



## Sample Pages

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Flame Retardants for Plastics and Textiles

Practical Applications

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## Preface to the First Edition

For several years, the authors taught a short course on flame retardancy under the auspices of the Business Communications Corp. This course was found useful by people entering the field of flame retardancy or those who were facing particular problems of flame retarding plastics. One of us (S.V.L.) presented an introductory lecture discussing mode of action of main classes of flame retardants and one of us (E.D.W.) presented a survey of flame retardants in commercial use or in advanced development, discussing polymer class by polymer class. This book is a much-extended coverage of flame retardants from the same practical point of view. It is intended to be of use especially by plastics compounders, textile technologists, and by R&D workers in this field. Our coverage is mainly chemistry-oriented. "Cook-book" directions cannot be given although we do suggest starting point formulations in some cases.

We have only briefly touched on flame retardant coatings, "firestops" and fire-barrier materials, flame retardants for wood or paper, and inherently flame resistant polymers.

We have made an effort to reference much of the information presented, but in the many cases where references are not cited, it can be assumed that the source is suppliers' literature, supplemented by discussions with suppliers where needed. Each chapter has an independent bibliography; duplications will occur and the same flame retardant may be discussed in several relevant chapters. We have made a particular effort to have a thorough index by compound, trade name, supplier, and application to facilitate convenient access to all mentions of a substance or topic.

## ■ Comments on the Second Edition

Since the manuscript of the first edition was completed in late 2008, much has transpired in flame retardancy up to the completion date (early 2015) of the 2<sup>nd</sup> edition manuscript, both in terms of new commercial or serious developmental products, and in the removal of several major established products. We decided to indicate withdrawn products but not remove our discussion of these discontinued materials. In this regard, we have made an effort to indicate plausible replacements or alternative approaches to satisfy the flame retardancy requirements.

References from the first edition were not renumbered although patent application numbers were replaced by the corresponding issued patent numbers. New references were interposed with number-letter designations.

# 3

## Flame Retardants in Commercial Use or Development for Polystyrenes, Thermoplastic Styrene Copolymers, and Blends

### ■ 3.1 Introduction

In the present chapter, we will cover each main type of styrenic polymers, foamed, high impact polystyrene (HIPS), acrylonitrile-butadiene-styrene (ABS), and HIPS-polyphenylene oxide (PPO). Styrene-butadiene elastomers are discussed in the chapter on polyolefins and diene elastomers. Other reviews on styrenic flame retardancy published in recent years have encompassed the theory as well as mixing and test methods, rather than focusing on the flame retardant chemicals [1, 2]. In a journal article, the present authors have reviewed this topic with inclusion of theory and academic or noncommercialized industrial work [3].

Since most of the styrenic polymers are not good char-formers and tend to depolymerize to release fuel, the vapor-phase-effective brominated flame retardants have been predominant. However, due to a few that posed environmental concerns, this group of products has seen major changes since the 2009 edition of this book.

The leading brominated additives actually used (or recently being discontinued) are discussed in the following sections. In replacing brominated flame retardants, which have been discontinued or have a risk of discontinuance, polymeric or oligomeric brominated products are being brought to prominence by the industry. A concise 2012 overview [3a] shows the toxicological and environmental advantages of these higher molecular weight brominated flame retardants, without, however, being specific as to the polymers in which they can be used.



## ■ 3.2 General Comments on the Fire Properties of Styrenic Polymers

Polystyrene and its copolymers have the tendency to depolymerize when exposed to fire temperatures, and the volatile products are materials of high fuel value, namely styrene monomer, styrene dimers and related hydrocarbons such as benzene, lower-alkylbenzenes, and a few percent of oxygen-containing related aromatics [2]. Polystyrenes by themselves form little or no char [4]. The volatiles burn with copious soot formation. In the vicinity of a polystyrene fire, visibility can be nil. Moreover, while depolymerization is taking place, melt flow and drip are also occurring, and the drips may be capable of igniting other flammable objects. If flaming drips are allowed by the test standard, as in the UL 94 V2 rating, then the dripping may actually be useful to meet lenient flame retardancy requirements.

It is possible to retard the vapor phase combustion reaction by means of additives which reach the flame zone. These will be discussed in more detail next. For many uses of styrenics, especially electrical equipment, the requirements are to prevent a small source of ignition, such as a hot or sparking wire, from igniting the item, or if the polymer is ignited, causing it to self-extinguish quickly. The UL 94 test is the dominant one in the U.S. and indeed in much of the world, although other small scale tests are also significant.

Styrenic polymers fall into several main subclasses: polystyrene itself as molding resin; high impact (rubber modified) styrene copolymers (two main subclasses: HIPS and ABS), and expandable polystyrene (foam). Two other types of styrenic polymers, namely, the blends of HIPS with PPO and the blends of ABS with polycarbonates (PC), have their own combustion characteristics, mainly because of the char-forming PPO or PC components. We will discuss the ABS-PC blends in Chapter 7 because the PC is the larger fraction; the PPO-styrenic blends are discussed later in the present chapter because the styrenic component typically predominates in those blends.

## ■ 3.3 Crystal Polystyrene

This is nearly pure polystyrene and is primarily used in food containers, CD boxes, and miscellaneous housewares where flame retardancy is not required. Where flame retardancy is required, a soluble bromine compound has been usually used, typically hexabromocyclododecane. This compound, more commonly used in expanded polystyrene, is now being discontinued and alternatives are discussed next. An ICL patent [5] also suggests the use of pentabromobenzyl bromide.

## ■ 3.4 Expandable Polystyrene and Extruded Polystyrene Foam

There are two main technologies used to make polystyrene foams. One, expandable or expanded polystyrene (EPS), involves the preparation of polystyrene beads containing a blowing agent, typically a lower alkane; the beads are then placed in a mold or void to be filled, and expanded by heat. The second, extruded polystyrene foam (XPS), involves the expansion of a melt, containing the blowing agent and any other desired additives such as a flame retardant, through a slotted die from whence it expands. The processing temperature is somewhat higher for XPS than for EPS so that a somewhat higher stability requirement is imposed on the flame retardant system for XPS. In the EPS process, it is convenient to add the flame retardant to the styrene before polymerization, but it must not interfere with polymerization and cause a high concentration of residual styrene, or, in the case of XPS, it must have enough thermal stability to survive the extrusion process.

Expanded polystyrene foam, both EPS and XPS, is a major material used for insulation of buildings, refrigerators, and the like. The maximum flame spread and smoke-developed ratings at a maximum 4" (102 mm) thickness and 4.0 lbs./cu.ft. (64 kg/m<sup>3</sup>) density are less than 25 and 450, respectively, as measured by the ASTM E-84 25-ft. tunnel test. This and various other standards allow styrenic foam to melt and flow when exposed to fire, provided that the drips are not burning, and melt-flow can be exploited as a mode of flame retardancy. Because some pyrolysis to monomer and dimer takes place, aside from the flow of molten polymer out of the fire zone, a vapor-phase flame retardant is usually needed. The patent literature shows that this retardant is almost always a brominated aliphatic compound, which can readily release HBr (a vapor-phase flame retardant). A recent study by Dow [5a] showed that the essential mechanism of flame retardant action is fast depolymerization of polystyrene by Br· radicals, which leads to easy melting and retreat of the foam from the flame. At the relatively low processing temperatures for foamable polystyrenes, these aliphatic bromine compounds are generally stable enough. And, with fast enough HBr release, a synergist such as antimony trioxide may not be needed.

### 3.4.1 Hexabromocyclododecane (HBCD)

This additive has been discontinued for environmental reasons in Europe as of mid-2015. Although the "sunset date" has not been defined at the time of our writing, North America will probably follow. We will include HBCD in our discussion because of "legacy" products still in place containing it, and possible application of

its formulation information to its replacements. HBCD (Albemarle's Saytex HP900 powder or granules), HP900HG granules, and 9006L ground particles or ICL's FR-1206 (both powder and granules), are made by addition of bromine to cyclododecatriene, a product of catalyzed trimerization of butadiene. It is a mixture of three diastereoisomers, with one (the gamma-isomer) comprising 80% of the technical product. The structures have been elucidated by NMR and X-ray crystallography [6]. There are some differences in stability of the isomers [7] and presumably the manufacturers have tried to optimize the process to favor stability at processing temperatures. This matter is complicated by the fact that there is a thermal isomerization process that interconverts the diastereoisomers [8] and this conversion occurs during compounding. Generally, HBCD has not been used with antimony trioxide.

There have been many special grades of hexabromocyclododecane. Saytex HP900SG and BC-70HS were blended with stabilizers for use in XPS and injection molded HIPS, and allowed better color stability of the resin. BC-70HS appears to be more highly stabilized and was targeted for use in HIPS (see next section on HIPS). Some of the stabilizer systems for HBCD have been disclosed and claimed in patents, for example, a combination of a dialkyltin dicarboxylate and a barium or cadmium carboxylate [9]. The use of a zeolite in the range of 0.6–1.2%, optionally with a zinc salt of a fatty acid, is also patented as a means for stabilizing a HBCD formulation [10]. The systematic development of an optimized stabilized HBCD using two stabilizers is described by ICL (former Dead Sea Bromine Group) [11]. Other stabilizers mentioned [2] are metal hydroxides, metal oxides, and hydrotalcite. The HBCD stabilizers may be applicable to its replacements.

HBCD has shown persistence and bioaccumulation tendencies in the environment [12–14]. Risk assessments under EU and U.S. EPA auspices showed environmental issues and have led to the discontinuance of this product as of 2014–2015 [14a]. Prior to this action, the brominated flame retardant industry implemented a Voluntary Emissions Control Action Program (VECAP) that helped decrease the emission of HBCD in the environment [15]. However, this did not overcome the objections to the continued use of HBCD. Research by users and flame retardant manufacturers has led to satisfactory substitutes (discussed next).

Thermal stabilization of brominated aliphatic or cycloaliphatic flame retardant additives, such as HBCD for styrenics, is shown to be accomplished by addition of a thermoplastic acrylate or methacrylate in the 5–10% range relative to the bromine compound. Optionally, hydrotalcite is also added [17].

### 3.4.2 Other Bromine Flame Retardants in Expanded Polystyrene

Chloropentabromocyclohexane was made by addition of bromine to chlorobenzene (Dow 651). This was at one time a leading additive for expanded polystyrene foam, but has been discontinued. Likewise, discontinued are tetrabromocyclooctane, Albemarle's Saytex BC-48, and dibromoethyldibromocyclohexane (Saytex BC-462). Foam with these additives is likely to be still in place.

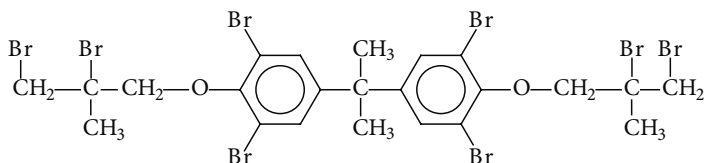
Certain other brominated compounds were in use for a time but appear to have been discontinued, such as cinnamalacetophenone tetrabromide [18]. A few other bromine compounds have been recommended for this use in patents. The authors are not aware of the commercial use of these additives. For instance, hexabromohexene [19], 1,2-dibromoalkyl ketones [20], 1,1,2,3,4,4-hexabromo-2-butene [21], dialkyl tetrabromophthalate [22], bis(2,3-dibromopropyl) tetrabromophthalate [23], 2,4,6-tribromophenyl allyl ether [24] and bis(allyl ether) of tetrabromobisphenol A [25] are mentioned here because they may be found in "legacy" foam insulation.

A tentative substitute for hexabromocyclododecane, judging from a series of Albemarle patent applications, is N-(2,3-dibromopropyl)-4,5-dibromotetrahydrophthalimide [26-28].

For a time, tris(2,3-dibromopropyl) phosphate was used, but this compound was shown to be a mutagen and was discontinued in the mid-1970s.

More recently, it was shown that nonbrominated polystyrene can be blended with a flame retardant amount of a brominated anionic styrene polymer plus a bis-(pentabromophenyl) ethane to make extruded flame retarded foam [29]. The use of a polymeric or oligomeric additive is usually considered more environmentally benign than the use of migration-capable small molecular weight additives, and this strategy has led to Dow's successful development of brominated butadiene-styrene copolymer, shown next.

As an alternative to HBCD, Dai-Ichi Kogyo Seiyaku (DKS) is marketing 2,2-bis[4-(2,3-dibromo-2-methylpropoxy)3,5-dibromophenyl]propane [29a] (SR-130) for both XPS and EPS applications.

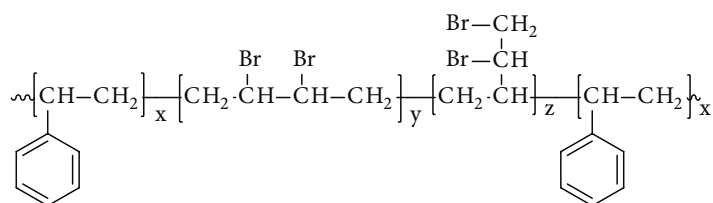


A substantial amount of patent literature exists on the use of combinations of brominated additives in expandable polystyrene. Combinations of the usual bromoaliphatic flame retardant with a more thermally stable bromine compound with an

aromatic, vinylic, or neopentyl structure may permit a lower total loading of flame retardant [30].

### 3.4.3 Brominated Styrene-Butadiene Polymer

After a major multicompany research effort that assessed many bromine, phosphorus, bromine-phosphorus and phosphorus-sulfur compounds, and brominated vegetable oils, an acceptable replacement was found by Dow [14a, 30a], manufactured by them as PolyFR, and licensed to several producers. The structure is as follows:



The product of ICL is FP-122P, a white to off-white powder, softening range 120–140 °C, bromine content 65%, and molecular weight in the 100,000 range. This polymer has no tendency to migrate or penetrate living cells or to bioaccumulate, and is environmentally benign. It is a satisfactory replacement for HBCD. For stabilization of FR-122P in the XPS application, ICL recommends using a brominated epoxy oligomer (F-2200HM), which is also a part of the active flame retardant [30b]. It can be further enhanced with hydrotalcite. A similar product is Chemtura's (Great Lakes Solutions') Emerald Innovation 3000, which is reported to contain 64% Br and has a molecular weight of 100,000–160,000. Chemtura has done research leading to a stabilizer package for the foam containing this additive, which appears to be an optimized combination of a hindered phenol, an organophosphite, and an epoxy cresol novolac [30c]. Albemarle's corresponding product, licensed from Dow, is marketed as GreenCrest Polymeric FR Solution.

A thorough review of the development, chemistry, properties, and safety evaluation of this additive has been published by Dow [30d].

### 3.4.4 The Flammability Effect of the Expanding Agent

The expanding agent is often a flammable hydrocarbon, such as pentanes. Some combinations of fluoro- or fluorochloroethanes with alkanes have been patented for this application [31].

A patent to Hoechst [32] suggests that there may be need for a storage period of extruded polystyrene foam, depending on dimensions, before it will pass flammability tests, and they claim shortening this period by use of less than 1.9–3% of the propane/butane mixture.

### 3.4.5 Synergists with Brominated Additives in Polystyrene Foam

For use with the brominated additives, synergists are known, and may be relevant to both the old and the new formulations. An early example is 0.25–0.75 phr of an allyl ether, said to aid in the decomposition of the bromine compound during burning [33]. Various peroxy compounds or other free-radical-forming compounds having labile C–C, O–O, or N=N bonds, enhance the action of the brominated flame retardants and have been used for a long time, following early work of Dow [34–38]. An advantage in efficiency was claimed by applying a radical-generating synergist to the outside of the polystyrene particles while having the bromine compound distributed throughout [39]. Peroxides useful in this mode of synergism must not decompose at processing temperatures, but must decompose at the temperatures reached by the molten polymer when exposed to flame: examples are dicumyl peroxide ( ), 1,3-bis[2-*t*-butylperoxy]isopropyl]benzene (Akzo Nobel's Perkadox<sup>®</sup> 14), or 2,5-dimethyl-2,5-di(*t*-butylperoxy)hexane (Arkema's Luperox 101). With care to feed the peroxide so as not to decompose it prematurely, combinations of HBCD (and presumably its replacements) with di-*tert*-butyl peroxide (Trigonox<sup>®</sup> B) or *tert*-butyl hydroperoxide (Trigonox<sup>®</sup> A80) can be used [40].

A nonperoxide synergist that can be used is one that undergoes C–C bond cleavage to produce tertiary-alkyl radicals, such as 2,3-dimethyl-2,3-diphenylbutane (Akzo Nobel's Perkadox<sup>®</sup> 30).

Another type of synergist to be used along with the usual brominated flame retardant is a waxy material such as a chloroparaffin, melting above the foaming temperature of the EPS, at about 0.1–2% [41].

### 3.4.6 Phosphorus-Bromine Combinations in Polystyrene Foam

Combinations of HBCD with a wide variety of phosphorus compounds, with examples showing the use of triphenyl phosphate, were claimed by Dow as flame retardants permitting lower levels of bromine [42]. Although HBCD is being withdrawn, it is useful to retain our discussion of HBCD combinations for their possible extension to HBCD replacements.

Combinations of HBCD (or many other Br additives) with triphenyl phosphate or with encapsulated red phosphorus are disclosed as flame retardants in foamed

styrene copolymers with specified olefins [43]. In extruded styrenic foam, combinations of a halogenated flame retardant with a phosphorus-nitrogen compound (such as APP), various tetrazoles and triazines, boron compounds, or phosphoric esters (such as triphenyl phosphate) are disclosed for the production of flame retardant foam with certain cell dimensions [44, 45].

Achievement of improved flame retardancy of an EPS while reducing the amount of HBCD to less than 2.5% was made possible by the use of 0.1–4% of a phosphorus co-additive, exemplified by triphenyl phosphate and various other phosphates, phosphonates, phosphinates, phosphines, phosphonium compounds, or phosphine oxides [42].

It will be of practical interest to discover synergists for the newer brominated styrene-butadiene replacement for HBCD.

### 3.4.7 Nonbromine Systems in Polystyrene Foam

Alternatives to the bromine additives have been disclosed in the patent literature. Very high loadings of ATH, such as 150–250 phr, afford a flame retardant foam and it is surprising that densities lower than 100 g/l can be thus obtained. Further addition of phosphorus or brominated flame retardants was recommended to reach adequate flame retardancy [46].

The use of heat-set flame-resistant carbonaceous fibers derived from polyacrylonitrile or pitch has been claimed as a flame retardant method for foams, including polystyrene foams. The fibers appear to collect at and protect the surface [47]. The use of carbonaceous particles or platelets is similarly disclosed by the same inventors in a later patent [48].

Recently more advanced EPS foams containing small graphite particles were developed in Europe. Because of its distinct silver gray color, this foam is called “gray foam” (e.g., the Neopor<sup>®</sup> line of products from BASF [48a]). Due to the presence of graphite, better heat reflective properties are achieved. Typically graphite particles of 1–50 micron size are used, however, presence of graphite require a higher percentage of a Br additive to obtain flame retardant EPS foam with density equal or less than 35 g/l [49].

Expandable graphite plus a halogenated flame retardant and optionally antimony oxide are disclosed for flame retarding EPS [50]. A combination of 1–12% expanded graphite and a phosphorus flame retardant (exemplified by red phosphorus and/or triphenyl phosphate) can enable EPS of 20–200 g/l and a cross-sectional area of at least 50 cm<sup>2</sup> to be self-extinguishing [51].

A combination of less than 2% elemental sulfur plus a phosphorus additive (exemplified by triphenyl phosphate, triphenylphosphine oxide or sulfide, or hexaphe-

noxytriphosphazene) afford higher oxygen indices, up to 26, in polystyrene such as the type used in foam production [52].

A 2008 patent application [53] with many Dow inventors covers a wide range of thiophosphates and dithiophosphates, most examples having neopentylene rings.

The applications exemplified were in foamed polystyrene, and not commercialized as far as the present authors know. Later, BASF patented a number of phosphine sulphides, which further amended with elemental sulfur pass DIN 4102 B-2 rating in EPS foam at similar loading as HBCD [53a]. Apparently this technology has not reached commercialization.

Extruded polystyrene foam can be made flame retardant by use of a blend with an epoxy resin containing reacted-in DOPO (9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide) and phosphoric acid [54]. Another development which showed promise is sulphur bridged bis-DOPO-thiophosphinates [54a]. Another means for increasing flame resistance of EPS is to make a board with a flame-resistant barrier, such as a coating, laminate or foil [55]. Polystyrene foam board with aluminum foil facing is widely available and the radiant-heat reflective foil contributes to the thermal insulation effect.

A novel way of making a flame retardant polystyrene foam is to coat the beads before expansion with boric acid plus a binder. After expanding, the boric acid forms a vitreous coating when the foam is exposed to flame [56].

A patent application by Owens-Corning inventors [57] claims that nanoclays such as sodium montmorillonite in a carrier such as water are useful to improve fire performance of a foamed polystyrene.

### **3.4.8 The Recommended Fire-Safe Use of Extruded Polystyrene Insulation**

Ample directions are available from Dow and other manufacturers regarding the safe use of polystyrene foam insulation.

Some of the key points are:

- Polystyrene foam boards should be separated from the interior of a building by a code-compliant thermal barrier such as 1/2" thick (12.7 mm) gypsum wall board applied with code-compliant fasteners. More details as to the various styrene foam insulation boards and their recommended applications are available from Dow and ICC [58].
- Expanded polystyrene foam is highly useful for rooftop applications, withstanding foot traffic and weathering. In order to be used directly on steel roof decks, the proposed configuration must pass UL 1256 or FM 4450. Large scale tests have been done by Underwriters Laboratories with favorable results [59].



- At least one application has had an adverse fire experience and is advised against. The U.S. Coast Guard recommends that foamed polystyrene insulation not be used in commercial fishing vessels, for example, as engine box covers, because in this high heat location, the foam could ignite and spread fire rapidly with dense smoke [60].

## ■ 3.5 High Impact Polystyrene

Polystyrene produced by polymerization, with some grafting, in the presence of rubber latex particles, is known as “high impact polystyrene” (HIPS). HIPS is used widely in equipment enclosures such as TV sets, computers, business equipment housings, and other electrical equipment. The TV set use is possibly the largest application. In the U.S., this application requires a UL 94 V0 rating, whereas in Europe a typical level of flame retardancy was about in the range of a UL 94 HB [61]. Publicity about the high rate of fire deaths from TV fires in Europe (contrasting with a low rate in the U.S., where fire standards for TV sets are more stringent) stimulated major international TV producers to use a higher UL 94 V0 level of flame retardancy in Europe. Flame retardant HIPS has an attractive balance of mechanical properties, processability, and cost.

### 3.5.1 Bromine-Containing Flame Retardants for HIPS

Approximately 10 wt% Br in combination with antimony oxide is required to pass UL94 V0 requirements. The exact amount will vary with the rubber content of the HIPS. HIPS is usually processed at 220–230 °C, so thermally stable bromine compounds are favored.

Antimony trioxide is normally used as a synergist with bromine- and chlorine-based flame retardants. The theoretical ratio would be 3 atoms of halogen to one atom of antimony, assuming that an antimony trihalide is the desired active species but the optimum ratio is best determined experimentally. Antimony regulatory aspects and antimony replacements are discussed in the Polyolefin chapter.

An antimony replacement by calcium borate on a carrier, specifically for styrenics (HIPS and ABS exemplified), is disclosed in a 2013 patent to Levchik et al. [61a].

### 3.5.1.1 Decabromodiphenyl Oxide (Ether) in HIPS

DBDPO, Albemarle's Saytex 102, ICL's FR-1210, and Chemtura's DE-83 (believed to be still available from Chinese manufacturers) has been the most widely used flame retardant for HIPS. In view of an over 10 years long risk assessment study in Europe and numerous academic publications, DBDPO is also the most studied flame retardant from human health and environmental points of view [61b]. Although no significant risk was found DBDPO has still succumbed in Europe and North America to environmental concerns and is now discontinued, we chose to keep information about it in this edition because it is still used in the rest of the world and many surviving plastics contain it. Also, formulation information may be relevant to substitutes. Its high bromine content (83%) and low cost made it a favorite choice. It is a stable solid up to its melting point of 305 °C. It is soluble in hot molten HIPS and only partly phase-separates on cooling, thus reduces impact only slightly but lowers HDT somewhat more [62]. It does have a tendency to yellow when exposed to ultraviolet light (such as sunlight) and therefore was mainly used in painted or dark pigmented plastic. DBDPO has typically been used at about 12% with about 4–5% antimony trioxide as a synergist. A formulation in HIPS with 6% rubber content with 12% DBDPO and 5% antimony trioxide was reported to have a LOI of 25.3 and a UL 94 rating of V0, notched impact of 43 kJ/m (without fire retardant., 85.9) and Vicat softening point of 91.5.

This photosensitivity not only causes discoloration, but interaction of DBDPO (and in the same way, tetrabromobisphenol A) with wavelengths in the 260–280 and 300 nm range, can also provoke polymer chain degradation to some degree [63]. Light stabilizers can make some improvement; in one extensive study [64], 0.25% of a chlorobenzotriazole photostabilizer plus 0.25% of a particular hindered phenol radical inhibitor were found most effective in preventing darkening, and could be further boosted by an epoxy cresol novolac, zinc stearate, or tin maleate. A hindered amine photostabilizer also was effective in such combinations.

However, the use of dark pigments was the most effective and economical means. If this approach was unsuitable, then it was possible to turn to the more expensive more light-stable brominated additives, such as Albemarle's ethylenebis(tetrabromophthalimide) (BT-93), to light-stabilized Saytex® 8010, to ICL's tris (2,4,6 tribromophenoxy)triazine (FR-245) or to tribromophenyl end capped brominated epoxy oligomers discussed next.

### 3.5.1.2 Decabromodiphenylethane

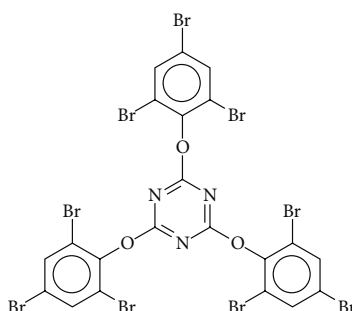
This compound is the closest replacement for decabromodiphenyl ether which encountered concerns (65) resulting in manufacturing discontinuance, a topic discussed at greater length in Chapter 2.

This compound is Albemarle's Saytex® 8010, Chemtura's Firemaster® 2100, and ICL's FR-1410. It is a high melting solid (melting point 350 °C) that has a use pat-

tern similar to that of DBDPO. This is useful for customers who wish to have formulations free of DBDPO, but are seeking a “drop in” substitute with a close Br content (82% compared to 83% for DBDPO). It has some other advantages, such as superior UV resistance relative to DBDPO and low blooming tendency. It is suitable for HIPS systems where recycling is anticipated. A disadvantage relative to DBDPO is somewhat poorer impact in HIPS, but this can be corrected for by either adding an impact modifier or starting with a higher impact grade of HIPS.

### 3.5.1.3 2, 4, 6-Tris(2, 4, 6-Tribromophenoxy)-1, 3, 5-Triazine

This compound is also a high melting (230 °C) but is a melt-blendable solid with a pattern of utility rather similar to DBDPE but with advantages of better melt flow, impact and light stability [67, 68, 68a]. It also allows use of less antimony trioxide synergist. Some comparative data is shown in Table 3.1.



**Table 3.1** V0 and V2 Formulations of FR-245 in HIPS (from [66])

Composition (wt%)	HIPS, no FR	HIPS V2	HIPS V0
HIPS	100	88.4	77.8
FR-245	—	8.9	14.9
Sb <sub>2</sub> O <sub>3</sub>	—	2.4	4
UV absorber	—	0.3	0.3
TiO <sub>2</sub>	—	—	3
Bromine content %	—	6	10
FR UL 94	not rated	V2	V0
GWT 960C	fail	pass	pass
Melt flow, g/10 min 200 °C 5 kg	5	8	15
Maximum strength, MPa	19	21	22
Elongation at break,%	61	53	47
Modulus, MPa	1600	1700	1800
Izod notched impact, J/m	130	120	115
HDT (1.82 MPa, annealed)	78	76	76
UV stability, AE 300 hr xenon	8	10	11

A combination of this melt-blendable flame retardant (which aids melt flow) with a filler-like flame retardant such as Saytex 8010 (which can reduce melt flow) enables molding of large dimension housings of electronic equipment made of HIPS, without the problems of melt flow and poor impact (Table 3.2).

**Table 3.2** Melt Flow and Impact Properties of FR-245-Based HIPS

Flame retardant	MFI at 200 °C, 5 kg	Gardner impact kg-cm
FR-245	10	100
1:1 FR-245/Saytex 8010	8	81
Saytex <sup>®</sup> 8010	5	62

Another advantageous combination of FR-245, as claimed by Dai Ichi, is with tris-(2,3-dibromopropoxy) cyanurate [69].

An important feature of FR-245 is that it can be used to reach UL 94 V1 in HIPS with little or no antimony trioxide, with the assistance of a very small amount of PTFE to retard drips. For example, 17.9% of FR-245, 1.5% ATO, and 0.1% PTFE allows ABS to reach a V0 rating [69a]. An overview is available by ICL authors [69b] on reducing or avoiding the need for ATO by choice of the brominated flame retardant, especially FR-245.

#### 3.5.1.4 Modified Brominated Epoxy Oligomers and Polymers

These are melt-blendable additives especially suitable for HIPS and ABS. They provide excellent flow properties with good light stability. However, for demanding applications with large dimensions, high impact styrenic copolymers flame retarded with brominated epoxies have rather inadequate impact. Partially replacing brominated epoxy with FR-245 retains the good melt flow but greatly improves the impact.

A series of tetrabromobisphenol A epoxy oligomers and polymers (the epoxy group has been reacted out) end capped with 2,4,6-tribromophenoxy groups is available from ICL as the F-3000 series. One of these, F-3010 with MW 2000, 56% Br, and softening range 105–120°C, is preferred for HIPS. It provides a good balance of processability, thermal, and UV stability, heat distortion, and impact. Dust-free blends of lower and higher molecular weight, brominated epoxy oligomers have been optimized by ICL for reaching the V0 rating in HIPS with antimony trioxide synergist, and with good physical properties.

#### 3.5.1.4.1 Polybrominated Polyphenylene Ethers

This family of additive oligomers has been developed by Chemtura (Great Lakes Solutions) and marketed under the trade name Emerald<sup>®</sup> Innovation 1000 as an environmentally and toxicologically benign product [69c]. It is an off-white powder, average particle size 5–6  $\mu\text{m}$ , with 78% Br. It is thermally stable and loses 5% weight at 410°C. This additive in HIPS at 11.5% Br can provide a V0 rating with good impact and heat distortion, with some impairment of melt flow.

Combinations of Emerald<sup>®</sup> Innovation1000 and poly-2,6-dimethylphenylene oxide in HIPS have been shown to be able to reach UL 94 V0 ratings without antimony trioxide. [69d, 69e, 69f].

#### 3.5.1.5 Brominated Lower Molecular Weight Polystyrenes

While ring-brominated higher-molecular-weight polystyrenes have been on the market for decades, these have found use mostly in polyamides and thermoplastic polyesters, but lack suitable physical properties for use in HIPS. Albemarle has brought to the market a patented family of ring-brominated low molecular weight anionically-polymerized styrenes, GreenArmor<sup>®</sup>, which have good compatibility with HIPS and other styrenic plastics [69f]. These have the advantage, characteristic of polymers, of environmental safety and absence of toxicity.

#### 3.5.1.6 Tetrabromobisphenol A

TBBA (Albemarle Saytex RB-100 or CP-2000 or ICL's FR-1524) is a lower melting solid, often used as a reactant in epoxies or polycarbonates, but also used as an additive to a limited extent in HIPS and mostly in ABS. Because of its lower melting point, it melts into the polymer aiding melt flow considerably and allowing impact to be maintained. Another advantage is low cost. On the negative side, it is poor on color stability and tends to be used only in dark formulations. It also tends to leach out of plastics and becomes distributed in the environment. Because of this, flame retardant producers currently recommend using oligomeric and polymeric flame retardants instead.

#### 3.5.1.7 Tetrabromobisphenol A Bis(2,3-Dibromopropyl Ether)

This product (Albemarle Saytex HP800A or the granular version HP800AG, Great Lakes' PE-68<sup>™</sup> and ICL's FR-720) is a relatively low melting solid, melting point 108–120 °C, rather soluble in HIPS and suitable for reaching UL 94 V2 ratings. It has minimal effect on impact strength. Probably, its larger uses are in polypropylene.

### 3.5.1.8 Light-Stable Bromine Additives: 1, 2-Ethylenebis(tetrabromophthalimide) and Tris(tribromoneopentyl) Phosphate

Ethylenebis(tetrabromophthalimide), Albemarle BT-93, may be used when photo-discoloration must be minimized. UV stability is its outstanding feature, but it has a slight yellow color to begin with. A colorless version is a purified grade, BT-93W. BT-93 has a melting point above the processing temperature of HIPS and is quite insoluble in HIPS. It acts like an inert filler, and therefore tends not to lower HDT, but on the other hand, is somewhat detrimental to impact strength [62]. An alternative way to achieve good UV stability in a flame retardant styrenic is to use ICL's FR-245 or DBDPE with a good UV stabilizer system.

Tris(tribromoneopentyl) phosphate (ICL's FR-370), solid with melting point of 181°C, is recommended for V2 UL 94 rating in styrenics with excellent stability to visible and ultraviolet light, probably attributable to its totally aliphatic structure.

### 3.5.1.9 Comparison of Typical Formulations of Brominated Flame Retardants in HIPS

Table 3.3 shows a comparison of several of the above-discussed bromine-based flame retardants for HIPS.

**Table 3.3** Performance of Some Brominated Flame Retardants in HIPS

Additive	Control	BT-93 ethylenebis (tetrabromophthalimide)	Saytex 8010 Decabromodiphenylethane	CP-2000 tetrabromobisphenol A
<b>Flame Retardant:</b>	<b>0.0</b>	<b>18.0</b>	<b>14.6</b>	<b>20.4</b>
% Antimony trioxide	0.0	4.0	4.0	4.0
% Bromine	0.0	12.1	12.0	12.1
<b>Physical Properties</b>				
Izod Impact 3.2 mm, ft-lb/in	3.3	0.6	2.0	1.7
Izod Impact 3.2 mm, J/cm	176	32	107	91
DTUL 3.2 mm°C	79	82	80	68
MFI 230 °C/3800 g	5.8	3.3	2.6	14.0
<b>UV Stability</b>				
100 hr Xenon arc	7.9	7.3	21.1	nd (bad)
300 hr Xenon arc	9.2	9.4	25.3	34.3

(from Albemarle)

Various other polybrominated additives are shown in recent patents to be useful in HIPS, bis(pentabromobenzyl) ether for example [70].

### 3.5.1.10 The Dripping Problem and Antidripping Additives

Many thermoplastics, and HIPS is no exception, can have flaming drips. In some end products, it is acceptable to have drips if they are nonflaming, but flaming drips can be tolerated only in those products allowed to pass UL 94 with a V2 rating. The contribution to fire safety of V2 standards is controversial. Where the stricter V1 or V0 standards are required, means must usually be found to prevent drip. The addition of very small amounts of polytetrafluoroethylene (PTFE) powder, at loadings generally in the 0.05–0.3% range, are often effective, probably because of a rheological effect. Uniform and continuous mixing of such small quantities of a powder is challenging.

As it was mentioned earlier, use of PTFE is crucial in low antimony trioxide formulations [69b], especially for V1 rating where the allowed burning time is longer and the risk of producing flaming drips is higher.

### 3.5.1.11 The Effect of Brominated Flame Retardants and Antimony Oxide on Mechanical Properties of HIPS

Even though DBDPO is largely discontinued, studies done on it are relevant to its substitutes. An academic study [72] showed that the impact strength of HIPS declined gradually and approximately linearly as the loading of DBDPO was increased. Antimony oxide, used as a synergist, did likewise, but it was found that  $\text{Sb}_2\text{O}_3$  particles at 0.08 micron size did not reduce impact strength, but in the range of 0.5–1.2 microns, severe impact strength loss occurred. Surprisingly, at 4–10 microns, no impact strength loss occurred. It was postulated that these larger particles served as craze terminators.

A study of the effect of particle size using representative solid additives showed that impact strength of a polystyrene can even be increased by particles several microns in diameter because of formation of voids, peeling layers, and extension of crazes [73]. However, some compounders believe it best to have smaller particles, and in the case of antimony oxide, often the flame retardant effect is improved by using smaller particles. The detrimental effect of powdered additives on impact strength can also be at least partially overcome by starting with a “superhigh” impact plastic or by adding an impact modifier, such as a chlorinated polyethylene (which also contributes to flame retardancy), a thermoplastic styrene-butadiene-styrene block polymer, or ethylene-vinyl acetate. Where the use of very fine particles adversely influences processing characteristics, a surface-modifying agent (“coupling agent”) may help.

Liquid flame retardants generally do not lessen the impact strength and may, in fact, often improve it.

A useful study was done at Monsanto addressing the quantitative relationship of solubility of decabromodiphenyl oxide in HIPS to the impact and processing char-

acteristics [74]. This might be a clue to the expected behavior of decabromodiphenylethane in HIPS.

Regarding electrical properties, usually the bromine-antimony systems have somewhat decreased loss factor and may be prone to leakage currents and diminished arc resistance, particularly on aging.

### 3.5.2 Nonhalogen Flame Retardants for HIPS

This is a challenge because of the need to avoid spoiling impact strength, heat distortion temperature, and cost, and also because of the poor char-forming propensity of HIPS. Noting that UL 94 V0 can be obtained for PPO-HIPS (discussed further, next), researchers have studied the use of smaller loadings of PPO plus char-forming phosphates. This system can also be aided by adding char-forming phenolic novolacs [75] or by adding small amounts of “nanoclay” (an exfoliatable layered montmorillonite). For example, a formulation of 80 parts by weight of HIPS, 20 parts PPO, 20 parts of resorcinol bis(diphenyl phosphate), and 3 parts of quaternary ammonium-treated montmorillonite can give a V0 rating [76]. The use of higher PPO blends with HIPS is an important approach because the PPO provides phosphorus-catalyzed charring capability. Such alloys, notably GE’s Noryl<sup>®</sup>, are discussed further in Section 3.7.

A patent shows the use of expandable graphite plus a phosphorus flame retardant, such as resorcinol bis(diphenyl phosphate) or triphenyl phosphate, plus a co-additive, such as 12 wt% of a polycarbonate, to prevent migration of the phosphorus compound to the surface of HIPS [77].

A U.S. patent application [78] by Israeli inventors shows HIPS flame retarded to V0 by 15% expandable graphite and 15% melamine, with no halogen, no antimony oxide, and no phosphorus.

Where only a UL 94 V2 rating is required, efficient flame retardancy has been obtained in HIPS with a solid aromatic diphosphate, ICL’s Fyrolflex<sup>®</sup> Sol-DP. Two advantages are good melt flow and a dust-free product. A U.S. patent application [79] by Cheil Industries (Korea) shows HIPS flame retarded by a combination of an aryl phosphate, such as PX 200<sup>®</sup> and a phosphonate, such as Amgard<sup>®</sup> 1045.

A study [79a] recently showed that a synergistic combination of encapsulated red phosphorus and magnesium hydroxide at a total loading of 50% gave a strong V0 rating. The study addressed the mechanism of the synergism, which was attributed to condensed phase charring action. Obviously, physical properties are questionable at the 50% loading.



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