



## **ETHANOX™ and ETHAPHOS™ Antioxidants and ALBlend™ Additive Blends One Source For All Your Antioxidant Needs™**

ETHANOX and ETHAPHOS antioxidants and ALBlend additive blends are safe and effective for use in a wide variety of polymers—from thermoplastic elastomers and engineering plastics and alloys, to hot melt adhesives and tackifiers, as well as polyolefins. Their effectiveness and overall stability mean that their use in most polymers greatly improves your capacity in a wide range of processes without degradation and to help polymers stand up to demanding long-term end use applications.

Our trained sales, marketing, and technical service personnel are ready to help you in your formulations at any time. If you wish to know more about Albemarle's antioxidants and additive blends, please call or write to our office near you.

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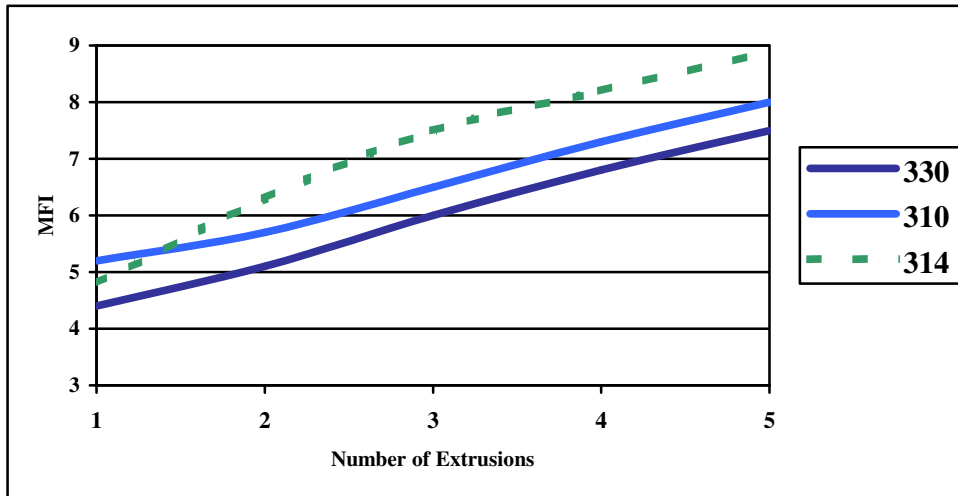
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## PROCESSING STABILITY

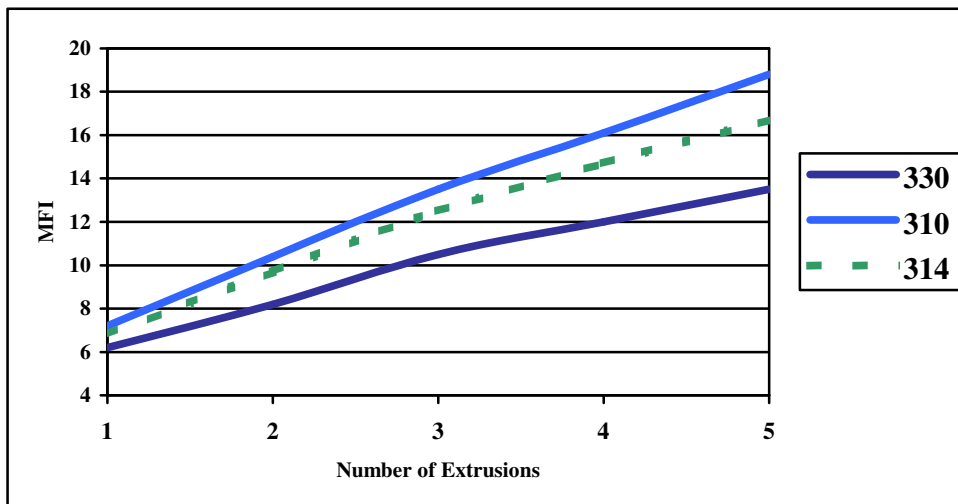
As heat and shear forces of polymer processing break the molecular chains of polypropylene into shorter segments during repeated passes through extruders, polypropylene tends to become less viscous. A function of an antioxidant in processing is to slow down this reduction in molecular weight loss, keeping the polymer as close as possible to its initial viscosity.

### Processing stability of *homopolymer* polypropylene containing 1500 ppm ETHANOX antioxidants



All ETHANOX antioxidants help control degradation during polymer processing. ETHANOX 330 antioxidant often proves superior to other high-molecular-weight antioxidants, as demonstrated by a smaller change in the polymer's melt flow index (MFI) through several extrusions of a 4<sup>th</sup> generation polypropylene produced by high-activity catalysts. All materials were processed in a 3/4-inch (1.9 cm) single-screw extruder (24/1 LD ratio) with a flat 260 °C temperature profile at 30 rpm. All samples contained 500ppm calcium stearate.

### Processing stability of *copolymer* polypropylene containing 1500 ppm ETHANOX antioxidants



Phosphite antioxidants enhance the processing stabilization of high molecular weight phenolic antioxidants. A comparison of various phenolic antioxidants and phosphites is shown below. The materials were processed in a 3/4-inch (1.9 cm) single screw extruder (24/1 LD ratio) with a flat 288°C high temperature profile at 30 rpm.

**Comparison of different phosphates at 750/750/550 ppm of ETHANOX antioxidants/phosphite/DHT-4A in polypropylene**

<b>MFI (Condition L)</b>		
	<b>First Pass</b>	<b>Fourth Pass</b>
ETHANOX 330 antioxidant / 626 phosphite	5.1	16.1
ETHANOX 310 / 626	5.5	16.5
ETHANOX 314 / 626	5.0	18.4
ETHANOX 330 antioxidant / P-EPQ phosphite	5.2	16.8
ETHANOX 310 / P-EPQ	6.2	19.5
ETHANOX 314 / P-EPQ	5.1	22.1
ETHANOX 330 antioxidant / ETHAPHOS 368 phosphite	5.1	23.9
ETHANOX 310 / ETHAPHOS 368	7.9	33.6
ETHANOX 314 / ETHAPHOS 368	5.2	27.6

## LONG-TERM HEAT AGING

ETHANOX phenolic antioxidants provide excellent long-term protection against oxidative degradation of polymers including polypropylene at normal end use application temperatures. The excellent long-term capability of Ethanox 330 and Ethanox 314 is often masked when testing in the laboratory by high-temperature-accelerated oven aging in the interest of speed and convenience. However, when antioxidants are compared over a range of evaluation temperatures, their relative performance ranking can change quite markedly.

The use of a thioester antioxidant synergist increases the effectiveness of phenolic antioxidants in minimizing the degradation of polymers including polypropylene. Polypropylene containing ETHANOX antioxidants with the synergist have superior aging resistance at ambient temperatures plus excellent resistance over a wide range of temperatures.

### Thermal stability of polypropylene (days to failure of 2- and 10-mil plaques, oven aged)

Antioxidant	AO Level (phr)	Without DSTDP				With 0.25 phr DSTDP				
		2 mil		10 mil		2 mil			10 mil	
		140°C	150°C	140°C	150°C	125°C	140°C	150°C	140°C	150°C
ETHANOX 310	0.10	34	13	52	22	88	73	29	104	56
	0.25	62	31	83	46	90	83	37	111	79
ETHANOX 314	0.10	6	3	6	2	130	55	14	97	31
	0.25	13	5	8	3	136	83	25	146	51
ETHANOX 330	0.10	38	16	34	16	110	55	35	104	51
	0.25	59	22	62	28	129	83	46	122	63
ETHANOX 376	0.10	8	4	13	6	--	34	15	57	26
	0.25	8	5	20	9	--	28	12	48	19

## EXTRACTION RESISTANCE

The case study polymer, polypropylene, was compounded with the additives formulations shown in the table below in a twin-screw, short-barrel extruder for initial compounding at a stock temperature of 200° C and 30-rpm extrusion speed. The multi-pass extrusion was then done on a 28:1 single-screw extruder using a stock temperature of 260° C and 30-rpm extrusion speed. The pellets from the first-pass extrusion were compression-molded into plaques, some of which were submerged in boiling distilled water for 7 days and 14 days, respectively. Three types of plaques were used to run the oven aging experiment: plaques that were not subject to boiling water (0 days), plaques submerged in boiling water for 7 days, and plaques submerged in boiling water for 14 days.

### Effect of boiling water exposure on ETHANOX antioxidants and other additives in polypropylene – 150°C oven aging from first-pass extrusion samples

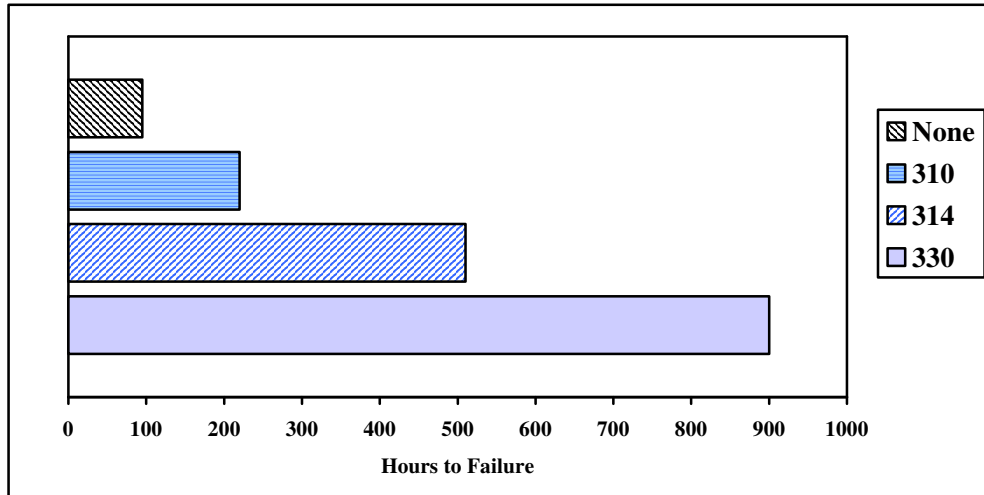
Additive Composition	Conc. (ppm)	Hours (Days) to Failure		
		0 Days*	7 Days*	14 Days*
ETHANOX 310 antioxidant / DSTDP	600/2500	1608 (67)	360 (15)	228 (9.5)
ETHANOX 310 /ETHAPHOS 368 phosphite / DSTDP	600/600/2500	1416 (59)	96 (4)	48 (2)
ETHANOX 310 / P-EPQ / DSTDP	600/600/2500	1416 (59)	96 (4)	48 (2)
ETHANOX 310 / ETHAPHOS 368	600/600	360 (15)	48 (2)	48 (2)
ETHANOX 330 / P-EPQ / DSTDP	600/600/2500	1008 (42)	240 (10)	192 (8)
ETHANOX 330 / DSTDP	600/2500	1104 (46)	456 (19)	--

Number of days in boiling distilled water before aging  
All samples contain 500 ppm of CaSt.

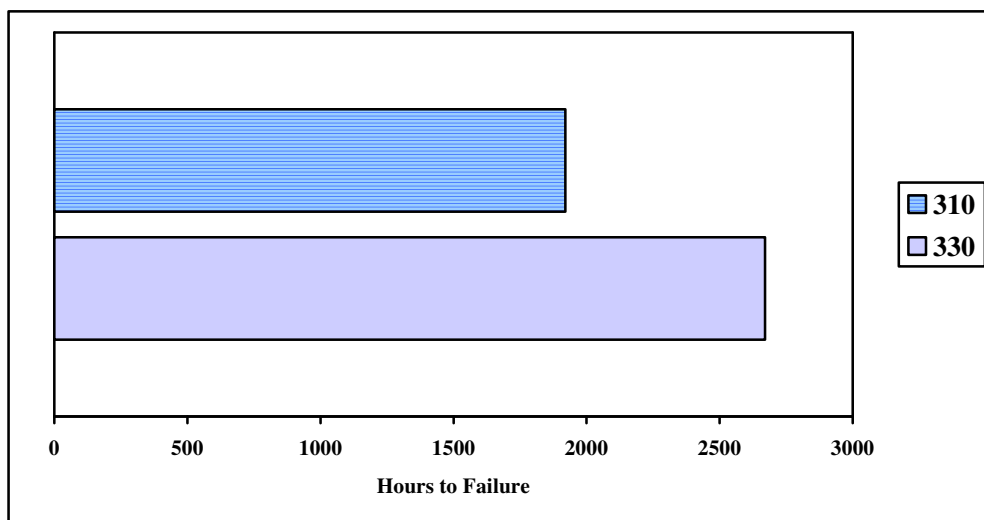
## ABSORPTION RESISTANCE

Fillers used in polypropylene tend to absorb phenolic antioxidants onto the surface of the filler keeping the antioxidant from solubilizing into the polymer. This is dependant on the relative solubility of the antioxidant in the filler and/or in the polymer. ETHANOX 330 antioxidant is very resistant to such absorption, while ETHANOX 314 is the next best candidate.

**ETHANOX antioxidants in polypropylene containing 25% Vermont talc; 25-mil plaques aged at 120°C, 2000 ppm AOX, 500-ppm calcium stearate, (CaSt)**



**ETHANOX antioxidants in polypropylene containing 40% calcium carbonate filler; 25-mil plaques aged at 120°C, 500 ppm AOX, 500 ppm CaSt**

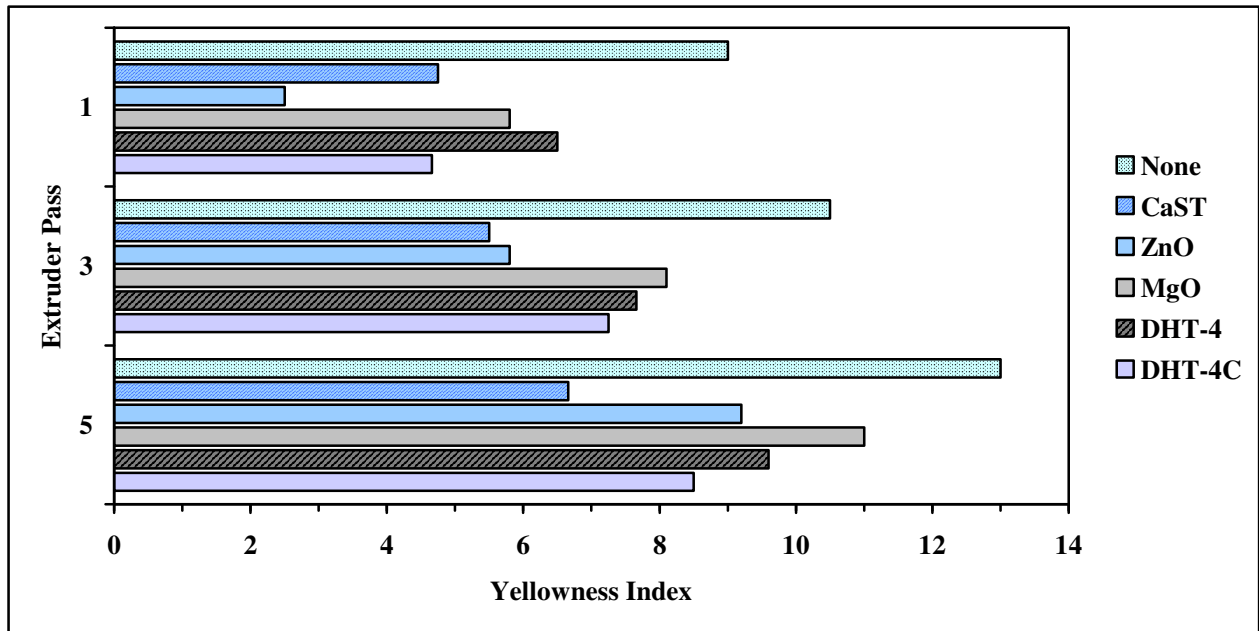


## COLOR

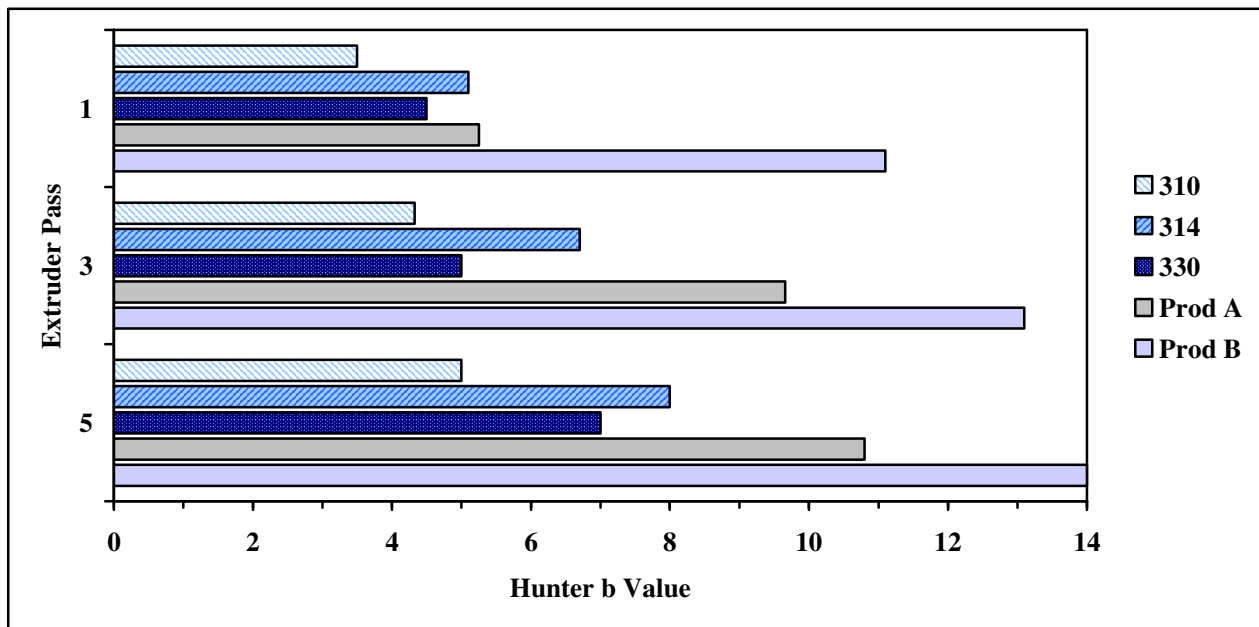
**Acid Neutralizer Comparison:** Polyolefin acidic polymerization catalyst residues are known for interacting with phenolic antioxidants resulting in low levels of highly colored species, which increase discoloration of the polymer. Proper acid neutralization in polyolefins with calcium stearate and other acid neutralizers helps control color formation.

Albemarle also has custom ALBlend™ additive blends that offer cost-effective color and thermal stabilization solutions.

### Color control with acid neutralizers in polypropylene with ETHANOX 330 antioxidant/neutralizer (500/600 ppm)



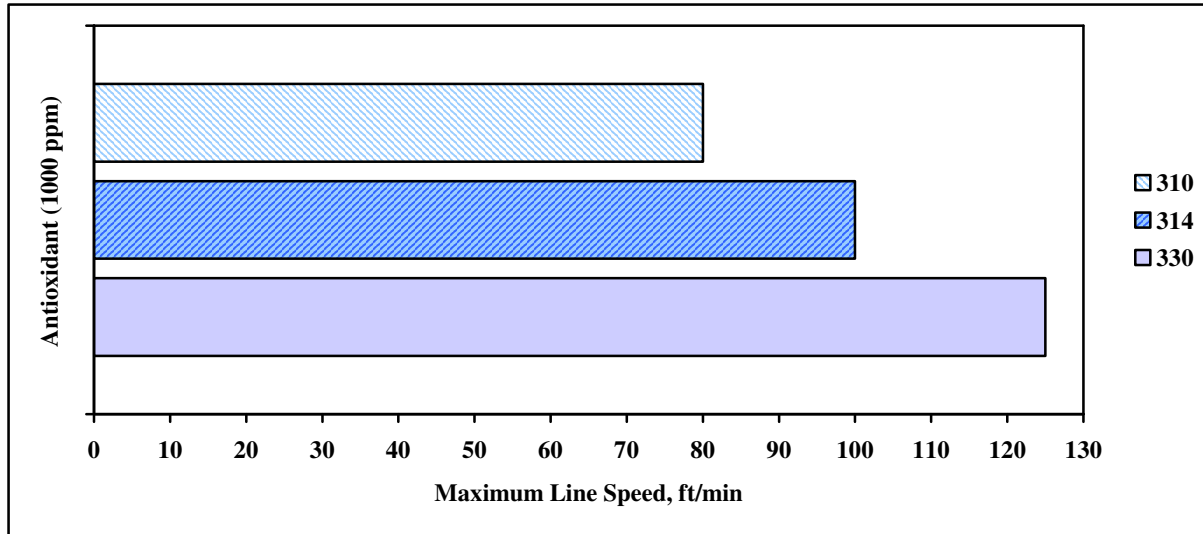
### Color comparison of ETHANOX and other antioxidants in polypropylene with calcium stearate (AOX/CaSt=500/600 ppm)



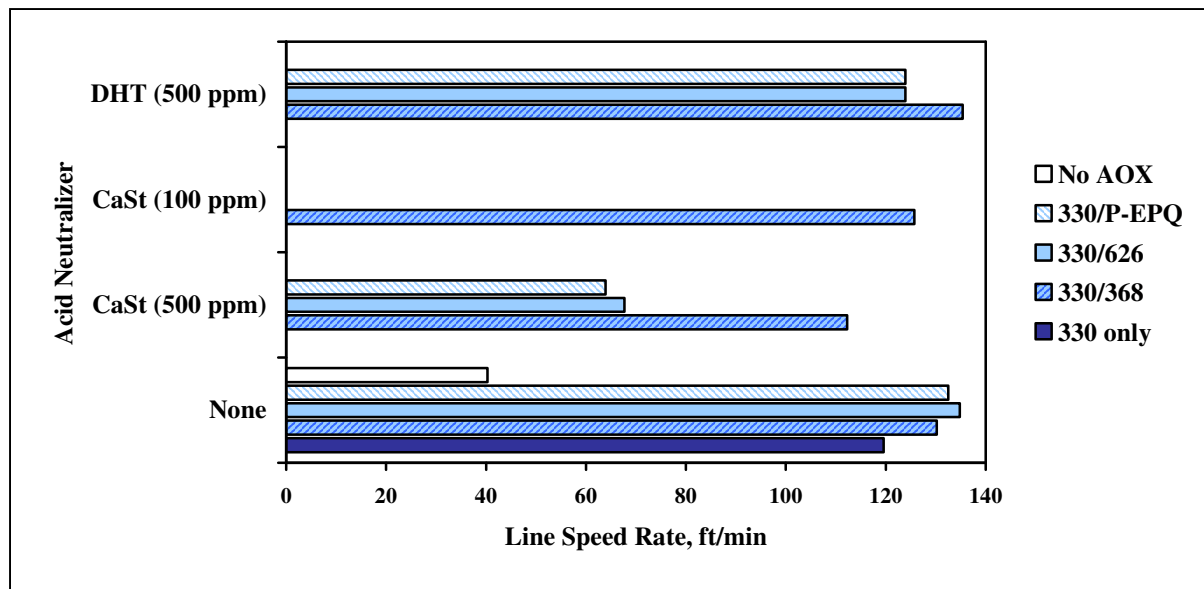
## WATER CARRYOVER

Most phenolic antioxidants tend to increase water carryover and thereby decrease maximum line speed in the production of water-quenched polypropylene films. ETHANOX 330 antioxidant helps produce high-quality film surfaces that provide maximum line speeds.

**Water carryover effect of ETHANOX antioxidants in 1-inch-wide (2.54 cm) polypropylene ribbon extruded at 275°C**



**Water carryover effect of ETHANOX 330 antioxidant with ETHAPHOS 368 phosphite and other phosphates when different acid neutralizers are used in polypropylene**



## CONTROLLED RHEOLOGY (CR)

Organic peroxides are used to enhance processability of polypropylene by decreasing the melt viscosity and narrowing the molecular weight distribution.

**Effect of antioxidants on the efficiency of Lupersol 101 peroxide to increase melts flow @  
Antioxidant/Peroxide/Calcium Stearate (CaSt) = 1000/440/500 ppm**

<b>MFI (Condition L)</b>		
	<b>Original</b>	<b>After Cracking</b>
ETHANOX 330 antioxidant	1.2	10.3
ETHANOX 310 antioxidant	1.2	10.5
ETHANOX 314 antioxidant	1.2	9.5
1,1,2 -Tris (2 methyl -4-hydroxy -5-tert-butylphenol ) butane	1.1	8.4
1,3,5 -Tris (4-tert-butyl -3-hydroxy -2,6-dimethyl benzyl) -s-triazine -2,4,6 - (1H,3H,5H) trione	1.3	7.8

**Effect of antioxidants in CRPP – Extruder condition A, polymer E with  
AOX/CaSt = 1000/500 ppm**

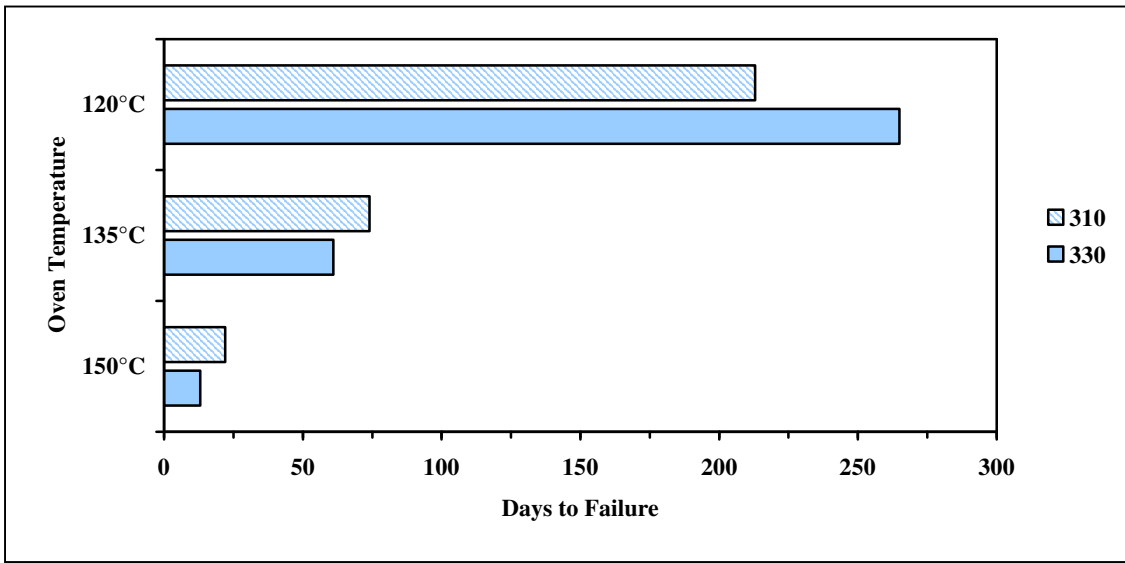
<b>MFI (Condition L)</b>			
	<b>L-101 Conc.</b>		
	<b>200 ppm</b>	<b>400 ppm</b>	<b>800 ppm</b>
ETHANOX 330 antioxidant	4.8	10.3	23.2
ETHANOX 310 antioxidant	5.4	10.5	23.9
ETHANOX 314 antioxidant	5.3	10.4	23.5
Topanol CA	5.1	7.8	17.4
Cyanox® 1790	3.6	8.4	17.3

ETHANOX antioxidants do not significantly interfere with peroxides used in processing.

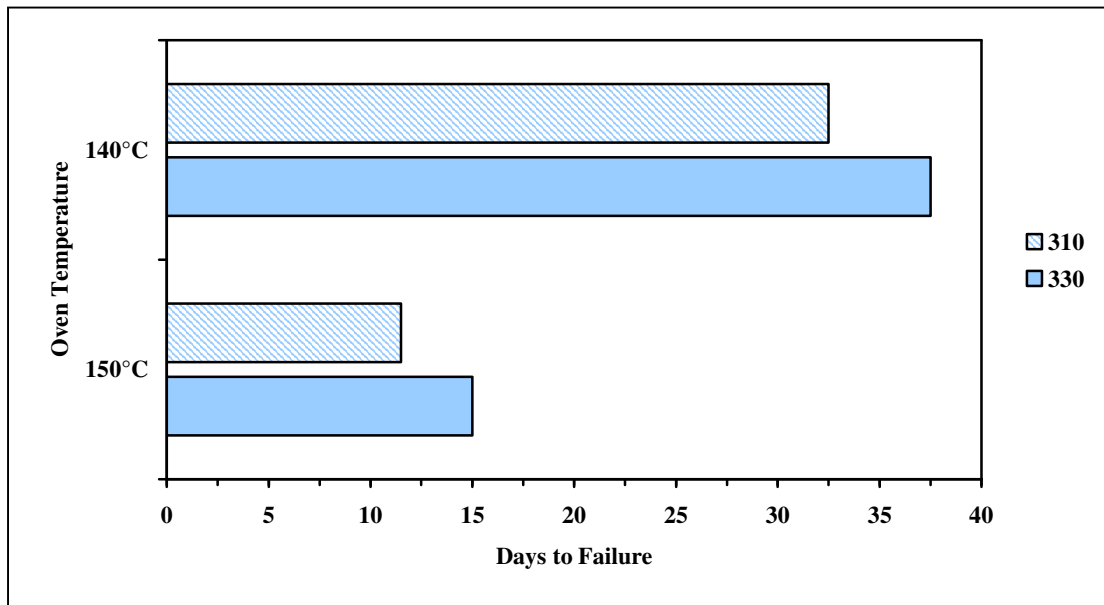
## LONG TERM THERMAL STABILITY TESTING

When ETHANOX antioxidants are compared at a range of test temperatures in a thick specimen. ETHANOX 330 antioxidant is clearly superior as test conditions begin to approach more normal use temperatures. Similar trends also are reported for ETHANOX 314 antioxidant. In thin (2 mil) specimens, ETHANOX 330 antioxidant also provides excellent protection even in high-temperature accelerated aging.

**Antioxidant/calcium stearate (CaSt) = 1000/300 ppm; 20-mil samples**



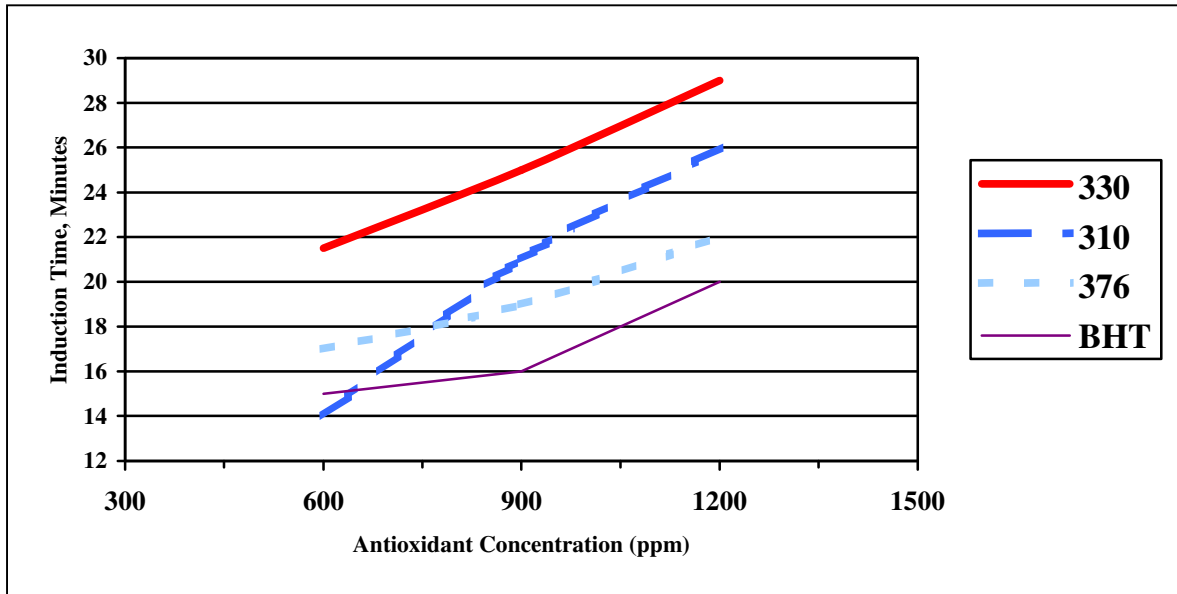
**Antioxidant/calcium stearate (CaSt) = 1000/300 ppm; 2-mil samples**



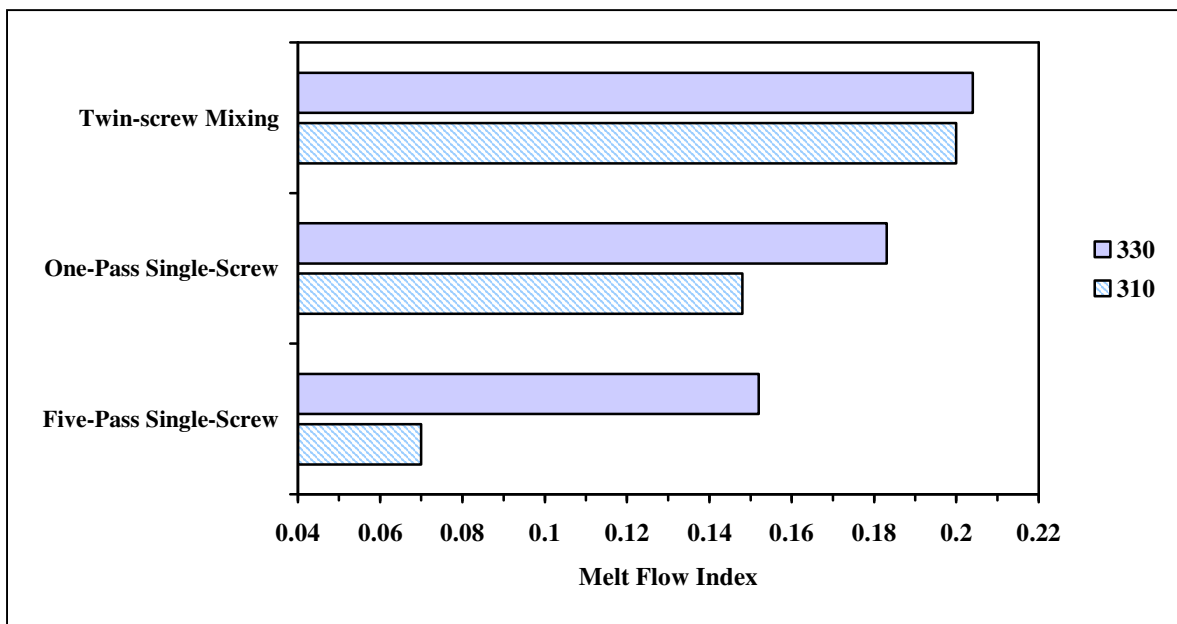
## PROCESSING STABILITY

In high-density polyethylene, the decreased viscosity resulting from degradation and the breaking of the polymer chains into shorter segments may be offset to varying degrees by the increasing viscosity resulting from crosslinking degradation. The Brabender Torque Rheometer measures the induction time to the onset of polymer crosslinking (first step of degradation) and thus measures the effect of such counteractivity.

### Brabender Rheometer (230°C 50 rpm) comparisons of ETHANOX antioxidants and BHT



### Processing stability of ETHANOX antioxidants in HDPE, 190°C / 2160 g (Antioxidant / 626 phosphite = 300/300 ppm)



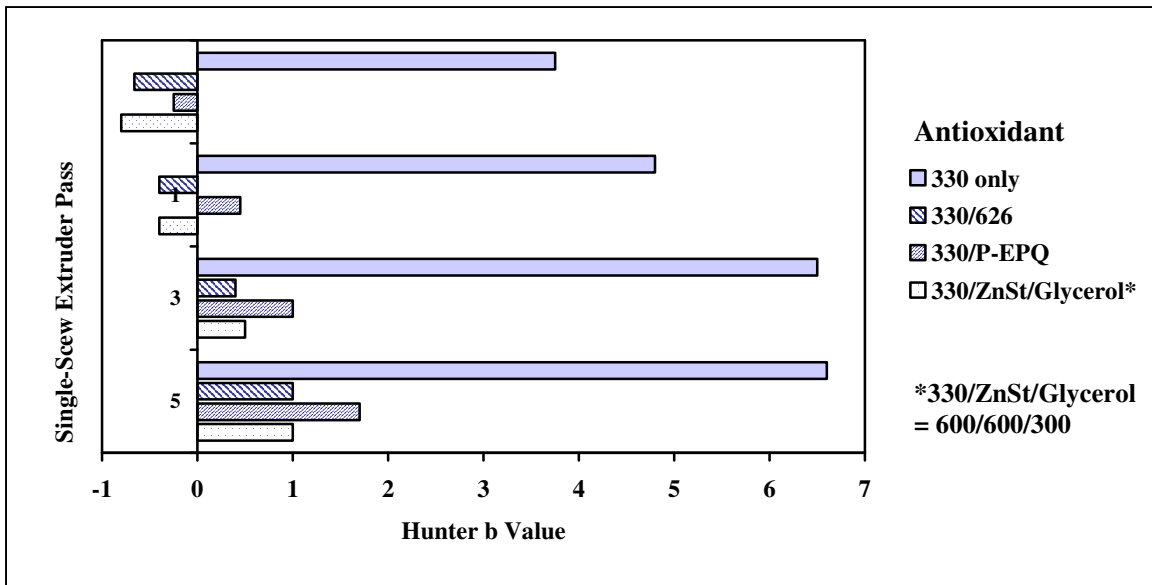
## USA OF ETHANOX ANTIOXIDANTS IN HIGH-DENSITY POLYETHYLENE

## COLOR

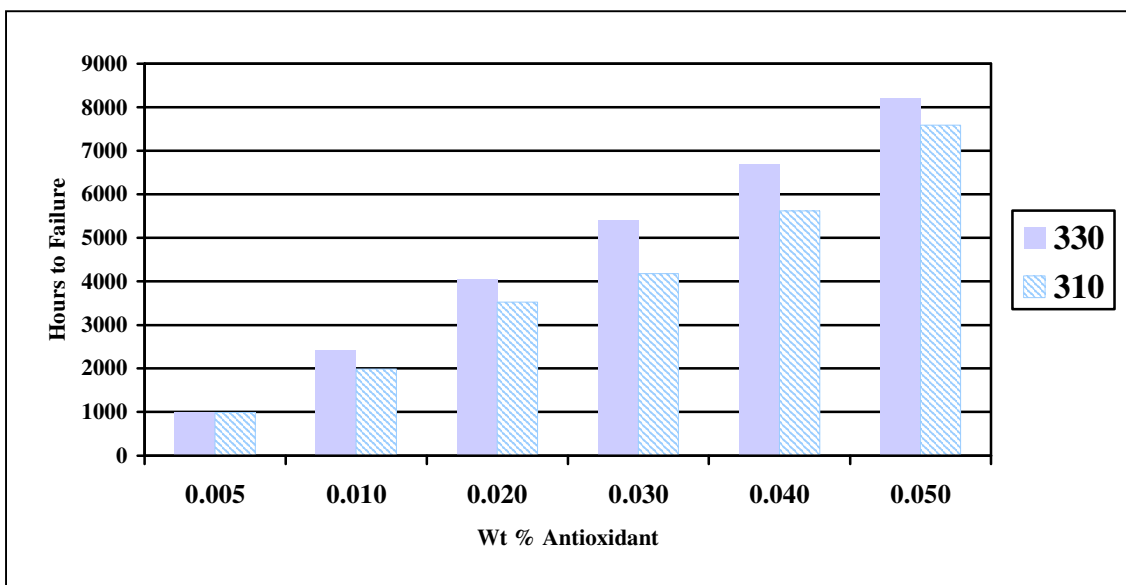
A darkening in color is generally associated with the use of phenolic antioxidants in HDPE. The color change can be controlled by the use of phosphites, polyhydric alcohols and acid neutralizers such as zinc stearate (ZnSt).

### Effect of Co-Additives on HDPE Color

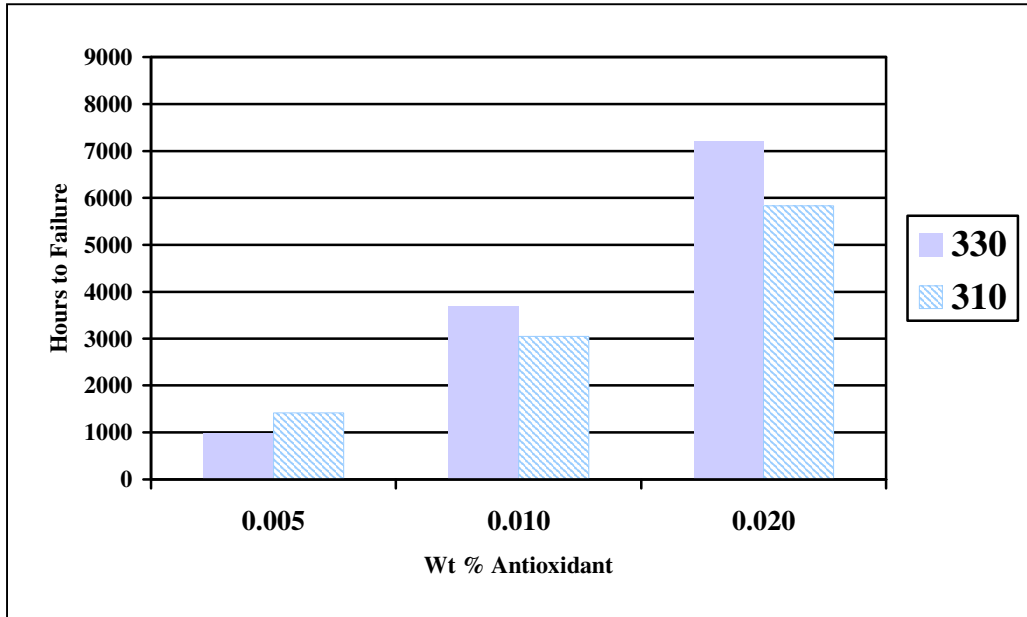
ETHANOX 330 Antioxidant/Co-additive = 600/300 ppm



Oven aging of HDPE containing ETHANOX antioxidants at 110°C. Chromium catalyst, 10-mil plaques



**Oven aging of HDPE containing ETHANOX antioxidants at 110°C. Chromium catalyst, 25-mil plaques**



**Aging of LLDP by oxygen induction time. 25-mil plaque, two passes through extruder; OIT @180°C in air. All samples contain DHT (500 ppm).**

Additive Composition (ppm)	Unaged Sample	Days to Failure		
		Oven Aged @ 105°C 14 Days	Boiled @ 100°C 7 Days	Boiled @ 100°C 14 Days
ETHANOX 330 antioxidant (1000)	23.7	23.2	21.4	27.5
ETHANOX 330 / 398 (500/500)	18.3	14.5	9.3	9.6
ETHANOX 310 antioxidant (1000)	25.4	24.4	4.9	4.0
ETHANOX 310 / ETHAPHOS 368 (500/500)	18.5	17.2	1.2	1.3

## NON-EXTRACTABILITY

In a medium-density polyethylene pipe compound, C14 -labeled ETHANOX 330 antioxidant was used as the stabilizer. Water was run through this pipe for 10 months under ambient conditions. No radioactivity was found in the water, and the results show that ETHANOX 330 antioxidant:

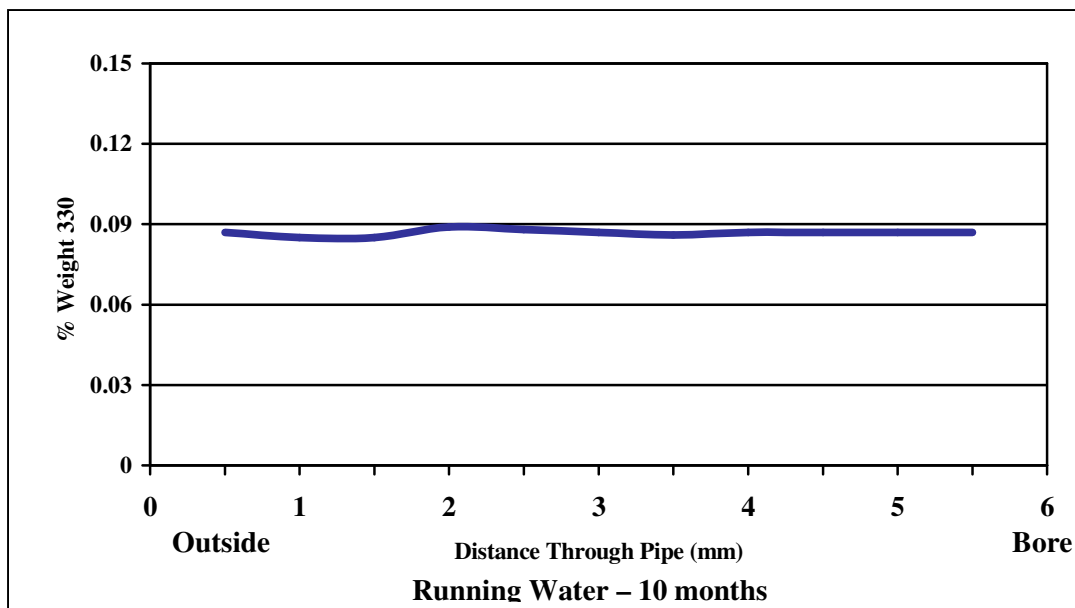
- Remains evenly distributed throughout the pipe.
- Does not migrate.
- Is not lost at either the air- or water-contacted surfaces.

Similar results were obtained in an accelerated three-month test at 80°C.

In polypropylene, when 12-mil strips containing 1000 ppm of C 14 -labeled ETHANOX 330 antioxidant were immersed in water at 120°F (49°C) and 250°F (121°C) and in 8% ethanol at 120°F (49°C) for 72 hours, no ETHANOX 330 antioxidant was found in any of the extracts (limit of detection 25 ppb).

ETHANOX 330 antioxidant has a unique resistance to extraction from polyolefins.

### Distribution of ETHANOX 330 antioxidant in HDPE pipe under ambient conditions



## THERMAL OXIDATION PREVENTION

ETHANOX antioxidants are extremely effective in polyethylene wire insulation, which requires long-term protection while exposed to copper. Samples of low-density polyethylene containing TiO<sub>2</sub> pigment and oxalic acid bis (benzylidene hydrazide), a copper deactivator, were placed on copper foil, and then oven-aged.

### Thermal Stability of LDPE in the presence of Copper, Oven Aged at 105°C

<b>Antioxidant</b>	<b>% Wt</b>	<b>Hours to Failure</b>
ETHANOX 330 antioxidant	0.025	1368
ETHANOX 310 antioxidant	0.025	1320
ETHANOX 330 antioxidant	0.1	4728
ETHANOX 310 antioxidant	0.1	4176

The formulation above contained 1% wt TiO<sub>2</sub> pigment and equal amounts of antioxidant and copper deactivator.

In an accelerated test using the Differential Scanning Calorimeter (DSC), samples of high-density polyethylene containing the copper deactivator were placed on copper foil. Electrically neutral ETHANOX antioxidants excel in polyethylene insulating materials.

### Thermal Stability of HDPE in the presence of Copper powder by D.S.C.

<b>Antioxidant</b>	<b>% Wt</b>	<b>Hours to Failure (200°C)</b>
None	0.5	3
ETHANOX 330 antioxidant	0.5	41
ETHANOX 310 antioxidant	0.5	35

The formulation above contained 1% wt TiO<sub>2</sub> pigment and 0.1% wt copper deactivator.

## NON-TOXICITY

Complete information on the use of ETHANOX antioxidants in FDA-regulated applications is available in our report titled, "ETHANOX Antioxidants - The Proven Choice in FDA Regulated Applications."



## ANTIOXIDANTS REFERRED TO IN FIGURES

**ETHANOX 310 Antioxidant** - Tetrakis [methylene-3- (3',5'-di-tert-butyl-4'-hydroxyphenyl) propionate] methane or Tetrakis [methylene (3,5-di-tert-butyl-4-hydroxyphenyl)drocinnamate] methane

**ETHANOX 314 Antioxidant** - 1,3,5-Tris (3,5-di-tert-butyl-4-hydroxybenzyl)-s-triazine-2,4,6- (1H,3H,5H) trione

**ETHANOX 330 Antioxidant** - 1,3,5-trimethyl-2,4,6-tris (3,5-di-tert-butyl-4-hydroxybenzyl) benzene

**ETHAPHOS 326 Antioxidant** - Bis(2,4-di-t-butylphenyl) pentaerythritol diphosphite

**ETHANOX 376 Antioxidant** - octadecyl 3,5-di-tert-butyl-4-hydroxyhydrocinnamate

**ETHAPHOS 368 Antioxidant** - tris- (2,4-di-t-butylphenyl) phosphite

BHT - 2,6,-di-tert-butyl-para-cresol

Product A - 1,1,2-Tris (2 methyl-4-hydroxy-5-tert-butylphenol) butane

Product B - 1,3,5-Tris (4-tert-butyl-3-hydroxy-2,6-dimethyl benzyl)-s-triazine-2,4,6- (1H,3H,5H) trione.

626 phosphite = Ultrinox® 626 Antioxidant, a product of Crompton Corporation

P-EPQ phosphonite = Sandostab® P-EPQ Antioxidant, a product of Sandoz, Ltd.

DHT-4A, DHT-4, and DHT-4C are products of Kyowa Chemical Industry Co., Ltd.

Luperox 101 is a product of Atofina Chemical Company.

9228 = Doverphos® 9228 Antioxidant, a product of Dover Chemical Corporation

## **ALBlend™ ADDITIVE BLENDS**

Albemarle knows polymers, especially polyolefins, from A to Z. We are a leading global manufacturer of polyolefin chemicals and additives including catalysts, co-catalysts and supported catalyst systems; antioxidants; and flame-retardants. So when other companies say they can't make an additive blend for you—or that your additives can't even be put into a blend—call Albemarle. We know how to use ingredients no one else does, giving you results no one else can. And we may save you money, too.

### **How Does Albemarle Do It?**

For its ALBlend additive packages, Albemarle starts with a blend of solid and/or liquid additives and a formulation-dependent solvent mix. We then run this blend through a patented pelletization, drying and screening process designed to deliver clean, consistent and uniform pellets of blended additive products with essentially no heat history. Every pellet, contains the same concentration of ingredients—100 percent additives, no binders and not a polymer master batch-giving plastics compounders and processors more consistent resins and end-use products.

### **Advantages of ALBlend Additive Packages:**

- 1 – No Heat History
- 2 – No Binders
- 3 – Processing Efficiency
- 4 – Less Waste
- 5 – Quality Control & Cost
- 6 – Improved Handling Safety

#### **1 – No Heat History**

The ALBlend proprietary pelletization process:

- Uses no melt or high compression processes
- Provides blends with minimized heat history of additives

#### **2 – No Binders**

ALBlend additive packages:

- Contain no binders to hold the additive components together
- Are 100% percent value-adding components

#### **3 – Processing Efficiency**

ALBlend additive packages benefit users by reducing their cost of capital initial, operations, maintenance and repairs. Plastic processors will enjoy:

- Reduced number of feeders
- Reduced downtime from feeding problems, especially pluggage from sticky additives and bridging or flow problems from powders
- Reduction in blend and storage hoppers, as there is no need for master batching

#### **4 – Less Waste**

Albemarle's polymer additive experts target optimum additive formulations and help improve customers' resins via:

- Reduced additive consumption
- Reduced additive loss from dusty powder
- Increased resin grade quality from more consistent additive blends
- Reduced cross contamination in additive feed hoppers
- Faster production grade changes

#### **5 – Quality Control & Cost Advantage**

ALBlend additive packages benefit users through:

- Reduced analytical cost from quality control
- Reduced ordering, shipping and handling of small quantities
- Reduced space and warehousing costs
- Reduced inventory costs of individual additives

#### **6 – Improved Handling Safety**

By eliminating powders and, thus, dust from in the workplace, ALBlend products:

- Improve workplace hygiene
- Improve transfer and handling of additives
- Limit exposure to contact and/or inhalation irritants
- Eliminate dust explosivity hazards

If your current supplier can't provide the technical expertise, the quality or the cost-effectiveness you need, try Albemarle and our ALBlend additive blends. Albemarle offers customer-specific solutions incorporating any of the "standard" components plus other cost saving components. Unique blend solutions for common problems, such as water carry-over in water-quenched raffia film are common at Albemarle.

If your supplier says they can't make a blend for you or that the additives you use can't even be put into a blend, call Albemarle. We make multi-additive blends with low-bulk density, low-melt or liquid additives routinely. If you have a color stability problem requiring additional expensive phosphite antioxidants, our ALBlend technology may save you money. If your production relies on first-or second-generation catalyst systems, our ALBlend technology may be able to save you money. If you want one source for all your antioxidant needs, call Albemarle.

# ALBlend™ ADDITIVE BLEND

## Case Study 1

### ALBlend Additive Packages Control Color, Lower Cost

**Issues:** A major polyethylene manufacturer, needed to improve the color of the finished resin, and they also wanted to reduce additive cost.

**Analysis:** The resin process used a complex catalyst system that actually contributed to color of the finished resin. To control color, the customer had resorted to a high concentration of an expensive secondary phosphite antioxidant.

**Action taken:** Albemarle's polymer additives experts created a statistical experimental design for the customer's resin to define the optimum formulation for the manufacturer's additive package.

Albemarle then reformulated the customer's additive package by replacing the expensive phosphite antioxidant with a lower-cost phosphite antioxidant plus proprietary chemistry to quench the process included active catalyst residue during compounding.

The ALBlend package improved the resin color and is more resistant to the process included alkaline internal environment of the resin, found both during resin production and handling by polymer manufacturer.

**Result:** By addressing critical characteristics of both the resin production process and catalyst system used, Albemarle helped improve the overall stability of the resin while reducing the additive package total cost. The ALBlend package improved the resin's process stability as measured by MFI long-term heat stability and color.

**Bottom line:** Albemarle greatly improved the customer's resin at a lower cost to the customer. The customer was so pleased with Albemarle's expert service and products that it now buys additional additive packages from Albemarle for resins used in sheet, injection and blow molding applications.

# ALBlend™ ADDITIVE BLEND

## Case Study 2

### ALBlend BOPP Additive Package Improve Productivity and Lower Cost

**Issues:** A major polypropylene (PP) manufacturer needed to change the formulation of its resin to reduce screen pack pluggage problems at its customers' film extrusion processes, or risk losing their business. The customer also wanted to improve the quality of its film products and reduce additive cost.

The resin producer's customers had to change screen packs in their film extruders seven times more often than when they had used a competitive PP grade.

**Analysis:** The PP contained high-cost secondary phosphite antioxidants plus a high-temperature, free-radical stabilizer.

Catalyst residue and the PP process itself contributed to the screen packing problems.

**Action taken:** Albemarle's polymer additives experts reformulated the customer's additive package by replacing the high-cost secondary phosphite antioxidant with a lower-cost secondary phosphite and removed the high-temperature additive, which Albemarle found was completely unnecessary to achieve the results the customer desired.

**Result:** Albemarle's formulation reduced screen pack pluggage problems by a factor of seven (7), equaling the performance of competitive resins.

The ALBlend additive helped our customer produce a resin of equal color and melt stability to its previous resin at a significant lower total cost.

**Bottom line:** Albemarle greatly improved the customer's resin by providing a lower-cost, better-performing additive package. Our customer's customers saved money too, and they no longer had the screen pack pluggage problems they had had with the previous resin.

# ALBlend™ ADDITIVE BLEND

## Case Study 3

### Improved Consistency, Feeding, Handling with ALBlend Additive Package

**Issues:** A major polypropylene (PP) resin manufacturer, wanted to improve the additive level consistency and additive feeding for its PP resin.

They also wanted to reduce dusting it experienced from its previous powder-form additives, which had resulted in some operator dermatological irritation.

**Analysis:** Customer Z's PP resin contained small particle sodium benzoate which led to handling problems and a dusty environment. Dispersion of this additive plus feed consistency were major headaches.

Customer Z's previous additive package was delivered in powder form, the dusting from which can potentially lead to poor workplace cleanliness, and contact or inhalation hazards.

**Action taken:** Albemarle's polymer additives ALBlend additive blend package technology replaced the powder additives with a highly consistent, pelletized blend of all the required additives including the sodium benzoate.

**Result:** Albemarle's new ALBlend additive technology helped Customer Z end the nagging quality problems it experienced with previous additive packages and improve the consistency of Customer Z's own resin.

Albemarle's ALBlend pelletized additive package improved the ease of feeding and ended the dusting problem associated with the customer's previous powder-form additive package.

[Overall Albemarle's ALBlend technology helped Customer Z produce a resin of improved quality, consistency and environmental characteristics].

**Bottom line:** Overall Albemarle's ALBlend technology helped Customer Z produce a resin of improved quality, consistency and environmental characteristics.