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Demetrius A. Kourtides

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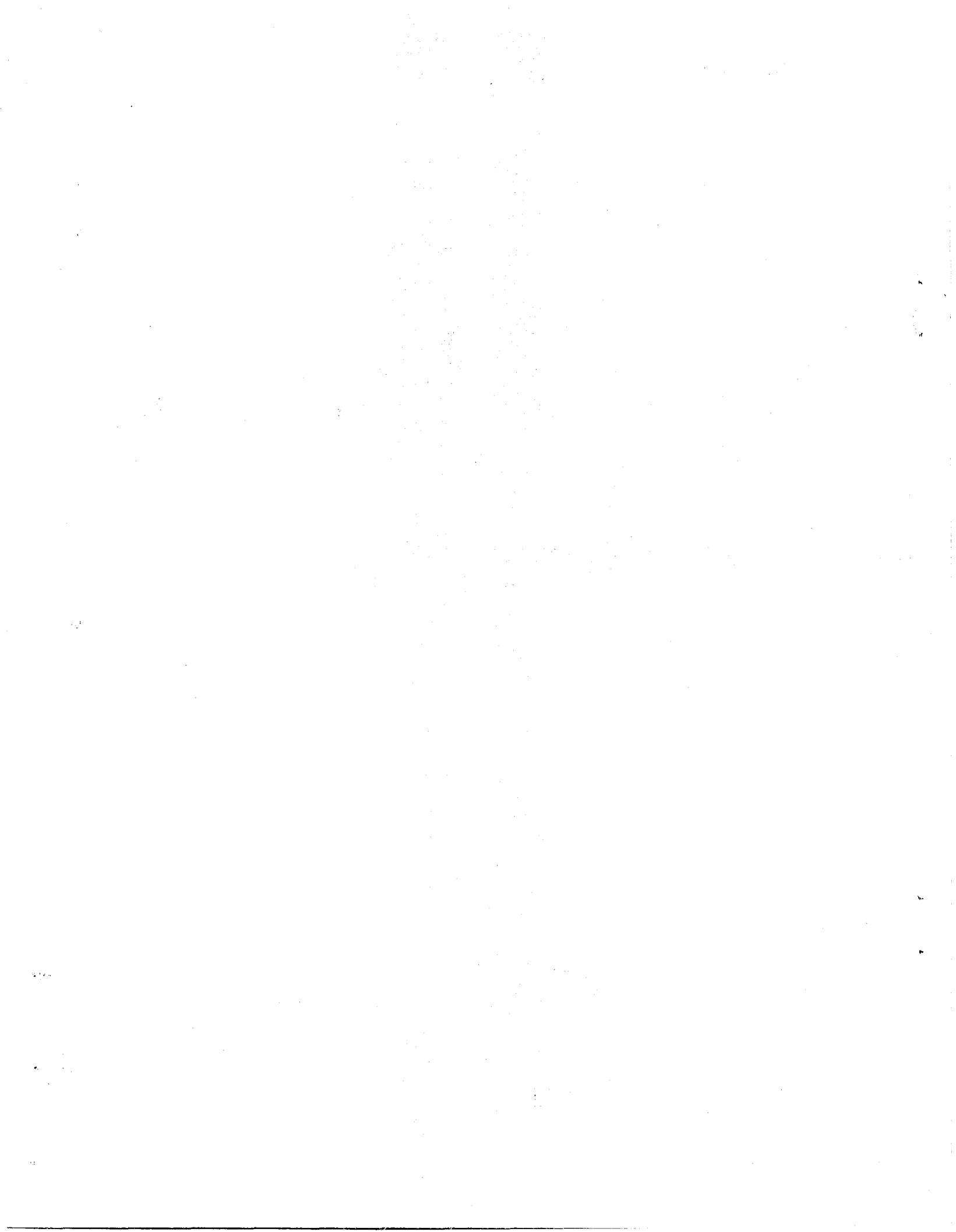National Aeronautics and
Space Administration

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Fire-Retardant Decorative Inks for Aircraft Interiors

Zohar Nir, Makhteshim, Ltd., Beersheba, Israel
John A. Mikroyannidis, University of Patras, Patras, Greece
Demetrius A. Kourtides, Ames Research Center, Moffett Field, California

FIRE-RETARDANT DECORATIVE INKS FOR AIRCRAFT INTERIORS

Zohar Nir,[*] John A. Mikroyannidis,[†] and Demetrius A. Kourtides

Ames Research Center

SUMMARY

Commercial and experimental fire retardants were screened for possible use with acrylic printing inks on aircraft interior sandwich panels. The fire retardants were selected according to their physical properties and thermostabilities. Thermostabilities were determined by the techniques of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). A criterion was then established for selecting the more stable agent. Results showed that some of the bromine-containing fire retardants are more thermostable than the acrylic ink, alone, used as a control. Also, the bromine-containing fire retardants yield even better limiting oxygen index values when tested after adding carboxy-terminated butadiene acrylonitrile (CTBN) rubber.

INTRODUCTION

In this study, candidates were screened to select fire-retardant additives for the acrylic inks used on aircraft interior sandwich panels. These candidates were to be additives that might not migrate (leach), such as reactive fire retardants, and those polymeric fire retardants in which migration is diffusion-controlled.

The following criteria were set up for candidate selection:

1. Soluble in MEK (the acrylic ink solvent)

2. Thermostable

3. Has a potential reaction site, or a polymeric backbone

4. Contains halogens or phosphorus, or both

5. Able to be processed with the acrylic ink

6. Compatible with the curing cycle of the ink

The first two formulations in table 1 were selected from a variety of available vinyl-terminated fire retardants that met the above criteria.

The F2001P (no. 3, table 1), a commercial brominated fire retardant (ref. 1), contains 50% Br. The family of these brominated polymeric additives (BPA) is available at various molecular weights ranging from 660 to 40,000. Chemically, the family has two glycidal groups, and includes aromatic bromine (which is very thermostable). The BPA (F2001P) which was selected is soluble in MEK, and has an epoxy equivalent (EE) of 545. This fire retardant was used as a control and a reference in a series of formulations (nos. 4-7, table 1), in which different modifications of the basic polymeric backbone were accomplished.

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[*]Currently at Makhteshim, Ltd., Beersheba, Israel.
[†]Currently at the University of Patras, Patras, Greece.

Recent research (ref. 2) showed that carboxy-terminated butadiene acrylonitrile (CTBN) rubber improves the fracture toughness of various thermosets (refs. 3 and 4); consequently, we wished to determine its effect on fire-retardant acrylic inks relative to a control.

In that research, rubber-toughened BPA (F2001P) was synthesized by reacting F2001P with liquid CTBN rubber, using ethyl triphenyl phosphonium iodide (ETPPI) or triphenyl phosphine (TPP) as a catalyst. This produced a concentration of prereacted rubber in BPA (ref. 2). Molecules with concentrations of up to 50% CTBN were synthesized to obtain a block polymer of the general type shown in figure 1. Two of the glycidyl groups were consumed, and the molecular weight increased accordingly. The CTBN was now entrapped between two BPA units, thus retaining its elastomeric properties. The resulting block polymer still had two glycidyl ether groups.

The introduction of CTBN rubber decreases the bromine content of the BPA; therefore, modification with brominated phenols is required to keep an acceptable bromine concentration. As a result, the two glycidyl end groups are further reacted with brominated phenols, using the same catalyst, to produce a polymer that is capped at both sides with the brominated phenoxy group (fig. 2). This procedure increases the bromine content, the extent of the increase being dependent on the selected brominated phenol and the loaded amount of the CTBN rubber.

We used two different models of brominated phenols: tribromophenol (TBP) and pentabromophenol (PBP). In a typical modification (fig. 3), the bromine content was increased to 58% and 64% with no rubber (no. 4, table 1) and to 53% with 12% rubber (no. 5, table 1).

Model compounds were synthesized, with and without CTBN rubber. In both cases, the F2001P was blocked through its diglycidyl end groups with the brominated phenols (fig. 3). The resulting products may serve as fire-retardant candidates, as well as impact modifiers, for both fire-retardant thermoplastics and thermosets.

The incorporation of these fire retardants was technically successful. However, in flame-spread tests, difficulties were encountered in evaluating the fire-retardant properties of the fire-retardant-coated Tedlar sheets and the acrylic films made from the acrylic ink.

MATERIALS

The following materials were used.

1. F2001P: brominated polymeric additive (BPA); epoxy equivalent (EE) 545; 50% bromine content; mp 60°-70°C; Makhteshim Chemical Works, Israel.

2. Tribromophenol (TBP): mp 95°-96°C; 72% Br; Great Lakes Chemical Corp.

3. Pentabromophenol (PBP): mp 229°-230°C; 82% Br; Great Lakes Chemical Corp.

4. Ethyl triphenyl phosphonium iodide (ETPPI): Thiokol/Garstab Corp.

5. Carboxy-terminated butadiene acrylonitrile (CTBN) rubber: Hycar 1300 × 13; B. F. Goodrich Chemical Co.

6. 2,3 Dibromopropylacrylate (AE-59): Great Lakes Chemical Corp.

7. Bis(β-chlorethyl)vinyl phosphonate: Stauffer Chemical Co.

8. Acrylic ink: K. C. Coatings, Inc.

9. Tedlar: E. I. du Pont de Nemours Co.

SYNTHESES

The various formulations (table 1) were calculated to contain the same loading of 10% bromine, or an equivalent analog amount of combined chlorine and phosphorus (no. 2, table 1). All formulations were adjusted to contain 60% w/v of solids as the original formulation of the acrylic ink.

Synthesis of Tribromophenol-Blocked Polymer of the Brominated Polymeric Additive (BPA): F2001P-TBP

A 2-liter resin kettle equipped with four standard inlets for nitrogen, mechanical stirrer, thermocouple, and chemicals loading was used as a standard reaction vessel. A 1090-g (2-eq) sample of F2001P (brominated polymeric diglycidyl with EE = 545) was introduced and heated until it was fully melted (70°-80°C). The temperature was then raised to 110°C, and 3.5 g (0.2%) of ethyl triphenyl phosphonium iodide was added in one portion to the melted reaction mixture. Tribromophenol (TBP) was then added in one portion, and the temperature in the reaction vessel fell to 90°C. The reaction continued at temperatures in the range of 100°-120°C for 150 min. The advancement of the reaction was monitored by titration to determine the acid number (AN) value of the residual phenolic groups. Aliquotes of the melted reaction mixture were taken every 40 min, dissolved in dioxane, and titrated with 0.1N KOH/EtOH using 1% alcoholic phenolphthalein as an indicator. At an AN of 0.75, the reaction was stopped by pouring the vessel's contents into an aluminum pan. The product was a glassy, amber, brittle solid. Its thermal properties are shown in no. 1, table 2.

Synthesis of Rubber-Toughened Brominated Polymeric Additive Blocked with TBP: F2001P-RTBP

The same equipment described in the preceding paragraph was used with the following materials.

1. F2001P, 1136.1 g (2.1 eq)

2. CTBN (Hycar 1300 × 13), 227.2 g (0.136 eq)

3. $EtPh_3P^+I^-$ (ETPPI), 2.7 g (0.2%) (first stage)

4. Tribromophenol (TBP), 349.1 g (1.96 eq)

5. ETPPI, 1.3 g (0.2%) (second stage)

The CTBN (a high-viscosity liquid) was heated in the reaction vessel under nitrogen to 80°C, to reduce its viscosity. At that temperature, the F2001P was added as

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fine powder. The mixture was heated to 100°C while being stirred continuously until fully homogenized.

The catalyst for the first stage was added, and the reaction was continued for 80 min. The reaction advancement was monitored by 0.1N KOH/EtOH titration to determine the equivalent per hundred grams (EPHR) of the residual carboxy group. The reaction continued until the EPHR-COOH (fig. 1) had reached at least 0.0012 (2% of the original EPHR).

Tribromophenol (TBP) was added in one portion, at temperatures in the range of 135°-140°C. TBP should be introduced carefully because it is subject to sublimation. An additional portion of the catalyst (ETPPI) was later added. Reaction time was 2 hr, and the temperature reached 150°C.

The reaction was terminated, after an acid number (AN) <1.0 was reached, by pouring the product into an aluminum pan. The product was a transparent, amber, brittle solid. Its thermal properties are shown in no. 2, table 2.

Synthesis of Pentabromophenol (PBP) blocked polymer of Brominated Polymeric Additive: F2001P-PBP

The same equipment previously described was used with the following materials: F2001P, 981 g (1.8 eq); PBP, 880 g (1.8 eq); and ETPPI, 2.79 g (0.15%). F2001P was introduced first, and heated until fully melted. At 110°C, small portions of PBP were introduced over a period of 10-15 min; temperature in the reaction vessel fell to 100°C.

The reaction was continued for 100 min, during which time the temperature was elevated to 170°C. The reaction was monitored by titration, as previously described. At an AN of <1.0 the reaction was stopped by pouring the product into an aluminum pan. The product was a transparent, amber, brittle solid. Its thermal properties are shown in no. 3, table 2.

Treatment of Synthesis Products

The above three products were brittle solids, and were treated separately as follows in order to reduce the particle size. The size reduction was accomplished by a ball mill, in the presence of 2% silica gel as a free-flowing agent. After the milling, the product became a free-flowing powder that showed no tendency to cake. If the reduction process was attempted without the anticaking agent, there was substantial caking, and the product stuck to the ball mill porcelain walls. Later on, it was discovered that the presence of the 2% fumed silica particles facilitates the phenomenon of afterglowing. Afterglowing is observed in flammability tests of specimens of the newly developed fire retardants in various thermoplastics.

In a different particle reduction process, the solid product was broken into smaller lumps which were hammer-milled to give a fine, powdery product. No caking was observed during the milling.

The yields of the syntheses were almost quantitative in the three products — F2001P-TBP, F2001P-RTBP, F2001P-PBP — although some difficulties were experienced in quantitatively taking the melted viscous product out of the reaction vessel. The

product was fully soluble in common organic solvents such as MEK, toluene, and dioxane.

FILM PREPARATION

Glass Plates

Films of acrylic ink, each containing a different fire-retardant additive, were cast on glass plates. At first the solvent was vacuum evaporated, and the films were then cured at 80°-100°C for 30 min in an air-draft oven.

The incorporation of the fire-retardant additives into the acrylic ink did not significantly affect the transparency of the casting films. However, as a result of adding the fire-retardant agent to the acrylic ink, some difficulties were experienced in preparing the casting films. In particular the casting films from the F2001P-RTPB, in both concentrations of CTBN rubber (nos. 5 and 7, table 1), adhered very tightly to the glass plates. (The films could not be peeled off without being destroyed.) In both cases, spraying the glass plates with a mold-release dry lubricant helped separate the ink films from the plates, but also reduced the films' transparency. Film no. 7 in table 1 was observed to be relatively brittle. After the inks were peeled off the plates, the thermochemical properties of the inks were examined by thermogravimetric analysis (TGA), and by differential scanning calorimetry (DSC).

Polyvinyl Fluoride (Tedlar) Sheets

By the same method used to prepare the glass plates, sheets of polyvinyl fluoride were covered by films of fire-retardant acrylic inks. Later, because no useful results were obtained by flame spread tests, the Tedlar sheets' flammability was evaluated by determining the limiting oxygen index (LOI) at room temperature (ref. 5). The smoke emission of the Tedlar films was also determined later.

THERMOCHEMICAL MEASUREMENTS

Thermogravimetric Analysis and Differential Scanning Calorimetry

Thermogravimetric analysis (fig. 4) and differential scanning calorimetry (fig. 5) techniques were used to evaluate relative thermostability of fire retardant formulations. These formulations were compared with an "as received" acrylic ink as a control. For ease of comparison, one should follow the line showing the acrylic ink control specimen.

Thermogravimetric analysis (TGA) was accomplished by using a du Pont 951 TGA cell and 1090 thermoanalyzer. Heating rates were 20°C/min, and the N_2 flow rate was 80 ml/min. On figure 4, the control starts to lose weight at 120°C, until the deflection point at 240°C. There, the specimens start to lose weight rapidly — 40% during a temperature interval of 40°C. These curves show that at temperatures above 120°C and below 240°C, these substances would contribute toxic gases; that is, in an actual fire situation, the observed weight loss would convert to a net contribution to the toxic gases. Figure 4 indicates that, in the 120°-240°C range, all the fire-retardant samples lose less weight (therefore are more stable) than the control (sample no. 0).

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The order of observed thermal stabilities, therefore, was established in descending order as shown on table 3. This relative order of stabilities shows the advantage of aromatic bromine versus aliphatic bromine, as observed by comparing F2001P derivatives and 2,3 dibromopropyl acrylate. The comparison of aromatic bromine with a combination of chlorine and phosphorus also favors the aromatic bromine.

In the differential scanning analysis (DSC) studies, the first thermal transformation for the acrylic ink occurs at 51°-52°C, as shown in figure 5. All formulations show a decomposition exotherm in the range of 210°-220°C, which is probably accompanied by the unzipping of the acrylic chain. The information gained from the DSC studies is, therefore, insufficient as a basis for judging the relative stability of the fire-retardant formulations.

Limiting Oxygen Index and Smoke Emission Tests

FAR 25-32 flame-spread tests (ref. 6) were used to evaluate the flammability properties of the polyvinyl fluoride sheets which were coated with fire-retardant acrylic inks. No useful results were obtained, however, because the sheets melted when the specimens were exposed to the flame. Different char lengths were obtained using the same material.

The flame-spread test according to the ASTM E162 test method (ref. 7) was also attempted. Again, the results were neither satisfactory nor reproducible. As soon as the flame started at the top, the polyvinyl fluoride sheet, coated with a fire-retardant acrylic ink, burned and contracted away from the flame source within a few seconds.

Because both of these flame-spread tests were ineffective for determining flammability, the limiting oxygen index (LOI) test was performed, and the established order of efficiency is shown in table 4.

In the order of efficiency, which differs from the order of thermal stability (table 3), the aliphatic bromine (with an LOI value of 31.2) is inferior relative to the chlorine and phosphorus combination. Only the F2001P has a lower LOI, namely, 30.2.

The smoke-emission characteristics of the candidate materials were determined by using the technique of smoke accumulation in an enclosure, and by testing the materials in the smoke density chamber (ref. 8). The apparatus was operated over a range of incident heat fluxes, 1.0 to 2.5 W/cm^2, to determine the response of the inks to various fire environments. The results are shown in table 5.

Stability of Fire Retardant Agents

The derivatives of F2001P show higher LOIs than the baseline acrylic ink. Remarkably, the higher the rubber content, the higher the LOI, the aliphatic chains of the CTBN rubber being potential reaction sites for the acrylic ink. This phenomenon was given further attention by doing TGA studies of the fire-retardant agents themselves (fig. 6). In this figure, it is apparent that the rubber-containing F2001P is less stable than either of the bromophenols-blocked F2001P, and the TBP-blocked F2001P is more stable than the PBP-blocked F2001P. CTBN rubber (as received) is very stable in comparison with the other three tested.

Thermostability correlated well with the LOI values — the less stable the aromatic bromine, the higher the LOI. It is likely that higher rubber concentrations would result in less stable polymers and would tend to supply more bromine to the flame zone. The aliphatic fire retardants are less stable and, therefore, do perform better than F2001P itself, which is more stable than its derivatives.

When the TGA results and the LOI results are compared, the following order in terms of fire-retardant effectiveness is established:

F2001P-RTBP (16% CTBN) > F2001P-PBP > F2001P-RTBP (12% CTBN)

The thermostabilities of other flame retardants do not compare favorably with that of the fire-retardant ink.

Further evaluation of mechanical and other properties of the ink (peel strength, ultraviolet resistivity, etc.) should be performed to determine the preferred fire-retardant formulation.

CONCLUSIONS

The fire resistance of the acrylic ink was enhanced by the use of the fire retardants. It was found that a modified brominated polymeric additive results in a more stable fire-retardant formulation. The fire-retardant acrylic-ink formulations are more thermally stable than the control, and higher by 4.3-4.9 limiting oxygen index units than the control.

It was also shown that the thermostability of a fire-retardant agent correlates with its LOI performance. Because this phenomenon is typical of certain polymers, an especially made formulation should be carefully adjusted to the polymer under investigation.

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TABLE 1.- FORMULATION OF FIRE RETARDANTS IN MEK CALCULATED FOR 100 ml INK
(60% SOLUTION w/v IN MEK)

Sample No.	Sample description	% hydrogen, Cl, P, or CTBN rubber	FR per 100 cm^3 ink MEK, g	Diluting[a] MEK, cm^3	% Br, (Cl), and (P)
1	2,3 Dibromopropyl acrylate	58.8 Br	12.3	15.3	10.0
2	Bis(β=chloroethyl)vinyl phosphonate	13.3 P 30.0 Cl	22.5	28.1	5.0 (P) 11.2 (Cl)
3	Brominated polymeric additive (F2001P)	50.0 Br	15.0	18.7	10.0
4	F2001P-Tribromophenol (TBP)	58.0 Br	12.5	15.6	10.0
5	F2001P-Rubberized tribromophenol (RTBP)[b]	53.0 Br +12.0 CTBN	13.9	17.4	10.0
6	F2001P-Pentabromophenol (PBP)	63.0 Br	11.3	18.8[c]	10.0
7	F2001P-Rubberized tribromophenol (RTBP)[d]	48.6 Br 16.2 CTBN	20.5	34.3	10.0

[a]Final ink concentration 60.0% w/v.
[b]CTBN rubber content 12.0%.
[c]50:50 dioxane:MEK.
[d]CTBN rubber content 16.2%.

TABLE 2.- THERMOGRAVIMETRIC (TGA) AND DIFFERENTIAL SCANNING CALORIMETRY (DSC)
CHARACTERIZATION OF REACTION PRODUCTS

Product	Br, %	MP DSC, °C	TGA (N$_2$ 80 ml/min, 20°C/min)			
			Onset, °C	Inflection, °C	Completion, °C	Char yield, %
(1) F2001P-TBP	58.0	80-81	369.7	391.9	413.0	10.4
(2) F2001P-RTBP	53.5	65-68	353.4	376.5	395.3	21.8
(3) F2001P-PBP	64.0	80	286.6	317.2	361.7	14.5

TABLE 3.- OBSERVED THERMAL STABILITIES

Order of thermal stability	Sample No.	Sample description
1	3	Brominated polymeric additive (F2001P)
2	5	F2001P — Rubberized tribromophenol (RTBP, 12.0% rubber)
3	7	F2001P — Rubberized tribromophenol (RTBP, 16.2% rubber)
4	4	F2001P — Tribromophenol (TBP)
5	6	F2001P — Pentabromophenol (PBP)
6	2	Bis(β-chloroethyl)vinyl phosphonate
7	1	2,3 Dibromopropyl acrylate
8	0	Acrylic ink

TABLE 4.- LIMITING OXYGEN INDEX (LOI) OF FIRE-RETARDANT INK (1-7) VERSUS CONTROL (0)

Sample No.	Sample description	LOI value	Δ LOI[a]
0	Acrylic ink (neat)	28.4	0
1	2,3 Dibromopropyl acrylate	31.2	2.8
2	Bis(β-chloroethyl)vinyl phosphonate	32.3	3.9
3	F2001P	30.2	1.8
4	F2001P-TBP	31.3	2.9
5	F2001P-RTBP[b]	32.7	4.3
6	F2001P-PBP	33.0	4.6
7	F2001P-RTBP[c]	33.3	4.9

[a]Percent difference with respect to sample 0.
[b]CTBN rubber content 12.0%.
[c]CTBN rubber content 16.2%.

TABLE 5.- SMOKE DENSITY OF FIRE-RETARDANT INK (1,2,3,5) VERSUS CONTROL (0)

Sample No.	Sample description	1 W/cm^2		2.5 W/cm^2	
		Ds-2 min	Dm	Ds-2 min	Dm
0	Acrylic ink (neat)	1.54	2.61	1.90	2.86
1	2,3 Dibromopropyl acrylate	1.96	8.44	3.52	8.98
2	Bis(β-chloroethyl)vinyl phosphonate	0.38	0.90	6.75	8.25
3	F2001P	0.41	0.51	5.38	5.83
5	F2001P-RTBP[a]	0.91	1.04	6.14	6.34

[a]CTBN (rubber content 12.0%).

O

△—[BPA]—△ O

+ HOOC—[(CH$_2$—CH=CH—CH$_2$)$_X$—(CH$_2$—CH)$_Y$—]$_Z$ COOH
                                                    |
                                                  C≡N

BROMINATED POLYMERIC ADDITIVE   **CARBOXY-TERMINATED BUTADIENE ACRYLONITRILE (CTBN) RUBBER**

ETHYL TRIPHENYL PHOSPHONIUM IODIDE (ETPPI)

         OH   OH   O
—CH$_2$—CH—[BPA]—CH—CH$_2$—O—C—[(CH$_2$—CH=CH—CH$_2$)$_X$—(CH$_2$—CH)$_Y$—]$_Z$ C—O—
                                                                    | ‖
                                                                   C≡N   O

BPA—CTBN—BPA TYPE REACTION PRODUCT

Figure 1.- Synthesis of rubber-toughened BPA.

O

△—[BPA]—[CTBN]—[BPA]—△ O

(1) BROMINATED PHENOLS:
(2) ETPPI (0.15%)

OH

Br$_n$

n = 3, 5

        OH   OH
⬡—O—CH—[BPA]—[CTBN]—[BPA]—CH—O—⬡
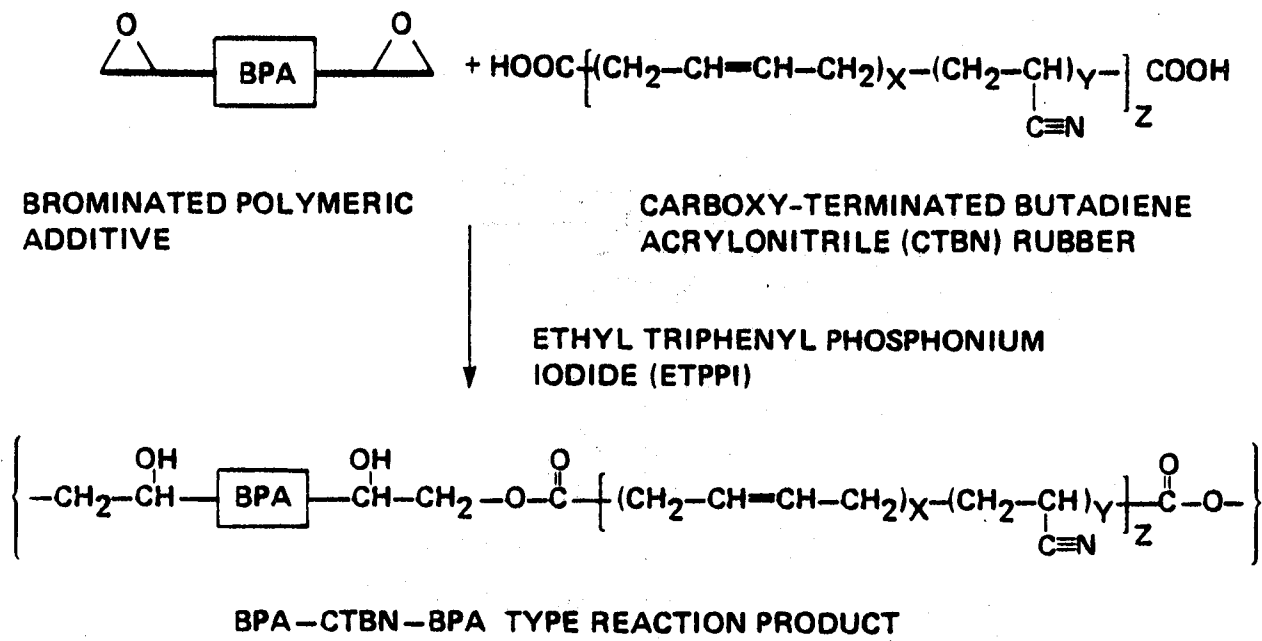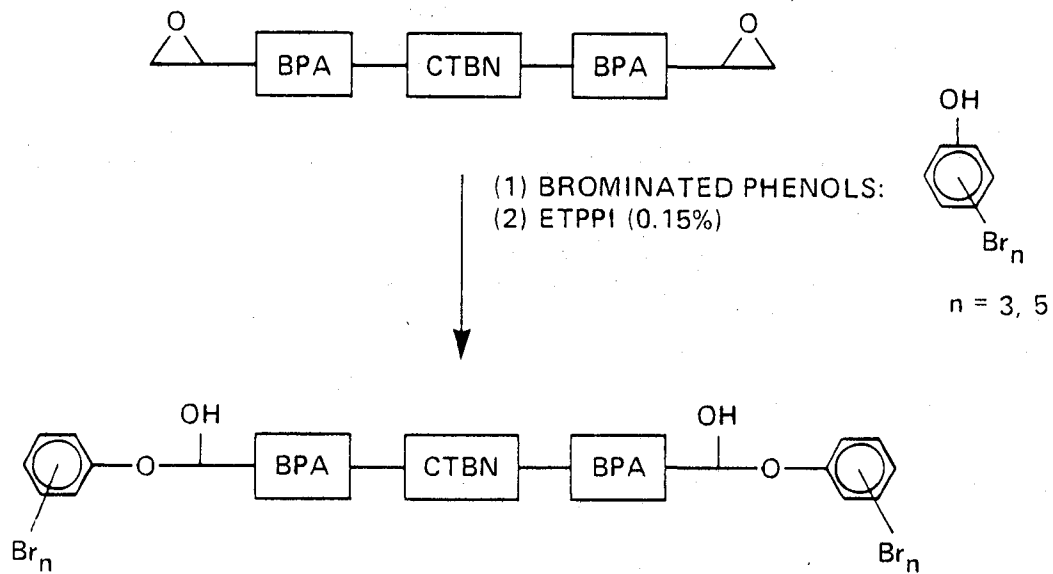| |
Br$_n$ Br$_n$

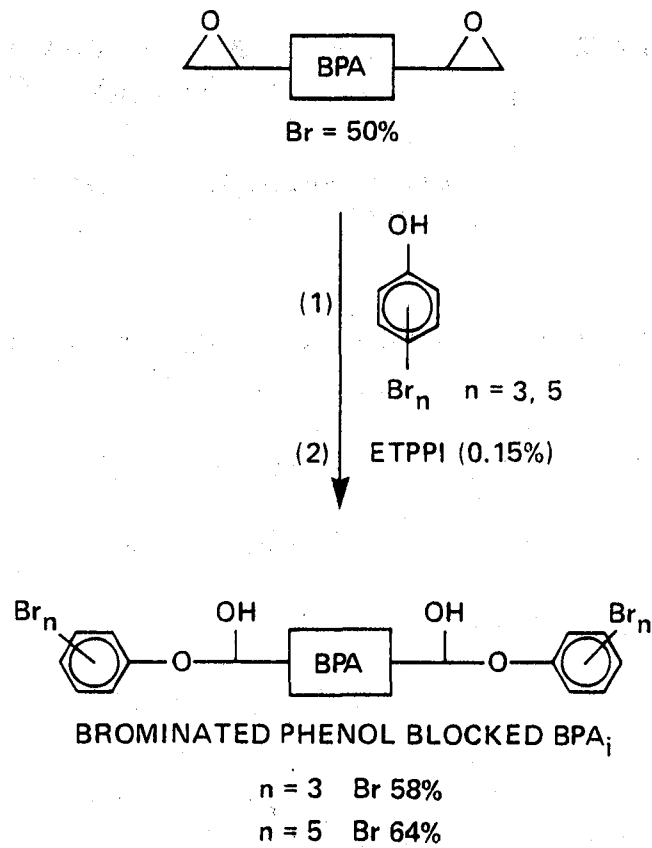Figure 2.- Synthesis of rubber-toughened, brominated phenols-blocked BPA.

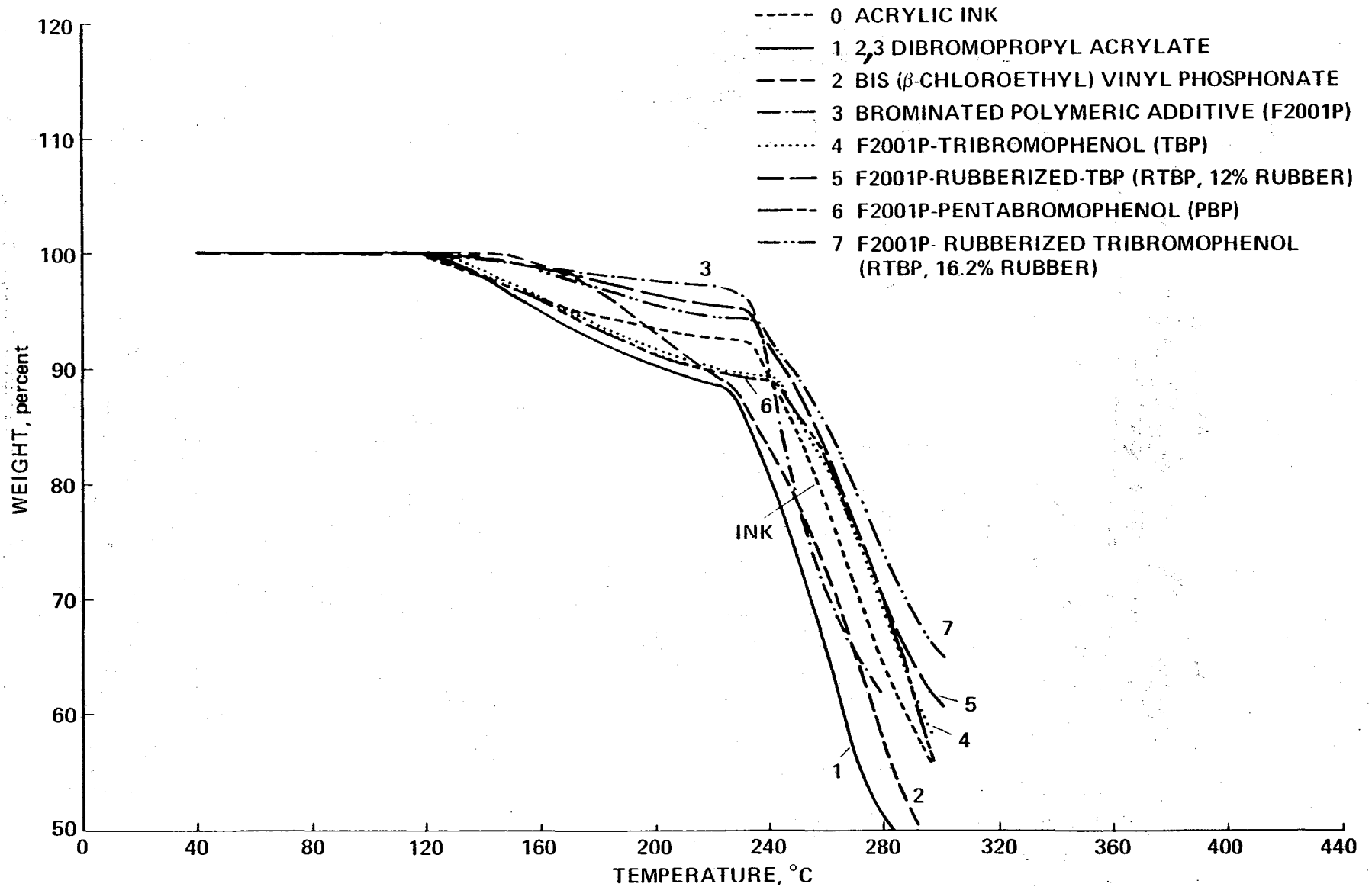Figure 3.- Synthesis scheme of brominated phenols-blocked BPA.

Legend

----- 0 ACRYLIC INK
_____ 1 2,3 DIBROMOPROPYL ACRYLATE
--- 2 BIS (β-CHLOROETHYL) VINYL PHOSPHONATE
—·— 3 BROMINATED POLYMERIC ADDITIVE (F2001P)
········ 4 F2001P-TRIBROMOPHENOL (TBP)
— — 5 F2001P-RUBBERIZED-TBP (RTBP, 12% RUBBER)
—·- 6 F2001P-PENTABROMOPHENOL (PBP)
—··— 7 F2001P- RUBBERIZED TRIBROMOPHENOL
(RTBP, 16.2% RUBBER)

Figure 4.- Thermogravimetric analysis of fire-retardant acrylic ink films (Nos. 1-7) versus control (0): nitrogen flow rate 80 ml/min; heating rate 20°C/min.
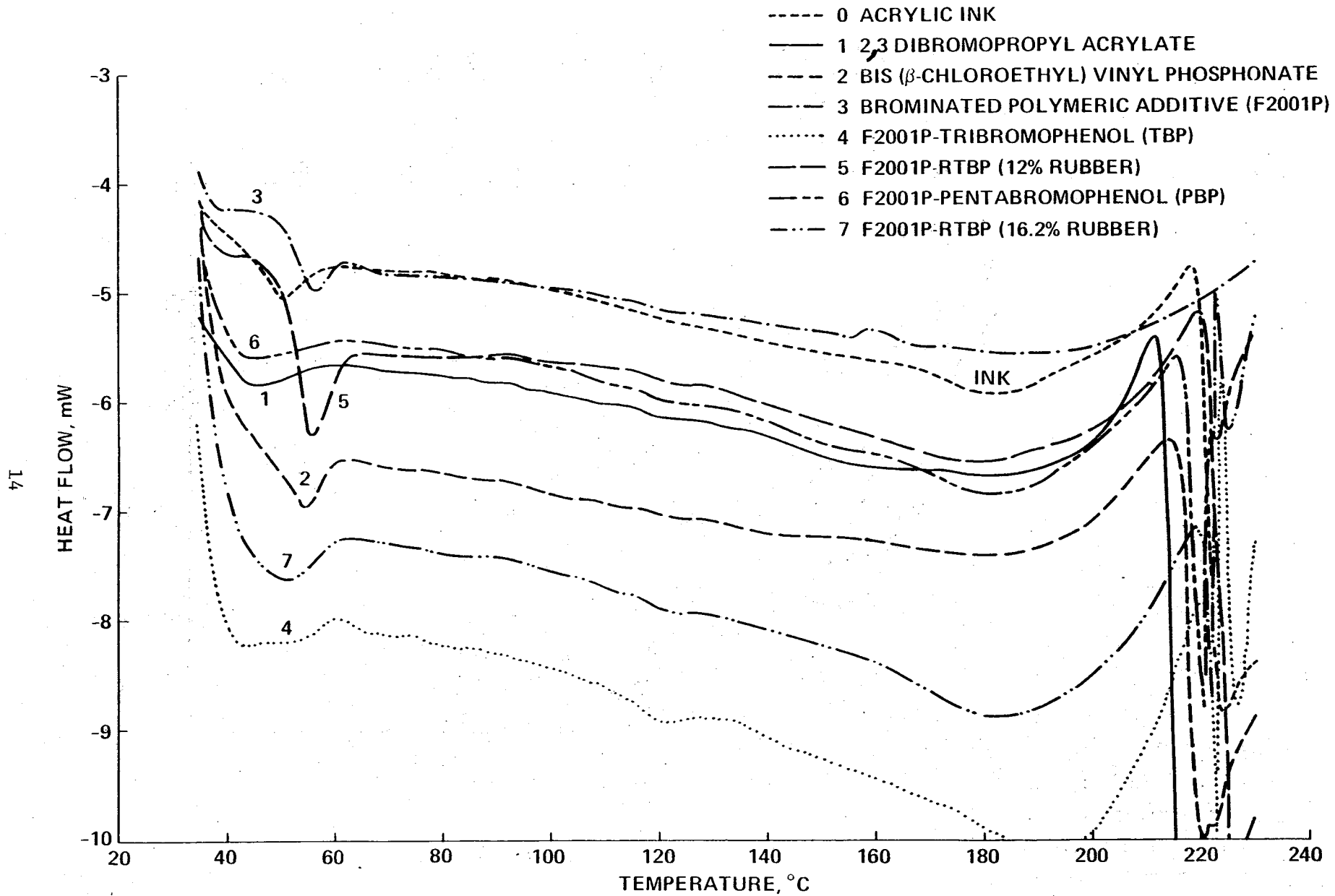
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Figure 5.– Differential scanning analysis of fire-retardant acrylic ink films (Nos. 1-7) versus control (0): nitrogen flow rate 80 ml/min; heating rate 20°C/min.
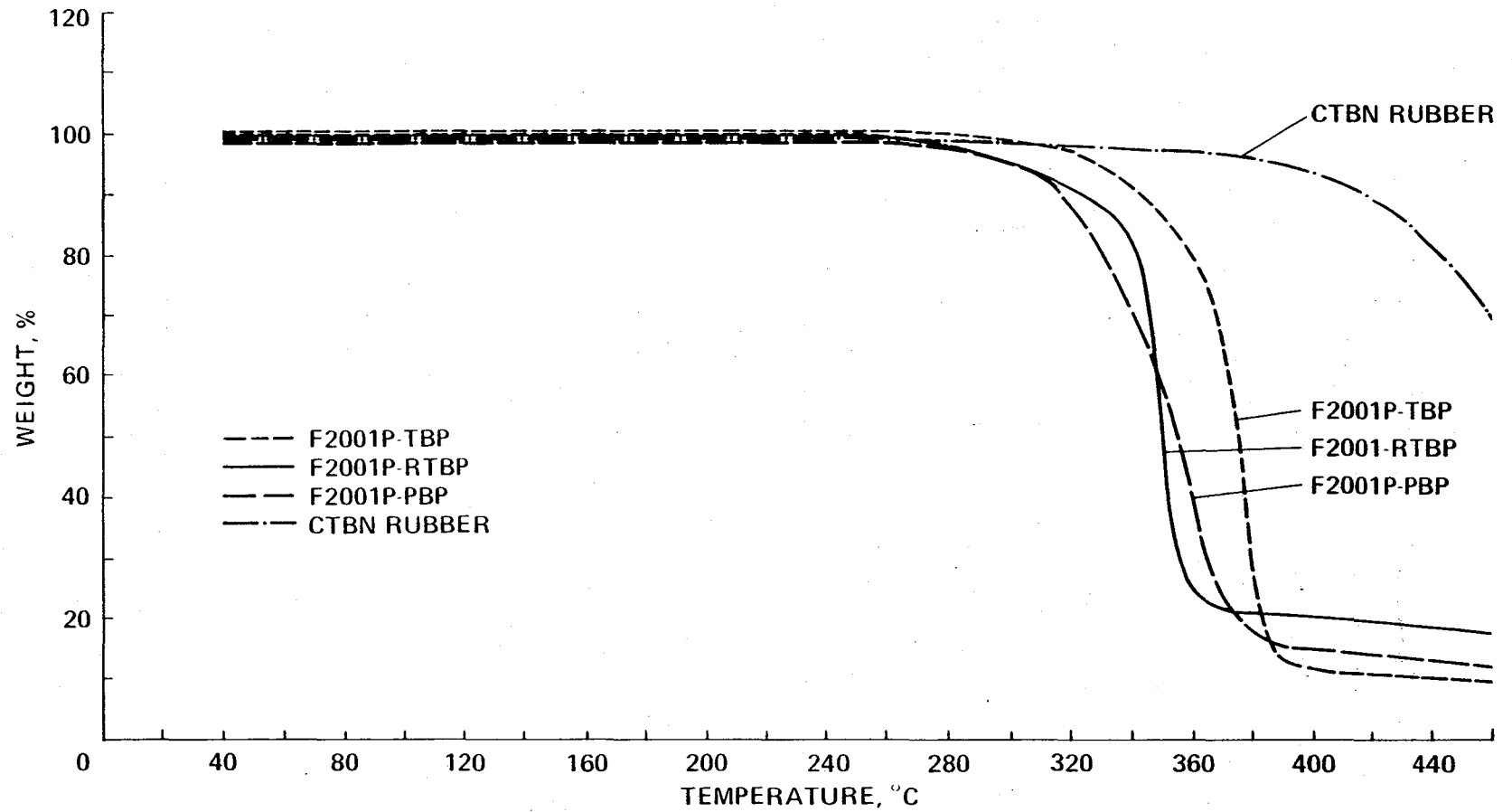
Figure 6.- Thermogravimetric analysis in nitrogen atmosphere of modified BPAs —
F2001P-TBP; F2001P-RTBP; F2001P-PBP — versus CTBN rubber: nitrogen flow rate
80 ml/min; heating rate 20°C/min.

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16. Abstract

Commercial and experimental fire retardants were screened for possible use with acrylic printing inks on aircraft interior sandwich panels. The fire retardants were selected according to their physical properties and thermostabilities. Thermostabilities were determined by the techniques of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). A criterion was then established for selecting the more stable agent. Results showed that some of the bromine-containing fire retardants are more thermostable than the acrylic ink, alone, used as a control. Also, the bromine-containing fire retardants yield even better limiting oxygen index values when tested after adding carboxy-terminated butadiene acrylonitrile (CTBN) rubber.

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