISCOSITY AT 60 rpm, cp

Elvacite 2041
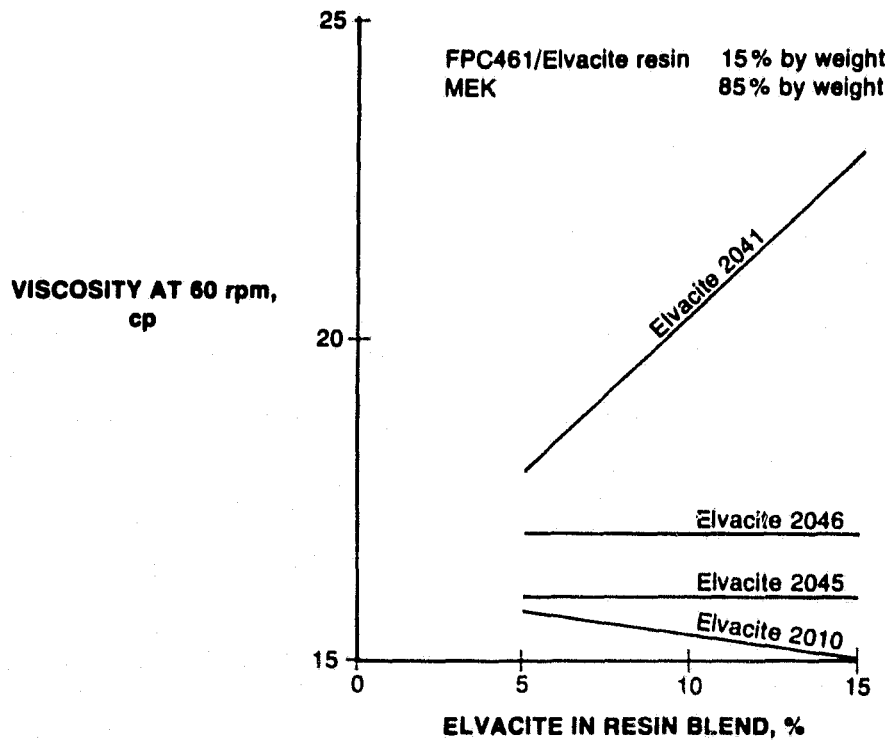
Elvacite 2046

Elvacite 2045

Elvacite 2010

ELVACITE IN RESIN BLEND, %

Figure 40.–Viscosity Versus Elvacite

74