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PLASTICIZED PHENOLPHTHALEIN POLYCARBONATE

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for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

FOREWORD

This report presents the work performed by Aerotherm Division of Acurex Corporation on NASA Contract NAS2-8012, Modification 2 entitled "Plasticized Phenolphthalein Polycarbonate" during the period 15 May 1975 to 30 June 1976. The program was sponsored by the Chemical Project Research Office, National Aeronautics and Space Administration, Ames Research Center, Moffett Field, California 94035. The program was under the administration of Dr. George Fohlen.

This program was conducted in the Nonmetallic Materials Department of Aerotherm Division under the direction of Mr. C. B. Delano. Mr. E. S. Harrison, Senior Chemist, was the principal investigator with Dr. D. N. Vincent, Consultant, Messrs. R. T. Rafter, Senior Chemist, and M. E. McKee, Chemical Technician assisting.

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ABSTRACT

Phenolphthalein polycarbonate was successfully plasticized with polychlorinated biphenyls (e.g., Aroclor 1231) or tricresyl phosphate and cast from tetrahydrofuran to give clear films without loss of fire resistance. At loadings of 20 to 30 percent plasticizer the T_g was lowered to approximately 100°C which would render phenolphthalein polycarbonate easily moldable. Although these materials had some mechanical integrity as shown by their film forming ability, the room temperature toughness of the plasticized polymer was not significantly improved over the unmodified polymer.

OBJECTVE

The objective of the program was to improve the moldability, toughness and abrasion resistance of phenolphthalein polycarbonate to make it more acceptable for use in thin film form as a decorative overlay in aircraft interiors without detracting from its excellent fire resistance.

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SECTION 1

INTRODUCTION

Phenolphthalein polycarbonate has recently been shown (Reference 1) to give much greater char yield than bisphenol-A polycarbonate. For example, the phenolphthalein system results in a 54 percent char yield while the bisphenol-A polycarbonate yields only 20 percent residual char at 800°C under anaerobic conditions. This property makes phenolphthalein polycarbonate an excellent candidate for use as a film in aircraft interior decorative overlays. However, phenolphthalein polycarbonate presents some difficulty in processing as the thermal decomposition temperature is only slightly higher than the temperature necessary to process the polymer. An additional problem is that while commercial polycarbonate has excellent toughness, phenolphthalein polycarbonate does not. Even though high molecular weight polymer has been prepared, films made from it are brittle.

The addition of a plasticizer to a polymer system results in the effective lowering of the PMT and T_g resulting in greater ease of processing and could, as is the case in many polymer systems, give increased toughness. If a flame retardant plasticizer could be used, the fire resistant properties of the material should be maintained.

SECTION 2

TECHNICAL DISCUSSION

2.1 ESTIMATION OF THE SOLUBILITY PARAMETER

Based on the theoretical structure of phenolphthalein polycarbonate, the solubility parameter, δ, was calculated from molar-attraction constants by the method of Small, yielding a value of 9.9 $(cal/cc)^{1/2}$. This value is in agreement with the observed solubility behavior, reported previously (Reference 2) and summarized in Table 1. Morgan reports that phenolphthalein polycarbonate, and the phthalein polyesters in general, are subject to acid-base associations with solvents. Thus, while the polymer is soluble in dichloromethane, a hydrogen donor, and in tetrahydrofuran, a hydrogen acceptor, the addition of one of these solvents to a solution of the polymer in the other causes polymer precipitation.(Reference 3).

2.2 POTENTIAL PLASTICIZERS (REFERENCES 4 AND 5)

The following table (Table 2) lists a number of potential plasticizers for phenolphthalein polycarbonate. The solubility parameters were calculated from the heats of vaporization which were estimated from reported boiling points. In the absence of the required boiling point data, molar attraction constants were used to estimate the solubility parameter from the molecular structure.

2.3 PREPARATION OF PHENOLPHTHALEIN POLYCARBONATE

Using the procedures of Morgan (Reference 3) phenolphthalein polycarbonate was prepared with inherent viscosities in the range of 1.3 - 1.8 (0.5 percent CH_2Cl_2). When the techniques developed for the small scale reactions were translated into larger scale (one gallon blender, 80 g. monomer) the inherent viscosities dropped to near 0.6 and the yields fell from approximately 90 to 60 percent. This was due to the viscosity buildup brought about by molecular weight increase which severely restricts adequate stirring, essential for efficient interfacial polymerization. Increased stirrer speed is effective during the earlier stages of the reaction but as molecular weight increases, the stirrer speed must be increased to a very high level to adequately disperse the

TABLE 1. SOLUBILITY PARAMETERS

Poorly H-Bonding Solvents		Moderately H-Bonding Solvents		Strongly H-Bonding Solvents	
Benzene	9.2 I	Tetrahydrofuran	9.1 S	Piperidine	8.7 S
Chloroform	9.3 S	Methyl ethyl ketone	9.3 I	M-cresol	10.2 S
Methylene chloride	9.7 S	Acetone	9.9 I	Formic acid	12.1 I
1,2-dichloroethane	9.8 S	Cyclohexanone	9.9 S		
		1,4-dioxane	10.0 S		
		Dimethyl acetamide	10.8 S		
		Dimethyl sulfoxide	12.0 S		

Note: S = Soluble

I = Insoluble

TABLE 2. CALCULATED SOLUBILITY PARAMETERS FOR VARIOUS PLASTICIZERS

Material	B.P.(mm)	Density	M.W.	δ
Triphenyl phosphate	413.5(760)	1.2055	326	9.05
Tri p-tolyl phosphate	244 (3.5)	1.247	368	8.92
Tri m-tolyl phosphate	260 (15)	1.150	368	8.34
Tri 2,5-xylyl phosphate	263 (8)	1.197	410	8.26
Tri phenyl phosphite	360 (76)	1.1844	310	8.62
Tri o-tolyl phosphite	238 (11)	1.1423	352	8.29
Tri p-tolyl phosphite	253 (10	1.1313	352	8.45
1-chloronaphthalene	259.3(760)	1.1938	163	10.40
2-chloronaphthalene	256 (76)	1.1377	163	10.11
1,2-dichloronaphthalene	297 (76)	1.3147	197	10.48
1,7-dichloronaphthalene	285 (76)	1.2611	197	10.09
Tris (2-chloroethyl) phosphate	203 (10)	1.425	285	9.78
Tris (2,3-dibromopropyl) phosphate		2.2	697	10.8
40% hydrogenated terphenyl	368 (76)	1.004	237	9.17
40% chlorinated paraffin		1.25	972	10.6
60% chlorinated paraffin		1.45	1213	11.2
N-methyl benzene sulfonamide	225 (12)	1.26	171	12.0
Poly (methyl α-chloroacrylate)				10.1
Poly (vinyl chloride)				9.7
Poly (vinyl chloride/vinyl acetate) 87/13				10.5
Poly (vinyl chloride/propylene) 97/3				10.1
Poly (vinyl chloride/propylene) 90/10				9.1
Poly (vinylidine chloride/ethyl acrylate) 90/10				

reactants. This produces a large amount of heat. The resultant temperature rise brings about several other problems:

a. Hydrolysis rate (depolymerization) is apparently increased

b. Solvent (dichloroethane) is lost even through the attached reflux condenser

c. Most important, an inverse temperature effect is observed, i.e., polymer solution viscosity increases with increasing temperature

By readjusting concentration, addition rates, stirrer speeds, etc., reproducibly good yields of high molecular weight material ($\eta_{inh} > 1.3$, MW > 155,000" (Reference 1), in >85 percent yield) were successfully obtained. The procedures are described in the Experimental Section.

2.4 PREPARATION OF FILMS FROM PHENOLPHTHALEIN POLYCARBONATE

Films (2 mil thick) were cast on cleaned glass plates (doctor blade setting 0.023 inch) using a solution of phenolphthalein polycarbonate (13.5 weight percent) in tetrachloroethane and dried overnight under ambient conditions, followed by a step-wise heatup to 175°C (25° increments after 1 hour exposures). At this point the clear film still held 17 percent residual solvent as shown by thermogravimetric analysis. Figure 1 shows the TGA scan obtained from the high molecular weight ($\eta_{inh} = 1.66$) polymer. It shows the loss of solvent beginning at 175°C and complete at 352°C.

Penetration studies showed the deflection temperature under load (DTUL) to occur at ~174°C for the unplasticized film dried at 175°C for 1 hour, at ~210°C for the same film dried an additional 15 minutes at 225°C and at ~268°C after an additional 20 minutes at 300°C (all drying >175°C done under N_2). After the final exposure the film had darkened slightly and was found be only about 90 percent soluble in dichloromethane, indicating degradation and crosslinking had taken place. Figure 2 shows the DTUL scans under a 10 gram load (~45 psi) at a scan rate of 15°C/min. The reported T_g of 270°C for phenolphthalein polycarbonate corresponds quite well with the DTUL after the 300°C dry.

The film was soaked in hexane for a week with essentially no change in solvent content (residual solvent content of ~15 percent compared to the original 17 percent). Figure 3 shows the TGA scan after this exposure.

2.5 PLASTICIZATION OF PHENOLPHTHALEIN POLYCARBONATE

Films of plasticized phenolphthalein polycarbonate were cast as described in the previous section. A polymer solution of known concentration was prepared and to this a weighed amount of

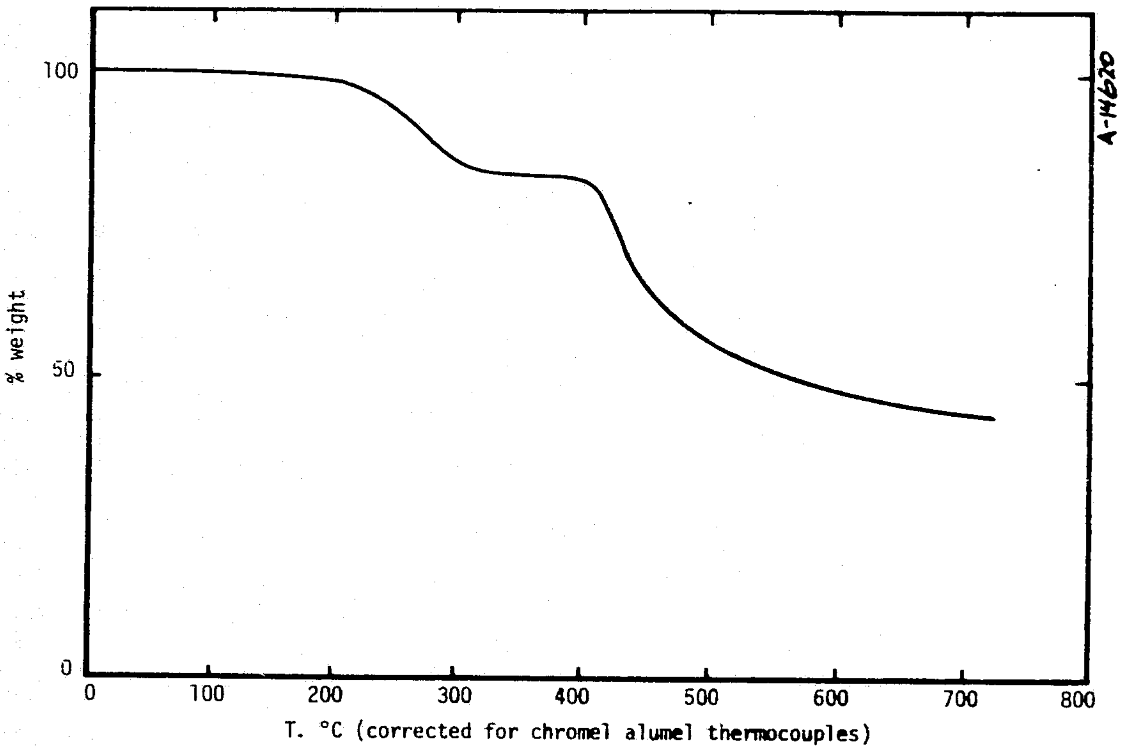

Figure 1. TGA of phenolphthalein polycarbonate.

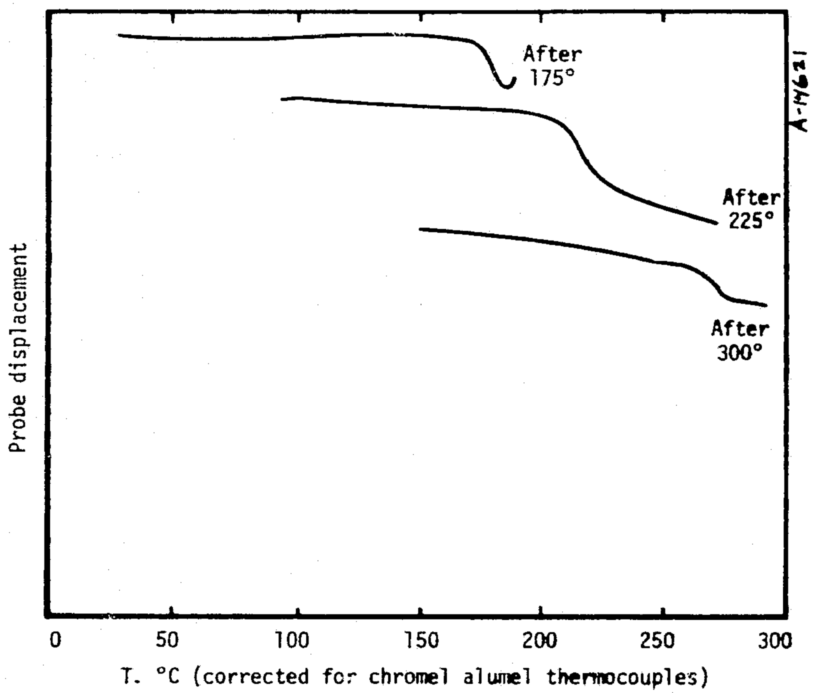

Figure 2. Penetration of phenolphthalein polycarbonate film.

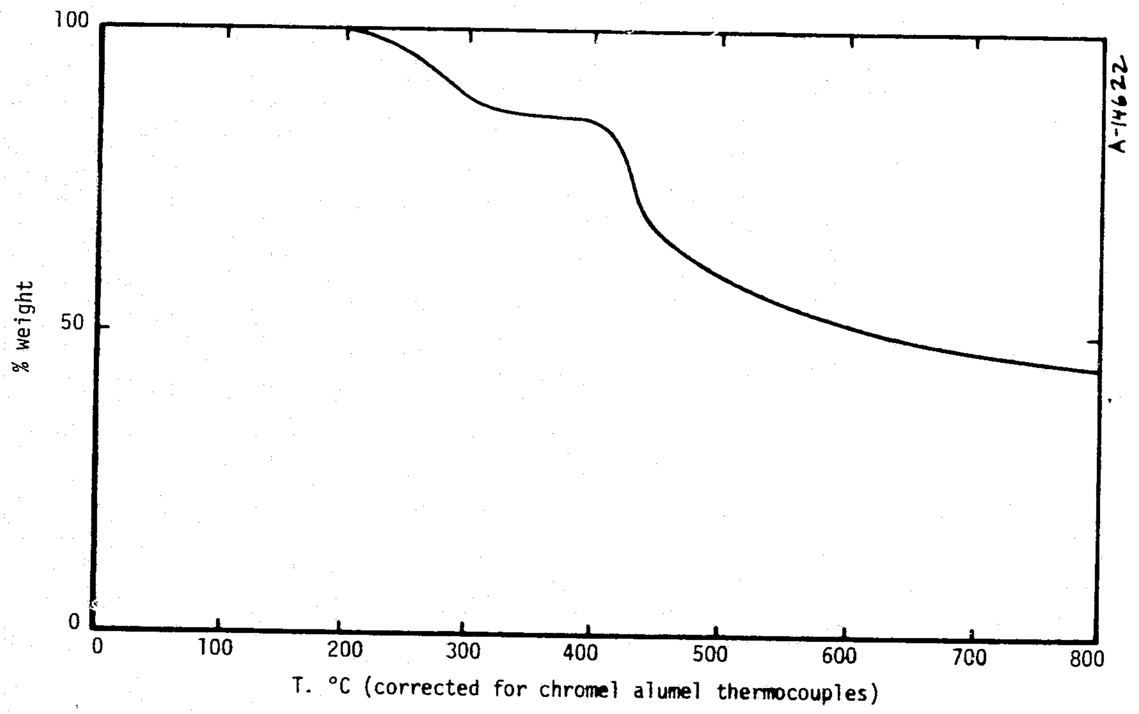

Figure 3. TGA of phenolphthalein polycarbonate film after hexane extraction.

plasticizer dissolved in the same solvent was added to the weighed stock solution. Films were then cast from generally three different concentrations (percent plasticizer/polymer weight), allowed to dry overnight and then heated over a relatively long period (perhaps 8 hours) to the final drying temperature.

Because of the difficulty in removing chlorinated solvent such as tetrachlorethane, dichloroethane and chloroform, other solvents were examined which resulted in the use of tetrhydrofuran or dioxane. THF is removed to be less than 1 percent residual concentration by heating for a few hours at 150°C, dioxane on the other hand requires a few hours at 180°C. Even though the relatively high volatility of these solvents makes preparation of quality films difficult (skins rapidly), we used these two solvents almost exclusively during the remaining studies.

Volatility of the various candidate plasticizers also makes close control of plasticizer content difficult using this procedure which was to serve only as a preliminary screening method.

The flammability requirements of the plasticizer severely limited the available choices and the best candidates seemed to be polychlorinated biphenyls (Aroclors) and phosphates (Santicizers and Kronitox 100).

A series of polychlorinated biphenyls (PCB) were obtained from Monsanto (Aroclor 1231, 1232, 1242, 1248, 1254, and 1260), with higher numbers corresponding to higher chlorine content. Aroclors 1231 through 1248 were compatible with the polymer up to 30 percent, but 1254 and 1260 were not compatible with the polymer in the absence of solvent as evidenced by haze, nonuniform patches and sometimes lack of film integrity at higher loadings (Table 3) (Reference 4).

The residual plasticizer content after drying was established by measuring weight loss to 340°C under nitrogen after solvent removal. Generally a plateau was established by that temperature and the polymer had not yet begun decomposition. Figure 4 is a TGA of Aroclor 1231 and Figure 5 is a TGA of a film cast from dioxane containing Aroclor 1231 at 30 percent by weight of the polymer dried at 180°C for 3 hours. At 340°C a weight loss of 18 percent is experienced which is attributed to plasticizer content after solvent removal.

The cast films were all given qualitative evaluation. All were brittle in that recreasing was impossible even if creasable initially. This lack of toughness prompted us to only determine softening point (by penetration at 45 psi compressive load) and total plasticizer content on a very few of the samples. The softening point measurement showed that in all cases the T_g was lowered greatly with values as low as 86°C. This means that fabrication would be quite easy compared with unplasticized phenolphthalein polycarbonate, but molding efforts were not carried out due to lack of toughness in the films.

TABLE 3. PLASTICIZED PHENOLPHTHALEIN POLYCARBONATES

Plasticizer	Casting Concentration	Solvent	Qualitative Clarity	Film Thickness	Qualitative Toughness	Tg by TMA	Measured Concentration by TGA	Remarks
"Aroclors"[a]								
1231	20%	DIOX.	Clear	0.0034"	Not creasable			Slight burning in flame/self-extinguishing
1231	20%	THF	Clear	0.0033"	Fingernail creasable, but brittle	~89°		Burning in flame — continues to burn outside of flame for brief period
1231	30%	DIOX.	Clear	0.0052"	Fingernail creasable, but brittle		~18% wt loss @ 340°C	
1231	30%	THF	Gen. clear, patchy haze	0.0040"	Fingernail creasable, brittle			
1231	70%	DIOX.	Clear, mottled	0.0030"	Fingernail creasable brittle to edge tear		~21% wt loss @ 340°C	
1231	70%	THF	Cloudy, slimey					
1232	20%	DIOX.	Clear	0.0034"	Not creasable, brittle			Burning in flame — contines to burn outside of flame for brief period
1232	20%	THF	Clear, patchy haze	0.0041"	Not creasable, brittle			
1232	30%	DIOX.	Clear	0.0046"	Fingernail creasable, but brittle	~86°C		
1232	30%	THF	Patchy haze gen. clear	0.0038"	Fingernail creasable, brittle			

[a] Chlorinated biphenyls, Monsanto Chemical

TABLE 3. Continued

Plasticizer	Casting Concentration	Solvent	Qualitative Clarity	Film Thickness	Qualitative Toughness	Tg by TMA	**Measured Concentration by TGA**	Remarks
1232	70%	DIOX.	Patchy haze, mottled	0.0060"	Cheesy			Same as above, but flame appeared longer and more violent
1232	70%	THF						
1242	20%	DIOX.	Clear	0.0049"	Brittle, not creasable			
1242	20%	THF	Clear	0.0037"	Brittle, not creasable	~94°		
1242	30%	DIOX.	Clear	0.0043"	Brittle, not creasable			Self-extinguishing/ burning outside <1 sec
1242	30%	THF	Patchy haze (air bubbles)	0.0050"	Fingernail creasable brittle			Self-extinguishing, no after burn
1242	70%	DIOX.	Haze	0.0182	Cheesy			Stronger flame than lower concentration, but self-extinguishing
1242	20%	THF						
1248	20%	DIOX.	Clear	0.0047"	Brittle, not creasable			Self-extinguishing, no after burn
1248	20%	THF	Clear with bubble haze	0.0035"	Brittle, not creasable			Self-extinguishing with slight after burn <1 sec
1248	20%	DIOX.	Clear	0.0044"	Brittle, not creasable			Self-extinguishing with slight after burn <1 sec
1248	30%	THF	Clear with bubble haze	0.0073"	Brittle, not creasable			No after burn
1248	70%	DIOX.	Cloudy	0.0143"	Cheesy			Lots of flames, and smoke self-extinguishing
1248	70%	THF						

TABLE 3. Continued

Plasticizer	Casting Concentration	Solvent	Qualitative Clarity	Film Thickness	Qualitative Toughness	Tg by TMA	**Measured Concentration by TGA**	Remarks
1254	70%	DIOX.	Clear	0.0048"	Brittle, not creasable			Self-extinguishing
1254	20%	THF	Hazy	0.0056"	Creasable			Self-extinguishing
1254	30%	DIOX.	Clear	0.0062"	Brittle, not creasable		~28% wt loss @ 340°C	Self-extinguishing, brief after flame <1 sec
1254	30%	THF	Hazy		Brittle			Self-extinguishing, brief after flame <1 sec
1254	70%	DIOX.	Hazy	0.092"	Brittle, not creasable		~56% wt loss @ 340°C	Self-extinguishing, no after flame, much smoke
1254	70%	THF						
1260	20%	DIOX.	Clear with bubbles	0.0064"	Brittle, not creasable			Self-extinguishing, after flame <1 sec
1260	20%	THF	Translucent haze	0.0046"	Fingernail creasable tears, brittle			Self-extinguishing, no **after flame**
1260	30%	DIOX.	Clear with large bubble		Brittle			
1260	30%	THF	Translucent haze		Fingernail creasable tears, brittle			
Tri-phenyl phosphite	20%	DIOX.	Hazy		No integrity whatsoever			
Tri-phenyl phosphite	30%	DIOX.	Hazy					
Tri-phenyl phosphite	40%	DIOX.	Hazy					

TABLE 3. Continued

Plasticizer	Casting Concentration	Solvent	Qualitative Clarity	Film Thickness	Qualitative Toughness	Tg by TMA	Measured Concentration by TGA	Remarks
TCP[a]	20%	DIOX.	Clear, mottled	0.0036	Fingernail creasable brittle	~89°C	16% wt loss at 350°C	9% wt loss @ 240°C self-extinguishing, after flame
TCP	20%	THF	Clear with bubbles	0.0032	Fingernail creasable tears, brittle	~117°C		8% wt losss @ 340°C, self-extinguishign, after flame
TCP	30%	DIOX.	Clear, mottled	0.0054	Brittle nor creasable	~112°C		Self-extinguishing, no after flame
TCP	40%	DIOX.	Clear with bubbles	0.0041	Fingernail creasable tears, brittle	~103°C	13% wt loss @ 340°C	
TCP	40%	DIOX.	Clear, mottled	0.0034	Fingernail creasable tears, brittle	~88°C		
TCP	40%	THF	Clear, mottled with bubbles	00.41	Fingernail creasable tears, brittle		4% wt loss @ 340°C	
Kronitox 100[b]	20%	DIOX.	Clear, mottled	0.0035	Brittle not creasable	~97°C		
Kronitox 100	20%	THF	Clear with bulles	0.0023	Fingernail creasable tears, brittle			
Kronitox 100	30%	DIOX.	Hazy, mottled		No integrity			
Kronitox 100	30%	THF	Clear, patchy haze		Fingernail creasable tears, brittle		10% wt loss @ 340°C	
Kronitox 100	40%	DIOX.	Hazy, mottled		No integrity			

[a] Tricresyl phosphate

[b] Triisopropylphenyl phosphate, Monsanto

TABLE 3. Concluded

Plasticizer	Casting Concentration	Solvent	Qualitative Clarity	Film Thickness	Qualitative Toughness	Tg by TMA	Measured Concentration by TGA	Remarks
Kronitox 100	40%	THF	Hazy with bubbles		Cheesy			Self-extinquishing brief after burn <1 sec
DRAPEX 3.2[a]	20%	THF						Incompatible with polycarbonate
DRAPEX 3.2	10%	THF						Incompatible with polycarbonate
SANTICIZER 140[b]	20%	THG	Clear with bubbles		Fingernail creasable tears, brittle	9% wt loss at 340°C		
SANTICIZER 141[c]	20%	THF	Translucent with bubbles		Brittle, very little integrity			
SANTICIZER 148[d]	20%	THF	Opaque gray		Fingernail creasable brittle			Incompatible with polycarbonate
SANTICIZER S-22	20%	THF						
SANTICIZER P-95	20%	THF						

[a] Octyl epoxy stearate, Argus Chemical Co.

[b] Cresyl diphenyl phosphate, Monsanto

[c] Octyl diphenyl phosphate

[d] Octyl diphenyl phosphate

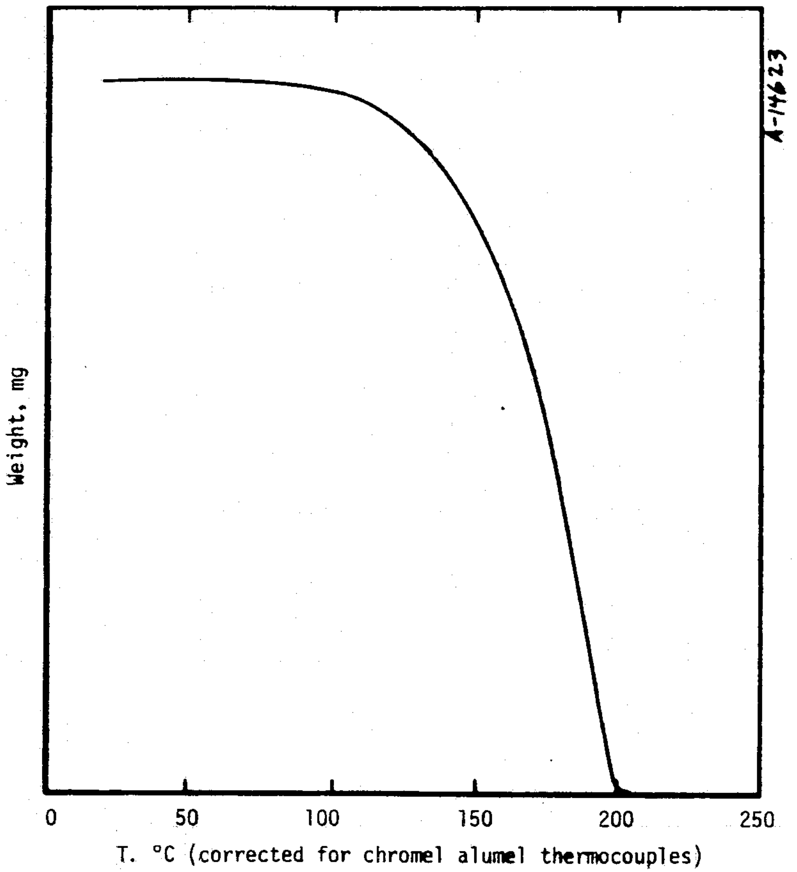

Figure 4. TGA of Aroclor 1231.

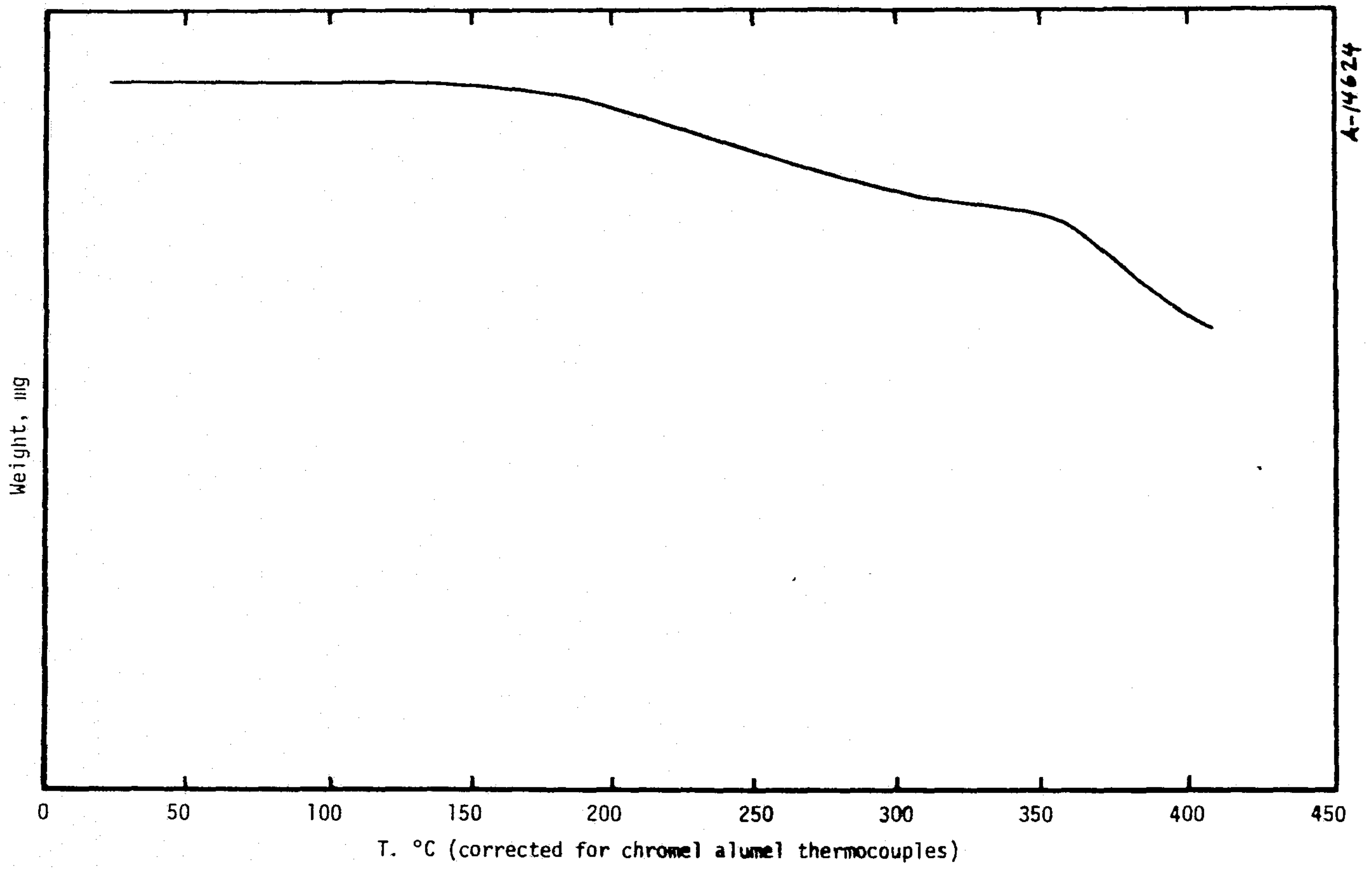

Figure 5. TGA of Phenolphthalein polycarbonate film cast with 30% Aroclor 1231 from dioxane and dried @ 180°C for 3 hours.

The flame resistance of the plasticized films was essentially unchanged from the unplasticized material and all were self-extinguishing.

At this point, it was obvious that plasticizer could be used for phenolphthalein polycarbonate to lower the processing temperature and still maintain the flame resistance. However, the goal of producing a tough film was still unaccomplished. The phosphate plasticizers were next chosen for study. Tricresyl phosphate (TCP), triisopropylphenyl phosphate (Kronitox 100), and cresyl diphenyl phosphate (Santicizer 140) were compatible with phenolphthalein polycarbonate while octyl diphenyl phosphate (Santicizer 141), isodecyl diphenyl phosphate (Santicizer 148) and triphenyl phosphate are not (Table 3). Figure 6 is the TGA of a phenolphthalein polycarbonate cast with 20 percent cresyl diphenyl phosphate (Santicizer 140) in THF and dried for 8 hours at 100°C. The TGA shows an 8 percent plasticizer content which is slowly lost from 100°C to 325°C. As in the case of the chlorinated biphenyls, the phosphate gave clear films which had low T_g's and good fire resistance, but poor toughness. Some increase in toughness over the unplasticized film was attained. but not enough to be of practical use.

Some plasticizers, particularly those with long aliphatic chains, were incompatible with phenolphthalein polycarbonate. For example, when a 15 percent solution of the polymer is mixed with a 10 percent solution of dioctylphthalate in THF, the polymer immediately precipitates. This is undoubtedly caused by the disparity between the cohesive energy density of the polymer and potential plasticizer. An incompatible plasticizer could sometimes be used with a plasticizer of similar structure which was compatible with the polymer. For example, good compatibility was achieved with 2/1, 1/1, and 1/2 mixtures of TCP and either ocyyl or isodecyl diphenyl phosphates (Santicizer 141) and 148).

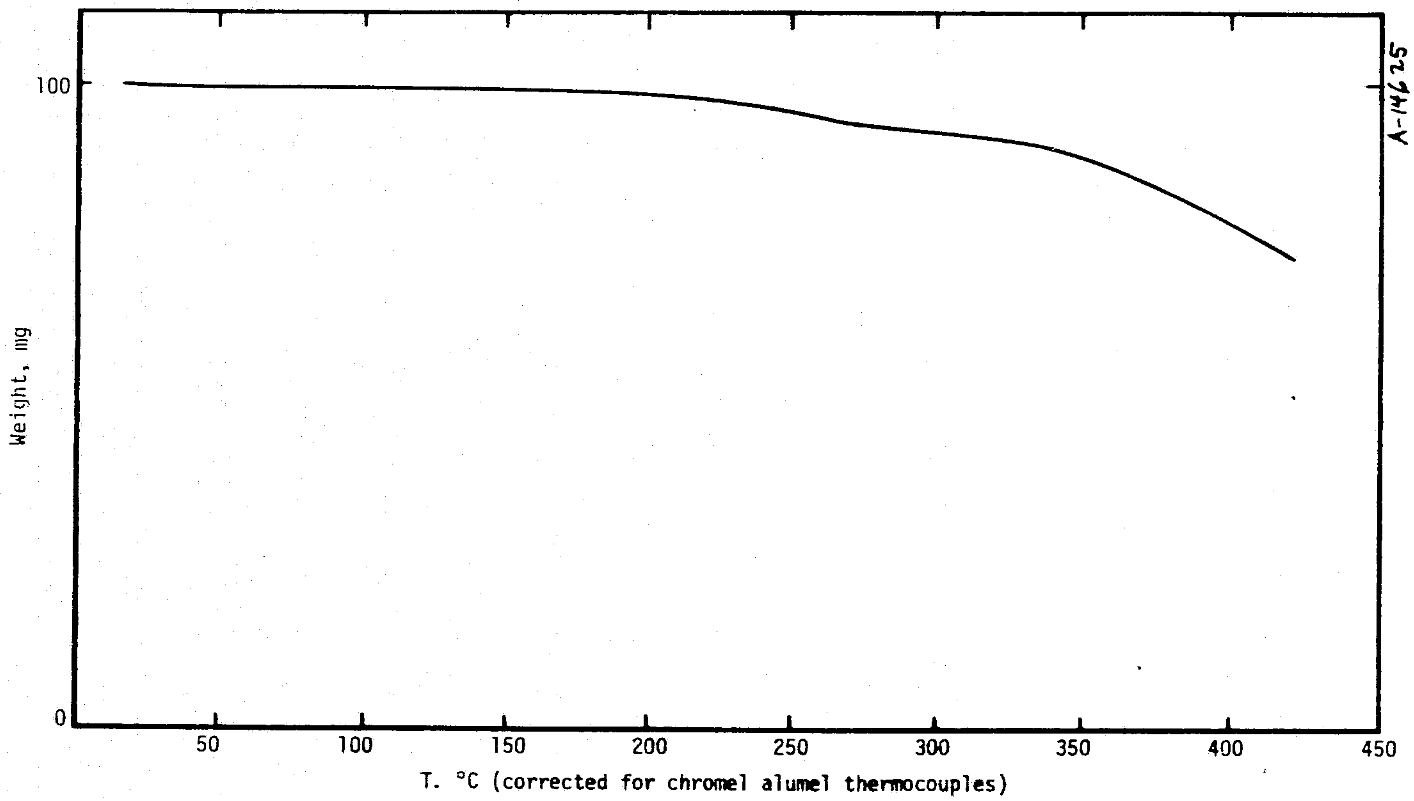

Figure 6. TGA of phenolphthalein polycarbonate film cast with 20% Santicizer 140 in THF and dried 8 hours @ 100°C.

SECTION 3

CONCLUSIONS AND RECOMMENDATION

The phenolphthalein moiety presents a very stiff chain segment with the three aromatic rings at roughly right angles to each other, sterically hindering chain motion. However, the carbonate linkage does provide relatively free rotation, leading to a very bulky, coiled molecule, but with some level of segmental mobility. The possibility of long range intramolecular attractions between the mildly electropositive aromatic lactone ring and the carbonate carbonyl can further restrict chain motion as can the possibility of crosslinking through opening of the phenolphthalein lactone. A bulky, stiff-chain polymer such as this would require much longer than normal chain lengths to develop a sufficient degree of chain entanglements to allow for high plasticizer loadings while maintaining reasonable mechanical strength. Plasticizer efficiency appears to be moderately good, as evidenced by the 160°C - 180°C decrease in T_g at the 10 - 30 percent level of either PCB or TCP. However, even with such a reduction, the T_g is still considerably above ambient temperature (85°C - 100°C) and the polymer is still in the glassy state. Although some toughening was observed by qualitative examination, practical levels of film flexibility and strength were not achieved.

It is recommended that other approaches to attaining tough, fire-resistant polycarbonate polymers with a higher heat distortion temperature than bisphenol-A polycarbonates be persued. Block and random copolymers and possibly polymer blends may hold such promise.

SECTION 4

EXPERIMENTAL

Preparation of Phenolphthalein Polycarbonate

A 1 gallon Waring blender was equipped with an ice-cooled water jacket, addition funnel containing 25 percent sodium hydroxide solution, reflux condenser protected by an inert atmosphere and phosgene bubbler. Phenolphthalein (Aldrich Chemical Co.) (80 g, 0.25 mole), sodium hydroxide (20 g, 0.5 mole), trimethyl ammonium chloride (20 g) as an emulsifying agent, sym-dichloroethane (1.0 ℓ) and distilled water (1.2 ℓ) were added and moderate agitation was begun. The water jacket on the stainless steel blender kept the reaction cool throughout. Stirrer speed was continuously adjusted to avoid over-heating but still provide adequate mixing. Phosgene gas was bubbled into the reactants at a rapid rate until the purple mixture became colorless (ca. 15 min.). Sodium hydroxide (10 ml, 25 percent solution) was added regenerating the purple color and phosgene addition resumed until the mixture was again colorless (ca. 2 minutes of phosgene gas required for subsequent additions).

This process of alternately adding phosgene and base was repeated 12 - 14 times* at which time the colorless reaction mixture after phosgene addition had attained a pH of 10 - 11. Further base addition at this point only turns the milk-white mixture a slight pink. Pyridine (5 ml) was then added and phosgene addition continued until the reaction was neutral to Litmus paper. The mixture was allowed to separate (usually required standing overnight) and the organic phase washed successively with water to remove salts, then with dilute base, dilute acid, and finally water until neutral.

The organic solution was then poured into churning acetone in a Waring blender to precipitate the polymer as a white powder. The phenolphthalein polycarbonate was collected on a filter, washed with hexane and dried in vacuo at 85°C. Yields were generally 65 g-75 g (ca. 90 percent) with η_{inh} of >1.3 (0.5 percent in CH_2Cl_2 at 25.0°C)

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*For lower molecular weight polycarbonate, this cycle should be repeated only 3 - 5 times, depending on D.P. sought.

Preparation of Phenolphthalein Polycarbonate Film

Films (2 mil thick) were cast on clean glass plates (doctor blade setting 0.023 inch) using a solution of phenolphthalein (ca. 10 percent) and plasticizer in an appropriate solvent and dried overnight at ambient conditions followed by additional drying at elevated temperatures.

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