Kirk-Othmer Encyclopedia of Chemical Technology.

ANTIOZONANTS

The term antiozonant, in its broadest sense, denotes any additive that protects an elastomer against ozone deterioration. Most frequently, the protective effect results from a reaction with ozone, in which case the term used is chemical antiozonant. Ozone is generated naturally by electrical discharge and also by solar radiation in the stratosphere (1). These sources produce ground-level ozone concentrations of 1–5 parts per hundred million (pphm). Ozone is also produced in urban centers by ultraviolet photolysis, which can yield concentrations up to ≈25 pphm. Only a few pphm ozone in air can cause rubber cracking, which may destroy the usefulness of elastomer products. Ozone attacks any elastomer with backbone unsaturation. Degradation results from the reaction of ozone with rubber double bonds. Unstretched rubber reacts with ozone but is not cracked. Ozone cracking in stretched rubber is always perpendicular to the direction of the applied stress. Commercial antiozonants have been available since the early 1950s. Since that time, the ozone degradation problem has worsened as atmospheric ozone concentrations have gradually increased, especially in urban industrial areas.

1. Chemistry of Ozone Attack

Ozone cracking is a physico-chemical phenomenon, and many factors are involved in explaining the effect of ozone attack on elastomers (2–7). From a chemical point of view, ozone attack on olefinic double bonds causes chain scission and the formation of the decomposition products shown in Figure 1 (8, 9). These chemical reactions are believed to be similar for both small olefins and unsaturated rubbers. The first step is the formation of a relatively unstable primary ozonide (or molozonide) (**1**), which cleaves to an aldehyde or ketone (**2**) and a carbonyl oxide (or zwitterion) (**3**). Subsequent recombination of (**2**) and (**3**) produces a secondary ozonide (or just ozonide) (**4**). In small olefins, ozonide formation is generally a facile process. In stretched rubber, however, ozonide formation is more difficult, since the cleaved intermediates (**2**) and (**3**) may be forcefully separated to relieve the stress. Ozonides are reasonably stable in neutral environments, but they decompose readily under the influence of heat or various reducing agents to yield such chain scission products as aldehydes, ketones, acids, and alcohols. Polymeric peroxides (**5**) may be formed initially from the carbonyl oxide, but these are unstable and eventually decompose to yield chain scission products. The rate of chain scission is increased in the presence of active hydrogen (eg, water), probably because of the reaction with carbonyl oxides to form reactive hydroperoxides (**6**).

1.1. Physical Factors

Unsaturated elastomers must be stretched for ozone cracking to occur. Elongations of 3–5% are generally sufficient. Crack growth studies (10–18) have shown that some minimum force, called the critical stress, rather than a minimum elongation is required for cracking to occur. Critical stress values are nearly the same for most unsaturated rubbers. However, polychloroprene has a higher critical stress value than other diene rubbers,

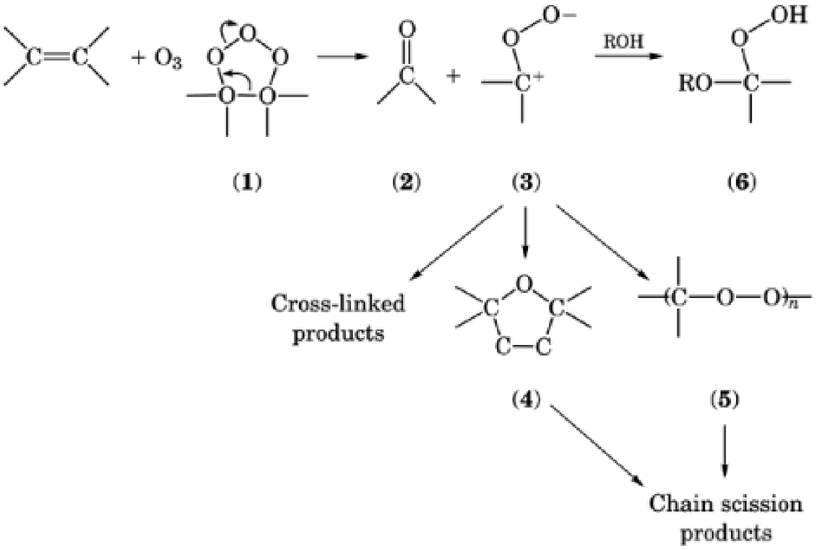


Fig. 1. Alkene–ozone reaction scheme.

consistent with its better ozone resistance. It has been found that temperature, plasticization, and ozone concentration have little effect on critical stress values.

Typically, ozone cracking initiates at sites of high stress (flaws) on the rubber surface. Thus, in general, rubber articles should be designed to minimize potential sites of high elongation such as raised lettering. Similarly, the use of clean molds helps to reduce the incidence of surface flaws.

2. Desirable Properties of Antiozonants

A physical antiozonant must provide an effective barrier against the penetration of ozone at the rubber surface. A chemical antiozonant, on the other hand, must first of all be extremely reactive with ozone.

The antiozonant should possess adequate solubility and diffusivity characteristics (19). Since ozone attack is a surface phenomenon, the antiozonant must migrate to the surface of the rubber to provide protection. The antiozonant should have no adverse effects on the rubber processing characteristics, eg, mixing, fabrication, vulcanization, or physical properties.

The antiozonant should be effective under both static and dynamic conditions.

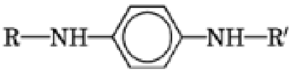
It should persist in the rubber over its entire life cycle. For noncarbon black filled rubbers, the antiozonant must be nondiscoloring and nonstaining. The antiozonant should have a low toxicity and should be nonmutagenic, and, the antiozonant should be acceptable economically, eg, have low manufacturing and end use costs.

3. Hydrocarbon Waxes

Waxes are one of the two general classes of commercial antiozonants. Waxes are derived from petroleum and are of two common types, paraffin and microcrystalline (20–23). Typical carbon numbers are $n = 20 - 50$ for paraffin waxes and $n = 30 - 70$ for microcrystalline materials. If a wax is present in a vulcanizate at a concentration exceeding its solubility, some of it will migrate to the rubber surface where it can form a physical barrier to prevent the penetration of ozone. Waxes, of course, are essentially unreactive towards ozone so that there is no appreciable chemical protection. Commercial waxes are usually blends of paraffin and microcrystalline waxes. Such blends provide protection over a wide temperature range ($\approx 10 - 60^\circ\mathrm{C}$). Advantages of waxes are their relatively low cost; they are nonstaining; and they generally have no adverse effects on rubber processing and vulcanization. Unfortunately, waxes have a number of shortcomings: they are ineffective under dynamic stress conditions; their protection capability is highly dependent on exposure temperature; and waxes may easily be lost during storage or use by embrittlement, scuffing, or delamination (6).

4. Chemical Antiozonants

Chemical antiozonants comprise the second general class of commercial antiozonants. Of the many compounds reported to be chemical antiozonants, nearly all contain nitrogen. Compound classes include derivatives of 1,2-dihydro-2,2,4-trimethylquinoline, *N*-substituted ureas or thioureas, substituted pyrroles, and nickel or zinc dithiocarbamate salts (see also Antioxidants). The most effective antiozonants, however, are derivatives of *p*-phenylenediamine [106-50-3] (*p*-PDA):



(see Amines-aromatic, phenylenediamines). The commercial materials fall into three classes (Table 1): *N,N′*-dialkyl-*p*-PDAs, *N*-alkyl-*N′*-aryl-*p*-PDAs, and *N,N′*-diaryl-*p*-PDAs.

The *N,N′*-dialkyl-*p*-PDAs (where the alkyl group may be 1-methylheptyl, 1-ethyl-3-methylpentyl, 1,4-dimethylpentyl or cyclohexyl) are the most effective in terms of their reactivity towards ozone (24–26). These derivatives increase the critical stress required for the initiation of crack growth, and they also reduce the rate of crack growth significantly (15). The *sec*-alkyl group is most active, for reasons that are as yet not completely clear. The drawbacks of these derivatives are: their rapid destruction by oxygen, ie, shorter useful lifetimes; their activity as vulcanization accelerators, and hence increased scorchiness; their tendencies to cause dark red or purple discoloration; and the difficulty in handling because they are liquids. The dialkyl-*p*-PDAs are seldom used alone in rubber compounds, although they can be used effectively when blended with *N*-alkyl-*N*-aryl-*p*-PDAs.

The *N*-alkyl-*N′*-aryl-*p*-PDAs (where the aryl is phenyl and the alkyl may be cyclohexyl, 1,3-dimethylbutyl or 1-methylethyl) are the most widely used *p*-PDAs. These derivatives reduce the rate of crack growth and also the number of cracks. The alkyl-aryl-*p*-PDAs are in general excellent antiozonants, particularly in dynamic environments. These derivatives are destroyed only slowly by oxygen and increase the scorchiness of the stock only slightly. These are intermediate in staining among the three classes of *p*-PDAs.

The *N,N′*-diaryl-*p*-PDAs (where the aryl group may be phenyl, methylphenyl, or napthalenyl) are only moderately active antiozonants, that can only be used at low concentrations (generally less than 2 phr) because of their poor solubility. However, they have minimal scorching effects, are the most stable towards oxygen, and stain the least. Their main advantage is high resistance to loss by consumption and vaporization (2), and in combination with more reactive antiozonants, they can offer a degree of increased protection in such long term applications as radial passenger tires (27).

The principal objection to *p*-PDA antiozonants is their staining characteristics. The lack of suitable alternative antiozonants for light-colored diene rubber articles is one of the outstanding problems in rubber technology. Few chemical antiozonants outside the class of *p*-PDAs have much commercial importance. One of the few exceptions to this rule is 6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline [91-53-2], one of the first commercial antiozonants.

Table 1. Commercial Antiozonants

p-Phenylenediamine[a]	CAS Registry Number	Abbreviation	Melting point, °C	Trade names[b]
N,N′-bis(1,4-dimethylpentyl)	[3081-14-9]	DMPPD	liquid	Eastozone 33, Santoflex 77, Flexzone 4L, UOP 788
N,N′-bis(1-ethyl-3-methylpentyl)	[139-60-6]	DEMPD	liquid	Flexzone 8L, UOP 88, Antozite 2
N,N′-bis(1-methylheptyl)	[103-96-8]	DMHPD or DOPPD	low melting	UOP 288, Antozite 1
N-(1,3-dimethylbutyl)-*N*′-phenyl	[61931-82-6]	HPPD or 6PPD	48–50	Antiozonant PD-2, Anto$_3$ "E", Antozite 67P, Flexzone 7L/7F, Permanax 6PPD, Santoflex 13/13F, UOP 588, Vulkanox 4020
N-(1-methylethyl)-*N*′-phenyl	[101-72-4]	MEPD or IPPD	70–78	Antiozonant PD-1, Anto$_3$ "H", Flexzone 3C, Permanax IPPD, Santoflex IP, UOP 388, Vanox 3C, Vulkanox 4010 NA
N,N′-dicyclohexyl	[4175-38-6]	DCHPD	106	UOP 26
N-cyclohexyl-*N*′-phenyl	[101-87-1]	CHPD	110	Antioxidant 4010, Flexzone 6H, UOP 36, Vanox 6H
mixed-*N,N*′-diaryl	[68953-84-4]	DTPD	90–115	Wingstay 100 [50864-70-5], Wingstay 200 [88026-77-1], Vanox 2
N,N′-diphenyl	[74-31-7]	DPPD	144–153	AgeRite DPPD, J-Z-F, Permanax DPPD
N,N′-bis(2-naphthalenyl)	[93-46-9]	DNPD	224–230	AgeRite White, Vulkanox DNP

[a] 1,4-Diaminobenzene [106-50-3].

[b] Manufacturers' trade names: Akron Chemical (Antiozonant), Eastman (Eastozone), Goodyear (Wingstay), Mobay (Vulkanox, Antioxidant), Monsanto (Santoflex), Pennwalt (Anto$_3$), Uniroyal (Flexzone, J-Z-F), Universal Oil Products (UOP), R. T. Vanderbilt (Antozite, AgeRite, Vanox), and Vulnax (Permanax).

5. Mechanism of Action of Chemical Antiozonants

Several theories have appeared in the literature regarding the mechanism of protection by *p*-PDA antiozonants. The scavenger theory states that the antiozonant diffuses to the surface and preferentially reacts with ozone, with the result that the rubber is not attacked until the antiozonant is exhausted (25, 28, 29). The protective film theory is similar, except that the ozone–antiozonant reaction products form a film on the surface that prevents attack (28). The relinking theory states that the antiozonant prevents scission of the ozonized rubber or recombines severed double bonds (14). A fourth theory states that the antiozonant reacts with the ozonized rubber or carbonyl oxide (**3**) in Fig. 1) to give a low molecular weight, inert self-healing film on the surface (3).

The literature suggests that more than one mechanism may be operative for a given antiozonant, and that different mechanisms may be applicable to different types of antiozonants. All of the evidence, however, indicates that the scavenger mechanism is the most important. All antiozonants react with ozone at a much higher rate than does the rubber which they protect. The extremely high reactivity with ozone of *p*-phenylenediamines, compared to other amines, is best explained by their unique ability to react free-radically. The chemistry of ozone–*p*-PDA reactions is known in some detail (30, 31). The first step is believed to be the formation of an ozone–*p*-PDA adduct (32), or in some cases a radical ion. Four competing fates for dissociation of the initial adduct have been described: amine oxide formation, side-chain oxidation, nitroxide radical formation, and amino radical formation.

Although all antiozonants must react rapidly with ozone, not all highly reactive materials are antiozonants. Something else in addition to the scavenging effect is required. The protective film theory contends that ozonized products, to a considerable extent, prevent ozone from reaching the rubber. There is visual and microscopic evidence for formation of a protective film on the rubber surface (33). Spectroscopic characterization has shown that this film consists of unreacted antiozonant and many of the same components observed in ozonized liquid antiozonant (30, 33, 34). The components of the film are polar and tend not to diffuse back into the rubber bulk.

The relinking (14) and self-healing film (3) theories require chemical interaction between the antiozonant and ozonized rubber. The evidence for these interactions is meager (35, 36). Overall, there seems to be no clear evidence in the literature for *p*-PDA derivatives becoming attached to rubber chains as a result of ozone attack. Much fundamental work in this area remains to be done, however. It seems clear at this point that any antiozonant–rubber interaction must be much less important than the scavenging effect of the antiozonant. In summary, the scavenger model is believed to be the principal mechanism of antiozonant action. Ozone–antiozonant reaction products form a surface film that provides additional protection (37).

6. Manufacture and Production

The *N,N′*-dialkyl-*p*-PDAs are manufactured by reductively alkylating *p*-PDA with ketones. Alternatively, these compounds can be prepared from the ketone and *p*-nitroaniline with catalytic hydrogenation. The *N*-alkyl-*N′*-aryl-*p*-PDAs are made by reductively alkylating *p*-nitro-, *p*-nitroso-, or *p*-aminodiphenylamine with ketones. The *N,N′*-dialkyl-*p*-PDAs are made by condensing various anilines with hydroquinone in the presence of an acid catalyst (see Amines-aromatic, phenylenediamines).

7. Alternatives to Antiozonants

Any diene rubber article subjected to flexing, bending, or folding requires protection against ozone. Chemical antiozonants, primarily *p*-PDAs, are used in general purpose commercial applications (mainly tires, hoses, flat belts, and transmission belts) where staining and discoloration are not serious problems. Waxes act as antiozonants for diene rubbers under conditions requiring little or no flexing, such as tie-down straps. Under these conditions, waxes are more effective than the *p*-PDA antiozonants (21). However, in applications involving dynamic stress where discoloration cannot be tolerated, alternatives to the traditional antiozonants must be used. Typical applications include white tire sidewalls, gaskets, weather stripping, gloves, and sporting goods. The two principal alternatives to antiozonants are ozone-resistant elastomers and blends of ozone-resistant elastomers and diene rubbers.

7.1.1. Ozone-resistant elastomers

which have no unsaturation are an excellent choice when their physical properties suit the application, for example, polyacrylates, polysulfides, silicones, polyesters, and chlorosulfonated polyethylene (38). Such polymers are also used where high ozone concentrations are encountered. Elastomers with pendant, but not backbone, unsaturation are likewise ozone-resistant. Elastomers of this type are the ethylene–propylene–diene (EPDM) rubbers, which possess a weathering resistance that is not dependent on environmentally sensitive stabilizers. Other elastomers, such as butyl rubber (IIR) with low double-bond content, are fairly resistant to ozone. As unsaturation increases, ozone resistance decreases. Chloroprene rubber (CR) is also quite ozone-resistant.

7.1.2. Blends

of diene and backbone-saturated rubbers are frequently used in applications where discoloration by chemical antiozonants cannot be tolerated, yet where cost is still a primary consideration (eg, white sidewalls of tires).

Disadvantages are that physical properties have to be compromised and the two rubbers usually differ greatly in their rates of vulcanization. Usually, at least a 25% replacement by the ozone-resistant rubber is needed for an appreciable enhancement in ozone protection (6).

8. Testing

Since antiozonants are affected by most compounding ingredients, each new rubber compound requires the development of a cost-effective antiozonant system. Outdoor as well as accelerated ozone-chamber tests are available (39–45). Laboratory tests involve exposing a statically or dynamically elongated test sample to ozone and measuring the time to crack formation, the severity of cracking, or the decay of 100% modulus with time. Cracking is affected by ozone concentration and flow rate, temperature, humidity, sample shape, and type of strain (static, dynamic, or both). Cracking is accelerated by increasing ozone concentration. Normal test concentrations are 10–25 pphm, but they can be as high as 50 pphm in accelerated tests. Above 70°C ozone decomposes, and therefore testing is usually carried out at 30–50°C. Humidity can accelerate ozone cracking, and the maximum recommended value is $\approx 65\%$.

Strain affects both the number and size of cracks. Low strain (10–20%) causes fewer but larger cracks; higher strain gives numerous small cracks. Testing is usually done at low strain. Most antiozonants are used in applications where flexing is involved, and dynamic tests are needed. However, since actual field conditions involve a combination of static and dynamic stress, an intermittent static–dynamic ozone exposure often provides a more realistic test regimen. Laboratory tests are best used as screening procedures for new additives and compounds. Only field testing of rubber articles can provide a true judgment of the protective capability of a particular formulation.

9. Health and Safety Factors

The first *p*-PDA antiozonants were low molecular weight *N,N*′-dialkyl-*p*-PDAs which caused skin irritations. Current higher molecular weight *N,N*′-dialkyl or *N*-alkyl-*N*′-aryl derivatives are not primary skin irritants. A notable exception is *N*-(1-methylethyl)-*N*′-phenyl-*p*-PDA, which causes dermatitis. However, since some individuals are more sensitive than others, antiozonants should always be handled with care (46). When skin contact does occur, the affected area should be washed with mild soap and water. In case of eye contact, flush well with water. Inhalation of rubber chemicals should be avoided, and respiratory equipment should be used in dusty areas.

10. Uses and Formulations

Chemical antiozonants are routinely used to protect diene rubbers (NR, IR, BR, SBR, NBR) against atmospheric ozone for extended periods of time. Large volumes are used in tire, belt, and hoseapplications. The *N*-alkyl-*N*′-aryl-*p*-PDAs have largely displaced the *N,N*′-dialkyl-*p*-PDAs as the materials of choice since they are less scorchy, more persistent, and more easily handled. The *N,N*′-diaryl-*p*-PDAs are more effective antiozonants for chloroprene rubber (CR). Antiozonants (*p*-PDAs) are added to raw rubber stocks during mixing at 1–5 phr concentrations and, most frequently, at 2–4 phr. Paraffin waxes (1–2 phr) may also be added to rubber formulations containing *p*-PDAs for increased static ozone protection (21). Certain fillers may absorb or oxidize *p*-PDA antiozonants, slightly increasing the antiozonant requirement. The *p*-PDA antiozonants are generally compatible with sulfur-curing systems. Extender oils and plasticizers increase the ozone cracking of unprotected rubbers. This is because of increased chain mobility and a more facile separation of the ozonized rubber fragments (14, 47). General purpose antioxidants extend the service life of antiozonants and reduce the overall cost of the rubber protective system. Water can leach antiozonants and shorten the life of the product (48, 49). Leaching of *N,N′*-dialkyl-*p*-PDAs decreases with increasing size of the alkyl group and becomes negligible for groups containing more than seven carbon atoms.

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