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INTUMESCENT COATINGS AS FIRE RETARDANTS
ARC-10099

Source: J. A. Parker, G. M. Fohlen,
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several papers
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As one Doc

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NASA TECH BRIEF

Intumescent Coatings as Fire Retardants

The development of fire-retardant coatings to protect surfaces which may be exposed to fire or extreme heat is a subject of intense interest to many industries. A fire-retardant paint has been developed which represents a new chemical approach for preparing intumescent coatings, and potentially, is very important to fire-prevention authorities.

The requirements for a superior coating include ease of application, suitability to a wide variety of surfaces and finishes, and stability over an extended period of time within a broad range of ambient temperature and humidity conditions.

These innovative coatings, when activated by the heat of a fire, react to form a thick, low-density, polymeric coating or char layer. Water vapor and sulphur dioxide are released during the intumescent reaction. Two fire-protection mechanisms thus become available: (1) the char layer retards the flow of heat, due to the extremely low thermal conductivity; and (2) water vapor and sulfur dioxide are released, providing fire-quenching properties. Still another mechanism functions in cases where the char, by virtue of its high oxidation resistance and low thermal conductivity, reaches a sufficiently high temperature to re-radiate much of the incident heat load.

The coatings consist of dispersions of selective salts of a nitro-amino-aromatic compound. Specifically, para-nitroaniline bisulfate and the ammonium salt of para-nitroaniline-ortho suphuric acid (2-amino-5-nitrobenzenesulphuric acid) are used. Suitable vehicles are cellulose nitrate of lacquer grade, a nitrile-phenolic modified rubber, or epoxy-polysulfide copolymer. Three separate formulations have been developed. A solvent is usually employed, such as methylethyl ketone, butyl acetate, or toluene, which renders the coatings suitably thin and which evaporates after the coatings are applied. Generally, the intumescent material is treated as insoluble in the vehicle, and is ground and dispersed in the vehicle and solvent like an ordinary coating pigment.

The char found on intumescence is better in terms of yield and physical properties than chars obtained from many previously known intumescent materials. Prior to intumescence, the coating has a density of 85 pounds per cubic foot. After intumescence, the density is approximately 0.3 pound per cubic foot. The linear expansion of the coatings ranges from 70 to 200 times the applied coating thickness.

Notes:

1. Despite the fact that the char yield has better physical properties than those of previously known intumescent materials, a problem of char retention in some environments still exists. High-velocity air flow and high acceleration can cause the very light char to fall off. One approach under study to resolve the problem is to include fibrous material in the coating mix.
2. One of the intumescent coatings has been shown to retain both mechanical and thermal properties when exposed to a high humidity/temperature environment. In addition, the other coatings retain some utility at limited humidity/temperature ranges.
3. A specific paint developed at Ames Research Center extended the time for the backface of a steel disk to reach a temperature of 400°F to more than 4 minutes. An unprotected disk reached 400°F in 21 seconds.
4. An interesting application of one of the coatings is in small compartments, where, in the event of a fire, the coating swells, filling the compartment or void completely. Experiments have shown that this technique will extinguish the fire.

(continued overleaf)

5. Requests for further information may be directed to:

Technology Utilization Officer
Ames Research Center
Moffett Field, California 94035
Reference: B70-10450

Patent status:

Inquiries about obtaining rights for the commercial use of this invention may be made to NASA, Code GP, Washington, D.C. 20546.

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(ARC-10099)

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FOREWORD

This document contains specifications for making and applying three different intumescent coatings developed at Ames Research Center of NASA. The specifications for each coating are divided into two units -- material specifications and process and application specifications. These specifications are not necessarily final, and should be regarded as only preliminary. They have been issued in this form because widespread interest in the coatings demanded prompt dissemination of working documents.

The three coating systems described are designated types 341, 410-1A, and 45B3. The first two types have an environmental limitation. After exposure to high humidity at high temperature for extended periods, the intumescent salt may break down, and the coatings may lose their ability to swell upon application of heat. For this reason, we do not recommend the use of these two coatings in environments where the relative humidity will exceed 60% at temperatures above 80°F. On the other hand, we know of no limitation to the use of coating type 45B3.

Chemical Research Projects Office
Ames Research Center
National Aeronautics and Space Administration
Moffett Field, California

May 1970

INTUMESCENCE: AN IN SITU APPROACH TO THERMAL PROTECTION

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ABSTRACT

The phenomenon of intumescence, the swelling of a substance on the application of heat, has been used for many years in the form of surface coatings as a means of protecting fire-sensitive building materials. From our experience in the areas of polymer chemistry and ablation technology for thermal protection, we have taken a new look at intumescent systems that may serve to protect a variety of heat-sensitive components, such as fuels, explosives, structural materials, and even man.

In this paper, the evolution of intumescent coatings, from the older carbohydrate-phosphoric acid systems through the nitroaniline bisulfate salts originally used by NASA to the newer nitroaniline-sulfonic acids, quinonedioxime-acid mixtures, and nitroanilinosulfones, is described. In the nitroaniline derivatives, the intumescence is modeled as a thermal self-polymerization of the aromatic compounds passing through plastic states and being expanded by evolving gaseous products to form fine textured, low-density foams. These foams have low thermal conductivity, high emissivity, and good resistance to ignition. The gases evolved during the polymerization reaction and injection into the fire zone further serve as flame quenchers. The resulting polymeric foams are polyheterocyclic structures similar to polyquinoxalines or polyphenoxazines.

The development of several useful coating systems to effect fire protection in a variety of applications is discussed. Potential applications ranging from the prevention of weapons cook-off to the improvement in the fire safety of building materials are illustrated in simulation tests.

INTRODUCTION

As a result of the disastrous fires aboard the Apollo spacecraft and on the aircraft carrier USS Forrestal in 1967, the attention of the Thermal Protection Group of NASA-Ames, whose previous tasks were the development of heat-shield materials and thermal-protection coatings for spacecraft, was directed to the study of protecting heat- and fire-sensitive materials in general.

One immediate goal was to develop a means of thermal protection for stored bombs to prevent detonation when bombs were subjected to heat of burning fuels. The bombs must be protected for a time sufficient to deploy fire fighting equipment or to jettison them. To accomplish this short-time thermal protection, it was desired to coat the bombs with a material that would swell on heating to form a thermally resistant insulating layer. This process or phenomenon of swelling with heat is termed *intumescence* (ref.1).

Many people are familiar with a form of intumescence in the novelty item known as Pharoah's Serpents. Another example of this phenomenon is the popular classroom demonstration used to introduce the subject of carbohydrate chemistry to college freshmen. Here the dehydrating action of concentrated sulfuric acid on sugar produces a voluminous amount of black, low-density, carbonaceous solid.

The use of the intumescent process in the form of coatings to provide thermal protection for flammable substrates is not new. As an example, such coatings have been applied to cellulosic ceiling tiles for nearly 20 years. Early versions of these coatings made use of a reaction similar to the sugar and acid mixture mentioned above, in that they consisted of a mixture of a carbohydrate such as starch as a char-forming material, and a dehydrating catalyst, such as ammonium bisulfate or ammonium phosphate, compounded with a binder. On heating, these materials released ammonia gas initially and the resulting sulfuric or phosphoric acid subsequently acted on the carbohydrate to yield the low-density, carbonaceous char, foamed by the action of the liberated ammonia and steam (ref. 2). Continuing efforts by chemical and coating manufacturers to improve the intumescent coatings, both in their performance as coatings and as thermal protectors, have led to the use of various other carbonific substances, drawn usually from the class of polyols such as pentaerythritol and dipentaerythritol. Other gas-producing materials and catalysts, such as ammonium polyphosphate, melamine phosphate, phosphoramide and dicyandiamide, have also been used in improved versions (refs. 1-3). The main drawback to the use of these previously developed coatings has been the lack of long-time stability to outdoor weathering. This deficiency has limited the use of those materials to interior or protected environments. What has been needed is a coating that is able to withstand continuous outdoor exposure—conditions of high humidity, rain, wide fluctuation in ambient temperature, and, for bomb application, exposure to salt spray.

With the advent of new high-temperature and oxidation resistant polymers, such as polybenzimidazoles, polythiazoles, polyphenoxazines, and polyquinoxalines (refs. 4-6), the possibility was suggested that new kinds of char-forming materials might be developed to secure improved intumescent coating systems with high-temperature resistance that would also be nonburning, nonsmoking, and nontoxic, if a method could indeed be found by which these polymers could be made thermally and in place. A general and idealized sequence of reactions required to obtain stable heterocyclic polymers (in sufficiently low density to be useful) formed by the process of intumescence is shown in figure 1. The authors' approach has been to select monomers or prepolymers, stable to at least 300° to 350°F, that thermally react at temperatures greater than 390°F to give the thermally and oxidatively stable aromatic heterocyclic polymers. During the process, the evolution of some and partial retention of a fraction of the gases that are by-products of the condensation polymerization should form a low density foam in situ. As indicated in figure 1, the polymerization reaction must proceed through a molten phase to secure the desired degree of plasticity during the expansion. To obtain efficient thermal protection, the polymerization and expansion reaction should occur preferably in the direction of the applied heating. Another important requirement of this polymerization process is that a sufficiently high degree of polymerization be obtained by the time the expansion is completed to obtain a mechanically coherent foam structure that will not collapse.

To secure a low thermal conductivity, the foam obtained by this process should be predominantly closed cell and of low density. It should be possible to select monomers or prepolymers that polymerize to give off gaseous by-products that, in addition to effecting the foaming of the polymer, can diffuse into the flame boundary to block convective heat transfer (ref. 1) and interact with and suppress free-radical chain carriers characteristic of the flame propagation process.

APPLICATION OF THE REACTIONS OF SUBSTITUTED NITRO-AROMATIC AMINES AS INTUMESCENT MATERIALS

Earlier, Alyea (ref. 7) described the formation of voluminous, black, spongy foam from the action of heat on a mixture of sulfuric acid and p-nitroacetanilide. In our study, a number of variously substituted nitro-aromatic amines were examined for intumescent properties (fig. 2).

It was found, as indicated in figure 2, that the expanded black polymer was formed from both ortho- and para-nitroanilines as well as their substituted derivatives. These materials gave excellent expansion of the order of 70 to 240 times their original volume. The process was found to occur over a temperature range of 390-500°F. Acetylation of the amino group did not seem to affect the intumescent process. It was observed qualitatively that the resultant dark polymeric foam was very resistive to the erosive action of an intense hydrocarbon gas flame. Originally, we believed that it was possible to cause this intumescent reaction of nitroanilines only with concentrated sulfuric acid, but we found subsequently that the use of concentrated phosphoric and polyphosphoric acids also promoted the reaction.

The observation of the effect of heat and sulfuric acid on p-nitroaniline led quickly to the development of its *bisulfate salt* as a dry compound, useful as an intumescent agent. This compound was easily prepared from inexpensive, readily available materials. The preparation and properties of this material are shown in figure 3 (ref. 8). On heating the p-nitroaniline bisulfate to above 430°F, a yield of black foam of about 50 to 54 percent by weight is obtained that is stable to temperatures above 1020°F.

To investigate in detail the reactions occurring during intumescence, three analytical techniques were employed. Thermogravimetric analysis of the weight loss of a sample of p-nitroaniline bisulfate was made, elemental analysis of the thermally reacted material obtained, and, in a separate experiment, the kinds of gases produced were determined by gas chomatography.

An 8-mg sample of the p-nitroaniline bisulfate described above was heated in dried, deoxidized N_2 at a rate of 5.4°F per minute. The weight loss was determined by means of a Cahn recording electro-balance and plotted as a function of sample temperature. The results are given in figure 4. The empirical formula for the initial salt can be written as shown at the right of the figure. The theoretical weights corresponding to the thermolytic process are also shown. Three distinct and sequential reactions can be seen to occur in going from room temperature to 430°F, at which point a sudden intumescence is initiated. The weight loss associated with each of these three distinct processes is equivalent, on a mole basis, to the loss of one molecule of water. This produces a change in the composition of the residue material as indicated in the empirical formulas at the right of the thermogram. The water produced during this induction phase, that is, before intumescence, does not contribute to the foaming process but rather is injected directly into the adjacent gas boundary. As indicated in the thermogram, intumescence occurs above 450°F with elimination of sulfur dioxide, and more water is eliminated at temperatures above 600°F.

Chromatographic analysis of the gases eliminated during the intumescence confirmed that sulfur dioxide and water, both good flame quenchers, were produced. Elemental analysis obtained on the black intumesced foam formed at 480°F was obtained as shown in figure 5. The analysis indicates that nearly all the sulfur is lost and all the nitrogen is retained.

Coatings were prepared utilizing the p-nitroaniline bisulfate compound as the intumescing agent, and they performed quite well in the proper environment, as will be described subsequently. However, because of the nature of the material—a salt of a weak base and a strong acid—it was easily affected by the presence of moisture, giving a very acidic milieu that both affected the vehicle in which it was combined and corroded metallic substrates on humid exposure.

To circumvent these deficiencies, other intumescing agents were desired that were neither easily hydrolyzed nor acidic.

One of the primary reactions between sulfuric acid and nitroaniline is sulfonation of the ring. By keeping mixtures of these ingredients at temperatures below that of intumescence, fair yields of p-nitroaniline-o-sulfonic acid were prepared, isolated as an almost white powder. This compound was found to intumesce quite easily at 450°F and to give a char yield of 50 percent. The product of intumescence resembles and is similar in analysis to the materials obtained from p-nitroaniline bisulfate. The ring sulfonation present in this second compound overcame the problem of hydrolytic stability encountered with p-nitroaniline bisulfate, but there still remained the problem of acidity. We found that the ammonium salt of this sulfonic acid would also intumesce but at a somewhat higher temperature, 572°F. This material is easily prepared from the commercially available sodium salt by reaction with ammonium chloride. The TGA of this material is shown in figure 6.

A detailed explanation of the intumescent reaction of the p-nitroaniline-o-sulfonic acid, or its ammonium salt, will not be entered into at this time. However, the sulfonation of the aromatic ring is probably not a necessary step in the intumescent reaction, and the ring-substituted sulfonic acid actually acts as an in situ source of sulfuric acid when this compound is heated; thus, the reaction is similar to that of p-nitroaniline and sulfuric acid.

Continuing research into the reactions of nitrogenous aromatic compounds has produced two other intumescent systems of interest. These are bis-(4-nitroanilino)-sulfone that intumesces when heated alone, and p-benzoquinone dioxine that intumesces when heated with concentrated sulfuric or phosphoric acid (ref. 10). In all these types of intumescent reactions, it appears that the role of the acid is primarily that of dehydration.

Although it has been speculated that the polymer formed during the intumescence of p-nitroaniline bisulfate may be a laddered polyquinoxaline or polyphenoxazine (ref. 9), no direct proof has yet been obtained for this. The materials obtained in the thermal reaction of all these classes of compounds are insoluble in all solvents tried, including concentrated sulfuric acid. Spectroscopic examinations have been inconclusive, and the scissioning reactions tried have thus far not yielded fragments that would aid in structure elucidation. The main investigative means used has been the elemental analysis of the black materials obtained, and has led to the speculation that structures of the products of the thermal reactions are combinations of recurring units shown in figure 7, all derivable from the starting materials by addition, dehydration, hydrolysis, rearrangement, and ring closure to varying degrees depending on the temperature to which the material is exposed. These structures are similar to those proposed by Szita and Marvel (ref. 11) and Stille and Freeburger (ref. 12) for polymers derived from more complex starting materials in nonintumescing systems.

COATINGS

Two intumescing agents described in the previous section, p-nitroaniline bisulfate and the ammonium salt of p-nitroaniline-o-sulfonic acid were formulated into practical, useful, coating systems. The key to the successful formulation of these coatings has been the selection of a binder system that does not interfere with the intumescent process, which is compatible with the intumescing agent and which will not contribute significantly to the flammability of the system. The vehicle should be either thermoplastic or easily degraded thermally in the same temperature range in which intumescence takes place.

Use of the p-nitroaniline bisulfate salt also required that the vehicles selected as binder be relatively stable to strong acid. Water-based vehicles commonly used in paints were also eliminated from consideration with this material because of their hydrolytic instability. Two vehicles were selected for use with the p-nitroaniline bisulfate: nitrocellulose and a phenolic resin modified acrylonitrilebutadiene polymer. The formulations are given in figures 8-11. The one using the ammonium salt of p-nitroaniline-o-sulfonic acid is shown as #45B3. This material did not have the rather strict limitations of vehicle selection as with the first salt, since it is neither acidic nor hydrolytically unstable. These coatings were prepared by common paint-making techniques and were suitable for conventional brush or spray applications. The air-dried coatings had good initial properties.

TESTS AND POSSIBLE APPLICATIONS OF COATINGS

Measurements in a typical JP-4 fuel fire with free convection showed that heating rates applied to surfaces immersed in such fires are about 30,000 Btu/hr/ft^2, about 90 percent in the form of radiation. This fact made it possible to run screening tests on candidate intumescent coatings in the beam of a solar simulator (fig. 11) capable of delivery 30,000 Btu/hr/ft^2 to a 2-in.-diameter specimen at a distance of about 4 in. from the focus.

This arrangement was used to screen various thicknesses of the intumescent coatings described above. The spectral distribution of the solar simulator beam, which has its peak in the visible portion of the spectrum, did not match that of the JP-4 fire, which peaks at about 2.5 microns; but the thermal protection process appears to be independent of this difference in spectral distribution.

Intumescent coating formulation 341 was applied in thicknesses from about 0.007 in. to about 0.065 in. on a cold-rolled, 0.060-in.-thick substrate. The test specimens were 1 in. in diameter. They were surrounded by thermal insulation to eliminate edge effects, and a thermocouple was mounted on the backface to provide the time-temperature history.

The test of an intumescent coating specimen in the solar simulator beam is more severe than a JP-4 fuel fire for two reasons. Although the average heating rate at the surface is 30,000 Btu/hr/ft^2, the intumescence causes the surface to grow outward toward the focus where the heating rate is higher. Furthermore, there is a radial energy gradient in the beam, and the irradiance is slightly higher on axis than the average value over the area of the test specimen.

A history of the substrate backface temperature during the test in the solar simulator is shown in figure 12. The bare metal without the coating reached 400°F in about 20 sec. On a similar metal substrate with the intumescent coating, the intumescence began within about 10 sec and was nearly complete within 20 sec.

The thermal insulation provided by the intumescence caused a much slower temperature rise of the metal substrate. The increasing slope that occurred later shows the effect of the intumescence growing closer to the focus of the solar simulator where it experiences a much higher heating rate.

The results of numerous tests in the solar simulator beam and in JP-4 fuel fires are given in figure 13. These results show how the time required for the substrate to reach a given temperature varies with intumescent coating thickness. Temperatures of 300°F and 400°F were chosen as limits for presenting these data. The relationship is linear as might be predicted, since the thickness of the intumescence after heating is directly proportional to the thickness of the coating before heating. As can be seen in the figure, the times to reach 300°F and 400°F are shorter for the solar simulator tests than for the fuel fires, demonstrating the greater severity of the solar simulator tests.

The fact that the coating formulation #341, using nitrocellulose as the binder, intumesces to give a thick, stable, closed-cell polymer that is highly resistant to heat leads immediately to the possibility of its application in such places as an airplane where fuel lines or hydraulic lines exist in fairly restricted volumes. A fire in such a location would be extinguished if the intumescence filled the available volume. In order to test this, a simulated section of fuselage was built, and two 2-in.-diameter tubes (simulating fuel lines) were installed through holes in the section. This test setup (fig. 14) was 12-in. wide and 5-1/2-in. deep. The ends and one side of this box were sprayed on the inside with the intumescent coating to a thickness of 0.040 in. A fuel pan containing 200 cc of JP-4 aircraft fuel was placed under the box and the fuel was ignited. The fire caused the intumescent coating on the surface to fill the available volume, thus extinguishing the fire before 30 cc of the fuel had been consumed.

Because of the nature of the p-nitroaniline bisulfate salt and its high loading in formulations #341 and #410-1A, these coatings did not retain their good properties on long exposure to humid environments. The acidity of this salt caused corrosion of metallic substrates and deterioration of the vehicles. However, in certain controlled or protected environments, or if suitably protected by a top coating, they have great utility as just described.

The intumescent coating, formulation 45B3, is composed of the ammonium salt of p-nitroaniline-o-sulfonic acid dispersed in a binder composed of equal parts of a polysulfide polymer and an epoxy resin. After application, the coating has a hard, glossy, abrasion-resistant surface. It has good adherence to the substrate.

The coating has been tested for its effectiveness in protecting a structure from fire. Figure 15 shows the heating of a steel plate 1/16-in. thick with and without the protection of the coating. The tests were made in a specially constructed furnace that exposed the samples to a controlled fire of JP-4 fuel. With no protection, the temperature of the plate rose to 400°F in less than 1 min. When the plate was protected with an 80-mil-thick coating, the heating of the plate to 400°F was delayed for 5 min.

The effect of a high-humidity, high-temperature environment on the thermal performance of the coating has been measured. The results of this test are also shown in figure 15. The temperature history of a coated steel plate that had been exposed for 30 days to high humidity (90 percent) at a temperature of 100°F is compared with the temperature history of the coated plate that had been conditioned in the laboratory atmosphere (50 percent humidity, 73°F). The comparison shows that exposure to the humid environment had no significant influence on the thermal protection afforded by the coating. The small difference between the curves is within the limits of experimental reproducibility for this test.

Samples of the coating also have been exposed for a 90-day period to the natural weather conditions on the roof of the laboratory. Coating samples on 3 x 6-in. steel plates were placed at a 45° angle above the horizontal, facing south. During the period of exposure, approximately 9.7 in. of rain fell. The coating was darkened by the exposure, but its intumescence was not affected. Figure 16 is a photograph comparing an exposed test sample with a control sample, both of which were heated in an oven to complete intumescence following the exposure. No important difference in the intumescent characteristics of the two samples is evident. The coating thus shows good stability under humid environments for at least a 90-day period. Two environmental tests are continuing.

A slightly modified version of the 45B3 formulation was applied to a simulated bomb casing consisting of a section of 5-in. pipe with 1/4-in. wall thickness. The time for the interior wall of the pipe to attain a temperature of 400°C, the critical temperature for the explosive charge, was about 8 min. The time for an uncoated section of pipe to reach the same temperature was about 30 sec. The intumesced coating appeared to have acceptable char integrity and adhesion, better than other coatings tried. A full-scale test using 500-lb bombs is currently in progress.

Many applications of intumescent coatings are possible; for example, on electric motor casings; in fuel and chemical plants and tank farms; and on engine canopys, ventilator grids, and building exteriors. At the present state of development, it is not recommended that these coatings be used in populated or unventilated areas because of the quantities of sulfur dioxide released.

Preliminary specifications for the various coating systems have been established and are available from NASA headquarters.

A research contract has been let to study further intumescent coating systems, especially the relationship of the properties of several classes of polymers as vehicles, in combination with certain selected NASA developed intumescing agents. The influence of polymer type, cross-link index, glass transition temperatures, and other properties on the intumescent process is expected to result from such a study.

Other ongoing research within NASA will study the chemistry of intumescence and the synthesis of other intumescing agents, with the objectives of developing materials more suitable for use in viable environments and developing a range of activation temperatures. Another possible result is the use of the intumescent materials as an inexpensive, refurbishable, heat shield material.

ACKNOWLEDGMENT

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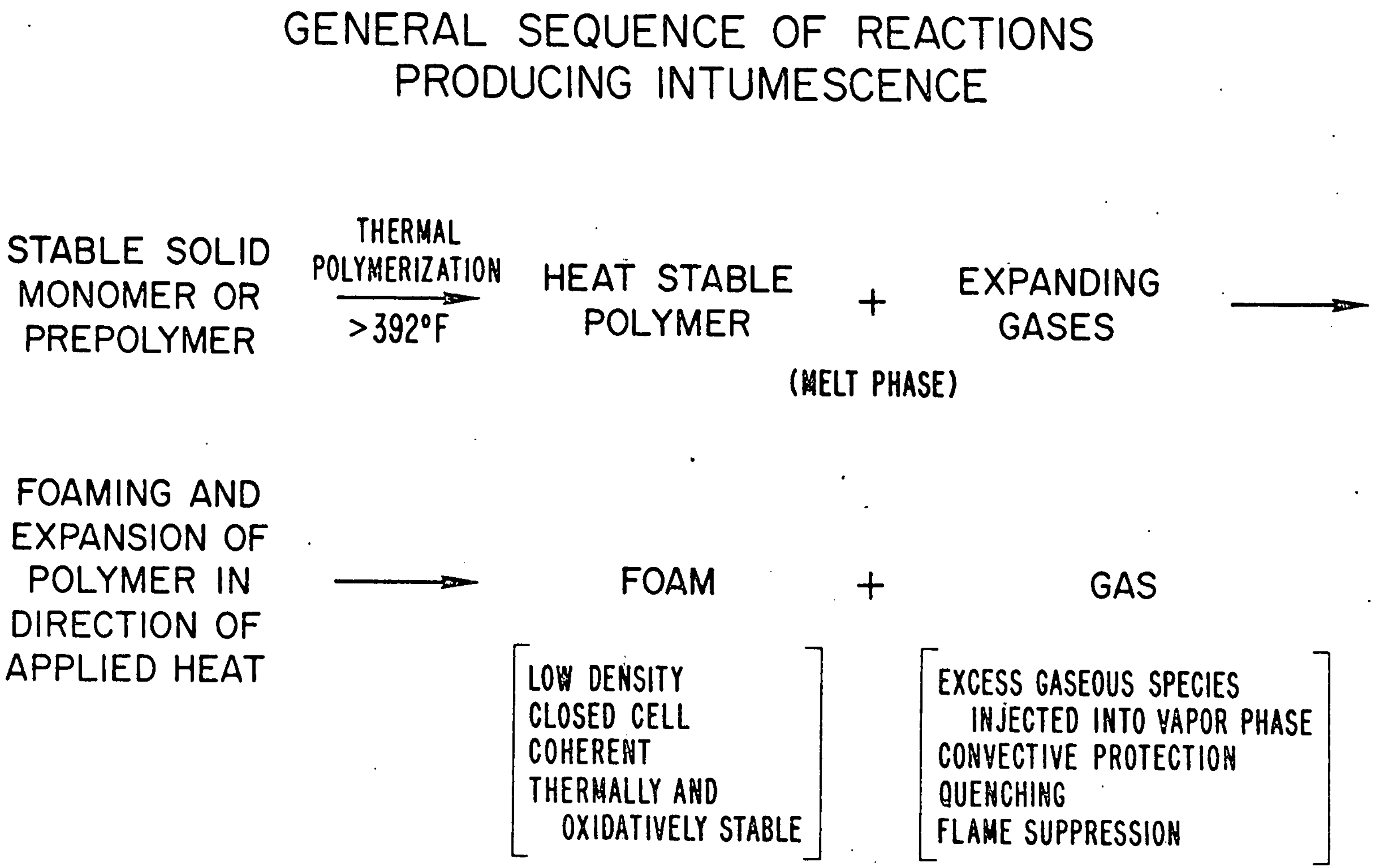

GENERAL SEQUENCE OF REACTIONS PRODUCING INTUMESCENCE
STABLE SOLID MONOMER OR PREPOLYMER
THERMAL POLYMERIZATION
>392°F
HEAT STABLE POLYMER
+
EXPANDING GASES
(MELT PHASE)
FOAMING AND EXPANSION OF POLYMER IN DIRECTION OF APPLIED HEAT
FOAM
+
GAS
LOW DENSITY
CLOSED CELL
COHERENT
THERMALLY AND OXIDATIVELY STABLE
EXCESS GASEOUS SPECIES INJECTED INTO VAPOR PHASE
CONVECTIVE PROTECTION
QUENCHING
FLAME SUPPRESSION

Figure 1.

INTUMESCENT REACTIONS OF NITRO SUBSTITUTED AROMATIC AMINES

THERMAL POLYMERIZATION PROCESS OCCURS WITH:

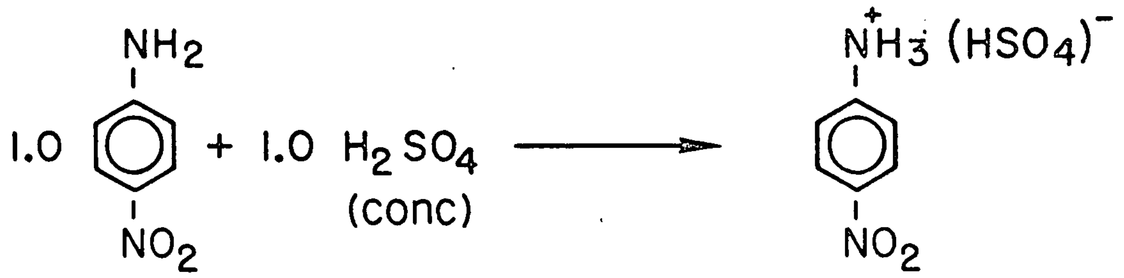
o-NITROANILINE
p-NITROANILINE
p-NITROACETANILIDE
5-NITROISATOIC ANHYDRIDE
etc

$$\xrightarrow[\text{CONCENTRATED SULFURIC OR POLYPHOSPHORIC ACID}]{\Delta \quad >392^\circ\text{F}}$$

SPONGY BLACK POLYMERIC FOAM + GAS

Figure 2.

SYNTHESIS AND CHARACTERIZATION OF INTUMESCENT INTERMEDIATE p-NITROANILINE BISULFATE



p-NITROANILINE		p-NITROANILINE BISULFATE
293°F	MELT POINT	392°F (DECOMP)
138	MOL WT	236 (CALCULATED) 236 (MEASURED)
YELLOW SOLID	APPEARANCE	LT YELLOW SOLID
		(SOLUBLE IN ALCOHOLS, KETONES INSOLUBLE IN BENZENE, TOLUENE, ETHER)

Figure 3.

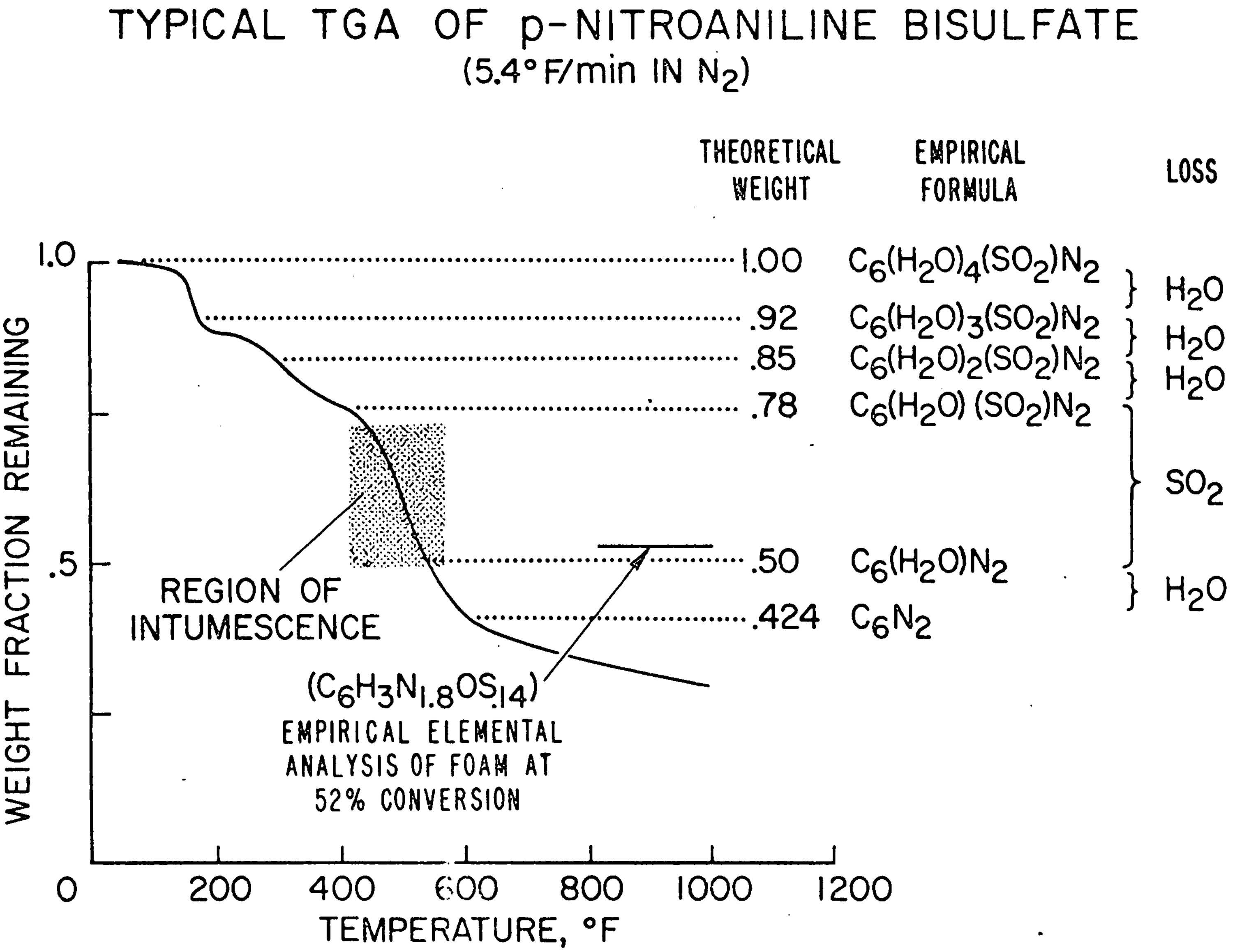

TYPICAL TGA OF p-NITROANILINE BISULFATE
(5.4° F/min IN N_2)
THEORETICAL WEIGHT
EMPIRICAL FORMULA
LOSS
1.00 $C_6(H_2O)_4(SO_2)N_2$
.92 $C_6(H_2O)_3(SO_2)N_2$
.85 $C_6(H_2O)_2(SO_2)N_2$
.78 $C_6(H_2O)(SO_2)N_2$
.50 $C_6(H_2O)N_2$
.424 C_6N_2
H_2O
H_2O
H_2O
SO_2
H_2O
REGION OF INTUMESCENCE
$(C_6H_3N_{1.8}OS_{.14})$
EMPIRICAL ELEMENTAL ANALYSIS OF FOAM AT 52% CONVERSION
WEIGHT FRACTION REMAINING
1.0
.5
0
200
400
600
800
1000
1200
TEMPERATURE, °F

Figure 4.

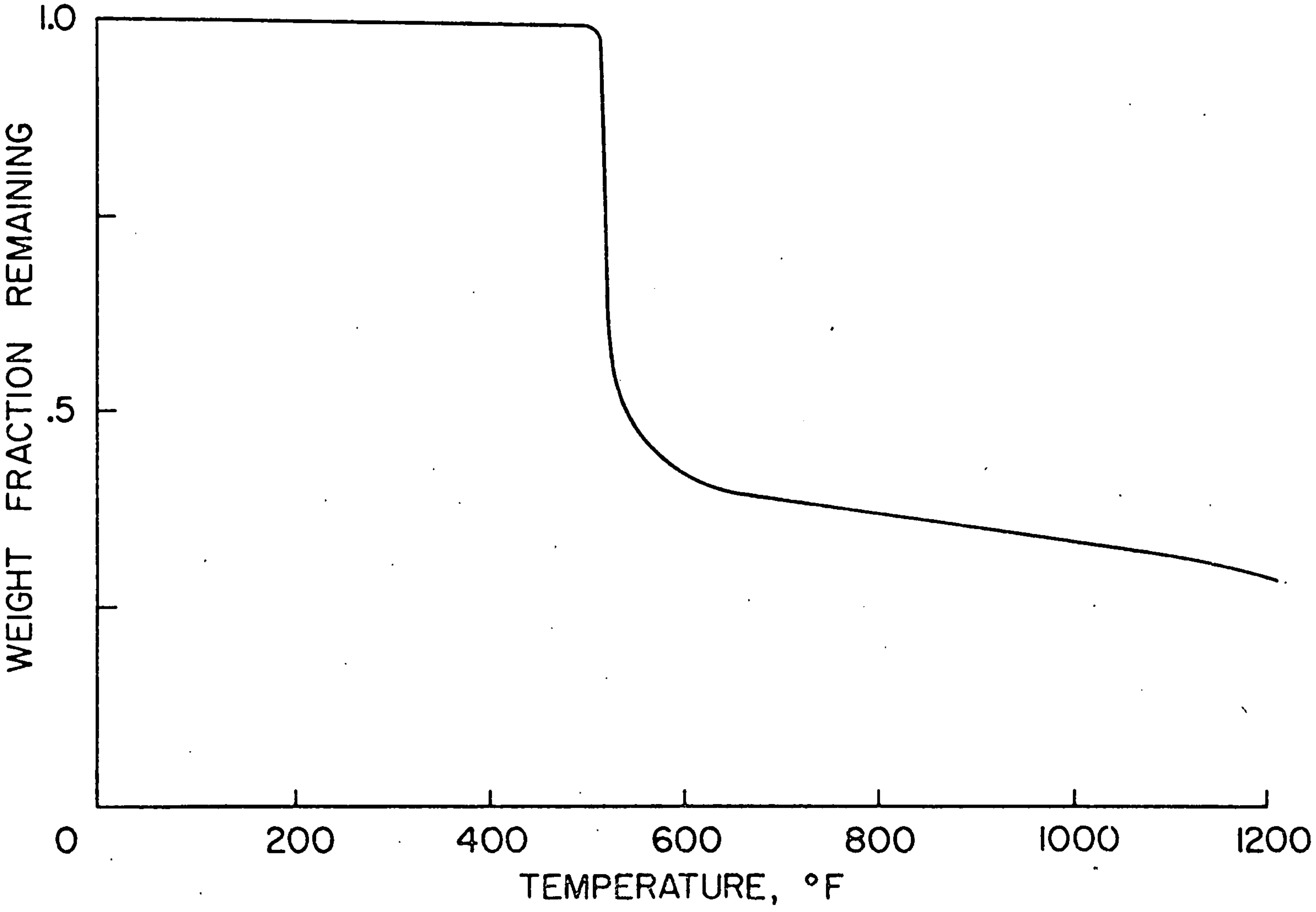
ELEMENTAL ANALYSIS OF BLACK INTUMESCED POLYMERIC FOAM FORMED FROM p-NITROANILINE BISULFATE AT 482°F

C	H	N	S	O
59.65	2.51	20.77	3.64	13.43 %

WHICH CALCULATES TO GIVE AN EMPIRICAL FORMULA:

$$C_6H_3N_{1.8}O_{1.0}S_{.14}$$

Figure 5.



TGA OF p-NITROANILINE – o-SULFONIC ACID, AMMONIUM SALT
(5.4 °F/min IN N_2)

Figure 6.

PROPOSED GENERALIZED MECHANISM FOR POLYMERIZATION

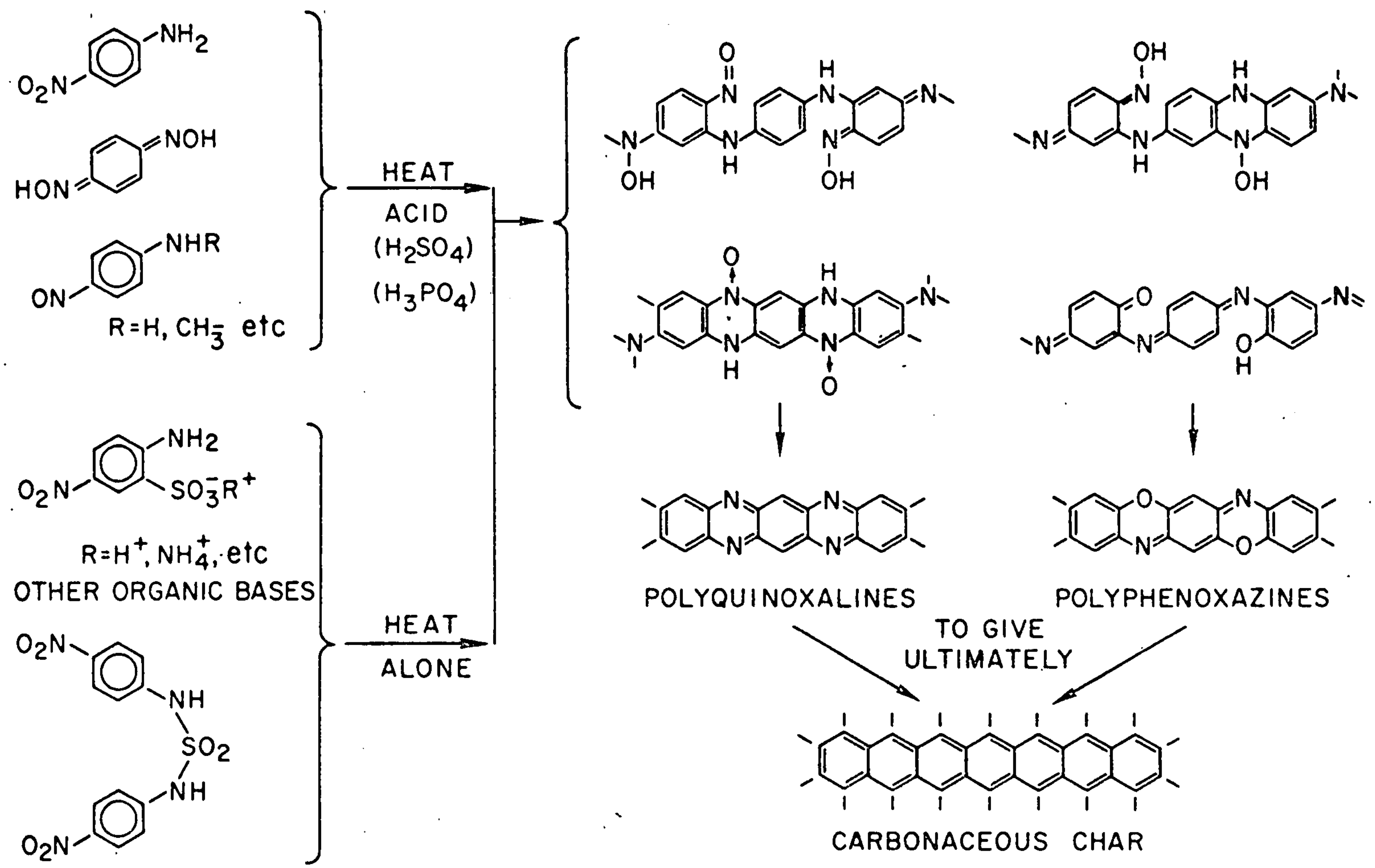


Figure 7.

VOID FILLING THERMAL PROTECTION COATING

FORMULATION NUMBER 341

	PARTS, wt
PART A	
NITROCELLULOSE, ETHANOL-WET (12% N)	8.0
METHYL ETHYL KETONE	28.6
PART B	
p-NITROANILINE BISULFATE	43.7
TOLUENE	19.7
BUTYL ACETATE	19.7

Figure 8.

THERMAL PROTECTION COATING

FORMULATION NUMBER 410-1A

	PARTS, wt
PHENOLIC MODIFIED ACRYLONITRILE-BUTADIENE RUBBER SOLUTION	29.4
p-NITROANILINE BISULFATE	44.0
CYCLOHEXANONE	14.5
METHYL ETHYL KETONE	12.15

Figure 9.

THERMAL PROTECTION COATING
FORMULATION NUMBER 45B3

PART A	PARTS, wt
POLYSULFIDE POLYMER	14.1
AMMONIUM p-NITROANILINE-o-SULFONATE	57.0
METHYL ETHYL KETONE	28.9
PART B	
EPOXY RESIN, LIQUID	14.1
TOLUENE	14.7
PART C	
TRI(DIMETHYLAMINOMETHYL)PHENOL	2.8
TOLUENE	2.8

Figure 10.

ARRANGEMENT FOR TESTS OF INTUMESCENT COATINGS IN SOLAR SIMULATOR BEAM

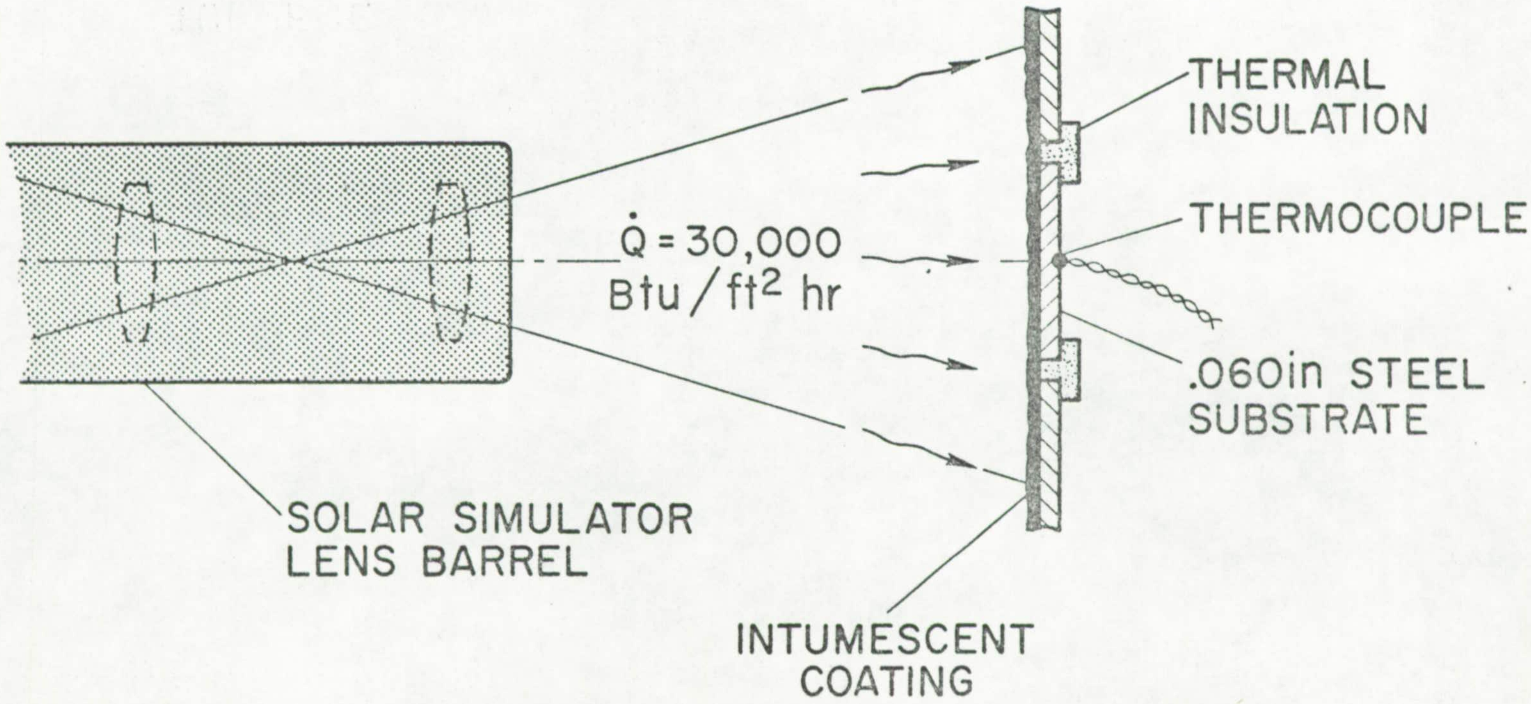
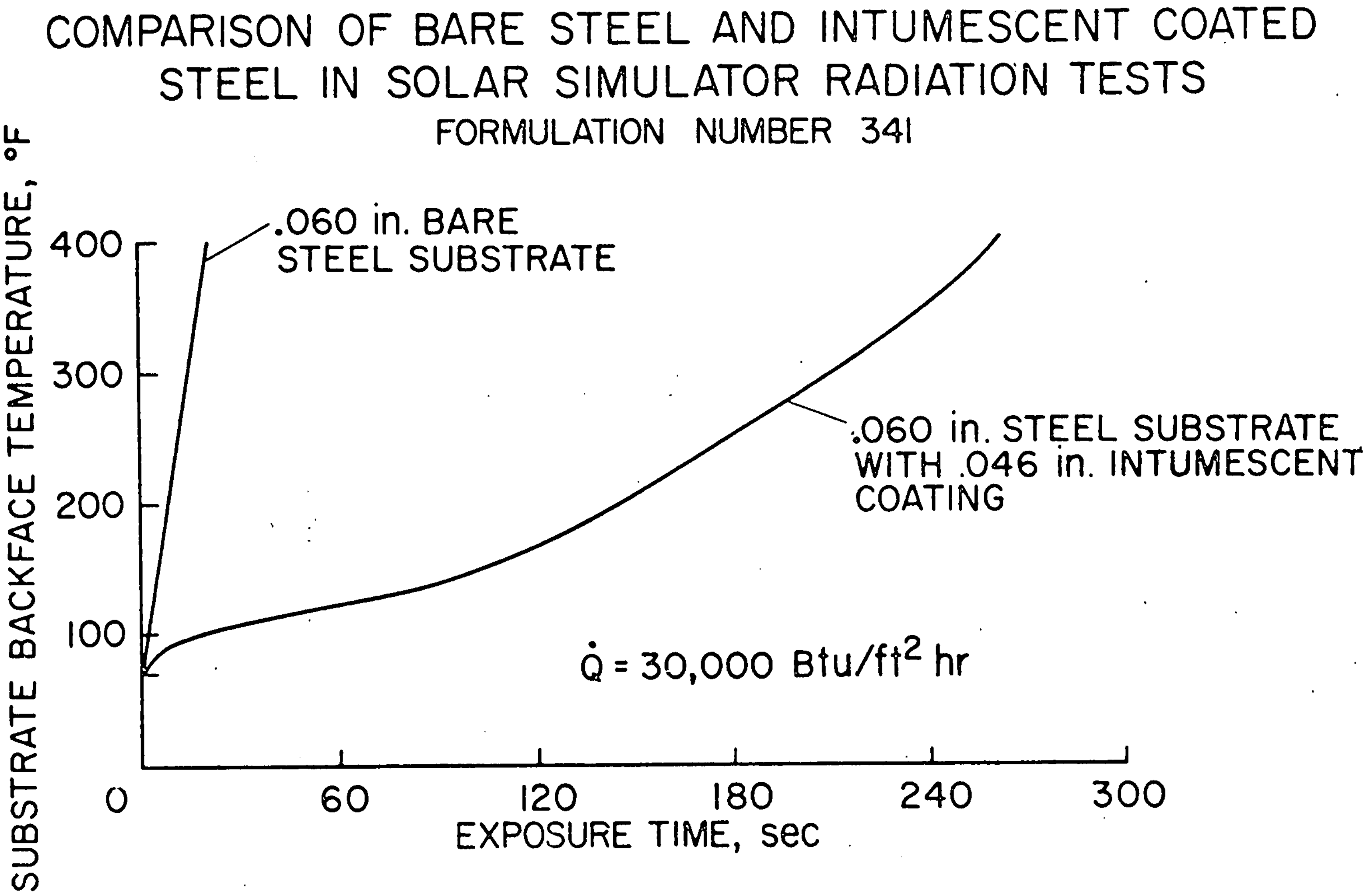


Figure 11.



COMPARISON OF BARE STEEL AND INTUMESCENT COATED STEEL IN SOLAR SIMULATOR RADIATION TESTS

FORMULATION NUMBER 341

Figure 12.

EFFECT OF INTUMESCENT COATING THICKNESS ON BACKFACE TEMPERATURE RISE

FORMULATION NUMBER 341

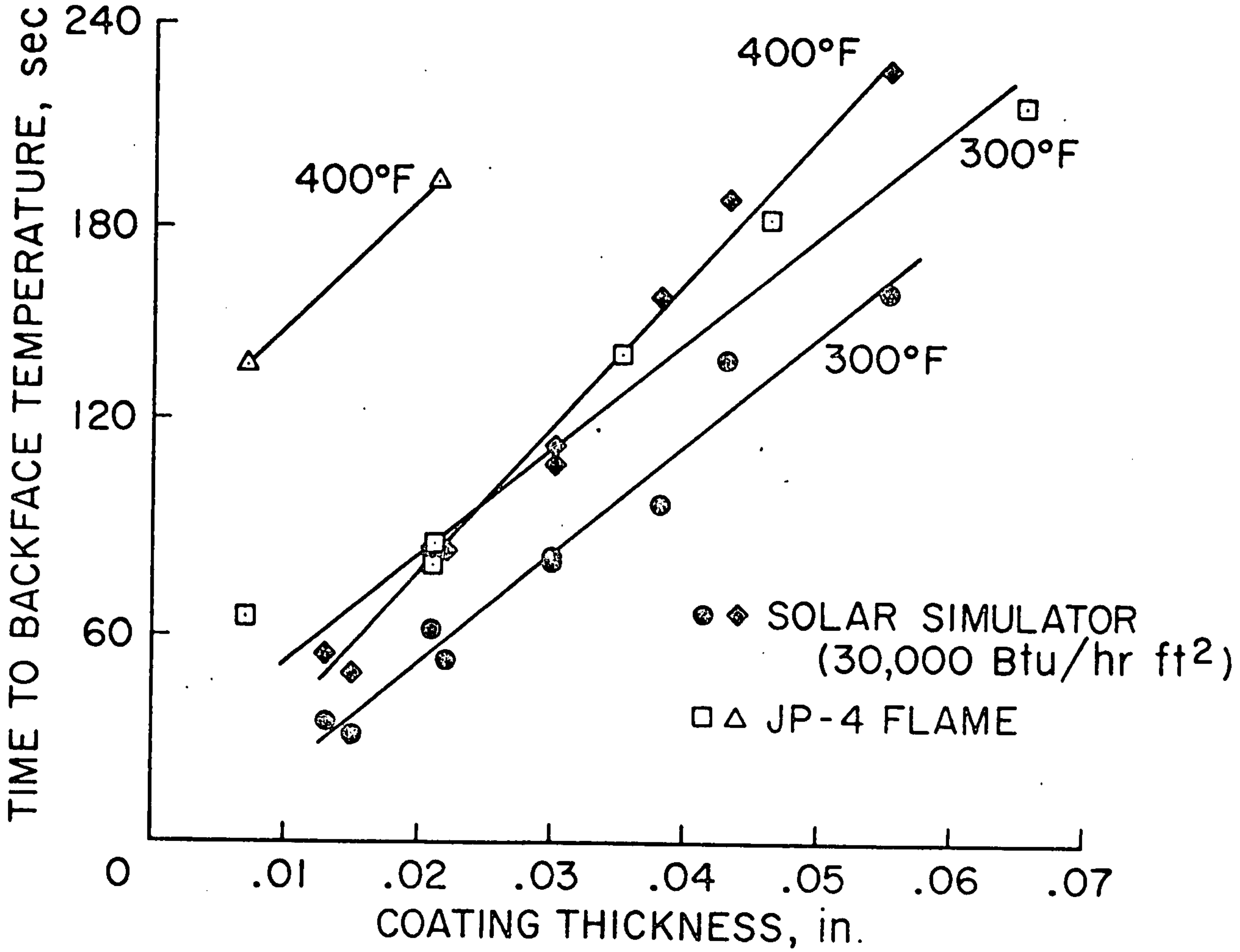
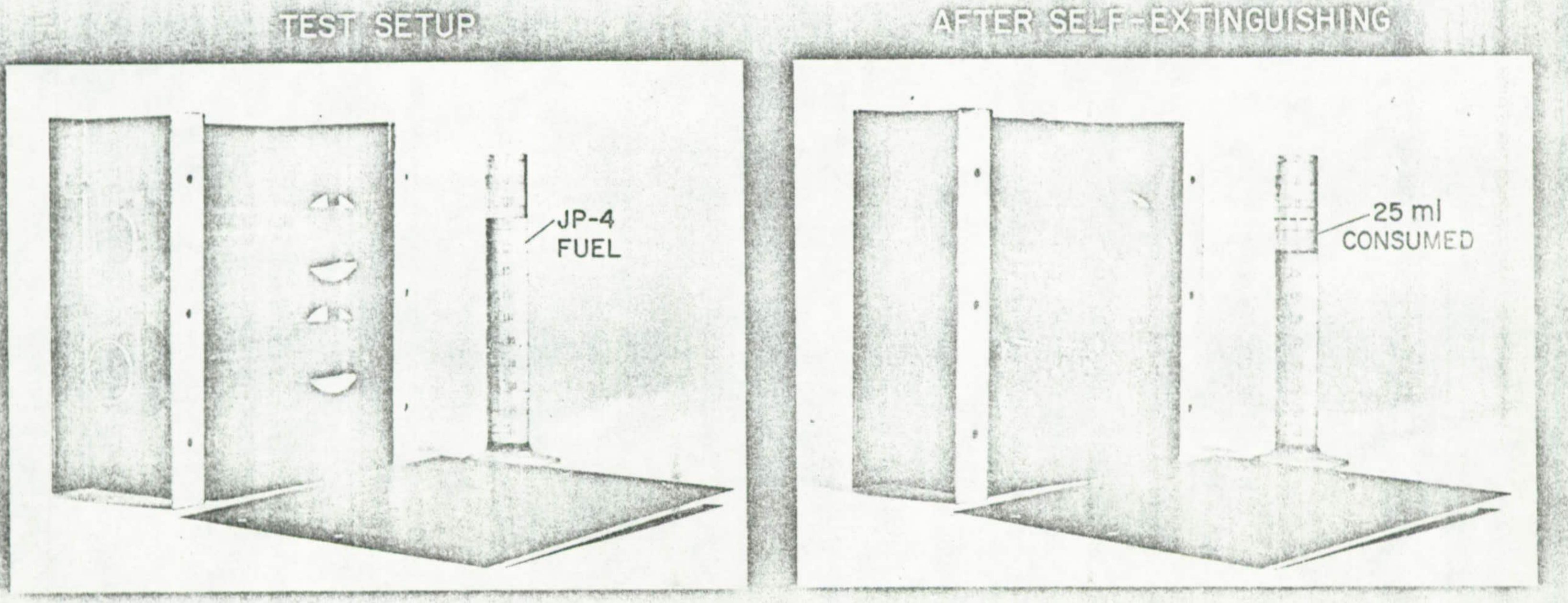


Figure 13.

EFFECT OF JP-4 FUEL FIRE ON SIMULATED FUSELAGE SECTION COATED WITH VOID FILLING THERMAL PROTECTION COATING

FORMULATION NUMBER 341


TEST SETUP

AFTER SELF-EXTINGUISHING

Figure 14

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER, MOFFETT FIELD, CALIFORNIA

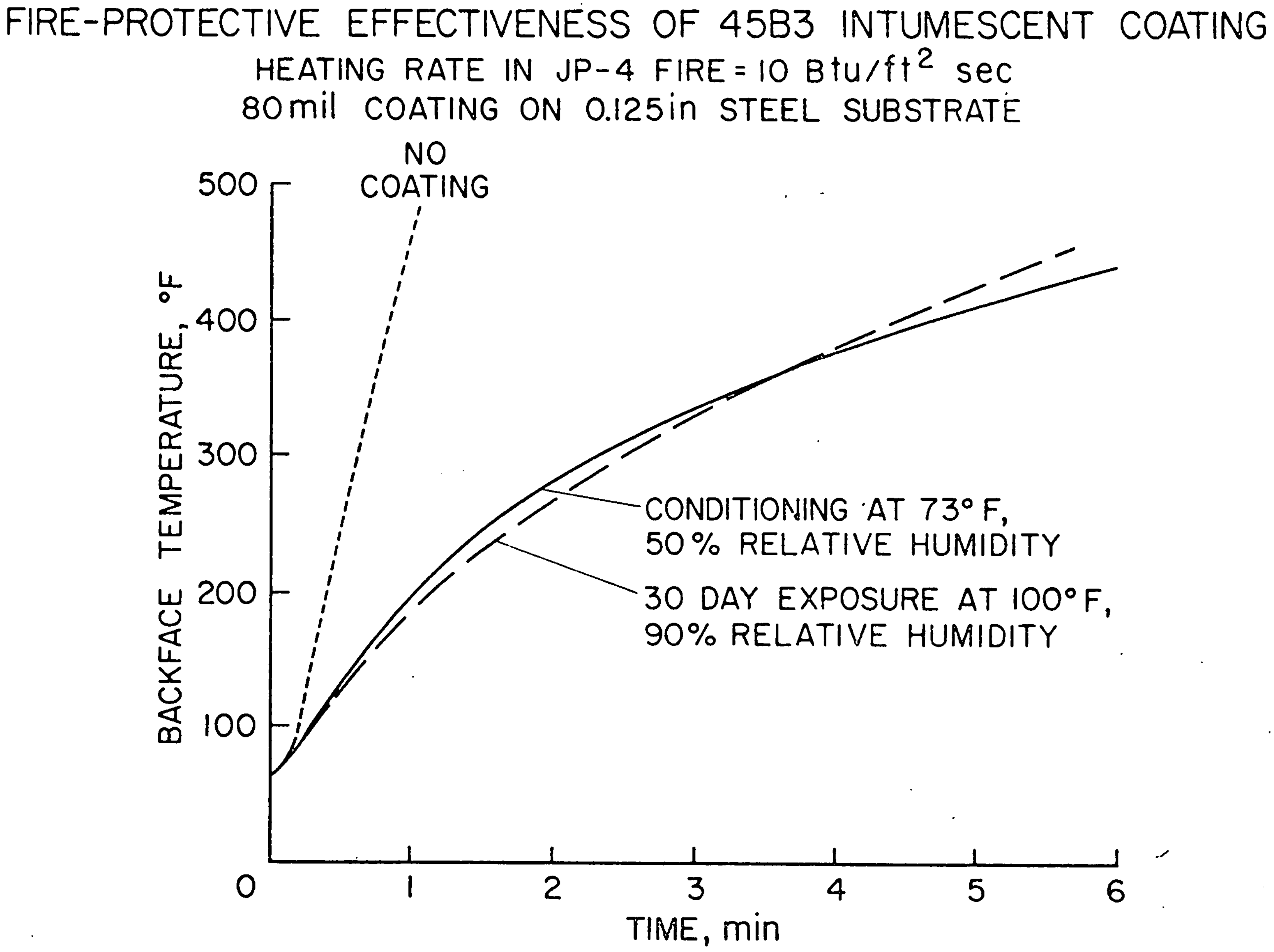
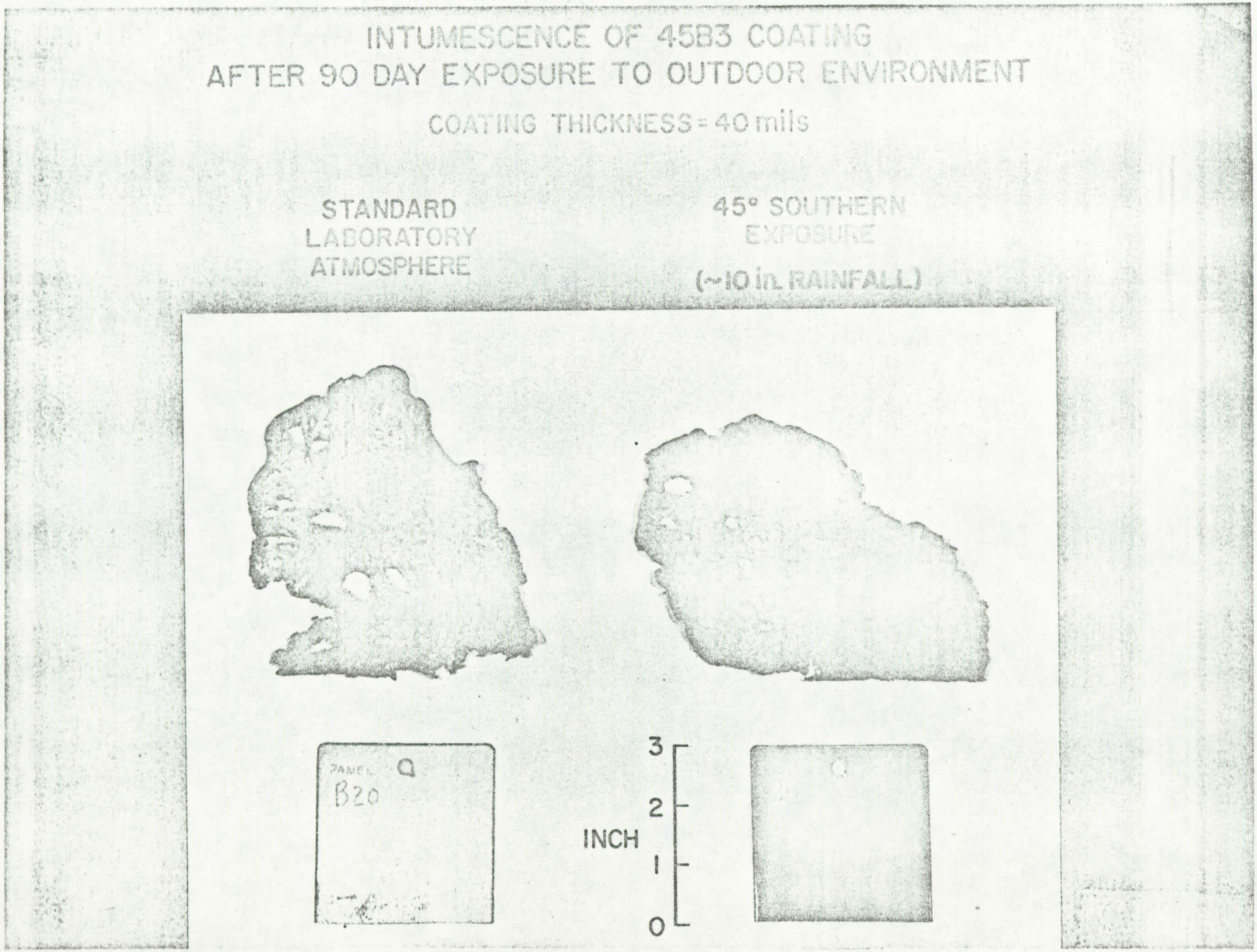

FIRE-PROTECTIVE EFFECTIVENESS OF 45B3 INTUMESCENT COATING
HEATING RATE IN JP-4 FIRE = 10 Btu/ft² sec
80 mil COATING ON 0.125 in STEEL SUBSTRATE
NO COATING
CONDITIONING AT 73° F, 50% RELATIVE HUMIDITY
30 DAY EXPOSURE AT 100° F, 90% RELATIVE HUMIDITY
BACKFACE TEMPERATURE, °F
500
400
300
200
100
0
1
2
3
4
5
6
TIME, min

Figure 15.



INTUMESCENCE OF 45B3 COATING
AFTER 90 DAY EXPOSURE TO OUTDOOR ENVIRONMENT

COATING THICKNESS = 40 mils

STANDARD
LABORATORY
ATMOSPHERE

45° SOUTHERN
EXPOSURE

(~10 in. RAINFALL)

Figure 16

PRELIMINARY MATERIAL SPECIFICATION

for

341 VOID FILLING THERMAL PROTECTION COATING

Chemical Research Projects Office

NASA - Ames Research Center

Moffett Field, California 94035

FOREWORD

This specification has been prepared for an intumescent coating system developed at Ames Research Center that appears to be useful in fire-protection applications. Because of the potential usefulness of the coating system, we are releasing this specification for limited distribution, even though it is only a preliminary draft.

Chemical Research Projects Office
Ames Research Center
National Aeronautics and Space Administration

February 1970

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PRELIMINARY MATERIAL SPECIFICATION

for

341 VOID FILLING THERMAL PROTECTION COATING

1.0 SCOPE

This specification covers the components for producing 341 Void Filling Thermal Protection Coating.

2.0 FORMULATION

Components	Parts Weight	Source
2.1 Part A		
Nitrocellulose, ethanol wet	8.01	DuPont Company
Methyl Ethyl Ketone	28.63	Mallinckrodt
2.2 Part B		
Para-nitroaniline bisulfate	43.67	Burdick-Jackson
Toluene	19.69	Mallinckrodt
Butyl Acetate	19.69	Mallinckrodt

3.0 INGREDIENTS

3.1 Nitrocellulose, ethanol wet

3.1.1 Scope

3.1.1.1 Scope. This specification covers the minimum requirements for a nitrocellulose, ethanol wet intended for use as a constituent in the formulation of a void filling thermal protection coating.

3.1.2 Applicable Documents

3.1.2.1 The following documents form a part of this specification:

American Society for Testing and Materials

ASTM D 301	Specification and Tests for Soluble Cellulose Nitrate
ASTM D 1343	Test for Viscosity of Cellulose Derivatives by Ball-Drop Method

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

3.1.3 Requirements

3.1.3.1 Qualification. The nitrocellulose, ethanol wet material furnished under this specification shall be a product which has been tested and passed the qualification tests specified herein.

3.1.3.2 Material. The nitrocellulose, ethanol wet material furnished under this specification shall produce the Infrared (IR) Spectral configuration as indicated in Figure 1, when examined as specified.

3.1.3.3 Properties

3.1.3.3.1 Physical Properties

3.1.3.3.1.1 Appearance. The appearance of the nitrocellulose shall be an off-white,finely cubed form wetted with ethanol and free from suspended foreign particles when examined as specified.

3.1.3.3.1.2 Volatile Content. The volatile content of the nitrocellulose shall be 30 ± 2 percent when tested as specified.

3.1.3.3.1.3 Viscosity. The viscosity of the nitrocellulose material shall be 30 - 40 seconds when tested as specified.

3.1.3.3.2. Chemical Properties

3.1.3.3.2.1 Nitrogen Content. The nitrogen content of the nitrocellulose shall be 11.8 - 12.2 percent when tested as specified.

3.1.3.3.2.2 Heat Stability. The heat stability of the nitrocellulose shall be 30 minutes minimum when tested as specified.

3.1.3.4 Shelf Life. After storage for two months from the date of manufacture under specified conditions, the nitrocellulose shall be capable of passing all the quality conformance tests specified.

3.1.4 QUALITY ASSURANCE PROVISIONS

3.1.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.1.4.2 Classification of Examinations and Tests. The examination and testing of the nitrocellulose shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 2.

3.1.4.3 Sampling

3.1.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.1.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.1.4.3.2.1 Lot and Batch. All nitrocellulose manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.1.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of nitrocellulose. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.1.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.1.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.2.3.3.1.1, the IR spectra requirements of Paragraph 3.2.3.2.

Table 1

Examination and Tests

Examination or Test	Requirement (Section 3.1.3)	Test Method (Section 3.1.4)
Qualification Test		
Material	3.1.3.2	3.1.4.4.1
Appearance	3.1.3.3.1.1	3.1.4.4.1
Volatile Content	3.1.3.3.1.2	3.1.4.4.2
Viscosity	3.1.3.3.1.3	3.1.4.4.3
Nitrogen Content	3.1.3.3.2.1	3.1.4.4.4
Heat Stability	3.1.3.3.2.2	3.1.4.4.5

Table 1 continued

Examination and Tests

Examination or Test	Requirement (Section 3.1.3)	Test Method (Section 3.1.4)
Quality Conformance Tests		
Material	3.1.3.2	3.1.4.4.1
Volatile Content	3.1.3.3.1.2	3.1.4.4.2
Viscosity	3.1.3.3.1.3	3.1.4.4.3
Nitrogen Content	3.1.3.3.2.1	3.1.4.4.4
Heat Stability	3.1.3.3.2.2	3.1.4.4.5

3.1.4.4.2 Volatile Content: The volatile content of the nitrocellulose shall be determined as follows and conform to Paragraph 3.1.3.3.1.2:

Procedure: Place about 50 grams of a representative sample of nitrocellulose into a tared sample holder, cover with the top screen and immediately weigh to the nearest 0.1 gram. Since the nitrocellulose is ethanol-wet, pour about 100 ml of water on top of the sample and allow it to percolate through the sample. Place the sample holder in the blower tube and pass warm air through it for at least 30 minutes at 60 - 65°C. Weigh the holder and dry sample, determine the loss in weight on drying, and calculate the volatile content:

$$\frac{\text{Original sample weight - dry sample weight}}{\text{original sample weight}} \times 100 = \text{percent volatile content}$$

3.1.4.4.3 Viscosity. The viscosity of the nitrocellulose shall be determined in accordance to ASTM D 301 and ASTMD 1343 and the results shall conform to Paragraph 3.1.3.3.1.3.

3.1.4.4.4 Nitrogen Content. The nitrogen content of the nitrocellulose shall be determined in accordance with ASTM D 301 and the results shall conform to Paragraph 3.1.3.3.2.1.

3.1.4.4.5 Heat Stability. The heat stability of the nitrocellulose shall be determined in accordance with ASTM D 301 and the results shall conform to Paragraph 3.1.3.3.2.2.

3.1.3.4 Shelf Life. The shelf life of the nitrocellulose shall be determined after storage for two months from date of manufacture, in original sealed containers, at a temperature of 65 ± 15°F. The stored material shall be tested in accordance with Paragraph 3.1.4.4.1 and the results shall conform to 3.1.3.4.

3.2 Para-nitroaniline bisulfate

3.2.1 Scope

3.2.1.1 Scope. This specification covers the minimum requirements for para-nitroaniline bisulfate intended for use as a constituent material in the formulation of a void filling thermal protection coating.

3.2.2 Applicable Documents

3.2.2.1 The following documents form a part of this specification:

NONE

3.2.3 Requirements

3.2.3.1 Qualification. The para-nitroaniline bisulfate furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.2.3.2 Material. The para-nitroaniline bisulfate furnished under this specification shall produce the Infrared (IR) Spectral configuration as indicated in Figure 2, when examined as specified.

3.2.3.3 Properties

3.2.3.3.1 Physical Properties

3.2.3.3.1.1 Appearance. The appearance of the para-nitroaniline bisulfate shall be a pale gray-yellow, free flowing granular solid when examined as specified.

3.2.3.3.2 Chemical Properties

3.2.3.3.2.1 Equivalent Weight. The equivalent weight of the para-nitroaniline bisulfate shall be 236 ± 5 when examined as specified.

3.2.3.3.2.2 Decomposition Temperature. The decomposition temperature of the para-nitroaniline bisulfate shall be 200 ± 10°C when examined as specified.

3.2.3.3.2.3 Purity. The purity of the para-nitroaniline bisulfate shall be a minimum of 95 percent when tested as specified.

3.2.4 QUALITY ASSURANCE PROVISIONS

3.2.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.2.4.2 Classification of Examinations and Tests. The examination and testing of the para-nitroaniline bisulfate shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 2.

3.2.4.3 Sampling

3.2.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.2.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.2.4.3.2.1 Lot and Batch. All para-nitroaniline bisulfate manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.2.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of para-nitroaniline bisulfate. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.2.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of $75 \pm 5^{o}F$ with a relative humidity not exceeding 60 percent.

3.2.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.2.3.3.1.1, the IR spectra requirements of Paragraph 3.2.3.2.

3.2.4.4.2 Equivalent Weight. The equivalent weight of the para-nitroaniline bisulfate shall be determined as follows and the results shall conform to Paragraph 3.2.3.3.2.1:

Procedure: Weigh out 0.23 gm of para-nitroaniline bisulfate. Suspend or dissolve the material in 50 to 100 ml of aqueous methanol. Titrate with 0.1N NaOH using a recording electrometric titrator. From the volume of the titrant to reach end point, calculate the equivalent weight.

3.2.4.4.3 Decomposition Temperature. The decomposition temperature of the para-nitroaniline bisulfate shall be determined as follows and the results shall conform to Paragraph 3.2.3.3.2.2:

Table 2

Examination and Tests

Examination or Test	Requirement (Section 3.2.3)	Test Method (Section 3.2.4)
Qualification Tests		
Material	3.2.3.2	3.2.4.4.1
Appearance	3.2.3.3.1.1	3.2.4.4.1
Equivalent Weight	3.2.3.3.2.1	3.2.4.4.2
Decomposition Temperature	3.2.3.3.2.2	3.2.4.4.3
Purity	3.2.3.3.2.3	3.2.4.4.4
Quality Conformance Tests		
Material	3.2.3.2	3.2.4.4.1
Appearance	3.2.3.3.1.1	3.2.4.4.1
Equivalent Weight	3.2.3.3.2.1	3.2.4.4.2
Decomposition Temperature	3.2.3.3.2.2	3.2.4.4.3
Purity	3.2.3.3.2.3	3.2.4.4.4

Procedure: The decomposition temperature is the temperature range observed during maximum weight loss. A thermogravimetric analysis apparatus recording weight loss versus temperature at a heating rate of 3°C/minute to 300°C in nitrogen atmosphere will be required.

3.2.4.4.4 Purity. The purity of the para-nitroaniline bisulfate shall be determined by elemental Carbon, Hydrogen, Nitrogen (CHN) Analysis and the results shall conform to Paragraph 3.2.3.3.2.3.

3.3 Methyl Ethyl Ketone

3.3.1 Scope

3.3.1.1 Scope. This specification covers the minimum requirements for methyl ethyl ketone intended for use as a constituent material in the formulation of a void filling thermal protection coating.

3.3.2 Applicable Documents

3.3.2.1 The following document forms a part of this specification:

Interim Federal Specification

TT-M-00261c	Methyl Ethyl Ketone

(Application for copies should be addressed to the General Services Administration Regional Offices)

3.3.3 Requirements

3.3.3.1 Qualification. The methyl ethyl ketone furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.3.3.2 Properties

3.3.3.2.1 Physical Properties. The physical properties of the methyl ethyl ketone shall be as listed in Table 3.

3.3.3.2.2 Chemical Properties. The chemical properties of the methyl ethyl ketone are that the compound shall have the chemical formula $CH_3COC_2H_5$ with a molecular weight of 72.10.

Table 3

Property	Requirement
Appearance	Clean and free from sediment and suspended matter
Alcohol Content, %	0.7 max. as secondary butyl alcohol
Specific Gravity, 20°/20°C	0.806 ± 0.001
Nonvolatile matter from 100 ml., gram	0.005 max.
Acidity, mg of KOH per gram of sample	0.047 max.
Distillation:	
Below 78.5	none
Above 81°C	none
Color, on the platinum cobolt scale	20
Water, percent by weight	0.3

3.3.4 QUALITY ASSURANCE PROVISIONS

3.3.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.3.4.2 Classification of Examinations and Tests. The examination and testing of the methyl ethyl ketone shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 4.

3.3.4.3 Sampling

3.3.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.3.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.3.4.3.2.1 Lot and Batch. All methyl ethyl ketone manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

Table 4

Examination and Tests

Examination or Test	Requirement (Section 3.3.3)	Requirement (Section 3.3.4)
Appearance	3.3.3.2.1	3.3.4.4.1
Alcohol Content	3.3.3.2.1	3.3.4.4.2
Specific Gravity	3.3.3.2.1	3.3.4.4.2
Nonvolatiles	3.3.3.2.1	3.3.4.4.2
Acidity	3.3.3.2.1	3.3.4.4.2
Distillation	3.3.3.2.1	3.3.4.4.2
Color	3.3.3.2.1	3.3.4.4.2
Water	3.3.3.2.1	3.3.4.4.2
Chemical Formula	3.3.3.2.2	3.3.4.4.2
Molecular Weight	3.3.3.2.2	3.3.4.4.2

3.3.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of methyl ethyl ketone. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.3.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.3.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.3.3.2.1.

3.3.4.4.2 Tests for Physical and Chemical Properties. All tests for physical and chemical properties shall be determined in accordance with Interim Federal Specification TT-M-00261c and the results shall conform to Paragraph 3.3.3.2.1 and Paragraph 3.3.3.2.2.

3.4. Toluene

3.4.1 Scope

3.4.1.1 Scope. This specification covers the minimum requirements for toluene intended for use as a constituent material in the formulation of a thermal protection coating.

3.4.2 Applicable Documents

3.4.2.1 The following document forms a part of this specification:

	Federal Specification
TT-T-548c	Toluene, Technical

(Application for copies should be addressed to the General Services Administration Regional Offices)

3.4.3 Requirements

3.4.3.1 Qualification. The toluene furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.4.3.2 Properties

3.4.3.2.1 Physical Properties. The physical properties of the toluene shall be as listed in Table 5.

Table 5

Property	Requirement
Appearance	Clear and free from sediment and suspended matter
Acidity	No free acid
Color	No darker than 0.003 gram of reagent grade potassium dichromate in 1 liter distilled water
Sulfur Compounds	Free from hydrogen sulfide and sulfur dioxide
Specific Gravity, 20°/20°C	0.865 ± 0.005
Boiling Point at 760 mm, °C	109 min.
Dry Point at 760 mm, °C	113 max.
Total Distillate, % by volume	97 min.
Aromatic hydrocarbons, % by volume	96 min.

3.4.3.2.2 Chemical Properties. The chemical properties of the toluene are that the compound shall have the chemical formula $C_6H_5CH_3$ with a molecular weight of 92.13.

3.4.4 QUALITY ASSURANCE PROVISIONS

3.4.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.4.4.2 Classification of Examinations and Tests. The examination and testing of the toluene shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 6.

3.4.4.3 Sampling

3.4.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.4.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.4.4.3.2.1 Lot and Batch. All toluene manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.4.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of toluene. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.4.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.4.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.4.3.2.1.

Table 6

Examination and Tests

Examination or Tests	Requirement (Section 3.4.3)	Test Method (Section 3.4.4)
Qualification Tests		
Appearance	3.4.3.2.1	3.4.4.4.1
Acidity	3.4.3.2.1	3.4.4.4.2
Color	3.4.3.2.1	3.4.4.4.2
Sulfur Compounds	3.4.3.2.1	3.4.4.4.2
Specific Gravity	3.4.3.2.1	3.4.4.4.2
Boiling Point	3.4.3.2.1	3.4.4.4.2
Dry Point	3.4.3.2.1	3.4.4.4.2
Total Distillate	3.4.3.2.1	3.4.4.4.2
Aromatic Hydrocarbons	3.4.3.2.1	3.4.4.4.2
Chemical Formula	3.4.3.2.2	3.4.4.4.2
Molecular Weight	3.4.3.2.2	3.4.4.4.2

3.4.4.4.2 Tests for Physical and Chemical Properties. All tests for physical and chemical properties shall be determined in accordance with Federal Specification TT-T-548c and the results shall conform to Paragraph 3.4.3.2.1 and Paragraph 3.4.3.2.2

3.5 Butyl Acetate

3.5.1 Scope

3.5.1.1 Scope. This specification covers the minimum requirements for butyl acetate intended for use as a constituent material in the formulation of a thermal protection coating.

3.5.2 Applicable Documents

3.5.2.1 The following document forms a part of this specification:

Interim Federal Specification

TT-B-838a	Butyl Acetate, Normal

(Application for copies should be addressed to the General Services Administration Regional Offices)

3.5.3 Requirements

3.5.3.1 Qualification. The butyl acetate furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.5.3.2 Properties

3.5.3.2.1 Physical Properties. The physical properties of the butyl acetate shall be as listed in Table 7.

3.5.3.2.2 Chemical Properties. The chemical properties of the butyl acetate are that the compound shall have the chemical formula $CH_3CO_2CH_2CH_2C_2H_5$, with a molecular weight of 116.16.

3.5.4 QUALITY ASSURANCE PROVISIONS

3.5.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

Table 7

Property	Requirement
Appearance	Clean and free from sediment and suspended matter
Ester Content, %	90 min. as butyl acetate
Specific Gravity, 20°/20°C	.875 ± 0.001
Nonvolatile matter from 100 ml., gram	0.005 max.
Acidity, percent as acetic acid	0.01 max.
Distillation Range	
initial B.P., °C	118 min.
Dry Point, °C	128 max.
Color, on the platinum cobolt scale	10

3.5.4.2 Classification of Examinations and Tests. The examination and testing of the butyl acetate shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 8.

3.5.4.3 Sampling

3.5.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.5.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.5.4.3.2.1 Lot and Batch. All butyl acetate manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.5.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of butyl acetate. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

Table 8

Examination and Tests

Examination or Test	Requirement (Section 3.3.3)	Test Method (Section 3.3.4)
Appearance	3.3.3.2.1	3.3.4.4.1
Alcohol Content	3.3.3.2.1	3.3.4.4.2
Specific Gravity	3.3.3.2.1	3.3.4.4.2
Nonvolatiles	3.3.3.2.1	3.3.4.4.2
Acidity	3.3.3.2.1	3.3.4.4.2
Distillation	3.3.3.2.1	3.3.4.4.2
Color	3.3.3.2.1	3.3.4.4.2
Water	3.3.3.2.1	3.3.4.4.2
Chemical Formula	3.3.3.2.2	3.3.4.4.2
Molecular Weight	3.3.3.2.2	3.3.4.4.2

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cont

3.5.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.5.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.5.3.2.1.

3.5.4.4.2 Tests for Physical and Chemical Properties. All tests for physical and chemical properties shall be determined in accordance with Interim Federal Specification TT-B-838a and the results shall conform to Paragraph 3.5.3.2.1 and Paragraph 3.5.3.2.2.

Preliminary Process and Application Specification
for 341 Void-Filling
Thermal Protection Coating

Chemical Research Projects Office

NASA-Ames Research Center

Moffett Field, California 94035

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Process and Application Specification for 341 Void-Filling Thermal Protection Coating

1.0 Scope

This specification covers the basic techniques of production and application as well as the minimum requirements for a void-filling thermal protection coating.

2.0 Classification

The void-filling thermal protection coating covered by this specification shall be classified as follows:

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3.0 Applicable Documents

The following documents form a part of the specification:

Military

MIL-T-5624	Turbine Fuel, Aviation, Grades JP4 and JP5
MIL-A-8625	Anodic Coatings for Aluminum and Aluminum Alloys
MIL-C-5541	Chemicals, Films and Chemical Film Materials for Aluminum and Aluminum Alloys.

Federal

TT-P-666B	Primer Coating, Zinc Yellow for Aluminum and Magnesium Surfaces
TT-P-664b	Primer Coating, Synthetic Rust-Inhibiting, Lacquer-Resisting.
TT-P-86e, Type 3	Paint, Red Lead Base, Ready Mixed
TT-L-0058c, Type	Lacquer, Spraying Clear

Other

Preliminary Material Specification for 341 Void-Filling Thermal Protection Coating

Taber Abraser Model 503 Instruction Manual

American Society for Testing and Materials

ASTM D 2370 Elongation and Tensile Strength of Free Films of Paint, Varnish, Lacquer, and Related Products, with a Tensile Testing Apparatus

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103)

4.0 Requirements

4.1 Qualification. The void-filling thermal protection coating furnished under the specification shall be a product which has been tested and has passed the qualification tests specified herein.

4.2 Material. The void-filling thermal protection coating furnished under this specification shall conform and shall be prepared using materials approved under Preliminary Material Specification for 341 Void-Filling Thermal Protection Coating in accordance to the following formulation:

Materials	Parts by Dry Weight
Part A	
Nitrocellulose, Ethanol wet	8.01
Methyl Ethyl Ketone	28.63
Part B	
Para-nitroaniline bisulfate	43.67
Toluene	19.69
Butyl Acetate	19.69

4.3 Properties

4.3.1 Physical Properties

4.3.1.1 Appearance. The appearance of the void-filling thermal protection coating shall be a brownish yellow, hard, integral film when examined as specified.

4.3.1.2 Tensile Strength. The tensile strength of the void-filling thermal protection coating shall be 850 ± 100 psi when tested as specified.

4.3.1.3 Density. The density of the void-filling thermal protection coating shall be 1.71 ± 0.05 g/cc when tested as specified.

4.3.1.4 Temperature of Intumescence. The temperature of intumescence of the void-filling protection coating shall be 207 ± 10°C when tested as specified.

4.3.1.5 Char Yield. The char yield of the void-filling thermal protection coating at 600°C shall be 35 ± 2 percent when tested as specified.

4.3.1.6 Abrasion Resistance. The abrasion resistance of the void-filling thermal protection coating shall be 74 ± 10 milligram weight loss/1000 cycles on Taber Abraser when tested as specified.

4.3.1.7 Flexibility. The flexibility of the void-filling thermal protection coating shall be 0.66 ± 0.1 foot-pounds when tested as specified.

4.3.1.8 Linear Expansion (Intumescence). The linear expansion of the void-filling thermal protection coating conditioned at 23 ± 2°C and 50 ± 5% R.H. shall be a minimum of 200 times the original coating thickness when tested as specified.

4.3.1.9 Thermal Efficiency. The thermal efficiency of the void filling thermal protection coating shall be a minimum of 40 seconds at 0.030 + 0.002 inches coating thickness to reach a backface temperature of 205°C (400°F) when tested as specified.

5.0 Dispersion Equipment

Pebble Mill. A burundum fortified pebble mill, burundum cylinders as grinding media, and variable speed roll mill capable of dispensing the required batch sizes

Machine Mixing. A Kady Mill or equivalent kinetic dispersion unit.

6.0 Mixing Procedure

Pebble Mill: The grinding media of burundum cylinders is to occupy 50% of total pebble mill volume. Add ingredients of Paragraph 4.2, sufficient to occupy 40% of total pebble mill volume. Next, the critical speed (N_c) in RPM is calculated from following relationship:

$$N_c = \frac{76.6}{\sqrt{I.D.}}$$

where N_c = speed at which grinding media, unencumbered by any charge within mill, begin to centrifuge

I.D. = inside diameter of mill in feet

The pebble mill is placed on the variable speed roll mill at a mixing speed of 70% of Critical Speed (N_c) for proper action of grinding media. After mixing for 1/2 hour the pebble mill is re-opened and inspected for dry agglomerates. If none present, continue milling for 20 hours total mixing time. Drain contents of pebble mill into appropriate container. Strain the coating through a cheese cloth filter to remove any extraneous matter prior to any application.

Machine Mixing: Add ingredients of Paragraph 4.2 to the jacketed mixing vessel of the Kady Mill. Mix the components in a water cooled mixing vessel in the Kady Mill for 20 minutes. Drain the contents of the Kady Mill dispersion into an appropriate container. Strain the coating through a cheese cloth filter to remove any extraneous matter prior to any application.

7.0 Application Procedure

Substrate Preparation

Cold Rolled Steel: Follow standard good painting procedures. Be sure all surfaces are dry and clean. Remove loose dust, paint, grease, and oil residue from untreated surface. Surface may be sandblasted if desirable. Surface may be treated with corrosion resistant primer Zinc Chromate Primer meeting Federal Specification TT-P-664b or Red Lead Primer 13-R-51 from Mobil Chemical Company conforming to Federal Specification TT-P-86e Type 3.

Aluminum: Follow standard good painting procedures. Treat surface of aluminum by scrubbing with scouring powder and water. Flush liberally with water and observe for water break-free surface. If surface is not water break-free, repeat scrubbing and flushing procedure. When water break-free surface is obtained, flush surface with trichloroethylene and allow surface to air dry. Handle cleaned surface with care. Avoid contact with dust, oils, and greases. If corrosion resistance primer is desired, use Zinc Chromate Primer conforming to Federal Specification TT-P-666B. If anodizing of aluminum is required, the treatment shall conform to MIL-A-8625. For an alodine treated surface, the aluminum shall conform to MIL-C-5541.

Spray Method: Spray apply the 341 prepared from Paragraph 6.0 with a Devilbiss JGA 502 or equivalent at ambient conditions to the dry film thickness desired. The coating shall air dry at ambient conditions for 72 hours minimum. Spray apply two passes of a clear nitrocellulose lacquer conforming to Federal Specification TT-L-0058c, Type 1 as a topcoat. Allow to air dry for 24 hours minimum before proceeding with Quality Assurance Provisions.

Brush Method: Substrate preparation shall conform to Paragraph 7.0. Brushing may be done with a good quality soft bristled brush. Apply a full level coat avoiding excessive brush-out. Two brush coats or more may be required to conform to required dry film thickness. The coating shall airdry at ambient conditions for 72 hours minimum. Spray apply two passes of a clear nitrocellulose lacquer conforming to Federal Specification TT-L-0058c, Type 1 as a topcoat. Allow to air dry for 24 hours minimum before proceeding with Quality Assurance Provisions.

8.0 Quality Assurance Provisions

8.1 Responsibility for Inspection. Unless otherwise specified in the contract or the purchase order, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The processing activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

8.2 Classification of Examinations and Tests. All of the examinations and testing of the void-filling thermal protection coating shall be classified as qualification tests. Qualification tests shall be performed on samples submitted for approval as qualified products and shall be performed on individual lots of products submitted for acceptance. The qualification tests are listed in Table I. A certified test report from the supplier shall accompany each product submitted for approval and each lot of void-filling thermal protection coating submitted for acceptance.

8.3 Sampling. The sample for product qualification or for qualified lot acceptance shall be as specified in Paragraph 8.3.2.1 conforming to the requirements of Paragraph 4.2 herein. A test report from the supplier shall accompany each sample to be tested, and shall show the composition number and the test results for all requirements of this specification and shall refer specifically to the applicable paragraphs herein. All the void-filling thermal protection coating manufactured and formulated as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

8.3.1 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of void-filling thermal protection coating. If the results of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

Table 1

Examination or Test	Requirement	Test Method
Material	4.2	8.3.3.1
Appearance	4.3.1.1	8.3.3.1
Tensile Strength	4.3.1.2	8.3.3.2
Density	4.3.1.3	8.3.3.3
Temperature of Intumescence	4.3.1.4	8.3.3.4
Char Yield	4.3.1.5	8.3.3.5
Abrasion Resistance	4.3.1.6	8.3.3.6
Flexibility	4.3.1.7	8.3.3.7
Linear Expansion	4.3.1.8	8.3.3.8
Thermal Efficiency	4.3.1.9	8.3.3.9

8.3.2 Specimen. All samples to be tested shall be coated with the void-filling thermal protection coating at a minimum dry film thickness of 0.030 inches on cold rolled steel panels or as free films.

8.3.2.1 Size and Number of Specimens. The size and number of specimens for conformance to each requirement is indicated in Table 2.

8.3.3 Test Methods. Unless otherwise specified, all examinations and tests shall be performed with a void filling thermal protection coating aged for a minimum of 170 hours at a temperature of 23 ± 2°C and a relative humidity of 50 ± 5 percent.

8.3.3.1 Visual Inspection. Visual inspection shall be performed to determine the appearance requirements of Paragraph 4.3.1.1 and Paragraph 4.2.

8.3.3.2 Tensile Strength. The tensile strength of the void-filling thermal protection coating shall be determined in accordance with ASTM D 2370, except the coating will be sprayed on a suitable temporary substrate, and the results shall conform to Paragraph 4.3.1.2.

8.3.3.3 Density. The density of the void-filling thermal protection protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.3:

Procedure: Material remaining from Paragraph 8.3.3.2 shall be used to determine the coating density. The volume of coating is measured by air pycnometer and then the weight is determined from an analytical balance. The density of the coating in gm/cc is reported.

8.3.3.4 Temperature of Intumescence. The temperature of intumescence of the void-filling thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.4:

Procedure: The temperature of intumescence is determined by a melting point apparatus. Here glass capillary tubes containing small quantities of coating are raised in temperature until intumescence. After an initial rapid rate of temperature rise to determine approximate intumescing temperature, the determination is made in duplicate at no greater than 5°C/min temperature rise thru the intumescing range.

Table 2

Test Specimens

Test	Substrate Size, inches	Number of Specimens	Requirement	Test Method
Appearance	3 x 3 x 1/16	1	4.3.1.1	8.3.3.1
Tensile Strength	6 x 6 free films	2	4.3.1.2	8.3.3.2
Density	use above	-	4.3.1.3	8.3.3.3
Temperature of Intumescence		-	4.3.1.4	8.3.3.4
Char Yield		-	4.3.1.5	8.3.3.5
Abrasion Resistance	4 1/8 x 1/16 disc with 1/4 hole in center	6	4.3.1.6	8.3.3.6
Flexibility	use above	-	4.3.1.7	8.3.3.7
Linear Expansion	3 x 3 x 1/16	4	4.3.1.8	8.3.3.8
Thermal Efficiency	3 x 3 x 1/16	4	4.3.1.9	8.3.3.9

Care should be exercised that the heat transfer to the thermometer on the apparatus is at a maximum. This can be assured by maintaining an integral coating of 1-2 aqueous solution of collodial graphite (Aqua-DAG) on the thermometer bulb.

8.3.3.5 Char Yield. The char yield of the void-filling thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.5:

Procedure: Using a thermogravemetric analysis (TGA) apparatus, record the weight loss versus temperature for the coating at a heating rate of 3°C/minute in a nitrogen atmosphere to 650°C. Report the char yield (Y_c) as the percent weight fraction remaining at 600°C.

8.3.3.6 Abrasion Resistance. The abrasion resistance of the void-filling thermal protection coating shall be determined according to Taber Abraser Weight Loss Method and the results shall conform to Paragraph 4.3.1.6.

8.3.3.7 Flexibility. The flexibility of the void-filling thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.7:

Procedure: Using a Gardner Impact Test, a round nosed, two pound impact rod is allowed to fall a calibrated distance (measured in feet) upon the coated side of the specimen panel. The foot-pounds of energy is increased until coating adhesion failure occurs. The greatest energy tolerable is reported in foot-pounds.

8.3.3.8 Linear Expansion (Intumescence). The linear expansion of the void-filling thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.8:

Procedure: The edges of the test specimens as well as a 1/8 inch perimeter overlap of the coating and the exposed cold rolled steel substrate shall be brushed with a red lead primer meeting Federal Specification TT-P-86e, Type 3 to minimize corrosion of uncoated edges. The coating specimens are placed coating side up in an oven preheated to 350°C and allowed to remain for 30 minutes. Gases should be vented from the oven. Remove after 30 minute period, allow to cool, then calculate the linear expansion:

$$\text{Linear Expansion} = \frac{Tf - Ti}{Ti}$$

where Tf = Thickness of coating after intumescence, inches

Ti = initial coating thickness, inches.

8.3.3.9 Thermal Efficiency. The thermal efficiency of the void-filling thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.9:

Procedure: This test exposes the coating to a fire environment where a total flux of 10 BTU/Ft^2-sec is 90 percent radiative in a slightly fuel rich condition. Aviation fuel (JP-4) is used to fire the unit. The specimens are prepared on special 3" x 3" x 1/16" cold rolled steel substrates. That is, a 1" diameter hole in the center of the panel has a 15/16" diameter 1/16" thick cold rolled steel disc cemented in place with epoxy novolac. This provides a higher thermal resistance to reduce thermal conductance effects from the edges of the specimen panel. The coated panel thickness is measured and recorded and the panel preconditioned to paragraph 8.3.3, or other special environments and when ready to test is inserted in a transite mask or holder which allows the panel to rest on about 1/16" to 1/8" inch of the edges of the coated surface. At time, t = 0, the masked panel is placed over the fire box and a thermocouple in contact with the back of the panel records the temperature rise of the center disc in the panel. The flux is uniform over the entire panel and the center disc is about 8 - 10% of the exposed area and is representative of a larger, semi-infinite panel. From the time-temperature history thus obtained, and the thickness in mils of coating tested, one reports the time in seconds to reach a given temperature and the mil thickness coating. Since the flame environment is a more severe test of the coatings, the intumescent thickness is also reported after this test.

PRELIMINARY MATERIAL SPECIFICATION

for

410-1A THERMAL PROTECTION COATING

CHEMICAL RESEARCH PROJECTS OFFICE
NASA - AMES RESEARCH CENTER
MOFFETT FIELD, CALIFORNIA 94035

FOREWORD

This specification has been prepared for an intumescent coating system developed at Ames Research Center that appears to be useful in fire-protection applications. Because of the potential usefulness of the coating system, we are releasing this specification for limited distribution, even though it is only a preliminary draft.

Chemical Research Projects Office
Ames Research Center
National Aeronautics and Space Administration

February 1970

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PRELIMINARY

MATERIAL SPECIFICATION

for

410-1A THERMAL PROTECTION COATING

1.0 SCOPE - This specification covers the components for producing 410-1A thermal protection coating.

2.0 FORMULATION

Components	Parts Weight	Source
Acrylonitrile-butadiene, phenolic modified compounded rubber solution (Plastilock 605-4)	29.37	B. F. Goodrich Company
Para-nitroaniline bisulfate	44.04	Burdick-Jackson Company
Cyclohexanone	14.44	DuPont Corp.
Methyl Ethyl Ketone	12.15	Mallinckrodt Chemical

3.0 INGREDIENTS

3.1 Acrylonitrile-butadiene, phenolic modified compounded rubber solution.

3.1.1. Scope

3.1.1.1 Scope. This specification covers the minimum requirements for an acrylonitrile-butadiene, phenol modified compounded rubber solution intended for use as a constituent material in the formulation of a thermal protection coating.

3.1.2 Applicable Documents

3.1.2.1 The following documents form a part of this specification:

American Society for Testing and Materials

ASTM D 1644 Tests for Nonvolatile Content of Varnishes

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

Federal Specifications

Federal Standard 141, Method 4184.1. Weight Per Gallon

(Application for copies should be addressed to General Services Administration Regional Offices.)

3.1.3 Requirements

3.1.3.1 Qualification. The acrylonitrile-butadiene, phenolic modified compounded rubber solution furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.1.3.2 Properties

3.1.3.2.1 Physical Properties

3.1.3.2.1.1 Appearance. The appearance of the acrylonitrile butadiene, phenolic modified compounded rubber solution shall be a black, pourable, methyl ethyl ketone thinner base solution when examined as specified.

3.1.3.2.1.2 Nonvolatiles. The nonvolatiles of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be 33% minimum when tested as specified.

3.1.3.2.1.3 Weight Per Gallon. The weight per gallon of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be 8.3 ± 0.2 when tested as specified.

3.1.3.2.1.4 Viscosity. The viscosity of the acrylonitrile butadiene, phenolic modified compounded rubber solution shall be 7000 ± 1000 centipoises when tested as specified.

3.1.3.2.2 Chemical Properties

3.1.3.2.2.1 Phenolic Resin Content. The phenolic resin content of the total dry solids of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be 17.5 ± 1 percent when tested as specified.

3.1.3.2.2.2 Nitrile Rubber Content. The nitrile rubber content of the total dry solids of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be 54.8 ± 1 percent when tested as specified.

3.1.3.2.2.3 Filler Content. The filler content of the total dry solids of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be 27.7 ± 1 percent when tested as specified.

3.1.3.3 Shelf Life. After storage for two months from date of manufacture under specified conditions, the acrylonitile-butadiene, phenolic modified compounded rubber solution shall be capable of passing all the quality conformance tests specified.

3.1.4 Quality Assurance Provisions

3.1.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.1.4.2 Classification of Examinations and Tests. The examination and testing of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 1.

3.1.4.3 Sampling

3.1.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.1.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

TABLE I

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.1.3)	Test Method (Section 3.1.4)
Qualification Tests		
Appearance	3.1.3.2.1.1	3.1.4.4.1
Nonvolatiles	3.1.3.2.1.2	3.1.4.4.2
Weight Per Gallon	3.1.3.2.1.3	3.1.4.4.3
Viscosity	3.1.3.2.1.4	3.1.4.4.4
Phenolic Resin Content	3.1.3.2.2.1	3.1.4.4.5
Nitrile Rubber Content	3.1.3.2.2.2	3.1.4.4.6
Filler Content	3.1.3.2.2.3	3.1.4.4.7
Quality Conformance Tests		
Appearance	3.1.3.2.1.1	3.1.4.4.1
Viscosity	3.1.3.2.1.4	3.1.4.4.4
Phenolic Resin Content	3.1.3.2.2.1	3.1.4.4.5
Nitrile Rubber Content	3.1.3.2.2.2	3.1.4.4.6
Filler Content	3.1.3.2.2.3	3.1.4.4.7

3.1.4.3.2.1 Lot and Batch. All acrylonitrile-butadiene, phenolic modified compounded rubber solution manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.1.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of acrylonitrile-butadiene, phenolic modified compounded rubber solution. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.1.4.4. Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.1.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.1.3.2.1.1.

3.1.4.4.2 Nonvolatiles. The nonvolatiles of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be determined in accordance with ASTM D 1644 and the result shall conform to the requirements of Paragraph 3.1.3.2.1.2.

3.1.4.4.3 Weight Per Gallon. The weight per gallon of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be determined in accordance with Federal Standard 141, Method 4184.1 and the result shall conform to the requirement of Paragraph 3.1.3.2.1.3.

3.1.4.4.4 Viscosity. The viscosity of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be determined by a Brookfield Viscometer, Model RVF or equivalent, using a number 3 spindle at a speed of 10 revolutions per minute. The result shall conform to the requirement of Paragraph 3.1.3.2.1.4.

3.1.4.4.5 Phenolic Resin Content. The phenolic resin content of the total dry solids of the acrylonitrile-butadiene, phenolic modified rubber solution shall be determined as follows and the results shall conform to Paragraph 3.1.3.2.2.1:

Procedure: A two (2) to three (3) gram specimen of the rubber solution is weighed in a beaker on an analytical balance to the nearest 0.1 mg. and diluted to 150 cc total volume with methyl ethyl ketone. After stirring to a uniform consistency, the dispersion was transferred quantitatively to a 1000 ml separatory funnel and an equal volume of 10% NaOH (aq.) added. The mixture was thoroughly shaken for 5 minutes and allowed to stand to effect phase separation. The clear lower aqueous layer was drained into a 250 ml Erlenmeyer flask and acidified to a pH of about 2 with concentrated HCl. A brownish emulsion immediately formed which finally broke after allowing it to stand for 24 hours. The phenolic resin, in the form of droplets on the bottom of the flask, was recovered by decantation of the supernatant liquid. The phenolic resin was washed with three (3) 100 ml alequots of hot distilled water, and after final decantation, was dried in an oven at 110°C for 15 minutes. The resin was then weighed on an analytical balance to the nearest 1 mg.

Calculation:

$$\text{\% Phenolic Resin (total basis)} = \frac{WP}{W} \times 100$$

$$\text{\% Phenolic Resin (dry solids basis)} = \frac{WP}{WS} \times 100$$

$$\text{\% Phenolic Resin (resin basis)} = \frac{WP}{W(S=F)} \times 100$$

where:

WP = weight of phenolic resin recovered by estraction

W = original weight of sample

$$S = \text{solids content fraction} = \frac{\text{\% solids content}}{100}$$

$$F = \text{filler content fraction} = \frac{\text{\% filler content}}{100}$$

3.1.4.4.6 Nitrile Rubber Content. The nitrile rubber content of the total dry solids of the acrylonitrile-butadiene, phenolic modified rubber solution shall be determined as follows and the results shall conform to Paragraph 3.1.3.2.2.2:

Procedure: The nitrile rubber content in the rubber solution formulation is determined by difference. The assumption made is that the only other major constituents of the formulation are inorganic filler, methyl ethyl ketone and phenolic resin.

Calculation:

$$\text{\% Nitrile Resin (total basis)} = (S-P-F)100$$

$$\text{\% Nitrile Resin (dry solids basis)} = \frac{(S-P-F)}{S}100$$

$$\text{\% Nitrile Resin (resin basis)} = \left(1 - \frac{P}{S-F}\right)100 = \frac{(S-P-F)}{(S-F)}100$$

where:

S= solids content fraction

P= phenolic resin content fraction (total basis)

F= filler content fraction (total basis)

3.1.4.4.7 Filler Content. The filler content of the total dry solids of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be determined as follows and the results shall conform to Paragraph 3.1.3.2.2.3:

Procedure: A two (2) to three (3) gram specimen of the formulation is weighed on an analytical balance to the nearest 0.1 mg and diluted to 100 cc total volume with methyl ethyl ketone. After stirring to a uniform consistency, the dispersion was filtered under vacuum through a pre-weighed and dried fritted glass crucible containing about 3/8" thickness of packed "Celite" (medium fineness diatomaceous silica) filter aid. The insoluble residue was then washed with three (3) 25cc aliquots of methyl ethyl ketone and dried in an oven at 110°C for 1 hour. The tared crucible, filter aid, and residue were then weighed on an analytical balance to the nearest 0./mg.

Calculation:

% Filler Content (insoluble) (total basis)

$$= \frac{WF}{W} \times 100$$

% Filler Content (dry solids basis) $= \frac{WF}{WS} \times 100$

where:

WF = weight of residue

W = original weight of sample

S = solids content fraction $= \frac{\text{\% solids content}}{100}$

3.1.4.5 Shelf Life: The shelf life of the acrylonitrile-butadiene, phenolic modified compounded rubber solution shall be determined after storage for two months from date of manufacture, in original sealed containers, at a temperature of 65 ± 15°F. An alternative method shall be to store the liquid under the same time and temperature conditions in an opened container with an inert atmosphere. The stored material shall be tested in accordance with Paragraphs 3.1.4.4.1, 3.1.4.4.4, 3.1.4.4.5, 3.1.4.4.6 and 3.1.4.4.7. The results shall conform to the requirements of Paragraph 3.1.3.3 herein.

3.2 Para-nitroaniline bisulfate

3.2.1 Scope

3.2.1.1 Scope. This specification covers the minimum requirements for para-nitroaniline bisulfate intended for use as a constituent material in the formulation of a thermal protection coating.

3.2.2 Applicable Documents

3.2.2.1 The following documents form a part of this specification:

None

3.2.3 Requirements

3.2.3.1 Qualification. The para-nitroaniline bisulfate furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.2.3.2 Material. The para-nitroaniline bisulfate furnished under this specification shall produce the Infrared (IR) Spectral configuration as indicated in Figure 1, when examined as specified.

3.2.3.3 Properties

3.2.3.3.1 Physical Properties

3.2.3.3.1.1 Appearance. The appearance of the para-nitro-aniline bisulfate shall be a pale gray-yellow, free flowing granular solid when examined as specified.

3.2.3.3.2 Chemical Properties

3.2.3.3.2.1 Equivalent Weight. The equivalent weight of the para-nitroaniline bisulfate shall be 236±5 when examined as specified.

3.2.3.3.2.2 Decomposition Temperature. The decomposition temperature of the para-nitroaniline bisulfate shall be 200 ± 5 when examined as specified.

3.2.3.3.2.3 Purity. The purity of the para-nitroaniline bisulfate shall be a minimum of 95 percent when tested as specified.

3.2.4 Quality Assurance Provisions

3.2.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.2.4.2 Classification of Examinations and Tests. The examination and testing of the para-nitroaniline bisulfate shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 2.

3.2.4.3 Sampling

3.2.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.2.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.2.4.3.2.1 Lot and Batch. All para-nitroaniline bisulfate manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.2.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of para-nitroaniline bisulfate. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.2.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of $75 \pm 5^{\circ}F$ with a relative humidity not exceeding 60 percent.

3.2.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.2.3.3.1.1, the IR spectra requirements of Paragraph 3.2.3.2.

3.2.4.4.2 Equivalent Weight. The equivalent weight of the para-nitroaniline bisulfate shall be determined as follows and the results shall conform to Paragraph 3.2.3.3.2.1:

TABLE II

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.2.3)	Test Method (Section 3.2.4)
Qualification Tests		
Material	3.2.3.2	3.2.4.4.1
Appearance	3.2.3.3.1.1	3.2.4.4.1
Equivalent Weight	3.2.3.3.2.1	3.2.4.4.2
Decomposition Temperature	3.2.3.3.2.2	3.2.4.4.3
Purity	3.2.3.3.2.3	3.2.4.4.4
Quality Conformance Tests		
Material	3.2.3.2	3.2.4.4.1
Appearance	3.2.3.3.1.1	3.2.4.4.1
Equivalent Weight	3.2.3.3.2.1	3.2.4.4.2
Decomposition Temperature	3.2.3.3.2.2	3.2.4.4.3
Purity	3.2.3.3.2.3	3.2.4.4.4

Procedure: Weigh out 0.23 gm of para-nitroaniline bisulfate. Suspend or dissolve the material in 50 to 100 ml of aqueous methanol. Titrate with 0.1N NaOH using a recording electrometric titrator. From the volume of the titrant to reach endpoint, calculate the endpoint.

3.2.4.4.3 Decomposition Temperature. The decomposition temperature of the para-nitroaniline bisulfate shall be determined as follows and the results shall conform to Paragraph 3.2.3.3.2.2:

Procedure: The decomposition temperature is the temperature range observed during maximum weight loss. A thermogravimetric analysis apparatus recording weight loss versus temperature at a heating rate of 3°C/minute to 300°C in nitrogen atmosphere will be required.

3.2.4.4.4 Purity. The purity of the para-nitroaniline bisulfate shall be determined by elemental carbon, hydrogen, nitrogen (CHN). Analysis and the results shall conform to Paragraph 3.2.3.3.2.3.

3.3 Methyl Ethyl Ketone

3.3.1 Scope

3.3.1.1 Scope. This specification covers the minimum requirements for methyl ethyl ketone intended for use as a constituent material in the formulation of a thermal protection coating.

3.3.2 Applicable Documents

3.3.2.1 The following document forms a part of this specification:

Interim Federal Specification

TT-M-00261c	Methyl Ethyl Ketone

(Application for copies should be addressed to the General Services Administration Regional Offices.)

3.3.3 Requirements

3.3.3.1 Qualification. The methyl ethyl ketone furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.3.3.2 Properties.

3.3.3.2.1 Physical Properties. The physical properties of the methyl ethyl ketone shall be as listed in Table 3.

TABLE III

Property	Requirement
Appearance	Clean and free from sediment and suspended matter
Alcohol Content, %	0.7 max. as secondary butyl alcohol
Specific Gravity, $20^\circ/20^\circ$	0.806 ± 0.001
Nonvolatile matter from 100 ml., gram	0.0005 max.
Acidity, mg of KOH per gram of sample	0.047 max.
Distillation:	
Below 78.5	None
Above 81°C	None
Color, on the platinum cobalt scale	20
Water, percent by weight	0.3

3.3.4 Quality Assurance Provisions

3.3.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.3.4.2 Classification of Examinations and Tests. The examination and testing of the methyl ethyl ketone shall be classified as follows:

a. Qualification Tests. The qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 4.

3.3.4.3 Sampling

3.3.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.3.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.3.4.3.2.1 Lot and Batch. All methyl ethyl ketone manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.3.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of methyl ethyl ketone. If the results of test of the second sample fails to meet the specification requirements, the entire lot

TABLE IV

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.3.3)	Test Method (Section 3.3.4)
Appearance	3.3.3.2.1	3.3.4.4.1
Alcohol Content	3.3.3.2.1	3.3.4.4.2
Specific Gravity	3.3.3.2.1	3.3.4.4.2
Nonvolatiles	3.3.3.2.1	3.3.4.4.2
Acidity	3.3.3.2.1	3.3.4.4.2
Distillation	3.3.3.2.1	3.3.4.4.2
Color	3.3.3.2.1	3.3.4.4.2
Water	3.3.3.2.1	3.3.4.4.2
Chemical Formula	3.3.3.2.2	3.3.4.4.2
Molecular Weight	3.3.3.2.2	3.3.4.4.2

shall be rejected.

3.3.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of $75 \pm 5^{\circ}$F with a relative humidity not exceeding 60 percent.

3.3.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.3.3.2.1.

3.3.4.4.2 Tests for Physical and Chemical Properties. All tests for physical and chemical properties shall be determined in accordance with Interim Federal Specification TT-M-0026/c and the results shall conform to Paragraph 3.3.3.2.1 and Paragraph 3.3.3.2.2.

3.4 Cyclohexanone

3.4.1 Scope

3.4.1.1 Scope. This specification covers the minimum requirements for cyclohexanone intended for use as a constituent material in the formulation of a thermal protection coating.

3.4.2 Applicable Documents

3.4.2.1 The following documents form a part of this specification:

American Society for Testing and Materials

ASTM D 1209	Test for Color of Clear Liquids
ASTM D 1078	Test for Distillation Range of Volatile Organic Liquids
ASTM D 1364	Test for Water in Lacquer Solvents and Diluents
ASTM D 891	Tests for Specific Gravity of Industrial Aromatic Hydrocarbons and Related Materials

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

Federal Specifications

Federal Standard 141, Method 4371. Refractive Index

(Application for copies should be addressed to General Services Administration Regional Offices.)

3.4.3 Requirements

3.4.3.1 Qualification. The cyclohexanone furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.4.3.2 Properties

3.4.3.2.1 Physical Properties

3.4.3.2.1.1 Appearance. The appearance of the cyclohexanone shall be a clear, water white to slight yellow liquid when examined as specified.

3.4.3.2.1.2 Color. The color of the cyclohexanone shall be A.P.H.A.3 when tested as specified.

3.4.3.2.1.3 Distillation Range. The distillation range of the cyclohexanone shall be 0.7°C when tested as specified.

3.4.3.2.1.4 Water. The water content of the cyclohexanone shall be 0.01% maximum when tested as specified.

3.4.3.2.1.5 Specific Gravity. The specific gravity of the cyclohexanone shall be 0.9448 ± 0.0001 when tested as specified.

3.4.3.2.1.6 Refractive Index. The refractive index of the cyclohexanone shall be 1.4476 ± 0.0004 when tested as specified.

3.4.3.3 Shelf Life. After storage for two months from date of manufacture under specified conditions, the cyclohexanone shall be capable of passing all the quality conformance tests required.

3.4.4 QUALITY ASSURANCE PROVISIONS

3.4.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.4.4.2 Classification of Examinations and Tests. The examination and testing of the cyclohexanone shall be classified as follows:

a Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 5.

TABLE V

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.4.3)	Test Method (Section 3.4.4.)
Qualification Tests		
Appearance	3.4.3.2.1.1	3.4.4:4.1
Color	3.4.3.2.1.2	3.4.4.4.2
Distillation Range	3.4.3.2.1.3	3.4.4.4.3
Water	3.4.3.2.1.4	3.4.4.4.4
Specific Gravity	3.4.3.2.1.5	3.4.4.4.5
Refractive Index	3.4.3.2.1.6	3.4.4.4.6
Quality Conformance Tests		
Appearance	3.4.3.2.1.1	3.4.4.4.1
Color	3.4.3.2.1.2	3.4.4.4.2
Distillation Range	3.4.3.2.1.3	3.4.4.4.3
Water	3.4.3.2.1.4	3.4.4.4.4
Specific Gravity	3.4.3.2.1.5	3.4.4.4.5
Refractive Index	3.4.3.2.1.6	3.4.4.4.6

3.4.4.3 Sampling

3.4.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.4.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.4.4.3.2.1 Lot and Batch. All cyclohexanone manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.4.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of cyclohexanone. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.4.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.4.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.4.3.2.1.1.

3.4.4.4.2 Color. The color of the cyclohexanone shall be determined in accordance with ASTM D 1209 and the result shall conform to the requirements of Paragraph 3.4.3.2.1.2.

3.4.4.4.3 Distillation Range. The distillation range of the cyclohexanone shall be determined in accordance with ASTM D 1078 and the results shall conform to the requirements of Paragraph 3.4.3.2.1.3.

3.4.4.4.4 Water. The water content of the cyclohexanone shall be determined in accordance with ASTM D 1364 and the results shall conform to the requirements of Paragraph 3.4.3.2.1.4.

3.4.4.4.5 Specific Gravity. The specific gravity of the cyclohexanone shall be determined in accordance with ASTM D 891 and the results shall conform to the requirements of Paragraph 3.4.3.2.1.5.

3.4.4.4.6 Refractive Index. The refractive index of the cyclohexanone shall be determined in accordance with Federal Standard 141, Method 4371 and the results shall conform to the requirements of Paragraph 3.4.3.2.1.6.

3.4.4.5 Shelf Life. The shelf life of the cyclohexanone shall be determined after storage for two months from date of manufacture, in original sealed containers, at a temperature of 65 ± 15°F. The stored material shall be tested in accordance with Paragraphs 3.4.4.4.1, 3.4.4.4.2, 3.4.4.4.4, 3.4.4.4.5, and 3.4.4.4.6. The results shall conform to the requirements of Paragraph 3.4.3.3 herein.

PRELIMINARY PROCESS AND APPLICATION SPECIFICATION

for

410-1A THERMAL PROTECTION COATING

CHEMICAL RESEARCH PROJECTS OFFICE
NASA - AMES RESEARCH CENTER
MOFFETT FIELD, CALIFORNIA 94035

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PRELIMINARY

PROCESS AND APPLICATION SPECIFICATION

for

410-1A THERMAL PROTECTION COATING

1.0 SCOPE - This specification covers the basic techniques of production and application as well as the minimum requirements for a thermal protection coating.

2.0 CLASSIFICATION - The thermal protection coating covered by this specification shall be classified as follows:

410-1A

3.0 APPLICABLE DOCUMENTS - The following documents form a part of this specification:

Military

MIL-T-5624	Turbine Fuel, Aviation, Grades JP4 and JP5
MIL-A-8625	Anodic Coatings for Aluminum and Aluminum Alloys
MIL-C-5541	Chemicals, Films, and Chemical Film Materials for Aluminum and Aluminum Alloys

Federal

TT-P-666B	Primer Coating, Zinc Yellow for Aluminum and Magnesium Surfaces
TT-P-664b	Primer Coating, Synthetic, Rust-Inhibiting, Lacquer-Resisting
TT-P-86e, Type 3	Paint, Red Lead Base, Ready Mixed
TT-L-0058e, Type 1	Lacquer, Spraying, Clear

Other

Preliminary Material Specification for 410-1A Thermal Protection Coating
Taber Abraser Model 503 Instruction Manual

American Society for Testing and Materials

ASTM D2370	Elongation and Tensile Strength of Free Films of Paint, Varnish, Lacquer, and Related Products, with a Tensile Testing Apparatus

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania, 19103).

4.0 REQUIREMENTS

4.1 Qualification. The thermal protection coating furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

4.2 Material. The thermal protection coating furnished under this specification shall conform and shall be prepared using materials approved under Preliminary Material Specification for 410-1A Thermal Protection Coating in accordance to the following formulation:

Materials	Parts by Weight
Acrylonitrile-butadiene, phenolic modified compounded rubber solution	29.37
Para-nitroaniline bisulfate	44.07
Cyclohexanone	14.44
Methyl Ethyl Ketone	12.15

4.3 Properties.

4.3.1 Physical Properties.

4.3.1.1 Appearance. The appearance of the thermal protection coating shall be a black, integral film when examined as specified.

4.3.1.2 Tensile Strength. The tensile strength of the thermal protection coating shall be 300 ± 25 psi when tested as specified.

4.3.1.3 Density. The density of the thermal protection coating shall be 1.34 ± 0.05 g/ce when tested as specified.

4.3.1.4 Temperature of Intumescence. The temperature of intumescence of the thermal protection coating shall be 165± 10°C when tested as specified.

4.3.1.5 Char Yield. The char yield of the thermal protection coating at 600°C shall be 50 ± 2 percent when tested as specified.

4.3.1.6 Abrasion Resistance. The abrasion resistance of the thermal protection coating shall be 105 ± 10 milligram weight loss/1000 cycles on Taber Abraser when tested as specified.

4.3.1.7 Flexibility. The flexibility of the thermal protection coating shall be 2.2 ± 0.1 foot-pounds when tested as specified.

4.3.1.8 Linear Expansion (Intumescence). The linear expansion of the thermal protection coating conditioned at 23 ± 2°C and 50 ± 5% R.H. shall be a minimum of 30 times the original coating thickness when tested as specified.

4.3.1.9 Thermal Efficiency. The thermal efficiency of the thermal protection coating shall be a minimum of 90 seconds at 0.030 + 0.002 inches coating thickness to reach a backface temperature of 205°C (400°F) when tested as specified.

5.0 DISPERSION EQUIPMENT

Pebble Mill. A burundum fortified pebble mill, burundum cylinders as grinding media, and variable speed roll mill capable of dispersing the required batch sizes.

Machine Mixing. A Kady Mill or equivalent kinetic dispersion unit.

6.0 MIXING PROCEDURE

Pebble Mill. The grinding media of burundum cylinders is to occupy 50% of total pebble mill volume. Add ingredients of Paragraph 4.2, sufficient to occupy 40% of total pebble mill volume. Next, the critical speed (N_c) in RPM is calculated from following relationship:

$$N_c = \frac{76.6}{\sqrt{\text{I.D.}}}$$

where N_c = speed at which grinding media, unencumbered by any change within mill, begin to centrifuge

I.D. = inside diameter of mill in feet

The pebble mill is placed on the variable speed roll mill at a mixing speed of 70% of Critical Speed (N_c) for proper action of grinding media. After mixing for 1/2 hour the pebble mill is re-opened and inspected for dry agglomerates. If none present, continue milling for 20 hours total mixing time. Drain contents of pebble mill into appropriate container. Strain the coating through a cheese cloth filter to remove any extraneous matter prior to any application.

Machine Mixing: Add ingredients of Paragraph 4.2 to the jacketed mixing vessel of the Kady Mill. Mix the components in a water cooled mixing vessel in the Kady Mill for 20 minutes. Drain the contents of the Kady Mill dispersion into an appropriate container. Strain the coating through a cheese cloth filter to remove any extraneous matter prior to any application.

7.0 APPLICATION PROCEDURE

Substrate Preparation

Cold Rolled Steel: Follow standard good printing procedures. Be sure all surfaces are dry and clean. Remove loose dust, paint, grease, and oil residue from untreated surface. Surface may be sandblasted if desirable. Surface may be treated with corrosion resistant primer Zinc Chromate Primer meeting Federal Specification TT-P-664b or Red Lead Primer 13-R-51 from Mobil Chemical Company conforming to Federal Specification TT-P-86e Type 3.

Aluminum: Follow standard good painting procedures. Treat surface of aluminum by scrubbing with scouring powder and water. Flush liberally with water and observe for water break-free surface. If surface is not water break-free, repeat scrubbing and flushing procedure. When water break-free surface is obtained, flush surface with trichloroethylene and allow surface to air dry. Handle cleaned surface with care. Avoid contact with dust, oils, and greases. If corrosion resistant primer is desired, use Zinc Chromate Primer conforming to Federal Specification TT-P-666B. If anodizing of aluminum is required, the treatment shall conform to MIL-A-8625. For an alodine treated surface, the aluminum shall conform to MIL-C-5541.

Spray Method: Spray apply 410-1A prepared from Paragraph 6.0 with a Devilbiss JGA 502 or equivalent at ambient conditions to the dry film thickness desired. The coating shall air dry at ambient conditions for 72 hours minimum. Spray apply two passes of a clear nitrocellulose lacquer conforming to Federal Specification TT-L-0058c, Type 1 as a topcoat. Allow to air dry for 24 hours minimum before proceeding with Quality Assurance Provisions.

Brush Method: Substrate preparation shall conform to Paragraph 7.0. Brushing may be done with a good quality soft bristled brush. Apply a full level coat avoiding excessive brush-out. Two brush coats or more may be required to conform to required dry film thickness. The coating shall air dry at ambient conditions for 72 hours minimum. Spray or brush apply two passes of a clear nitrocellulose lacquer conforming to Federal Specification TT-L-0058c, Type 1 as a topcoat. Allow to air dry for 24 hours minimum before proceeding with Quality Assurance Provisions.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection . Unless otherwise specified in the contract or the purchase order, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The processing activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

8.2 Classification of Examinations and Tests. All of the examinations and testing of the thermal protection coating shall be classified as qualification tests. Qualification tests shall be performed on samples submitted for approval as qualified products and shall be performed on individual lots of products submitted for acceptance. The qualification tests are listed in Table I. A certified test report from the supplier shall accompany each product submitted for approval and each lot of thermal protection coating submitted for acceptance.

Table 1

Examination or Test	Requirement	Test Method
Material	4.2	8.3.3.1
Appearance	4.3.1.1	8.3.3.1
Tensile Strength	4.3.1.2	8.3.3.2
Density	4.3.1.3	8.3.3.3
Temperature of Intumescence	4.3.1.4	8.3.3.4
Char Yield	4.3.1.5	8.3.3.5
Abrasion Resistance	4.3.1.6	8.3.3.6

Table 1 Continued

Examination or Test	Requirement	Test Method
Flexibility	4.3.1.7	8.3.3.7
Linear Expansion	4.3.1.8	8.3.3.8
Thermal Efficiency	4.3.1.9	8.3.3.9

8.3 Sampling. Samples for product qualification or for qualified lot acceptance shall be as specified in Paragraph 8.3.2. The material preparation shall conform to the requirements of Paragraph 4.2 herein. A test report from the supplier shall accompany each sample to be tested, and shall show the Composition number and the test results for all requirements of this specification and shall refer specifically to the applicable paragraphs herein. All the thermal protection coating manufactured and formulated as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

8.3.1 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of thermal protection coating. If the results of the second sample fails to the specification requirements, the entire lot shall be rejected.

8.3.2 Specimen. All samples to be tested shall be coated with the thermal protection coating at a minimum dry film thickness of 0.030 inches on cold rolled steel panels or as free films.

8.3.2.1 Size and Number of Specimens. The size and number of specimens for conformance to each requirement is indicated in Table 2.

8.3.3 Test Methods. Unless otherwise specified, all examinations and tests shall be performed with a thermal protection coating aged for minimum of 170 hours at a temperature of 23 ± 2°C and a relative humidity of 50 ±5percent.

8.3.3.1 Visual Inspection. Visual inspection shall be performed to determine the appearance requirements of Paragraph 4.3.1 and Paragraph 4.2.

Table 2

Test Specimens

Test	Substrate Size, inches	Number of Specimens	Requirement	Test Method
Appearance	3 x 3 x 1/16	1	4.3.1.1	8.3.3.1
Tensile Strength	6 x 6 free films	2	4.3.1.2	8.3.3.2
Density	use above	-	4.3.1.3	8.3.3.3
Temperature of Intumescence		-	4.3.1.4	8.3.3.4
Char Yield		-	4.3.1.5	8.3.3.5
Abrasion Resistance	4 1/8 x 1/16 disc with 1/4 hole in center	6	4.3.1.6	8.3.3.6
Flexibility	use above	-	4.3.1.7	8.3.3.7
Linear Expansion	3 x 3 x 1/16	4	4.3.1.8	8.3.3.8
Thermal Efficiency	3 x 3 x 1/16	4	4.3.1.9	8.3.3.9

8.3.3.2 Tensile Strength. The tensile strength of the thermal protection coating shall be determined in accordance with ASTM D 2370, except the coating will be sprayed on a suitable temporary substrate, and the results shall conform to Paragraph 4.3.1.2.

8.3.3.3 Density. The density of the thermal protection protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.3:

Procedure: Material remaining from Paragraph 8.3.3.2 shall be used to determine the coating density. The volume of coating is measured by air pycnometer and then the weight is determined from an analytical balance. The density of the coating in gm/cc is reported.

8.3.3.4 Temperature of Intumescence. The temperature of intumescence of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.4:

Procedure: The temperature of intumescence is determined by a melting point apparatus. Here glass capillary tubes containing small quantities of coating are raised in temperature until intumescence. After an initial rapid rate of temperature rise to determine approximate intumescing temperature, the determination is made in duplicate at no greater than 5°C/min temperature rise thru the intumescing range.

Care should be exercised that the heat transfer to the thermometer on the apparatus is at a maximum. This can be assured by maintaining an integral coating of 1-2 aqueous solution of collodial graphite (Aqua-DAG) on the thermometer bulb.

8.3.3.5 Char Yield. The char yield of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.5:

Procedure: Using a thermogravemetric analysis (TGA) apparatus, record the weight loss versus temperature for the coating at a heating rate of 3°C/minute in a nitrogen atmosphere to 650°C. Report the char yield (Y_c) as the percent weight fraction remaining at 600°C.

8.3.3.6 Abrasion Resistance. The abrasion resistance of the thermal protection coating shall be determined according to Taber Abraser Weight Loss Method and the results shall conform to Paragraph 4.3.1.6.

8.3.3.7 Flexibility. The flexibility of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.7:

Procedure: Using a Gardner Impact Test, a round nosed, two pound impact rod is allowed to fall a calibrated distance (measured in feet) upon the coated side of the specimen panel. The foot-pounds of energy is increased until coating adhesion failure occurs. The greatest energy tolerable is reported in foot-pounds.

8.3.3.8 Linear Expansion (Intumescence). The linear expansion of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.8:

Procedure: The edges of the test specimens as well as a 1/8 inch perimeter overlap of the coating and the exposed cold rolled steel substrate shall be brushed with a red lead primer meeting Federal Specification TT-P-86e, Type 3 to minimize corrosion of uncoated edges. The coating specimens are placed coating side up in an oven preheated to 350°C and allowed to remain for 30 minutes. Gases should be vented from the oven. Remove after 30 minute period, allow to cool, then calculate the linear expansion:

$$\text{Linear Expansion} = \frac{T_f - T_i}{T_i}$$

where T_f = Thickness of coating after intumescence, inches

T_i = initial coating thickness, inches.

8.3.3.9 Thermal Efficiency. The thermal efficiency of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.9:

Procedure: This test exposes the coating to a fire environment where a total flux of 10 BTU/Ft^2-sec is 90 percent radiative in a slightly fuel rich condition. Aviation fuel (JP-4) is used to fire the unit. The specimens are prepared on special 3" x 3" x 1/16" cold rolled steel substrates. That is, a 1" diameter hole in the center of the panel has a 15/16" diameter 1/16" thick cold rolled steel disc cemented in place with epoxy novolac. This provides a higher thermal resistance to reduce thermal conductance effects from the edges of the specimen panel. The coated panel thickness is measured and recorded and the panel preconditioned to paragraph 8.3.3, or other special environments and when ready to test is inserted in a transite mask or holder which allows the panel to rest on about 1/16" to 1/8" inch of the edges of the coated surface. At time, t = 0, the masked panel is placed over the fire box and a thermocouple in contact with the back of the panel records the temperature rise of the center disc in the panel. The flux is uniform over the entire panel and the center disc is about 8-10% of the exposed area and is representative of a larger, semi-infinite panel. From the time-temperature history thus obtained, the thickness in mils of coating tested, one reports the time in seconds to reach a given temperature and the mil thickness coating. Since the flame environment is a more severe test of the coatings, the intumescent thickness is also reported after this test.

PRELIMINARY MATERIAL SPECIFICATION

for

45B3 THERMAL PROTECTION COATING

Chemical Research Projects Office

NASA - Ames Research Center

Moffett Field, California 94035

FOREWORD

This specification has been prepared for an intumescent coating system developed at Ames Research Center that appears to be useful in fire-protection applications. Because of the potential usefulness of the coating system, we are releasing this specification for limited distribution, even though it is only a preliminary draft.

Chemical Research Projects Office
Ames Research Center
National Aeronautics and Space Administration

February 1970

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cont

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PRELIMINARY MATERIAL SPECIFICATION

for

45B3 Thermal Protection Coating

1.0 SCOPE - This specification covers the components for producing 45B3 thermal protection coating.

2.0 FORMULATION

Components	Parts Weight	Source
2.1 Part A		
Polysulfide Liquid Polymer, LP-3	14.1	Thiokol Chemical Corp.
Ammonium Salt of 4-nitro aniline-2-sulfonic acid	57.0	Dow Chemical Co.
Methyl Ethyl Ketone	28.9	Mallinckrodt Chemical
2.2 Part B		
Epoxy Resin, Liquid	14.1	Shell Chemical Co.
Toluene	4.7	Mallinckrodt
2.3 Part C		
Tri(dimethylamino methyl) phenol	2.8	Rohm and Haas Co.
Toluene	2.8	Mallinckrodt Chemical

3.0 INGREDIENTS

3.1 Polysulfide Polymer, Liquid

3.1.1 Scope

3.1.1.1 Scope. This specification covers the minimum requirements for a liquid polysulfide polymer intended for use as a constituent material in the formulation of a thermal protection coating.

3.1.2 Applicable Documents

3.1.2.1 The following document forms a part of this specification.

Product Specification, Code LF-241	Thiokol Chemical Corporation

(Application for copies should be addressed to Thiokol Chemical Corporation, Chemical Operations, 780 N. Clinton Avenue, Trenton, New Jersey 08607).

3.1.3 Requirements

3.1.3.1 Qualification. The liquid polysulfide polymer furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.1.3.2 Material. The liquid polysulfide polymer furnished under this specification shall produce the Infrared (IR) Spectral configuration as indicated in Figure 1, when examined as specified.

3.1.3.3 Properties.

3.1.3.3.1 Physical Properties

3.1.3.3.1.1 Appearance. The appearance of the liquid polysulfide polymer shall be a clear amber liquid, free of phase separation and suspended particles when examined as specified.

3.1.3.3.1.2 Specific Gravity. The specific gravity of the liquid polysulfide polymer shall be 1.260 to 1.283 when tested as specified.

3.1.3.3.1.3 Color. The color of the liquid polysulfide polymer shall be 12 maximum (Hellige Scale) when tested as specified.

3.1.3.3.1.4 Viscosity. The viscosity of the liquid polysulfide polymer at 80°F shall be 950±250 centipoises when tested as specified.

3.1.3.3.2 Chemical Properties.

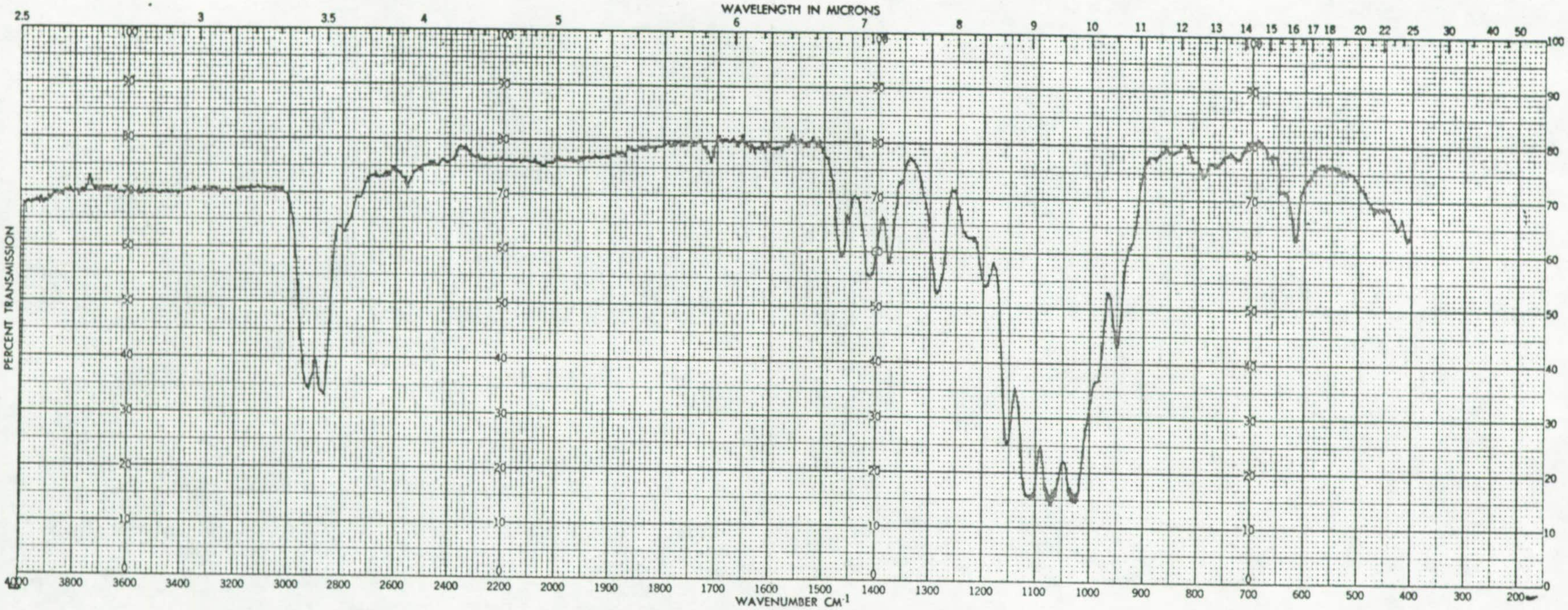

Figure 1. IR spectrum of liquid polysulfide polymer (as liquid between two salt plates).

3.1.3.3.2.1 pH (water extract). The pH of the liquid polysulfide polymer shall be in the range of 6.0 to 8.0 when tested as specified.

3.1.3.3.2.2 Moisture Content, %. The moisture content of the liquid polysulfide polymer shall be 0.1 maximum when tested as specified.

3.1.3.3.2.3 Mercaptan, %. The percent mercaptan of the liquid polysulfide polymer shall be 5.9 to 7.7 when tested as specified.

3.1.3.4 Shelf Life. After storage for two months from date of manufacture under specified conditions, the liquid polysulfide polymer shall be capable of passing all the quality tests specified.

3.1.4 Quality Assurance Provisions.

3.1.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity, of its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.1.4.2 Classification of Examinations and Tests. The examination and testing of the liquid polysulfide polymer shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table I.

3.1.4.3 Sampling.

3.1.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.1.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, lot number, date of sampling, and contract number.

3.1.4.3.2.1 Lot and Batch. All liquid polysulfide polymer manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.1.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of liquid polysulfide polymer. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.1.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F. with a relative humidity not exceeding 60 percent.

3.1.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.1.3.3.1.1, and the IR Spectra requirements of Paragraph 3.1.3.2. Visual inspection for appearance shall be performed by filling a small, clean, dry test tube with liquid polysulfide polymer and holding against a bright light.

3.1.4.4.2 Specific Gravity. The specific gravity of the liquid polysulfide shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform to the requirement of Paragraph 3.1.3.3.12.

3.1.4.4.3 Color. The color of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform to the requirement of Paragraph 3.1.3.3.1.3.

3.1.4.4.4 Viscosity. The viscosity of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform to the requirement of paragraph 3.1.3.3.1.3.

3.1.4.4.5. pH (water extract). The pH of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform to the requirement of paragraph 3.1.3.3.2.1.

TABLE I

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.1.3)	Test Method (Section 3.1.4)
Qualification Tests		
Material	3.1.3.2	3.1.4.4.1
Appearance	3.1.3.3.1.1	3.1.4.4.1
Specific Gravity	3.1.3.3.1.2	3.1.4.4.2
Color	3.1.3.3.1.3	3.1.4.4.3
Viscosity	3.1.3.3.1.4	3.1.4.4.4
pH (water extract)	3.1.3.3.2.1	3.1.4.4.5
Moisture Content	3.1.3.3.2.2	3.1.4.4.6
Mercaptan	3.1.3.3.2.3	3.1.4.4.7
Quality Conformance Tests		
Material	3.1.3.2	3.1.4.4.1
Appearance	3.1.3.3.1.1	3.1.4.4.1
Mercaptan	3.1.3.3.2.3	3.1.4.4.7

3.1.4.4.6 Moisture Content. The moisture content of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform to the requirement of Paragraph 3.1.3.3.2.2.

3.1.4.4.7 Mercaptan. The mercaptan percent of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the results shall conform to the requirement of Paragraph 3.1.3.3.2.3.

3.1.4.5 Shelf Life. The shelf life of the liquid polysulfide polymer shall be determined after storage for two months from date of manufacture, in original sealed containers, at a temperature of 65 ± 15°F. An alternative method shall be to store the liquid, polysulfide polymer, under the same time and temperature conditions in an opened container with an inert atmosphere. The stored material shall be tested in accordance with Paragraphs 3.1.4.4.1 and 3.1.4.4.7. The results shall conform to the requirements of Paragraph 3.1.3.4.

3.2 Ammonium salt of 4-nitroaniline-2-sulfonic acid

3.2.1 Scope

3.2.1.1 Scope. This specification covers the minimum requirements for an ammonium salt of 4-nitroaniline-2-sulfonic acid intended for use as a constituent material in the formulation of a thermal protection coating.

3.2.2 Applicable Documents

3.2.2.1 The following documents form a part of this specification.

NONE

3.2.3 Requirements

3.2.3.1 Qualification. The ammonium salt of 4-nitroaniline-2-sulfonic acid furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.2.3.2 Material. The ammonium salt of 4-nitroaniline-2-sulfonic acid furnished under this specification shall produce the Infrared (IR) Spectral configuration as indicated in Figure 2, when examined as specified.

3.2.3.3 Properties

3.2.3.3.1 Physical Properties

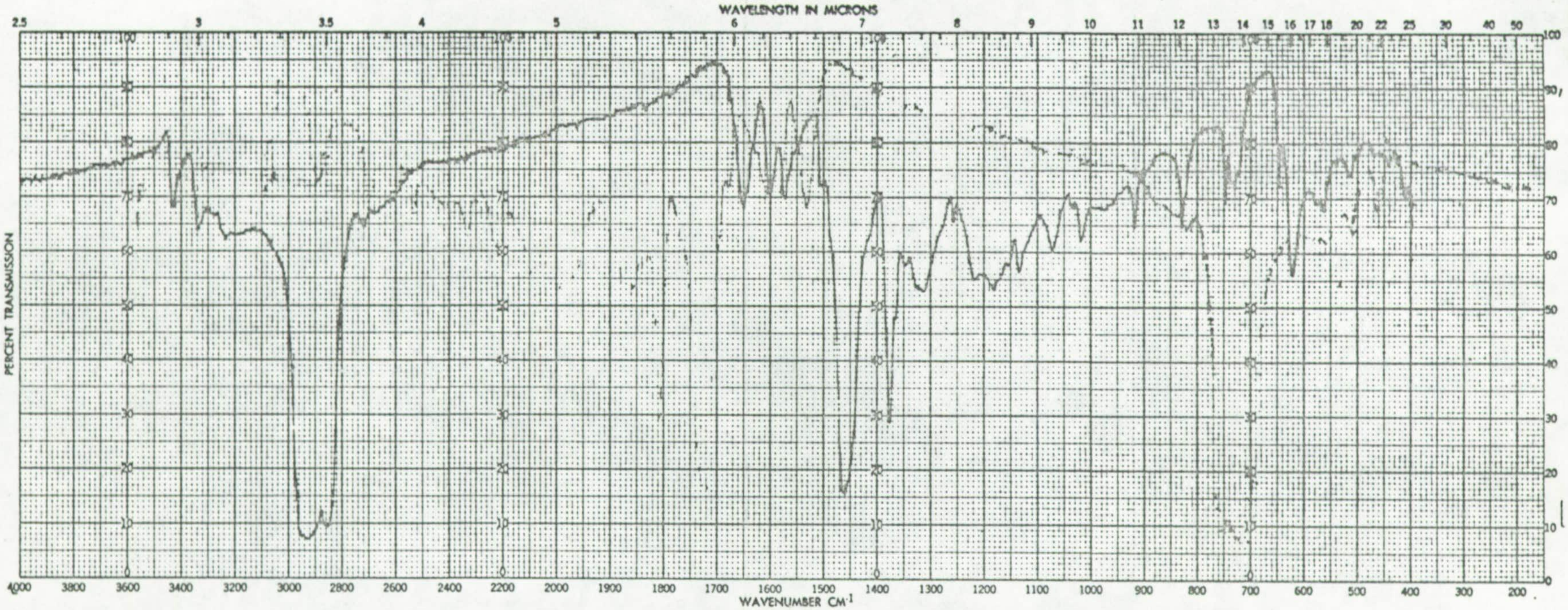

Figure 2. IR spectrum of ammonium salt of 4-nitroaniline-2-sulfonic acid (Nujol).

3.2.3.3.1.1 Appearance. The appearance of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be a yellow, free-flowing granular solid when examined as specified.

3.2.3.3.2 Chemical Properties

3.2.3.3.2.1 Equivalent Weight. The equivalent weight of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be 235 ± 5 when examined as specified.

3.2.3.3.2.2 Decomposition Temperature. The decomposition temperature of the ammonium salt of 4-nitroaniline-2- sulfonic acid shall be 325 ± 5°C when examined as specified.

3.2.3.3.2.3 Purity. The purity of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be a minimum of 95 percent when tested as specified.

3.2.4 Quality Assurance Provisions

3.2.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.2.4.2 Classification of Examinations and Tests. The examination and testing of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 2.

3.2.4.3 Sampling

3.2.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

TABLE 2

Examination and Tests

Examination or Test	Requirement (Section 3.2.3)	Test Method (Section 3.2.4)
Qualification Tests		
Material	3.2.3.2	3.2.4.4.1
Appearance	3.2.3.3.1.1	3.2.4.4.1
Equivalent Weight	3.2.3.3.2.1	3.2.4.4.2
Decomposition Temperature	3.2.3.3.2.2	3.2.4.4.3
Purity	3.2.3.3.2.3	3.2.4.4.4
Qualification Conformance Tests		
Material	3.2.3.2	3.2.4.4.1
Appearance	3.2.3.3.1.1	3.2.4.4.1
Equivalent Weight	3.2.3.3.2.1	3.2.4.4.2
Decomposition Temperature	3.2.3.3.2.2	3.2.4.4.3
Purity	3.2.3.3.2.3	3.2.4.4.4

3.2.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.2.4.3.2.1 Lot and Batch. All ammonium salt of 4-nitroaniline-2-sulfonic acid manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.2.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of ammonium salt of 4-nitroaniline-2-sulfonic acid. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.2.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.2.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.2.3.3.1.1 and the IR spectra requirements of Paragraph 3.2.3.2.

3.2.4.4.2 Equivalent Weight. Weigh out 0.2 to 0.3 gm of the ammonium salt of 4-nitroaniline-2-sulfonic acid. Suspend or dissolve the material in 50 to 100 ml of aqueous methanol. Titrate with 0.1N NaOH using a recording electrometric titrator. From the volume of the titrant to reach end point, calculate the equivalent weight. The result shall conform to Paragraph 3.2.3.3.2.1.

3.2.4.4.3 Decomposition Temperature. The decomposition temperature of the ammonium salt of 4-nitroaniline-2-sulfonic acid is the temperature range observed during the maximum weight loss. A thermogravimetric analysis apparatus recording weight loss versus temperature at a heating rate of 3°C/minute to 400°C in nitrogen atmosphere will be required. The result shall conform to the requirements of Paragraph 3.2.3.3.2.2.

3.2.4.4.4. Purity. The purity of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be determined by elemental Carbon, Hydrogen, Nitrogen (CHN) Analysis and the results shall conform to Paragraph 3.2.3.3.2.3.

3.3 Methyl Ethyl Ketone

3.3.1 Scope

3.3.1.1 Scope. This specification covers the minimum requirements for methyl ethyl ketone intended for use as a constituent material in the formulation of a thermal protection coating.

3.3.2 Applicable Documents

3.3.2.1 The following document forms a part of this specification:

Interim Federal Specification

TT-M-00261c	Methyl Ethyl Ketone

(Application for copies should be addressed to the General Services Administration Regional Offices.)

3.3.3 Requirements

3.3.3.1 Qualification. The methyl ethyl ketone furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.3.3.2 Properties

3.3.3.2.1 Physical Properties. The physical properties of the methyl ethyl ketone shall be as listed in Table 3.

3.3.3.2.2 Chemical Properties. The chemical properties of the methyl ethyl ketone are that the compound shall have the chemical formula $CH_3COC_2H_5$ with a molecular weight of 72.10.

3.3.4 Quality Assurance Provisions

3.3.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.3.4.2 Classification of Examinations and Tests. The examination and testing of the methyl ethyl ketone shall be classified as follows:

a. Qualification Tests. Qualification tests shall be be performed on samples submitted for approval as qualified products.

TABLE 3

Property	Requirement
Appearance	Clean and free from sediment and suspended matter
Alcohol Content, %	0.7 max. as secondary butyl alcohol
Specific Gravity, 20°/20°	0.806 ± 0.001
Nonvolatile matter from 100 ml., gram	0.0005 max.
Acidity, mg of KOH per gram of sample	0.047 max.
Distillation:	
Below 78.5	None
Above 81°C	None
Color, on the platinum cobalt scale	20
Water, percent by weight	0.3

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 4.

3.3.4.3 <u>Sampling</u>

3.3.4.3.1 <u>Qualification Sample</u>. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall how the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.3.4.3.2 <u>Quality Conformance Sample</u>. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.3.4.3.2.1 <u>Lot and Batch</u>. All methyl ethyl ketone manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.3.4.3.3 <u>Rejection and Retest</u>. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of methyl ethyl ketone. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.3.4.4 <u>Test Methods</u>. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.3.4.4.1 <u>Visual Inspection</u>. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.3.3.2.1.

3.3.4.4.2 <u>Tests for Physical and Chemical Properties</u>. All tests for physical and chemical properties shall be determined in accordance with Interim Federal Specification TT-M-00261c and the results shall conform to Paragraph 3.3.3.2.1 and Paragraph 3.3.3.2.2.

TABLE 4

Examination and Tests

Examination or Test	Requirement (Section 3.3.3)	Test Method (Section 3.3.4)
Appearance	3.3.3.2.1	3.3.4.4.1
Alcohol Content	3.3.3.2.1	3.3.4.4.2
Specific Gravity	3.3.3.2.1	3.3.4.4.2
Nonvolatiles	3.3.3.2.1	3.3.4.4.2
Acidity	3.3.3.2.1	3.3.4.4.2
Distillation	3.3.3.2.1	3.3.4.4.2
Color	3.3.3.2.1	3.3.4.4.2
Water	3.3.3.2.1	3.3.4.4.2
Chemical Formula	3.3.3.2.2	3.3.4.4.2
Molecular Weight	3.3.3.2.2	3.3.4.4.2

3.4 Toluene

3.4.1 Scope

3.4.1.1 Scope. This specification covers the minimum requirements for the toluene intended for use as a constituent material in the formulation of a thermal protection coating.

3.4.2 Applicable Documents

3.4.2.1 The following document forms a part of this specification:

Federal Specification

TT-T-548c	Toluene, Technical

(Application for copies should be addressed to the General Services Administration Regional Offices.)

3.4.3 Requirements

3.4.3.1 Qualification. The toluene furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.4.3.2 Properties

3.4.3.2.1 Physical Properties. The physical properties of the toluene shall be as listed in Table 5.

3.4.3.2.2 Chemical Properties. The chemical properties of the toluene are that the compound shall have the chemical formula $C_6H_5CH_3$ with a molecular weight of 92.13.

3.4.4 Quality Assurance Provisions

3.4.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.4.4.2 Classification of Examinations and Tests. The examination and testing of the toluene shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

TABLE 5

Property	Requirement
Appearance	Clear and free from sediment and suspended matter
Acidity	No free acid
Color	No darker than 0.003 gram of reagent grade potassium dichromate in 1 liter distilled water
Sulfur Compounds	Free from hydrogen sulfide and sulfur dioxide
Specific Gravity, 20°/20°C	0.865 ± 0.005
Boiling Point at 760 mm, °C	109 min.
Dry Point at 760 mm, °C	113 max.
Total Distillate, % by volume	97 min.
Aromatic hydrocarbons, % by volume	96 min.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 6.

3.4.4.3 <u>Sampling</u>

3.4.4.3.1 <u>Qualification Sample</u>. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.4.4.3.2 <u>Quality Conformance Sample</u>. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.4.4.3.2.1 <u>Lot and Batch</u>. All toluene manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.4.4.3.3 <u>Rejection and Retest</u>. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of toluene. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.4.4.4 <u>Test Methods</u>. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative himidity not exceeding 60 percent.

3.4.4.4.1 <u>Visual Inspection</u>. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.4.3.2.1.

3.4.4.4.2 <u>Tests for Physical and Chemical Properties</u>. All tests for physical and chemical properties shall be determined in accordance with Federal Specification TT-T-548c and the results shall conform to Paragraph 3.4.3.2.1 and Paragraph 3.4.3.2.2.

3.5 Epoxy Resin, Liquid

TABLE 6

Examination and Tests

Examination or Tests	Requirement (Section 3.4.3)	Test Method (Section 3.4.4)
Qualification Tests		
Appearance	3.4.3.2.1	3.4.4.4.1
Acidity	3.4.3.2.1	3.4.4.4.2
Color	3.4.3.2.1	3.4.4.4.2
Sulfur Compounds	3.4.3.2.1	3.4.4.4.2
Specific Gravity	3.4.3.2.1	3.4.4.4.2
Boiling Point	3.4.3.2.1	3.4.4.4.2
Dry Point	3.4.3.2.1	3.4.4.4.2
Total Distillate	3.4.3.2.1	3.4.4.4.2
Aromatic Hydrocarbons	3.4.3.2.1	3.4.4.4.2
Chemical Formula	3.4.3.2.2	3.4.4.4.2
Molecular Weight	3.4.3.2.2	3.4.4.4.2

3.5.1 Scope

3.5.1.1 Scope. This specification covers the minimum requirements for a liquid epoxy resin intended for use as a constituent material in the formulation of a thermal protection coating.

3.5.2 Applicable Documents

3.5.2.1 The following documents form a part of this specification:

Shell Chemical Company - Bulletin SC:60-146R

American Society for Testing and Materials

ASTM D 1544	Test for Color of Transparent Liquids
ASTM D 445	Test for Viscosity of Transparent and Opaque Liquids
ASTM D 1652	Test for Epoxy Content of Epoxy Resins
ASTM D 1217	Test for Density and Specific Gravity of Liquids by Bingham Pycnometer
ASTM D 1747	Test for Refractive Index of Viscous Materials.
ASTM D 1310	Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

3.5.3 Requirements

3.5.3.1 Qualification. The liquid epoxy resin furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.5.3.2 Material. The liquid epoxy resin furnished under this specification shall produce the Infrared (IR) Spectral configuration as indicated in Figure 3, when examined as specified.

3.5.3.3 Properties

3.5.3.3.1 Physical Properties

3.5.3.3.1.1 Appearance. The appearance of the liquid epoxy resin shall be a light colored liquid, free of phase separation and suspended particles when examined as specified.

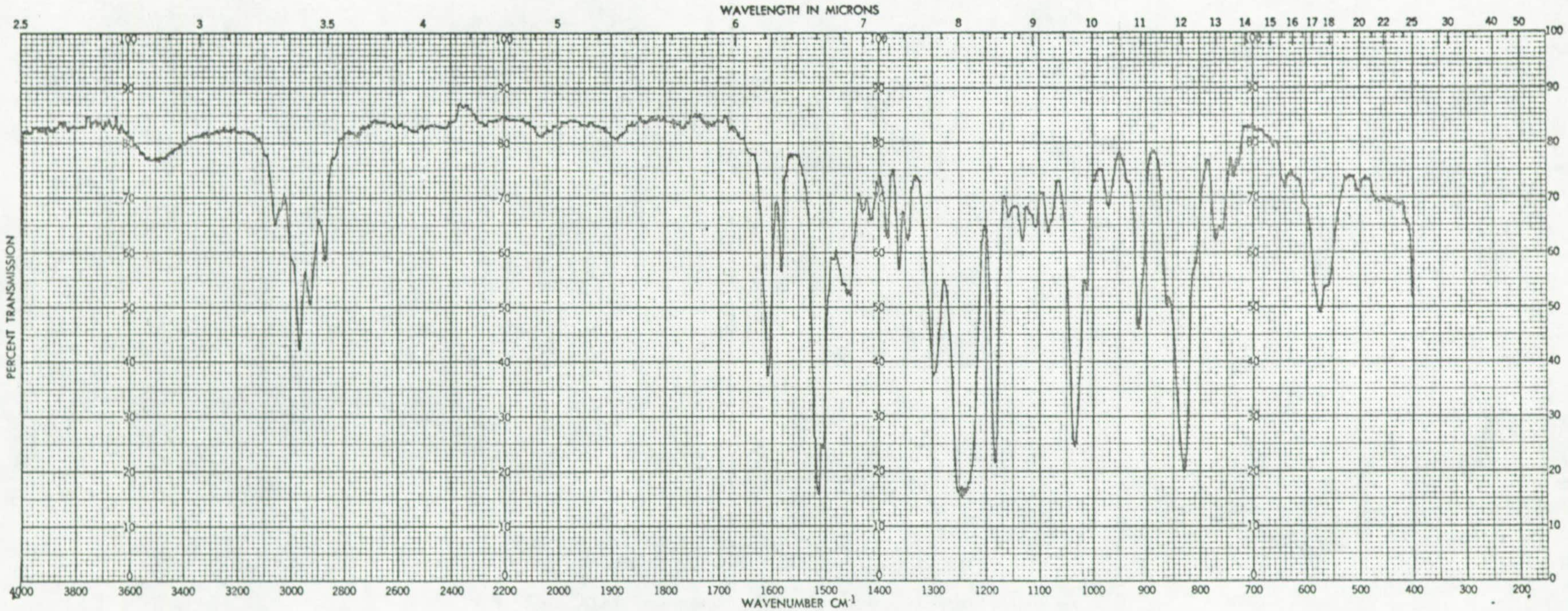

Figure 3. IR spectrum of liquid epoxy resin (as liquid between two salt plates).

3.5.3.3.1.2 Color. The color of the liquid epoxy resin shall be a Gardner 4 maximum when tested as specified.

3.5.3.3.1.3 Viscosity. The viscosity of the liquid epoxy resin shall be 100-160 poises when tested as specified.

3.5.3.3.1.4 Density. The density of the liquid epoxy resin shall be 1.168 ± 0.005 when tested as specified.

3.5.3.3.1.5 Refractive Index. The refractive index of the liquid epoxy shall be 1.570-1.575 when tested as specified.

3.5.3.3.1.6 Flash Point. The flash point of the liquid epoxy resin shall be greater than 175°F when tested as specified.

3.5.3.3.2 Chemical Properties

3.5.3.3.2.1 Epoxide Equivalent. The epoxide equivalent of the liquid epoxy resin shall be 185-192 when tested as specified.

3.5.3.3.2.2 Average Molecular Weight. The average molecular weight of the liquid epoxy resin shall be 380 (approx.) when tested as specified.

3.5.3.3.2.3 Chemical Type Structure. The chemical type structure of the liquid epoxy resin shall be the epichlorohydrin/bisphenol A-type when examined as specified.

3.5.3.4 Shelf Life. After storage for two months from date of manufacture under specified conditions, the liquid epoxy resin shall be capable of passing all the quality conformance tests specified.

3.5.4 Quality Assurance Provisions

3.5.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.5.4.2 Classification of Examinations and Tests. The examination and testing of the liquid epoxy resin shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 7.

3.5.4.3 Sampling

3.5.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.5.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.5.4.3.2.1 Lot and Batch. All liquid epoxy resin manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.5.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of liquid epoxy resin. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.5.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

3.5.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.5.3.3.1.1, the IR spectra requirements of Paragraph 3.5.3.2. Visual inspection for appearance shall be performed by filling a small, clean, dry test tube with liquid epoxy resin and holding against a bright light.

3.5.4.4.2 Color. The color of the liquid epoxy resin shall be determined in accordance with ASTM D 1544 and the result shall conform to the requirement of Paragraph 3.5.3.3.1.2.

3.5.4.4.3 Viscosity. The viscosity of the liquid epoxy resin shall be determined in accordance with ASTM D 445 and the result shall conform to the requirement of Paragraph 3.5.3.3.1.3.

TABLE 7

Examination and Tests

Examination or Test	Requirement (Section 3.5.3)	Test Method (Section 3.5.4)
Qualification Tests		
Material	3.5.3.2	3.5.4.4.1
Appearance	3.5.3.3.1.1	3.5.4.4.1
Color	3.5.3.3.1.2	3.5.4.4.2
Viscosity	3.5.3.3.1.3	3.5.4.4.3
Density	3.5.3.3.1.4	3.5.4.4.4
Refractive Index	3.5.3.3.1.5	3.5.4.4.5
Flash Point	3.5.3.3.1.6	3.5.4.4.6
Epoxide Equivalent	3.5.3.3.2.1	3.5.4.4.7
Average Molecular Weight	3.5.3.3.2.2	3.5.4.4.8
Chemical Type Structure	3.5.3.3.2.3	3.5.4.4.9
Quality Conformance Tests		
Material	3.5.3.2	3.5.4.4.1
Appearance	3.5.3.3.1.1	3.5.4.4.1
Epoxide Equivalent	3.5.3.3.2.1	3.5.4.4.7
Chemical Type Structure	3.5.3.3.2.3	3.5.4.4.9

3.5.4.4.4 Density. The density of the liquid epoxy resin shall be determined in accordance with ASTM D 1217 and the result shall conform to the requirement of Paragraph 3.5.3.3.1.4.

3.5.4.4.5 Refractive Index. The refractive index of the liquid epoxy resin shall be determined in accordance with ASTM D 1747 and the result shall conform to the requirements of Paragraph 3.5.3.3.1.5.

3.5.4.4.6 Flash Point. The flash point of the liquid epoxy resin shall be determined in accordance with ASTM D 1310 and the result shall conform to the requirements of Paragraph 3.5.3.3.1.6.

3.5.4.4.7 Epoxide Equivalent. The epoxide equivalent of the liquid epoxy resin shall be determined in accordance with ASTM D 1652 and the result shall conform to the requirements of Paragraph 3.5.3.3.2.1.

3.5.4.4.8 Average Molecular Weight. The average molecular weight of the liquid epoxy resin shall be determined in accordance with Shell Chemical Company Bulletin SC:60-146R and the result shall conform to the requirements of Paragraph 3.5.3.3.2.2.

3.5.4.4.9 Chemical Type Structure. The chemical type structure of the liquid epoxy resin shall be determined in accordance with Shell Chemical Company Bulletin SC:60-146R and the result shall conform to Paragraph 3.5.3.3.2.3.

3.5.4.5 Shelf Life. The shelf life of the liquid epoxy resin shall be determined after storage for two months from date of manufacture, in original sealed containers, at a temperature of 65 ± 15°F. An alternate method shall be to store the liquid epoxy resin under the same time and temperature conditions in an opened container with an inert atmosphere. No stored material shall be tested in accordance with Paragraphs 3.5.4.4.1, 3.5.4.4.7, and 3.5.4.4.9. The results shall conform to the requirements of Paragraph 3.5.3.4.

3.6 Tri(dimethylamino methyl)phenol

3.6.1 Scope

3.6.1.1 Scope. This specification covers the minimum requirements for tri(dimethylamino methyl)phenol intended for use as a constituent material in the formulation of a thermal protection coating.

3.6.2 Applicable Documents

3.6.2.1 The following document forms a part of this speicification.

Rohm and Haas Co.

Technical Bulletin	Technical Bulletin CO-25

(For copies of technical bulletin contact Rohm and Haas Co., Independence Mall West, Philadelphia, Pennsylvania 19105.)

ASTM E-203	Test for Water using Karl Fischer Reagent
ASTM D 941	Density and Specific Gravity of Liquids

3.6.3 Requirements

3.6.3.1 Qualifications. The 2, 4, 6 tri(dimethylamino methyl) phenol furnished under this specification shall be a product which has been tested and has passed the qualification tests specified.

3.6.3.2 Material. The 2, 4, 6 tri(dimethylamino methyl) phenol furnished under this specification shall produce the Infrared (IR) Spectral configuration as indicated in Figure 4.

3.6.3.3 Properties

3.6.3.3.1 Physical Properties

3.6.3.3.1.1 Appearance. The appearance of the 2, 4, 6 tri(dimethylamino methyl) phenol shall be clear, dark red liquid, free of phase separation and suspended particles.

3.6.3.3.1.2 Density. The density of tri (dimethylamino methyl) phenol shall be 0.973 g/cc when tested.

3.6.3.3.1.3 Flash Point. The flash point of tri (dimethylamino methyl) phenol shall be not less than 315° F when tested.

3.6.3.3.2 Chemical Properties

3.6.3.3.2.1 Water Content. The water content of the tri (dimethylamino methyl) phenol shall be 0.06 percent by weight maximum, when tested.

3.6.4 Quality Assurance Provisions

3.6.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The processing activity, or its representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.6.4.2 Classification of Examination and Tests. The examination and testing of the tri (dimethyl amino methyl) phenol solution shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 8.

3.6.4.3 Sampling

3.6.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.6.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, lot number, date of sampling, and contract number.

3.6.4.3.2.1 Lot and Batch. All material manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.6.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of material. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

3.6.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60 percent.

36.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.6.3.3.1.1, and the IR Spectral requirements of Paragraph 3.6.3.2. Visual inspection for appearance shall be performed by filling a small, clean, dry test tube with tri (dimethylamino methyl) phenol and holding against a bright light.

3.6.4.4.2 Density. The density of the material shall be determined in accordance with ASTM D 941 and the result shall conform to the requirement of Paragraph 3.6.3.3.1.2.

3.6.4.4.3 Flash Point. The flash point of the material shall be determined in accordance with ASTM D 56 and the result shall conform to the requirement of Paragraph 3.6.3.3.1.3.

3.6.4.4.4 Water Content. The water content of the material shall be determined in accordance with ASTM E 203 and the result shall conform to the requirement of Paragraph 3.6.3.3.2.1.

TABLE 8

Examinations and Tests

Examination or Test	Requirement	Test Method
Qualification Tests		
Material	3.6.3.2	3.6.4.4.1
Appearance	3.6.3.3.1.1	3.6.4.4.1
Density	3.6.3.3.1.2	3.6.4.4.2
Flash Point	3.6.3.3.1.3	3.6.4.4.3
Water Content	3.6.3.3.2.1	3.6.4.4.4
Quality Conformance Tests		
Material	3.6.3.2	3.6.4.4.1
Appearance	3.6.3.3.1.1	3.6.4.4.1
Water Content	3.6.3.3.2.1	3.6.4.4.4

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cont

PRELIMINARY

PROCESS AND APPLICATION SPECIFICATION

for

45B3 THERMAL PROTECTION COATING

Chemical Research Projects Office
NASA - Ames Research Center
Moffett Field, California 94035

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      8.3.3.9 Thermal Efficiency

4.1 Qualification. The thermal protection coating furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

4.2 Material. The thermal protection coating furnished under this specification shall conform and shall be prepared using materials approved under Preliminary Material Specification for 45B3 Thermal Protection Coating in accordance with the following formulation:

Material	Parts by Weight
Part A	
Polysulfide Polymer, Liquid	14.1
Ammonium salt of 4-nitroaniline -2-sulfonic acid	57.0
Methyl Ethyl Ketone	28.9
Part B	
Epoxy Resin, Liquid	14.1
Toluene	4.7
Part C	
Tri(dimethylamino methyl) phenol	2.8
Toluene	2.8

4.3 Properties

4.3.1 Physical Properties

4.3.1.1 Appearance. The appearance of the thermal protection coating shall be a brownish yellow, hard, integral film when examined as specified.

4.3.1.2 Tensile Strength. The tensile strength of the thermal protection coating shall be 1600 ± 100 psi when tested as specified.

4.3.1.3 Density. The density of the thermal protection coating shall be 1.42 ± 0.05 g/cc when tested as specified.

4.3.1.4 Temperature of Intumescence. The temperature of intumescence of the thermal protection coating shall be 275 ± 10°C when tested as specified.

4.3.1.5 Char Yield. The char yield of the thermal protection coating at 600°C shall be 42 ± 2 percent when tested as specified.

PRELIMINARY

PROCESS AND APPLICATION SPECIFICATION

for

45B3 THERMAL PROTECTION COATING

1.0 SCOPE - This specification covers the basic techniques of production and application as well as the minimum requirements for a thermal protection coating.

2.0 CLASSIFICATION - The thermal protection coating covered by this specification shall be classified as follows:

45B3

3.0 APPLICABLE DOCUMENTS - The following documents form a part of this specification:

Military

MIL-T-5624	Turbine Fuel, Aviation, Grades JP4 and JP5
MIL-A-8625	Anodic Coatings for Aluminum and Aluminum Alloys
MIL-C-5541	Chemicals, Films, and Chemical Film Materials for Aluminum and Aluminum Alloys

Federal

TT-P-666B	Primer Coating, Zinc Yellow for Aluminum and Magnesium Surfaces
TT-P-664b	Primer Coating, Synthetic, Rust-Inhibiting, Lacquer-Resisting
TT-P-86e,	Type 3 Paint, Red Lead Base, Ready Mixed

Other

Preliminary Material Specification for 45B3 Thermal Protection Coating

Taber Abraser Model 503 Instruction Manual

American Society for Testing and Materials

ASTM D 2370	Elongation and Tensile Strength of Free Films of Paint, Varnish, Lacquer and Related Products, with a Tensile Testing Apparatus

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

4.0 REQUIREMENTS

4.3.1.6 Abrasion Resistance. The abrasion resistance of the thermal protection coating shall be 205 ± 10 milligram weight loss/1000 cycles on the Taber Abraser when tested as specified.

4.3.1.7 Flexibility. The flexibility of the thermal protection coating shall be 1.3 ± 0.1 foot-pounds when tested as specified.

4.3.1.8 Linear Expansion (Intumescence). The linear expansion of the thermal protection coating conditioned at 23 ± 2°C and 50 ± 5% R.H. shall be a minimum of 60 times the original coating thickness when tested as specified. The linear expansion of the thermal protection coating conditioned for 30 days to a warm-humid atmosphere of 38 ± 2°C and 90 ± 2% R.H. shall be a minimum of 60 times the original coating thickness when tested as specified.

4.3.1.9 Thermal Efficiency. The thermal efficiency of the thermal protection coating shall be a minimum of 95 seconds at 0.030 + 0.002 inches coating thickness to reach a backface temperature of 205°C(400°F) when tested as specified.

5.0 DISPERSION EQUIPMENT

Pebble Mill. A burundum fortified pebble mill, burundum cylinders as grinding media, and variable speed roll mill capable of dispersing the required batch size.

Machine Mixing. A Kady Mill or equivalent kinetic dispersion unit.

6.0 MIXING PROCEDURE

Pebble Mill: The grinding media of burundum cylinders is to occupy 50% of total pebble mill volume. Add ingredients of Paragraph 4.2, Part A, sufficient to occupy 40% of total pebble mill volume. Next, the critical speed (N_e) in RPM is calculated from the following relationship:

$$N_c = \frac{76.6}{\text{I.D.}}$$

where N_c = speed at which grinding media, unencumbered by any charge within mill, begins to centrifuge

I.D. = inside diameter of mill in feet

The pebble mill is placed on the variable speed roll mill at a mixing speed of 70% of Critical Speed (N_c) for proper action of grinding media. After mixing for 1/2 hour the pebble mill is re-opened and inspected for dry agglomerates. If none present, continue milling for 2 hours total mixing time. Drain contents of pebble mill into appropriate mixing vessel. Next, add the proportional amount of the solution of Paragraph 4.2, Part B, using mechanized agitation to insure thorough mixing. Finally, add proportional amount of the solution of Paragraph 4.2, Part C, as the activator portions, using mechanical agitation again to insure thorough mixing. The blended components are now prepared for application with a working pot life of 4 hours. Strain the blended components through a cheese cloth filter to remove any extraneous matter prior to any application.

Machine Mixing: Add ingredients of Paragraph 4.2, Part A, to the jacketed mixing vessel of the Kady Mill. Mix the components in a water cooled mixing vessel in the Kady Mill for 20 minutes. Drain the contents of the Kady Mill dispersion into an appropriate mixing vessel. Next, add the proportional amount of the solution of Paragraph 4.2, Part B, using mechanical agitation to insure thorough mixing. Finally, add proportional amount of the solution of Paragraph 4.2, Part C, as the activator portion, using mechanical agitation again to insure thorough mixing. The blended components are now prepared for application with a working pot life of 4 hours. Strain the blended components through a cheese cloth filter to remove any extraneous matter prior to any application.

7.0 APPLICATION PROCEDURE

Substrate Preparation

Cold Rolled Steel: Follow standard good painting procedures. Be sure all surfaces are dry and clean. Remove loose dust, paint, grease, and oil residue from untreated surface. Surface may be sandblasted if desirable, Surface may be treated with corrosion resistant Zinc Chromate Primer meeting Federal Specification TT-P-664b or Red Lead Primer 13-R-51 from Mobil Chemical Company conforming to Federal Specification TT-P-86e Type 3.

Aluminum: Follow standard good painting procedures. Treat surface of aluminum by scrubbing with scouring powder and water. Flush liberally with water and observe for water break-free surface. If surface is not water break-free, repeat scrubbing and flushing procedure. When water break-free surface is obtained, flush surface with trichloro-ethylene and allow surface to air dry. Handle cleaned surface with care. Avoid contact with dust, oils, and greases. If corrosion resistant primer is desired, use Zinc Chromate Primer conforming to Federal Specification TT-P-666B. If anodizing of aluminum is required, the treatment shall conform to MIL-A-8625. For an alodine treated surface, the aluminum shall conform to MIL-C-5541.

Spray Method: Spray apply the 45B3 prepared from Paragraph 6.0 with a DeVilbiss JGA 502 or equivalent at ambient conditions to the dry film thickness desired. The coating shall air dry at ambient conditions for 72 hours minimum before proceeding with Quality Assurance Provisions.

Brush Method: Substrate preparation shall conform to Paragraph 7.0. Brushing may be done with a good quality soft bristled brush. Apply a full level coat avoiding excessive brush-out. Two brush coats or more may be required to conform to required dry film thickness. The coating shall air dry at ambient conditions for 72 hours before proceeding with Quality Assurance Provisons.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection. Unless otherwise specified in the contract or the purchase order, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The processing activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

8.2 Classification of Examinations and Tests. All of the examinations and testing of the thermal protection coating shall be classified as qualification tests. Qualification tests shall be performed on samples submitted for approval as qualified products and shall be performed on individual lots of products submitted for acceptance. The qualification tests are listed in Table 1. A certified test report from the supplier shall accompany each product submitted for approval and each lot of thermal protection coating submitted for acceptance.

8.3 Sampling. Samples for product qualification or for qualified lot acceptance shall be as specified in Paragraph 8.3.2. The material preparation shall conform to the requirements of Paragraph 4.2 herein. A test report from the supplier shall accompany each sample to be tested, and shall show the composition number and the test results for all requirements of this specification and shall refer specifically to the applicable paragraphs herein. All the thermal protection coating manufactured and formulated as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

8.3.1 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of thermal protection coating. If the results of test of the second sample fails to meet the specification requirements, the entire lot shall be rejected.

8.3.2 Specimen. All samples to be tested shall be coated with the thermal protection coating at a minimum dry film thickness of 0.030" on cold rolled steel panels or as free films.

8.3.2.1 Size and Number of Specimens. The size and number of specimens for conformance to each requirement is indicated in Table 2.

8.3.3 Test Methods. Unless otherwise specified, all examinations and tests shall be performed with a thermal protection coating aged for minimum of 170 hours at a temperature of 23 ± 2°C and a relative humidity of 50 ± 5 percent.

8.3.3.1 Visual Inspection. Visual inspection shall be performed to determine the appearance requirements of Paragraph 4.3.1.1 and Paragraph 4.2.

8.3.3.2 Tensile Strength. The tensile strength of the thermal protection coating shall be determined in accordance with ASTM D 2370, except the coating will be sprayed on a suitable temporary substrate, and the results shall conform to Paragraph 4.3.1.2.

8.3.3.3 Density. The density of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.3:

Procedure: Material remaining from Paragraph 8.3.3.2 shall be used to determine the coating density. The volume of coating is measured by air pycnometer and then the weight is determined from an analytical balance. The density of the coating in gm/cc is reported.

8.3.3.4 Temperature of Intumescence. The temperature of intumescence of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.4:

Procedure: The temperature of intumescence is determined by a melting point apparatus. Here glass capillary tubes containing small quantities of coating are raised in temperature until intumescence. After an initial rapid rate of temperature rise to determine approximate intumescing temperature, the determination is made in duplicate at no greater than 5°C/min temperature rise through the intumescing range.

Care should be exercised that the heat transfer to the thermometer on the apparatus is at a maximum. This can be assured by coating the thermometer bulb with colloidal graphite (Aqua-DAG).

8.3.3.5 Char Yield. The char yield of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.5:

Procedure: Using a thermogravimetric analysis (TGA) apparatus, record the weight loss versus temperature for the coating at a heating rate of 3°C/minute in a nitrogen atmosphere to 650°C. Report the char yield (Y_c) as the percent weight fraction remaining at 600°C.

8.3.3.6 Abrasion Resistance. The abrasion resistance of the thermal protection coating shall be determined according to Taber Abraser Weight Loss Method and the results shall conform to Paragraph 4.3.1.6.

8.3.3.7 Flexibility. The flexibility of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.7:

Procedure: Using a Gardner Impact Test, a round nosed, two pound impact rod is allowed to fall a calibrated distance (measured in feet) upon the coated side of the specimen panel. The foot-pounds of energy is increased until coating adhesion failure occurs. The greatest energy tolerable is reported in foot-pounds.

8.3.3.8 Linear Expansion (Intumescence). The linear expansion of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.8:

Procedure: The edges of the test specimen as well as a 1/8 inch perimeter overlap of the coating and the exposed cold rolled steel substrate shall be brushed with a red lead primer meeting Federal Specification TT-P-86e, Type 3 to minimize corrosion of uncoated edges. Two of the four specimens are further conditioned for 30 days at 38 ± 2°C and 90 ± 2% R.H. The coating specimens are placed coating side up in an oven preheated to 350°C and allowed to remain for 30 minutes. Gases should be vented from the oven. Remove after 30 minute period, allow to cool, then calculate the linear expansion:

$$\text{Linear Expansion} = \frac{Tf-Ti}{Ti}$$

where Tf = Thickness of coating after intumescence, inches

Ti = Initial coating thickness, inches

8.3.3.9 Thermal Efficiency. The thermal efficiency of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.9:

Procedure: This test exposes the coating to a fire environment where a total flux of 10 BTU/Ft^2-sec is 90 percent radiative in a slightly fuel rich condition. Aviation fuel (JP-4) is used to fire the unit. The specimens are prepared on special 3" x 3" x 1/16" cold rolled steel substrates. That is, a 1" diameter hole in the center of the panel has a 15/16" diameter 1/16" thick cold rolled steel disc cemented in place with epoxy novolac. This provides a higher thermal resistance to reduce thermal conductance effects from the edges of the specimen panel. The coated panel thickness is measured and recorded and the panel preconditioned to Paragraph 8.33 or other special environments and when ready to test is inserted in a transite mask or holder which allows the panel to rest on about 1/16" to 1/8" of the edges of the coated surface. At time, t=0, the masked panel is placed over the fire box and a thermocouple in contact with the back of the panel records the temperature rise of the center disc in the panel. The flux is uniform over the entire panel and the center disc is about 8-10% of the exposed area and is representative of a larger, semi-infinite panel. From the time-temperature history thus obtained, and the thickness in mils of coating tested, one reports the time in seconds to reach a given temperature and the mil thickness coating. Since the flame environment is a more severe test of the coatings, the intumescent thickness is also reported after this test.

TABLE 1

Examination or Test	Requirement	Test Method
Material	4.2	8.3.3.1
Appearance	4.3.1.1	8.3.3.1
Tensile Strength	4.3.1.2	8.3.3.2
Density	4.3.1.3	8.3.3.3
Temperature of Intumescence	4.3.1.4	8.3.3.4
Char Yield	4.2.1.5	8.3.3.5
Abrasion Resistance	4.3.1.6	8.3.3.6
Flexibility	4.3.1.7	8.3.3.7
Linear Expansion	4.3.1.8	8.3.3.8
Thermal Efficiency	4.3.1.9	8.3.3.9

TABLE 2

TEST SPECIMENS

Test	Substrate Size, Inches	Number of Specimens	Requirement	Test Method
Appearance	3x3x1/16	1	4.3.1.1	8.3.3.1
Tensile Strength	6x6 free films	2	4.3.1.2	8.3.3.2
Density		-	4.3.1.3	8.3.3.3
Temperature of Intumescence	Use above	-	4.3.1.4	8.3.3.4
Char Yield		-	4.3.1.5	8.3.3.5
Abrasion Resistance	4 1/8x 1/16 disc with 1/4 hole in center	6	4.3.1.6	8.3.3.6
Flexibility	Use above	-	4.3.1.7	8.3.3.7
Linear Expansion	3x3x 1/16	4	4.3.1.8	8.3.3.8
Thermal Efficiency	3x3x 1/16	4	4.3.1.9	8.3.3.9

Specification No. A-16839(C)

PRELIMINARY MATERIAL SPECIFICATION

For

313 THERMAL PROTECTION COATING

Chemical Research Projects Office

NASA-Ames Research Center

Moffett Field, California 94035

PRELIMINARY MATERIAL SPECIFICATION

For

313 THERMAL PROTECTION COATING

1.0 SCOPE

This specification covers the components for producing 313 Thermal Protection Coating.

2.0 FORMULATION

	Components	Parts Weight	Source
2.1	**Part A.**		
	Polysulfide Liquid Polymer, LP-3.	10.7	Thiokol Chemical Corp.
	Ammonium Salt of 4-nitroaniline-2-sulfonic acid.	43.3	Allied Chemical Corp.
	Methyl Ethyl Ketone.	22.0	Mallinckrodt Chemical
	Refrasil Fibers.	5.5	Hitco Materials Div.
2.2	**Part B.**		
	Epoxy Resin, Liquid.	10.7	Shell Chemical Company
	Toluene.	3.6	Mallinckrodt Chemical
2.3	**Part C.**		
	Tri (dimethylamino methyl) phenol.	2.1	Rohm and Haas Company
	Toluene.	2.1	Mallinckrodt Chemical

3.0 INGREDIENTS

3.1 Polysulfide Polymer, Liquid.

3.1.1 Scope.

3.1.1.1 Scope. This specification covers the minimum requirements for a liquid polysulfide polymer intended for use as a constituent material in the formulation of a thermal protection coating.

3.1.2 Applicable Documents.

3.1.2.1 The following document forms a part of this specification:

Product Specification, Code LF-241	Thiokol Chemical Corporation

(Application for copies should be addressed to Thiokol Chemical Corp., Chemical Operations, 780 N. Clinton Avenue, Trenton, New Jersey 08607.)

3.1.3 Requirements.

3.1.3.1 Qualifications. The liquid polysulfide polymer furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.1.3.2 Material. The liquid polysulfide polymer furnished under this specification shall produce the infrared (IR) spectral configuration as indicated in Figure 1, Page 4, when examined as specified.

3.1.3.3 Properties.

3.1.3.3.1 Physical Properties.

3.1.3.3.1.1 Appearance. The appearance of the liquid polysulfide polymer shall be a clear amber liquid, free of phase separation and suspended particles when examined as specified.

3.1.3.3.1.2 Specific Gravity. The specific gravity of the liquid polysulfide polymer shall be 1.260 to 1.283 when tested as specified.

3.1.3.3.1.3 Color. The color of the liquid polysulfide polymer shall be 12 maximum (Hellige Scale) when tested as specified.

FIGURE 1

INFRARED (IR) SPECTRAL CONFIGURATION FOR LIQUID POLYSULFIDE POLYMER

3.1.3.3.1.4 Viscosity. The viscosity of the liquid polysulfide polymer at 80°F shall be 950 ± 250 centipoises when tested as specified.

3.1.3.3.2 Chemical Properties.

3.1.3.3.2.1 pH (Water Extract). The pH of the liquid polysulfide polymer shall be in the range of 6.0 to 8.0 when tested as specified.

3.1.3.3.2.2 Moisture Content, %. The moisture content of the liquid polysulfide polymer shall be 0.1 maximum when tested as specified.

3.1.3.3.2.3 Mercaptan, %. The percent mercaptan of the liquid polysulfide polymer shall be 5.9 to 7.7 when tested as specified.

3.1.3.4 Shelf Life. After storage for two months from date of manufacture under specified conditions, the liquid polysulfide polymer shall be capable of passing all the quality tests specified.

3.1.4 Quality Assurance Provisions.

3.1.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.1.4.2 Classification of Examinations and Tests. The examination and testing of the liquid polysulfide polymer shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 1 , Page 7.

3.1.4.3 Sampling.

3.1.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany the required

3.1.4.3.1 Qualification Sample. (Continued) samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.1.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, the specification number, lot number, date of sampling, and contract number.

3.1.4.3.2.1 Lot and Batch. All liquid polysulfide polymer manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.1.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of liquid polysulfide polymer. If the results of test of the second sample fail to meet the specification requirements, the entire lot shall be rejected.

3.1.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5° F with a relative humidity not exceeding 60%.

3.1.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.1.3.3.1.1., and the IR spectral requirements of Paragraph 3.1.3.2. Visual inspection for appearance shall be performed by filling a small, clean, dry test tube with liquid polysulfide polymer and holding it against a bright light.

3.1.4.4.2 Specific Gravity. The specific gravity of the liquid polysulfide shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform with the requirement of Paragraph 3.1.3.3.1.2.

3.1.4.4.3 Color. The color of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform to the requirement of Paragraph 3.1.3.3.1.3.

3.1.4.4.4 Viscosity. The viscosity of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform to the requirement of Paragraph 3.1.3.3.1.4.

3.1.4.4.5 <u>pH (Water Extract)</u>. The pH of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the result shall conform to the requirements of Paragraph 3.1.3.3.2.1.

3.1.4.4.6 <u>Moisture Content</u>. The moisture content of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241, and the result shall conform to the requirement of Paragraph 3.1.3.3.2.2.

3.1.4.4.7 <u>Mercaptan</u>. The mercaptan percent of the liquid polysulfide polymer shall be determined in accordance with Thiokol Product Specification Code LF-241 and the results shall conform to the requirement of Paragraph 3.1.3.3.2.3.

3.1.4.5 <u>Shelf Life</u>. The shelf life of the liquid polysulfide polymer shall be determined after storage for two months from the date of manufacture, in original sealed containers, at a temperature of 65 ± 15°F. An alternative method shall be to store the liquid polysulfide polymer under the same time and temperature conditions in an opened container with an inert atmosphere. The stored material shall be tested in accordance with Paragraphs 3.1.4.4.1 through 3.1.4.4.7. The results shall conform to the requirements of Paragraph 3.1.3.3.

TABLE 1

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.1.3)	Test Method (Section 3.1.4)
Qualification Tests:		
Material	3.1.3.2	3.1.4.4.1
Appearance	3.1.3.3.1.1	3.1.4.4.1
Specific Gravity	3.1.3.3.1.2	3.1.4.4.2
Color	3.1.3.3.1.3	3.1.4.4.3
Viscosity	3.1.3.3.1.4	3.1.4.4.4
pH (Water Extract)	3.1.3.3.2.1	3.1.4.4.5
Moisture Content	3.1.3.3.2.2	3.1.4.4.6
Mercaptan	3.1.3.3.2.3	3.1.4.4.7
Quality Conformance Tests:		
Material	3.1.3.2	3.1.4.4.1
Appearance	3.1.3.3.1.1	3.1.4.4.1
Mercaptan	3.1.3.3.2.3	3.1.4.4.7

3.2 Ammonium Salt of 4-nitroaniline-2-sulfonic acid.

3.2.1 Scope.

3.2.1.1 Scope. This specification covers the minimum requirements for an ammonium salt of 4-nitroaniline-2-sulfonic acid intended for use as a constituent material in the formulation of a thermal protection coating.

3.2.2 Applicable Documents.

3.2.2.1 The following document forms a part of this specification:

Preparation of Ammonium-p-nitroaniline-o-sulfonate - (See Page 9.)

3.2.3 Requirements.

3.2.3.1 Qualification. The ammonium salt of 4-nitroaniline-2-sulfonic acid furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.2.3.2 Material. The ammonium salt of 4-nitroaniline-2-sulfonic acid furnished under this specification shall produce the infrared (IR) spectral configuration as indicated in Figure 2, Page 10, when examined as specified.

3.2.3.3 Properties.

3.2.3.3.1 Physical Properties.

3.2.3.3.1.1 Appearance. The appearance of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be a yellow free-flowing granular solid with a maximum particle size of 200 microns when examined as specified.

3.2.3.3.2 Chemical Properties.

3.2.3.3.2.1 Equivalent Weight. The equivalent weight of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be 235 ± 12 when examined as specified.

3.2.3.3.2.2 Initial Decomposition Temperature. The initial decomposition temperature of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be $545^{\circ} \pm 27^{\circ}F$ when examined as specified.

3.2.3.3.2.3 Purity. The elemental analysis of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be C, 30.7 ± 1.2%; H, 3.9 ± 0.2%; N, 17.9 ± 0.8%, when examined as specified.

PREPARATION OF

Ammonium p-nitroaniline-o-sulfonate

1. 143# of the sodium salt of p-nitroaniline-o-sulfonic acid (Allied Chemical Brand name: p-nitroaniline-o-sulfonic acid) is dissolved in 34 gallons of boiling water.

2. Maintain solution of salt at 200°F and filter debris from the solution.

3. Slowly add and stir 55# of solid ammonium chloride to the 200°F salt solution.

4. Continue stirring solution and cool to 50°F. On cooling, ammonium-p-nitroaniline-o-sulfonate will precipitate.

5. Filter precipitated ammonium p-nitroaniline-o-sulfonate by centrifuge or suction filtration.

6. Prepare 7.5 gallons of 10% by weight ammonium chloride aqueous solution and cool to 50°F.

7. Wash filtered ammonium p-nitroaniline-o-sulfonate solids with 3 equal portions (2.5 gallons each) of 50°F ammonium chloride solution.

8. Wash filtered ammonium p-nitroaniline-o-sulfonate solids with 4 equal portions (1.2 gallons each) of 40°F water.

9. Dry the salt (ammonium p-nitroaniline-o-sulfonate) to constant weight in an air or a circulating oven at 120°F.

Approximate Yield: 120#

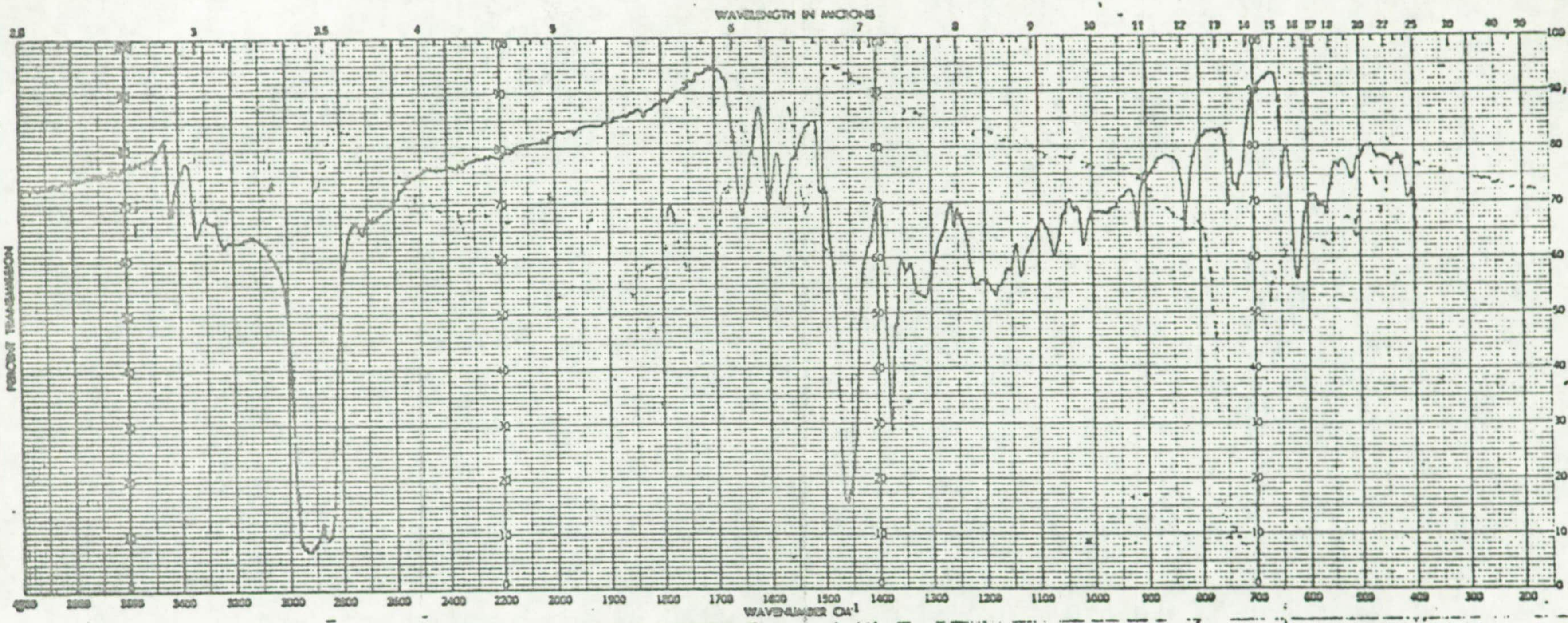

Figure 2. IR spectrum of ammonium salt of 4-nitroaniline-2-sulfonic acid (Nujol).

3.2.4 Quality Assurance Provisions.

3.2.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.2.4.2 Classification of Examinations and Tests. The examination and testing of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 2, Page 13.

3.2.4.3 Sampling

3.2.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany all of the required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.2.4.3.2 Qualify Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, the specification number, date of sampling, and contract number.

3.2.4.3.2.1 Lot and Batch. All ammonium salt of 4-nitroaniline-2-sulfonic acid manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.2.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens, prepared from the same lot of ammonium salt of 4-nitroaniline-2-sulfonic acid. If the results of the test of the second sample fail to meet the specification requirements, the entire lot shall be rejected.

3.2.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60%.

3.2.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.2.3.3.1.1 and the IR spectralrequirements of Paragraph 3.2.3.2.

3.2.4.4.2 Equivalent Weight. Weigh out 2.0 to 3.0 gm of the ammonium salt of 4-nitroaniline-2-sulfonic acid. Suspend or dissolve the material in 50 to 100 ml of 50% by volume aqueous methanol. Titrate with 1.0N NaOH using a recording electrometric titrator. From the volume of the titrant to reach end point, calculate the equivalent weight. The result shall conform to Paragraph 3.2.3.3.2.1.

3.2.4.4.3 Initial Decomposition Temperature. The decomposition temperature of the ammonium salt of 4-nitroaniline-2-sulfonic acid is the temperature range observed during the maximum weight loss. A thermogravimetric analysis apparatus recording weight loss versus temperature at a heating rate of 5°F/minute to the temperature of 750°F in nitrogen atmosphere will be required. The result shall conform to the requirement of Paragraph 3.2.3.3.2.2.

3.2.4.4.4 Purity. The purity of the ammonium salt of 4-nitroaniline-2-sulfonic acid shall be determined by elemental Carbon, Hydrogen, Nitrogen (CHN) Analysis, and the results shall conform to Paragraph 3.2.3.3.2.3.

TABLE 2

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.2.3)	Test Method (Section 3.2.4)
Qualification Tests:		
Material	3.2.3.2	3.2.4.4.1
Appearance	3.2.3.3.1.1	3.2.4.4.1
Equivalent Weight	3.2.3.3.2.1	3.2.4.4.2
Decomposition Temperature	3.2.3.3.2.2	3.2.4.4.3
Purity	3.2.3.3.2.3	3.2.4.4.4
Quality Conformance Tests:		
Material	3.2.3.2	3.2.4.4.1
Appearance	3.2.3.3.1.1	3.2.4.4.1
Equivalent Weight	3.2.3.3.2.1	3.2.4.4.2
Decomposition Temperature	3.2.3.3.2.2	3.2.4.4.3
Purity	3.2.3.3.2.3	3.2.4.4.4

3.3 Methyl Ethyl Ketone.

3.3.1 Scope.

3.3.1.1 Scope. This specification covers the minimum requirements for methyl ethyl ketone intended for use as a constituent material in the formulation of a thermal protection coating.

3.3.2 Applicable Documents.

3.3.2.1 The following document forms a part of this specification:

Interim Federal Specification:

TT-M-00261c	Methyl Ethyl Ketone

(Application for copies should be addressed to the General Services Administration Regional Offices.)

3.3.3 Requirements.

3.3.3.1 Qualification. The methyl ethyl ketone furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.3.3.2 Properties.

3.3.3.2.1 Physical Properties. The physical properties of the methyl ethyl ketone shall be as listed in Table 3, Page 16.

3.3.3.2.2 Chemical Properties. The chemical properties of the methyl ethyl ketone are that the compound shall have the chemical formula $CH_3COC_2H_5$ with a molecular weight of 72.10.

3.3.4 Quality Assurance Provisions.

3.3.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.3.4.2 Classification of Examinations and Tests. The examination and testing of the methyl ethyl ketone shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 4, Page 17.

3.3.4.3 Sampling.

3.3.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.3.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.3.4.3.2.1 Lot and Batch. All methyl ethyl ketone manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.3.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of methyl ethyl ketone. If the results of the test of the second sample fail to meet the specification requirements, the entire lot shall be rejected.

3.3.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60%.

3.3.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance to appearance requirements of Paragraph 3.3.3.2.1.

3.3.4.4.2 Tests for Physical and Chemical Properties. All tests for physical and chemical properties shall be determined in accordance with Interim Federal Specification TT-M-00261c and the results shall conform to Paragraph 3.3.3.2.1 and Paragraph 3.3.3.2.2.

TABLE 3

METHYL ETHYL KETONE PHYSICAL PROPERTIES

Property	Requirement
Appearance	Clean and free from sediment and suspended matter
Alcohol Content, %	0.7 maximum as secondary butyl alcohol
Specific Gravity, 68°/68°F	0.806 ± 0.001
Nonvolatile matter from 100 ml., gram	0.0005 maximum
Acidity, mg of KOH per gram of sample	0.047 maximum
Distillation:	
Below 172°F	None
Above 180°F	None
Color, on the platinum cobalt scale	20
Water, % by weight	0.3

TABLE 4

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.3.3)	Test Method (Section 3.3.4)
Appearance	3.3.3.2.1	3.3.4.4.1
Alcohol Content	3.3.3.2.1	3.3.4.4.2
Specific Gravity	3.3.3.2.1	3.3.4.4.2
Nonvolatiles	3.3.3.2.1	3.3.4.4.2
Acidity	3.3.3.2.1	3.3.4.4.2
Distillation	3.3.3.2.1	3.3.4.4.2
Color	3.3.3.2.1	3.3.4.4.2
Water	3.3.3.2.1	3.3.4.4.2
Chemical Formula	3.3.3.2.2	3.3.4.4.2
Molecular Weight	3.3.3.2.2	3.3.4.4.2

3.4 Toluene.

3.4.1 Scope.

3.4.1.1 Scope. This specification covers the minimum requirements for the toluene intended for use as a constituent material in the formulation of a thermal protection coating.

3.4.2 Applicable Documents.

3.4.2.1 The following document forms a part of this specification:

Federal Specification

TT-T-548c	Toluene, Technical

(Application for copies should be addressed to the General Services Administration Regional Offices.)

3.4.3 Requirements.

3.4.3.1 Qualification. The toluene furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.4.3.2 Properties.

3.4.3.2.1 Physical Properties. The physical properties of the toluene shall be as listed in Table 5, Page 20.

3.4.3.2.2 Chemical Properties. The chemical properties of the toluene are that the compound shall have the chemical formula $C_6H_5CH_3$ with a molecular weight of 92.13.

3.4.4 Quality Assurance Provisions.

3.4.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.4.4.2 Classification of Examinations and Tests. The examination and testing of the toluene shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

The qualification and quality conformance tests are listed in Table 6, Page 21.

3.4.4.3 Sampling.

3.4.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. This report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.4.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.4.4.3.2.1 Lot and Batch. All toluene manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.4.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of toluene. If the results of test of the second sample fail to meet the specification requirements, the entire lot shall be rejected.

3.4.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75° ± 5°F with a relative humidity not exceeding 60%.

3.4.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.4.3.2.1.

3.4.4.4.2 Tests for Physical and Chemical Properties. All tests for physical and chemical properties shall be determined in accordance with Federal Specification TT-T-548c, and the results shall conform to Paragraph 3.4.3.2.1 and Paragraph 3.4.3.2.2.

TABLE 5

TOLUENE PHYSICAL PROPERTIES

Property	Requirement
Appearance	Clear and free from sediment and suspended matter
Acidity	No free acid
Color	No darker than 0.003 gram of reagent grade potassium dichromate in 1 liter distilled water
Sulfur Compounds	Free from hydrogen sulfide and sulfur dioxide
Specific Gravity, 68°/68°F	0.865 ± 0.005
Boiling Point at 760 mm, °F	228 Minimum
Dry Point at 760 mm, °F	235 Maximum
Total Distillate, % by Volume	97 Minimum
Aromatic hydrocarbons, % by volume	96 Minimum

TABLE 6

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.4.3)	Test Method (Section 3.4.4)
Appearance	3.4.3.2.1	3.4.4.4.1
Acidity	3.4.3.2.1	3.4.4.4.2
Color	3.4.3.2.1	3.4.4.4.2
Sulfur Compounds	3.4.3.2.1	3.4.4.4.2
Specific Gravity	3.4.3.2.1	3.4.4.4.2
Boiling Point	3.4.3.2.1	3.4.4.4.2
Dry Point	3.4.3.2.1	3.4.4.4.2
Total Distillate	3.4.3.2.1	3.4.4.4.2
Aromatic Hydrocarbons	3.4.3.2.1	3.4.4.4.2
Chemical Formula	3.4.3.2.2	3.4.4.4.2
Molecular Weight	3.4.3.2.2	3.4.4.4.2

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cont.

3.5 Liquid Epoxy Resin.

3.5.1 Scope.

3.5.1.1 Scope. This specification covers the minimum requirements for a liquid epoxy resin intended for use as a constituent material in the formulation of a thermal protection coating.

3.5.2 Applicable Documents.

3.5.2.1 The following documents form a part of this specification:

Shell Chemical Company: Bulletin SC:60-146R.

American Society for Testing and Materials:

ASTM D 1544 - Test for Color of Transparent Liquids

ASTM D 445 - Test for Viscosity of Transparent and Opaque Liquids

ASTM D 1652 - Test for Epoxy Content of Epoxy Resins

ASTM D 1217 - Test for Density and Specific Gravity of Liquids by Bingham Pycnometer

ASTM D 1747 - Test for Refractive Index of Viscous Materials

ASTM D 1310 - Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

3.5.3 Requirements.

3.5.3.1 Qualification. The liquid epoxy resin furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.5.3.2 Material. The liquid epoxy resin furnished under this specification shall produce the infrared (IR) spectral configuration as indicated in Figure 3, page 23, when examined as specified.

FIGURE 3

INFRARED (IR) SPECTRAL CONFIGURATION
FOR LIQUID EPOXY RESIN

3.5.3.3 Properties.

3.5.3.3.1 Physical Properties.

3.5.3.3.1.1 Appearance. The appearance of the liquid epoxy resin shall be a light colored liquid, free of phase separation and suspended particles when examined as specified.

3.5.3.3.1.2 Color. The color of the liquid epoxy resin shall be a Gardner 4 maximum when tested as specified.

3.5.3.3.1.3 Viscosity. The viscosity of the liquid epoxy resin shall be 100-160 poises when tested as specified at 77°F.

3.5.3.3.1.4 Density. The density of the liquid epoxy resin shall be 1.168 ± 0.005 when tested as specified.

3.5.3.3.1.5 Refractive Index. The refractive index of the liquid epoxy resin shall be 1.570-1.575 when tested as specified.

3.5.3.3.1.6 Flash Point. The flash point of the liquid epoxy resin shall be greater than 175°F when tested as specified.

3.5.3.3.2 Chemical Properties.

3.5.3.3.2.1 Epoxide Equivalent. The epoxide equivalent of the liquid epoxy resin shall be 185-192 when tested as specified.

3.5.3.3.2.2 Average Molecular Weight. The average molecular weight of the liquid epoxy resin shall be approximately 380 when tested as specified.

3.5.3.3.2.3 Chemical Type Structure. The chemical type structure of the liquid epoxy resin shall be the epichlorohydrin/bisphenol A-type when examined as specified.

3.5.3.4 Shelf Life. After storage for two months from date of manufacture under specified conditions, the liquid epoxy resin shall be capable of passing all the quality conformance tests specified.

3.5.4 Quality Assurance Provisions.

3.5.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his

3.5.4.1 Responsibility for Inspection. (Continued) own facility or any commercial laboratory acceptable to ARC. The procuring activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.5.4.2 Classification of Examinations and Tests. The examination and testing of the liquid epoxy resin shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

Qualification and quality conformance tests are listed in Table 7, Page 27.

3.5.4.3 Sampling.

3.5.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.5.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.5.4.3.2.1 Lot and Batch. All liquid epoxy resin manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.5.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of liquid epoxy resin. If the results of the test of the second sample fail to meet the specification requirements, the entire lot shall be rejected.

3.5.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75° ± 5°F with a relative humidity not exceeding 60%.

3.5.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with the appearance requirements of Paragraph 3.5.3.3.1.1, the IR spectral requirements of Paragraph 3.5.3.2. Visual inspection for appearance shall be performed by filling a small, clean, dry test tube with liquid epoxy resin and holding against a bright light.

3.5.4.4.2 Color. The color of the liquid epoxy resin shall be determined in accordance with ASTM D 1544 and the result shall conform to the requirement of Paragraph 3.5.3.3.1.2.

3.5.4.4.3 Viscosity. The viscosity of the liquid epoxy resin shall be determined in accordance with ASTM D 445 and the result shall conform to the requirement of Paragraph 3.5.3.3.1.3.

3.5.4.4.4 Density. The density of the liquid epoxy resin shall be determined in accordance with ASTM D 1217 and the result shall conform to the requirement of Paragraph 3.5.3.3.1.4.

3.5.4.4.5 Refractive Index. The refractive index of the liquid epoxy resin shall be determined in accordance with ASTM D 1747 and the result shall conform to the requirements of Paragraph 3.5.3.3.1.5.

3.5.4.4.6 Flash Point. The flash point of the liquid epoxy resin shall be determined in accordance with ASTM D 1310 and the result shall conform to the requirements of Paragraph 3.5.3.1.6.

3.5.4.4.7 Epoxy Equivalent. The epoxide equivalent of the liquid epoxy resin shall be determined in accordance with ASTM D 1652 and the result shall conform to the requirements of Paragraph 3.5.3.3.2.1.

3.5.4.4.8 Average Molecular Weight. The average molecular weight of the liquid epoxy resin shall be determined in accordance with Shell Chemical Company Bulletin SC:60-146R and the result shall conform to the requirements of Paragraph 3.5.3.3.2.2.

3.5.4.4.9 Chemical Type Structure. The chemical type structure of the liquid epoxy resin shall be determined in accordance with Shell Chemical Company Bulletin SC:60-146R and the result shall conform to Paragraph 3.5.3.3.2.3.

3.5.3.5 Shelf Life. The shelf life of the liquid epoxy resin shall be determined after storage for two months from date of manufacture, in original sealed containers, at a temperature of 65 ± 15°F. An alternate method shall be to store the liquid epoxy resin under the same time and temperature conditions in an opened container with an inert atmosphere. The stored material shall be tested in accordance with Paragraphs 3.5.4.4.1, 3.5.4.4.7, and 3.5.4.4.9. The results shall conform to the requirements of Paragraph 3.5.3.4.

TABLE 7

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.5.3)	Test Method (Section 3.5.4)
Qualification Tests:		
Material	3.5.3.2	3.5.4.4.1
Appearance	3.5.3.3.1.1	3.5.4.4.1
Color	3.5.3.3.1.2	3.5.4.4.2
Viscosity	3.5.3.3.1.3	3.5.4.4.3
Density	3.5.3.3.1.4	3.5.4.4.4
Refractive Index	3.5.3.3.1.5	3.5.4.4.5
Flash Point	3.5.3.3.1.6	3.5.4.4.6
Epoxide Equivalent	3.5.3.3.2.1	3.5.4.4.7
Average Molecular Weight	3.5.3.3.2.2	3.5.4.4.8
Chemical Type Structure	3.5.3.3.2.3	3.5.4.4.9
Quality Conformance Tests:		
Material	3.5.3.2	3.5.4.4.1
Appearance	3.5.3.3.1.1	3.5.4.4.1
Epoxide Equivalent	3.5.3.3.2.1	3.5.4.4.7
Chemical Type Structure	3.5.3.3.2.3	3.5.4.4.9

3.6 Tri (dimethylamino methyl) phenol.

3.6.1 Scope.

3.6.1.1 Scope. This specification covers the minimum requirements for tri (dimethylamino methyl) phenol intended for use as a constituent material in the formulation of a thermal protection coating.

3.6.2 Applicable Documents.

3.6.2.1 The following documents form a part of this specification:

*Rohm and Haas Company: Technical Bulletin CO-25

American Society for Testing Materials:

ASTM E 203 - Test for Water Using Karl Fischer Reagent

ASTM D 941 - Density and Specific Gravity of Liquids

*(For copies of technical bulletin contact Rohm and Haas Company, Independence Mall West, Philadelphia, Pennsylvania 19105.)

3.6.3 Requirements.

3.6.3.1 Qualifications. The 2, 4, 6 tri (dimethylamino methyl) phenol furnished under this specification shall be a product which has been tested and has passed the qualification tests specified.

3.6.3.2 Material. The 2, 4, 6 tri (dimethylamino methyl) phenol furnished under this specification shall produce the infrared (IR) spectral configuration as indicated in Figure 4, Page 29.

3.6.3.3 Properties.

3.6.3.3.1 Physical Properties.

3.6.3.3.1.1 Appearance. The appearance of the 2, 4, 6 tri (dimethylamino methyl) phenol shall be a clear amber liquid, free of phase separation and suspended particles.

3.6.3.3.1.2 Density. The density of tri (dimethylamino methyl) phenol shall be 0.973 g/cc when tested.

3.6.3.3.1.3 Flash Point. The flash point of tri (dimethylamino methyl) phenol shall be not less than 315°F when tested.

FIGURE 4

INFRARED (IR) SPECTRAL CONFIGURATION FOR TRI (DIMETHYLAMINO METHYL) PHENOL

3.6.3.3.2 Chemical Properties.

3.6.3.3.2.1 Water Content. The water content of the tri (dimethylamino methyl) phenol shall be 0.06% by weight maximum, when tested.

3.6.4 Quality Assurance Provisions.

3.6.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity, or its representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.6.4.2 Classification of Examination and Tests. The examination and testing of the tri (dimethylamino methyl) phenol solution shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

Qualification and quality conformance tests are listed in Table 8, Page 31.

3.6.4.3 Sampling.

3.6.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.6.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, lot number, date of sampling, and contract number.

3.6.4.3.2.1 Lot and Batch. All material manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.6.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of material. If results of test of second sample fail to meet specification requirements, the entire lot shall be rejected.

3.6.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75° ± 5°F, with a relative humidity not exceeding 60%.

3.6.4.4.1 Visual Inspection. Visual inspection shall be performed to determine conformance with appearance requirements of Paragraph 3.6.3.3.1.1, and the IR spectral requirements of Paragraph 3.6.3.2. Visual inspection for appearance shall be performed by filling a small, clean, dry test tube with tri (dimethylamino methyl) phenol and holding it against a bright light.

3.6.4.4.2 Density. The density of the material shall be determined in accordance with ASTM D 941 and the result shall conform to the requirement of Paragraph 3.6.3.3.1.2.

3.6.4.4.3 Flash Point. The flash point of the material shall be determined in accordance with ASTM D 56 and the results shall conform to the requirement of Paragraph 3.6.3.3.1.3.

3.6.4.4.4 Water Content. The water content of the material shall be determined in accordance with ASTM E 203 and the result shall conform to the requirement of Paragraph 3.6.3.3.2.1.

TABLE 8

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.6.3)	Test Method (Section 3.6.4)
Qualification Tests:		
Material	3.6.3.2	3.6.4.4.1
Appearance	3.6.3.3.1.1	3.6.4.4.1
Density	3.6.3.3.1.2	3.6.4.4.2
Flash Point	3.6.3.3.1.3	3.6.4.4.3
Water Content	3.6.3.3.2.1	3.6.4.4.4
Quality Conformance Tests:		
Material	3.6.3.2	3.6.4.4.1
Appearance	3.6.3.3.1.1	3.6.4.4.1
Water Content	3.6.3.3.2.1	3.6.4.4.4

3.7 Refrasil Fibers.

3.7.1 Scope.

3.7.1.1 Scope. This specification covers the minimum requirements for Refrasil fibers intended for use as a constituent material in the formulation of a thermal protection coating.

3.7.2 Applicable Documents.

3.7.2.1 The following documents form a part of this specification:

Product Bulletin

Code PB1-2B	Hitco Materials Division
Code PB1-2C	Hitco Materials Division

3.7.3 Requirements.

3.7.3.1 Qualifications. The Refrasil fibers furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein. No other (substitude fibers) shall be accepted.

3.7.3.2 Properties.

3.7.3.2.1 Physical Properties.

3.7.3.2.1.1 Appearance. The appearance of the Refrasil fiber shall be a white, free flowing solid ranging from 0.003 to 0.03 inches in length and 0.0004 to 0.00005 inches in diameter. The fiber length distribution shall be approximately 10% each at the extremes of 0.003 and 0.03 inch lengths with 80% having a nominal length of 0.01 inches.

3.7.4 Quality Assurance Provisions.

3.7.4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The procuring activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

3.7.4.2 Classification of Examination and Tests. The examination and testing of Refrasil fibers shall be classified as follows:

a. Qualification Tests. Qualification tests shall be performed on samples submitted for approval as qualified products.

b. Quality Conformance Tests. Quality conformance tests shall be performed on individual lots of products submitted for acceptance.

Qualification and quality conformance tests are listed in Table 9, Page 34.

3.7.4.3 Sampling.

3.7.4.3.1 Qualification Sample. The qualification sample shall consist of sufficient material to perform all of the qualification tests specified herein. A test report from the supplier shall accompany required samples of each product for which qualification is desired. The report shall show the product test results for all requirements of this specification and shall refer specifically to the applicable paragraphs in this specification.

3.7.4.3.2 Quality Conformance Sample. The quality conformance sample shall be selected at random from each lot of material to be supplied to ARC and shall consist of sufficient material to perform the quality conformance tests required for acceptance. The sample shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, date of sampling, and contract number.

3.7.4.3.2.1 Lot and Batch. All Refrasil fibers manufactured as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

3.7.4.3.3 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of refrasil fibers. If the results of test of the second sample fail to meet the specification requirements, the entire lot shall be rejected.

3.7.4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of 75 ± 5°F with a relative humidity not exceeding 60%.

3.7.4.4.1 Visual Inspection. Visual inspection shall be performed with the aid of a microscope to determine conformance with the appearance requirements of Paragraph 3.7.3.2.1.1.

TABLE 9

EXAMINATION AND TESTS

Examination or Test	Requirement (Section 3.7.3)	Test Method (Section 3.7.4)
Appearance	3.7.3.2.1.1	3.7.4.4.1
Color	3.7.3.2.1.1	3.7.4.4.1
Size	3.7.3.2.1.1	3.7.4.4.1

Specification No. A-16839(D)

PRELIMINARY PROCESS AND APPLICATION SPECIFICATION

For

313 THERMAL PROTECTION COATING

Chemical Research Projects Office

NASA-Ames Research Center

Moffett Field, California 94035

PRELIMINARY PROCESS AND APPLICATION SPECIFICATION

For

313 THERMAL PROTECTION COATING

1.0 SCOPE

This specification covers the basic techniques of production and application as well as the minimum requirements for a thermal protection coating.

2.0 CLASSIFICATION

The thermal protection coating covered by this specification shall be classified as follows:

313

3.0 APPLICABLE DOCUMENTS

The following documents form a part of this specification:

Military

MIL-T-5624 - Turbine Fuel, Aviation, Grades JP4 and JP5
MIL-A-8625 - Anodic Coatings for Aluminum and Aluminum Alloys
MIL-C-5541 - Chemicals, Films, and Chemical Fil Materials for Aluminum and Aluminum Alloys

Federal

TT-P-666B - Primer Coating, Zinc Yellow, for Aluminum and Magnesium Surfaces
TT-P-664b - Primer Coating, Synthetic, Rust-Inhibiting, Lacquer-Resisting
TT-P-86e - Type 3 Paint, Red Lead Base, Ready Mixed

American Society for Testing and Materials

ASTM D 2370 - Elongation and Tensile Strength of Free Films of Paint, Varnish, Lacquer and Related Products, with a Tensile-Testing Apparatus

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.)

Other

Preliminary Material Specification for 313 Thermal Protection Coating
Taber Abraser Model 503 Instruction Manual

4.0 REQUIREMENTS

4.1 Qualification. The thermal protection coating furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

4.2 Material. The thermal protection coating furnished under this specification shall conform to and shall be prepared using materials in accordance with the following formulation:

Material	Parts by Weight
Part A.	
Polysulfide Liquid Polymer, LF-3.	10.7
Ammonium Salt of 4-nitroaniline-2 sulfonic acid.	43.3
Methyl Ethyl Ketone.	22.0
Refrasil Fibers.	5.5
Part B.	
Epoxy Resin, Liquid.	10.7
Toluene.	3.6
Part C.	
Tri (dimethylamino methyl) phenol.	2.1
Toluene.	2.1

4.3 Properties.

4.3.1 Physical Properties.

4.3.1.1 Appearance. The appearance of the thermal protection coating shall be a yellow, hard, integral film when examined as specified.

4.3.1.2 Tensile Strength. The tensile strength of the thermal protection coating shall be 1800 ± 200 psi when tested as specified.

4.3.1.3 Density. The density of the thermal protection coating shall be 1.50 ± 0.05 g/cc when tested as specified.

4.3.1.4 Char Yield and Temperature of Intumescence. The char yield of the thermal protection coating at 1112°F shall be 46 ± 3 percent, and the temperature of intumescence shall be 545° ± 27°F, when tested as specified.

4.3.1.5 Abrasion Resistance. The abrasion resistance of the thermal protection coating shall be 100 ± 20 milligram weight loss/1000 cycles on the Taber Abraser when tested as specified.

4.3.1.6 Flexibility. The flexibility of the thermal protection coating shall be 1.3 ± 0.1 foot-pounds when tested as specified.

4.3.1.7 Linear Expansion (Intumescence). The linear expansion of the thermal protection coating conditioned at 73.5° ± 3.5°F and 50 ± 5% R.H. for seven days shall be a minimum of sixty times the original coating thickness when tested as specified. The linear expansion of the thermal protection coating conditioned for seven days to a warm-humid atmosphere of 100° ± 3.5°F and 90 ± 2% R.H. shall be a minimum of sixty times the original coating thickness when tested as specified.

4.3.1.8 Thermal Efficiency. The thermal efficiency of the thermal protection coating shall be a minimum of 95 seconds at 0.030 + 0.002 inches coating thickness to reach a backface temperature of 400°F when tested as specified.

5.0 DISPERSION EQUIPMENT

Machine Mixing. A Kady Mill or equivalent kinetic dispersion unit.

6.0 MIXING PROCEDURE

Fiber Preparation. Place 1/16" Refrasil fibers in an oven for 24 hours. Remove fibers from the oven and allow to cool to ambient temperature. Add the fibers to a hammer mill, using an 0.050 mesh screen.

6.0 MIXING PROCEDURE (Continued)

Machine Mixing: Add ingredients of Paragraph 4.2, Part A to the jacketed mixing vessel of the Kady Mill. Mix the components in a water cooled mixing vessel in the Kady Mill for 20 minutes. Sample the dispersion until a Hegeman reading of 1+ is obtained. Strain the dispersion through a cheese cloth filter, test for % non-volatiles, and add the requisite methyl ethyl ketone for proper solvent balance. Return the dispersion to the Kady Mill, add the fibers slowly, and disperse for an additional 2 to 3 minutes. Drain the contents of the Kady Mill dispersion into an appropriate mixing vessel. Next, add the proportional amount of the solution of Paragraph 4.2, Part B, using mechanical agitation to ensure thorough mixing. Finally, add proportional amount of the solution of Paragraph 4.2, Part C, as the activator portion, using mechanical agitation again to ensure thorough mixing. The blended components are now prepared for application with a working pot life of 4 hours. The Morehouse Mill may be substituted for the Kady Mill for initial grinding of Part A before fiber addition. The following technique shall be adhered to if the Morehouse Mill is used: one pass at a 3-mil stone separation followed by two passes at a 1-mil stone separation. Fiber addition shall be completed only after the dispersion has been adjusted with methyl ethyl ketone and transferred to the Kady Mill as described earlier in the paragraph.

7.0 WET COATING REQUIREMENTS

7.1 Qualifications. The wet coating furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

7.2 Part A, 313 Thermal Protection Coating.

7.2.1 Viscosity. The viscosity of Part A of the 313 thermal protection coating before fiber addition shall be 600 - 1200 cps and after fiber addition shall be 2500 - 3200 cps when tested as specified.

7.2.2 Weight Per Gallon. The weight per gallon of Part A of the 313 thermal protection coating shall be 10.7 ± .1 pounds/gallon when tested as specified.

7.2.3 Non Volatiles %. The non-volatiles of Part A of the 313 thermal protection coating shall be 74 ± 1 when tested as specified.

7.2.4 Weight % Polysulfide. Weight % LP-3 polysulfide extracted from Part A shall be 13 ± 1 gm when tested as specified.

7.2.5 Weight % Solids. The combined weight % ammonium-p-nitroaniline-o-sulfonate and Refrasil filtered from Part A shall be 60 ± 3 gms when tested as specified.

7.2.6 Equivalent Weight. The equivalent weight of the sample from 7.2.5 shall be 235 ± 12 based upon an adjustment for a theoretical value of 88.8% ammonium salt and 11.2% fibers when examined as specified.

7.2.7 Test for Intumescence. A sample from 7.2.5 shall intumesce when tested as specified.

7.3 Part B, 313 Thermal Protection Coating.

7.3.1 Viscosity. The viscosity of Part B of the thermal protection coating shall be 80 - 100 cps when tested as specified.

7.3.2 Weight Per Gallon. The weight per gallon of Part B of the thermal protection coating shall be 8.9 ± .1 pounds/gallon when tested as specified.

7.4 Part C, 313 Thermal Protection Coating.

7.4.1 Viscosity. The viscosity of Part C of the thermal protection coating shall be 10 - 15 cps when tested as specified.

7.4.2 Weight Per Gallon. The weight per gallon of Part C of Thermal Protection Coating shall be 7.6 ± .1 pounds/gallon when tested as specified.

8.0 QUALITY ASSURANCE PROVISIONS

8.1 Responsibility for Inspection. Unless otherwise specified in the contract or the purchase order, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The processing activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to the prescribed requirements.

8.2 Classification of Examinations and Tests. All of the examinations and testing of the thermal protection coating shall be classified as qualification tests. Qualification tests shall be performed on samples submitted for approval as qualified products and shall be performed on individual lots of products submitted for acceptance. The qualification tests are listed in Table 1, Page 9. A certified test report from the supplier shall accompany each product submitted for approval and each lot of thermal protection coating submitted for acceptance.

8.3 Sampling. The material preparation shall conform to the requirements of Paragraph 4.2 herein. A test report from the supplier shall accompany each sample to be tested, and shall show the composition number and the rest results for all requirements of this specification and shall refer specifically to the applicable paragraphs herein. All the thermal protection coating manufactured and formulated as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

8.3.1 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of thermal protection coating. If the results of test of the second sample fail to meet the specification requirements, the entire lot shall be rejected.

8.4 Part A, 313 Thermal Protection Coating.

8.4.1 Viscosity. Use a Model RVT Brookfield Viscometer with #2 spindle at 10 RPM or a Model LVT Brookfield Viscometer with #2 spindle at 12 RPM and a temperature of 68°F. The result shall conform to Paragraph 7.2.1. However, viscosity measurements have been made on only a limited number of samples and acceptable ranges relative to application have yet to be determined. See report attached.

8.4.2 Weight Per Gallon. The test method shall be as described in Federal Test Methods Standard #141A, Method 4184.1. The result shall conform to Paragraph 7.2.2.

8.4.3 Non Volatiles %. The test method shall follow ASTM D 1644, with the following modification. The heating schedule shall be 194°F at 10 inches mercury for 30 minutes followed by 194°F at 30 inches mercury for 1 hour. The result shall conform to Paragraph 7.2.3.

8.4.4 Weight % Polysulfide. After careful stirring, weigh out a 100 gm sample of Part A and mix in approximately 100 ml toluene. Separate the slurry by suction filtration and wash the solids with 2 additional portions (100 ml) of

8.4.4 Weight % Polysulfide. (Continued)
toluene. Collect the filtrate and evaporate the solvent in a tared flask using a rotary evaporator with gentle heating. Calculate the residue as % polysulfide. Theory 13.1 gms.

8.4.5 Weight % Solids. Dry the solids collected from 8.4.4 on a tared filter paper to constant weight at 167°F in a circulating oven. Theory 60.0 gma combined salt-Refrasil.

8.4.6 Equivalent Weight. Weigh out 3.0 gms solids from 8.4.5 and adjust to theoretical weight of ammonium salt (x 0.88). Adjusted weight is 2.65 gms. Suspend the material in 100 ml of 50% by volume aqueous methanol and titrate with 1.0 NaOH using a recording electrometric titrator. From the volume of the titrant to reach end point, calculate the equivalent weight. The result shall conform to Paragraph 7.2.6.

8.4.7 Linear Expansion (Intumescence). Place a dry sample from 8.4.5 into an oven at 660°F. Material shall exhibit linear expansion.

8.5 Part B, 313 Thermal Protection Coating.

8.5.1 Viscosity. Use a Model RVT Brookfield Viscometer with #2 spindle at 50 RPM and a temperature of 68°F. The result shall conform to Paragraph 7.3.1.

8.5.2 Weight Per Gallon. The test method shall conform to Paragraph 8.4.2 and the result to Paragraph 7.3.2.

8.6 Part C, 313 Thermal Protection Coating.

8.6.1 Viscosity. Use a Model RVT Brookfield Viscometer with #2 spindle at 100 RPM and a temperature of 68°F. The result shall conform to Paragraph 7.4.1.

8.6.2 Weight Per Gallon. The test method shall conform to Paragraph 8.4.2 and the result to Paragraph 7.4.2.

TABLE 1

WET COATING REQUIREMENTS

Part A	Requirement	Test Method
Viscosity	7.2.1	8.4.1
Weight Per Gallon	7.2.2	8.4.2
% Non Volatiles	7.2.3	8.4.3
% Polysulfide	7.2.4	8.4.4
% Solids	7.2.5	8.4.5
Equivalent Weight	7.2.6	8.4.6
Intumescence	7.2.7	8.4.7
Part B		
Viscosity	7.3.1	8.5.1
Weight Per Gallon	7.3.2	8.5.2
Part C		
Viscosity	7.4.1	8.6.1
Weight Per Gallon	7.4.2	8.6.2
Part [illegible]		
W [illegible]		

9.0 APPLICATION PROCEDURE

Substrate Preparations.

Cold Rolled Steel. Follow standard good painting procedures. Be sure all surfaces are dry and clean. Remove loose dust, paint, grease, and oil residue from untreated surface. If sand-blasted, use Crystal Amber No. 3, sand, fresh water washed stock #5350-543-4398, and 80 - 100 psi air pressure. No scale should be present on the surface. Final treatment utilizes a methyl ethyl ketone wash followed by rinsing with Freon TF degreaser (Miller-Stephenson 180 or equivalent). Surfaces are allowed to air-dry and, if necessary, they may be treated with corrosion resistant zinc chromate primer meeting Federal Specification TT-P-664b or Red Lead Primer 13-k-51 from Mobile Chemical Company, conforming to Federal Specification TT-P-86e, Type 3.

Aluminum. Follow standard good painting procedures. Treat surface of aluminum by scrubbing with scouring powder and water. Flush liberally with water and observe for water break-free surface. If surface is not water break-free, repeat scrubbing and flushing procedure. When water break-free surface is obtained, flush surface with trichloro-ethylene and allow surface to air dry. Handle cleaned surface with care. Avoid contact with dust, oils, and grease. If corrosion resistant primer is desired, use zinc chromate primer conforming to Federal Specification TT-P-666B. If anodizing of aluminum is required, the treatment shall conform to MIL-A-8625. For an alodine treated surface, the aluminum shall conform to MIL-C-5541.

Glass. Follow standard good painting procedures. Glass is cleaned by first wiping the surface with methyl ethyl ketone, followed by a rinse with Freon TF degreaser (Miller-Stephenson 180 or equivalent).

Spray Method.

Spray apply the 313 Thermal Protection Coating prepared from Paragraph 6.0 with a Devilbiss #MBC-546 equipped with a fluid nozzle #AV632-D and aircap #MB-4039-69. Spray coating using 19.5 CFM with pot pressure of 4 - 6 psi and line pressure of 45 psi. The gun is held level at all times between 6 - 8 inches away from the panels, with no fanning motion permitted. Avoid spraying closer than 6 inches or material will run, giving an uneven coating. If the gun is held too far away, dry spraying occurs, causing a mottled appearance with concomitantly poor coating adherence to the substrate. Each pass will give a thickness of approximately 0.005 inches. Allow the solvent to evaporate in between each pass to prevent sagging or floating before applying subsequent coating. In general, the thicker the coating, the longer drying period required between passes. The coating shall air-dry at ambient conditions for 72 hours minimum before

Spray Method. (Continued)

proceeding with quality assurance provisions. Note: there is one exception, in that the tensile specimens are peeled from the glass substrate after 18 hours and before 24 hours after spraying. Air-drying for a total of 72 hours will then continue.

10.0 QUALITY ASSURANCE PROVISIONS

10.1 Responsibility for Inspection. Unless otherwise specified in the contract or the purchase order, the supplier is responsible for the performance of all the inspection requirements as specified herein. The supplier may utilize his own facility or any commercial laboratory acceptable to ARC. The processing activity, or its designated representative, reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

10.2 Classification of Examinations and Tests. All of the examinations and testing of the thermal protection coating shall be classified as qualification tests. Qualification tests shall be performed on samples submitted for approval as qualified products and shall be performed on individual lots of products submitted for acceptance. The qualification tests are listed in Table 2, Page 14. A certified test report from the supplier shall accompany each product submitted for approval and each lot of thermal protection coating submitted for acceptance.

10.3 Sampling. Samples for product qualification or for qualified lot acceptance shall be as specified in Paragraph 10.3.2. The material preparation shall conform to the requirements of Paragraph 4.2 herein. A test report from the supplier shall accompany each sample to be tested, and shall show the composition number and the test results for all requirements of this specficiation and shall refer specifically to the applicable paragraphs herein. All the thermal protection coating manufactured and formulated as one batch shall be considered a lot and shall be numbered as such for purposes of inspection.

10.3.1 Rejection and Retest. Failure of any lot to conform to all applicable requirements of this specification shall be cause for retest. Property or properties in question shall be retested on new specimens prepared from the same lot of thermal protection coating. If the results of tests of the second sample fail to meet the specification requirements, the entire lot shall be rejected.

10.3.2 Specimens. All samples to be tested shall be coated with the thermal protection coating at a minimum dry film thickness of 0.020" on cold rolled steel panels or as free films.

10.3.2.1 Size and Number of Specimens. The size and number of specimens for conformance to each requirement is indicated in Table 3, Page 15.

10.3.3 Test Methods. Unless otherwise specified, all examinations and tests shall be performed with a thermal protection coating conditioned for a minimum of seven days, or 170 hours, at a temperature of 73.5 ± 3.5°F and a R.H. of of 50 ± 5%.

10.3.3.1 Visual Inspection. Visual inspection shall be performed to determine the appearance requirements of Paragraph 4.3.1.1 and Paragraph 4.2.

10.3.3.2 Tensile Strength. The tensile strength of the thermal protection coating shall be determined in accordance with ASTM D 2370, except that the coating will be sprayed on 6" X 6" glass plates. Preparation of the panels shall be as follows: Wash the surface with methyl ethyl ketone and apply four average coats of a fluorocarbon release agent (Miller-Stephenson 122 or equivalent). Note: Peel the coating free of the glass substrate within 18-24 hours and cut specimens for tensile testing at that time.

10.3.3.3 Density. The density of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.3.

Procedure: Material remaining from Paragraph 10.3.3.2 shall be used to determine the coating density. The volume of coating is measured by air pycnometer and then the weight is determined from an analytical balance. The density of the coating in gm/cc is reported.

10.3.3.4 Temperature of Intumescence and Char Yield. The temperature of intumescence and char yield of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.4.

Procedure: Using a thermogravimetric analysis (TGA) apparatus, record the weight loss versus temperature for the coating at a heating rate of 5°F/minute in a nitrogen atmosphere to 1200°F. Report the char yield (Y_c) as the percent weight fraction remaining at 1112°F, and the temperature of intumescence as the range where the weight loss is greatest.

10.3.3.5 Abrasion Resistance. The abrasion resistance of the thermal protection coating shall be determined according to Taber Abraser Weight Loss Method and the results shall conform to Paragraph 4.3.1.5.

10.3.3.6 Flexibility. The flexibility of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.6.

Procedure: Using a Gardner Impact Test, a round nosed, two pound impact rod is allowed to fall a calibrated distance (measured in feet) upon the coated side of the specimen panel. The foot-pounds of energy is increased until coating adhesion failure occurs. The greatest energy tolerable is reported in foot-pounds.

10.3.3.7 Linear Expansion (Intumescence). The linear expansion of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.7.

Procedure: The edges of the test specimen as well as a 1/8 inch perimeter overlap of the coating and the exposed cold rolled steel substrate shall be brushed with a red lead primer meeting Federal Specification TT-P-86e, Type 3, to minimize corrosion of the uncoated edges. Two of the four specimens are further conditioned for seven days at 100 ± 3.5°F and 90 ± 2% R.H. The coating specimens are placed coating side up in an oven preheated to 660°F and allowed to remain for 30 minutes. Gases should be vented from the oven. Remove after 30 minute period, allow to cool, then calculate the linear expansion:

$$\text{Linear Expansion} = \frac{Tf - Ti}{Ti}$$

where Tf = Average thickness of coating after intumescence, inches

Ti = Initial coating thickness, inches

10.3.3.8 Thermal Efficiency. Thermal efficiency of the thermal protection coating shall be determined according to the following procedure and the results shall conform to Paragraph 4.3.1.8.

Procedure: This test, to be performed at ARC, exposes the coating to a fire environment where a total flux of 10 BTU/Ft^2-sec is 90% radiative in a slightly fuel rich condition. Aviation fuel (JP-4) is used to fire the unit. The specimens are prepared on special 3" X 3" X 1/16" cold rolled steel substrates. That is, a 1" diameter hole in the center of the panel has a 15/16" diameter 1/16" thick cold rolled steel disc cemented in place with epoxy novolac. This provides a higher thermal resistance to reduce thermal conductance effects from the edges of the specimen panel. The coated panel thickness is measured and recorded and the panel preconditioned to Paragraph 10.3.3 or other special environments and when ready to test is inserted in a transite mask or holder which allows the panel to rest on about 1/16" to 1/8" of the edges of the coated surface. At time, t=0, the masked panel is placed over the fire box and a thermocouple in contact with the back of the panel records the temperature rise of the center disc in the panel. The flux is uniform over the entire panel and the center disc is about 8-10% of the exposed area and is representative of a larger, semi-infinite panel. From the time-temperature history thus obtained, and the thickness in mils of coating tested, one reports the time in seconds to reach a given temperature and the mil thickness coating. Since the flame environment is a more severe test of the coatings, the intumescent thickness is also reported after this test.

TABLE 2

QUALIFICATION TESTS

Examination or Test	Requirement	Test Method
Material	4.2	10.3.3.1
Appearance	4.3.1.1	10.3.3.1
Tensile Strength	4.3.1.2	10.3.3.2
Density	4.3.1.3	10.3.3.3
Temperature of Intumescense and Char Yield	4.3.1.4	10.3.3.4
Abrasion Resistance	4.3.1.5	10.3.3.5
Flexibility	4.3.1.6	10.3.3.6
Linear Expansion	4.3.1.7	10.3.3.7
Thermal Efficiency	4.3.1.8	10.3.3.8

TABLE 3

TEST SPECIMENS

Test	Substrate Size, Inches	Number of Specimens	Requirement	Test Method
Appearance	3 X 3 X 1/16 CRS	1	4.3.1.1	10.3.3.1
Tensile Strength	6 X 6 free films	2	4.3.1.2	10.3.3.2
Density	Use above	-	4.3.1.3	10.3.3.3
Temperature of Intumescence and Char Yield	Use above	-	4.3.1.4	10.3.3.4
Abrasion Resistance	4 1/8 X 1/16 disc CRS with 1/4 hole in center	6	4.3.1.5	10.3.3.5
Flexibility	Use above	-	4.3.1.6	10.3.3.6
Linear Expansion	3 X 3 X 1/16 CRS	4	4.3.1.7	10.3.3.7
Thermal Efficiency	3 X 3 X 1/16 CRS with center disc (See 10.3.3.8)	2	4.3.1.8	10.3.3.8