1955

Part I: Mechanisms of inhibited air oxidation of olefins. Part II: Reduction of nitro compounds with titanium III

Chester Eugene Hamilton
Iowa State College

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PART I. MECHANISMS OF INHIBITED AIR OXIDATION OF OLEFINs

PART II. REDUCTION OF NITRO COMPOUNDS WITH TITANIUM III

by

Chester E. Hamilton

A Dissertation Submitted to the Graduate Faculty in Partial Fulfillment of The Requirements for the Degree of DOCTOR OF PHILOSOPHY

Major Subject: Organic Chemistry

Approved:

Signature was redacted for privacy.

In Charge of Major Work

Signature was redacted for privacy.

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Signature was redacted for privacy.

Dean of Graduate College

Iowa State College

1955
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ACKNOWLEDGMENTS
PART I. MECHANISMS OF INHIBITED AIR OXIDATION OF OLEFINS
INTRODUCTION

The inhibition of air oxidation has been a problem extensively studied because of practical applications in preventing deterioration of many different materials between the time of manufacture and consumption of the various products.

Despite theories advanced by various workers in the field through the years and despite the great number of investigations carried forward (many of which had direct practical application) the manner in which a small amount of a certain material in the presence of another substance prevents attack on the latter by oxygen in the air had been and, in many cases, still is an uncertainty.

Since a knowledge of the mechanism by which inhibitors or antioxidants prevent air oxidation is of both theoretical and practical interest, this work was carried forward, with the purpose of obtaining some knowledge of the chemical reactions of inhibitors exposed to air in the presence of an easily oxidized material.

Since the products from the air oxidation of the inhibitor would, in conjunction with kinetic studies also being carried forward in these laboratories, give evidence of the mechanism by which inhibitors act, it was decided to attempt to isolate and identify these products.

In order to accomplish this purpose, a rather large amount of inhibitor in the presence of tetralin, a typical olefin whose air oxidation has been previously studied, or in an inert solvent, was air oxidized and work leading to separation and identification of products from the inhibitors was pursued.
HISTORICAL REVIEW

The first observation of an antioxidant effect followed by use of the antioxidant to prevent deterioration of a valuable product has been in all probability long since buried in antiquity. There has been a rather large number of reviews written concerning various aspects of oxidation inhibitors. Le Bras has attributed the first observation of an antioxidant effect to Boyle. Moureau and Dufraisse, who published the first comprehensive review of the field, attributed the first observation of antioxidant action to Berthollet, who observed that traces of sulfur vapor prevented the luminescence of phosphorus in dilute oxygen atmosphere. Other early observations were that of Deschamps who observed that lard containing gum benzoin or populin did not become rancid as quickly as ordinary lard, and that of Chevreul who observed that oak wood retards the drying of linseed oil.

In spite of these and other observations, the existence, much less the importance, of antioxidants, in both practical and theoretical aspects, was not widely recognized until Moureau and Dufraisse, in 1922, began to publish observations on antioxygen. Their entry into the field occurred October 21, 1917 during World War I, when in an effort to prevent acrolein polymerization, they added pyrogallol, another easily oxidizable substance, to the acrolein and found that oxygen absorption was inhibited and the acrolein did not polymerize. Following the war they continued to carry forward investigations of antioxidant substances and their properties. Recognizing that the formation of organic peroxides was associated with oxidative degradation, they postulated that antioxidants react with organic
peroxides, causing their decomposition while the antioxidant was regenerated. They also observed a close relationship between oxidation catalysis and inhibition and that in some cases this positive or negative catalysis depends on pH and substrate.

The practical success of Moureau and Dufrasse in preventing polymerization of acrolein and their theories concerning antioxidants spurred the efforts of other workers to make both practical and theoretical contributions to this field. The rubber industry was vitally interested and was the first to institute active research in the field. The petroleum industry, food processors, paint industry and others soon followed and the work has been increasing in an evergrowing flood to the present time.

One of the notable events of practical importance was in 1929 when Egloff and coworkers published the first description of gasoline antioxidants, followed later by qualitative data concerning the effect of a large number of antioxidants on the length of the induction period before gum formation occurred in the gasoline. In the years that followed, aromatic amines, phenolic and aminophenolic compounds were the only known effective gas antioxidants. These were used along with copper deactivators such as the condensation product of salicylaldehyde and primary amino compounds. These compounds form complexes with traces of copper ion which often contaminate substances and the complex formation prevents deleterious effects which would occur should the copper be allowed to catalyze air oxidation of the gasoline.

Workers in vegetable and animal fats and oils were also concerned with antioxidants. Theirs was a two-fold interest since they recognized the
existence of naturally occurring oxidation inhibitors which they desired to identify and evaluate and they were also interested in more effective antioxidants. After Olcott and Emerson's work with naturally occurring vitamin E and the establishment of its structure by Ferhols, Golumbic in 1943 was able to show that vegetable oils contained tocopherol (vitamin E) which oxidized rapidly to tocoquinone and then more slowly to chroman-5,6-quinone. It was shown later that γ-tocopherol gives the same quinone. It was during the slower oxidation that organoleptic rancidity occurred and before peroxide concentration increased rapidly. Thus the difference in stability of vegetable and animal fats was explained since animal fats contain very little vitamin E. Since there is an optimum concentration of tocopherol for maximum inhibition the fact that addition of tocopherol to vegetable oil had no beneficial effect, whereas the opposite is true with animal fats, is thus explained.

In addition to vitamins C and E, other naturally occurring substances or their derivatives have been fruitful sources of highly efficient antioxidants. Gossypol was obtained from cottonseed and described by Mattill in 1931. In 1941 Sabalitschka and Boehm first patented the alkyl esters of gallic acid as antioxidants and Boehm and Williams described the properties of propyl gallate in 1943. Nordihydroguaiaretic acid obtained from creosote bush was described by Lundberg, et al. in 1944. Those most recently reported were norconidendrene from western hemlock trees reported in 1947 and aca-catechin from a bush native to India reported in 1951.

Although the results of the investigations to find efficient antioxidants and of the determination of how and where to use them have been
quite successful, the theories so far advanced to explain antioxidant action have not been able to explain all the facts or forecast with any certainty the results to be expected from a given antioxidant in a given system. However these theories have proved of value in being a guide to further study. The theory of Moureau and Dufraisse that the organic peroxides formed by autoxidation of the substrate are decomposed by the antioxidants with a regeneration of the antioxidant soon came into question because it began to be recognized that the antioxidant was oxidized during the inhibition period. Also, if there was regeneration of the antioxidant, the inhibition period should be infinitely long, which was not the case.

In 1929, Alyea and Backstrom concluded from their kinetic studies that autoxidation was a chain reaction and that antioxidants interrupted the oxidation chain and were themselves oxidized in the process. They were noncommittal about the actual mechanism, referring to energy chains, but not specifying how the energy was transmitted. A modification of the Moureau and Dufraisse theory had also been suggested. Since it had been recognized from the beginning that an inhibitor must be an easily oxidized substance and it was now recognized that the inhibitor was oxidized during the inhibition period, it was suggested that the inhibitor reduced the organic peroxides formed. Largely on the basis of kinetic evidence this idea is not now widely accepted, although kinetic evidence has recently been imputed by Shelton and co-workers to imply that amines (and carbon black) do not break oxidation chains, but destroy peroxide and are probably different than phenols in rubber. However, Kuzminskii showed that 2-naphthylphenylamine does not destroy rubber peroxide at 80° under a nitrogen atmosphere. He reports, on the
other hand, that di-\textit{p}-hydroxyphenylamine, \textit{p}-hydroxyphenyl-2-naphthylamine and trihydroxybenzene react with stable rubber peroxides. He feels this effect may occur but is not the primary cause of inhibition. He states that a good inhibitor should be a substance that gives up mobile hydrogen to peroxide radicals, thus breaking the chain and that it is necessary that the product from the inhibitor must be stable and must not be a chain transfer agent. Also as recently as 1948, Michaelis\textsuperscript{685} suggested a sort of cyclic mechanism where peroxide oxidizes hydroquinone to semiquinone and then reverses the process in some cyclic mechanism so that the peroxide is so short-lived in this cycle that it has no chance to break down and induce the slower irreversible reactions which result in the oxidation of the fatty acid substrate. This would seem to be substantially a reproposal of the old theory of Moureau and Dufraissee\textsuperscript{727}.

Other material of theoretical interest has been the evaluation of oxidation-reduction potentials of quinhydrone-type compounds and the critical oxidation potentials of other antioxidants which do not go to an oxidation product by an easily reversible process. After data of this type was obtained, an attempt was made to correlate the data with the efficiency of antioxidant action of various compounds.\textsuperscript{133,326,620,693} The potentials of the best inhibitors were between 0.6 and 0.8 volts, although some compounds in this range were not good inhibitors. This being the case, oxidation-reduction potentials could not be relied on to evaluate a substance as an inhibitor, although theoretically this should have been the case since the ease with which an electron can be removed should be related to the ease with which the antioxidant can be oxidized. The fact that quinols, which are usually good inhibitors, form a semiquinone reversibly on oxidation
led to the suggestion that phenolic oxidation proceeds in two steps, the first a reversible step and the second not usually so.

The effect of structure has been of importance in attempts to explain antioxidant activity. In 1934, Olcott showed that polyhydroxybenzenes were good antioxidants, but that hexahydroxybenzene exhibited no antioxidant action in spite of being easily oxidized. This is illustrative that mere ease of hydrogen removal is no guarantee of an effective antioxidant.

However, because many good inhibitors do have labile hydrogens, there still exists an impression that ease of hydrogen removal is a necessity in a good inhibitor in order that peroxy radicals may abstract the hydrogen, thus breaking the chain, provided the product from the inhibitor is stable. The studies of Murphy, et al. with phenothiazine derivatives is another case in point. They found that the first oxidation product of phenothiazine was phenothiazine-5-oxide which is almost as efficient an inhibitor as the first compound. This will form phenothiazine-5-dioxide on further oxidation and is no longer a good antioxidant, although there is still hydrogen available on the nitrogen atom. Substitution of alkyl radicals for the hydrogen impair the antioxidant properties of the original compound although the N-octadecyl and N-benzyl derivatives are nearly as efficient.

Other authors have studied the effect of varying substituents on various phenols and aromatic amines on antioxidant efficiency, as well as structure of hydrocarbons acting as antioxidants. Substituents releasing electrons to the aromatic ring enhance antioxidant activity.
Recently Campbell and Coppinger\textsuperscript{181} reported that 2,6-di-t-butyl-p-cresol formed \(1\)-methyl-\(1\)-t-butoxy-3,5-di-t-butylcyclohexadienone-4 when di-t-butylperoxide was decomposed in its presence and proposed that two RO\(_2\) with the same phenol should give the same type compound. Cook\textsuperscript{223} objected to this as a general mechanism of antioxidant action on the basis that he had obtained a dimerization product, \(3\),\(3\)',\(5\),\(5\)'-tetra-t-butyl-4,4'-dihydroxy-diphenylethane, on oxidation of the phenol with PbO\(_2\) or H\(_2\)O\(_2\). He mentions that this product was still a fairly efficient antioxidant. He cites other like products isolated by other workers in an attempt to show the generality of dimer formation. Moore and Waters\textsuperscript{704} also report isolating the same product in small yield from a free radical oxidation reaction. However, Bickel and Koojman\textsuperscript{110b} isolated the suggested peroxide derived from the phenol and \((\text{CH}_3)_2\text{C(NC)O}_2\) in 1953. This would mean that two peroxy radicals react with the phenol as compared to the one which would be necessary from Cook's dimerization product, if the dimer oxidized no further. In the case of Cook's product, it does go to \(3\),\(3\)',\(5\),\(5\)'-tetra-t-butylstilbene-4,4'-quinone, which would require that three radicals react with each phenol molecule. Kinetic evidence on stoichiometry is in order.

Two authors other than Boozer and Hammond\textsuperscript{135} have suggested complex formation is related to antioxidant effect. Jacquemain and Berger\textsuperscript{496} postulated that "positive catalysis" occurs at low (inhibitor) concentration because the inhibitor is associated with reacting substance, while at high concentration the association occurs mostly between molecules of inhibitor, thus explaining the antioxidant property of phenol at high concentration and its oxidizing properties at low concentration. Chamberlain and Walsh\textsuperscript{193,1114} postulate that there is a complex formed in the gas phase.
between the inhibitor and the radical chain carriers since they observed a correlation between antiknock effect and the influence of side chains on the electronic properties of aromatic rings. Those substituents which released electrons to the rings promoted antiknock effect.

Another problem of theoretical and also practical interest is that of synergism. Lea states that true inhibitors are usually oxidized while synergists enhance the activity of phenolic antioxidants. Many substances are synergists of another substance and no general theory has yet been applied. However, ascorbic acid is widely used and Calkins and Mattill proposed that the antioxidant quinone was reduced by the synergist and the semiquinone then oxidized to quinone by the activated peroxide which was reduced in the process. Golumbic found that benzoquinone and α-tocoquinone were good antioxidants if phosphoric acid were present and stated these compounds were reduced to the hydroquinone form by the fat substrate since a biological assay showed vitamin E activity upon addition to a fat of α-tocoquinone and phosphoric acid. Le Bras found that phenyl-2-naphthylamine and mercaptobenzimidazole were synergists. He proposed that the amine stopped reaction chains, since it decreased oxygen absorption and that the other compound, which had slight effect on the oxygen absorption rate, deactivated the peroxides formed.

In protecting material from oxidation not only are antioxidants and synergists used but also substances which form inactive complexes with pro-oxidant metals. This is particularly true in lubricants. Very complex substances are used which not only perform one or all antioxidant functions but also impart other desirable properties as well. Such substances are various sulfides, complex thiophosphoric acids, and others. Such a
vast amount of work has been done in the field in the last ten years alone that it is staggering. These and other substances have been listed in the following table. An effort has been made to make as complete a literature survey as possible of the work done in the last ten years. Much of the material was read only as found in Chemical Abstracts. No doubt there are omissions and errors since there are so many references. A few patents were not included since they could not be relocated by patent number in Chemical Abstracts. Others were not included which seemed to be very similar to other patents previously noted. The abstracts were searched page by page from January through September 1954. No doubt pertinent information has been overlooked. It is hoped that the compilation might prove serviceable. Where no designation is made in the table concerning the use of the antioxidant, none was indicated in the abstract.

From the previous discussion of theoretical investigations, it will be observed that little is known of the reaction products obtained from the air oxidation of antioxidants in a simple substrate. Most investigations have ignored this possibility of gaining an insight into the mechanism of inhibitor action. It is for this reason that the following work has been carried forward.
Table 1
Antioxidant literature survey

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aAccompanying substances or synergists and their references.
bElastomers.
cGasoline and fuels.
dCarotene and vitamins.
eLubricating and mineral oils.
fPaint oils.
gEdible oils and fats and derivatives.
hFoods and flavors.
iSoaps and waxes.
jMiscellaneous.
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\[(5,6,7,5',6',7'-Hexahydroxy-3,3,3',3'-tetramethylbis-1,1'-spirohydrindene)\]

| gallic acid                | X |   |   |   |   |   |   |   |   |                        | 577    |
| Ester of above             | X |   |   |   |   |   |   |   |   |                        | 577    |
| Gallic acid                | X | X |   |   |   |   |   |   |   |                        | 158, 328 |
|                            |   |   |   |   |   |   |   |   |   | 624, 722                |       |
|                            |   |   |   |   |   |   |   |   |   | 744, 745                |       |
|                            |   |   |   |   |   |   |   |   |   | 828, 896                |       |
|                            |   |   |   |   |   |   |   |   |   | 897, 955                |       |
|                            |   |   |   |   |   |   |   |   |   | 983, 1011               |       |

<p>| Hexylgallate               | X | X |   |   |   |   |   |   |   |                        | 158, 1032 |
| Decylgallate               | X | X |   |   |   |   |   |   |   |                        | 158, 1072 |
| Laurylgallate              | X | X |   |   |   |   |   |   |   | Citric acid - 638      | 983    |</p>
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<td>p-Hydroxycoumarans (2,2-dimethyl-6-t-butyl-5-hydroxycoumaran)</td>
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<td>3-t-Butyl-4-hydroxyanisole</td>
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<td>F. Quinone derivatives</td>
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<td>9-Naphthyl, phenyl-p-aminophenol</td>
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<td>Bis(piperidinomethyl)-p-α-amylyphenol</td>
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<td>Amino phenols, phennylamine, diphenylamine, naphthylamines, phenylenediamines - 1122</td>
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<td>p-(2-Naphthylsulfonamido) phenol</td>
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Table 1 (cont.)

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<td>d-(p-Tolylsulfonamido) phenol</td>
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<td>5-(p-Tolylsulfonamido)-1-naphthol</td>
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<td>Vanilline (3,4,1)__/CH₃OC₂H₃(OH)CHO/</td>
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<td>Coumarin and hydroxy derivatives</td>
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<td>Salicylaldoxime</td>
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<td>α-Naphthol</td>
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<td>β-Naphthol</td>
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<td>Thymol</td>
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<td>Chloromethylphenols</td>
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<td>1-Alkyl ethers of naphthols</td>
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<td>Phenol S</td>
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<td>2-4-butyl-4-methylphenol</td>
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<td>Aromatic α-hydroxymono-</td>
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<td>basic acid (propyl-, butyl- and benzylhydroxybenzoates) (α-hydroxybenzoic acid)</td>
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<td>Aliphatic hydroxy- polybasic acid (tartaric, citric, malic) - 897</td>
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<td>Picric acid</td>
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<td>Tricresol</td>
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<td>m-Nitro-p-cresol</td>
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<td>Kamala dye</td>
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<td>Citric and tartaric acids - 274 Maleic acid, hydroquinone, oleic acid - 833</td>
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<td>Tumeric dye</td>
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<td>2,6-Di-α-butylphenol</td>
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### Table 1 (cont.)

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<td>2,6-Di-t-butyl-p-cresol (ionol)</td>
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<td>Butylated hydroxy-anisole</td>
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<td>Polyalkylbenzylphenols</td>
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Additional information:
- Acids, hydroquinone,
- Lecithin, thiodi-
- Propionic acid - 345, 431
- Citric acid - 260, 583, 900, 603
- (NaPO₃)₆ and Mandrell's salt - 637, 638
- Propylgallate and citric acid - 190, 292, 546
- Propylgallate, citric acid and lecithin - 293
Table 1 (cont.)

<p>| Type | Antioxidant for | R | G | C | L | P | E | F | S | M | A | Ref. |
|------|----------------|---|---|---|---|---|---|---|---|---|---|-----|------|
| 2,2',3,3'-Tetrahydroxybiphenyls (5,5'-dimethyl-2,2'-3,3'-tetrahydroxybiphenyl, also the 5,5'-dipropyl and the 5,5'-diethyl) | X | | | | | | | | | | | 36, 194 |
| Bis(2-hydroxy-3,5-dimethylphenyl) butane | X | | | | | | | | | | | 146 |
| Bis(4-hydroxy-2,5-dimethylphenyl) butane | X | | | | | | | | | | | 146 |
| α-Alkylhydroxyphenylalkanes | X | | | | | | | | | | | 147 |
| Reac. prod. of 2-hydroxy alcohols with phenols | X | | | | | | | | | | | 124 |
| 2,4-Dihydroxybenzaldehyde | X | X | | | | | | | | | | P-S&lt;sub&gt;5&lt;/sub&gt; isobutylene, reac. prod. | 869 |
| p-Methoxyphenol | X | | | | | | | | | | | 133 |
| Cond. prod. of heterocyclic hydroxyaromatic compds. and aldehyde and nitrogen compd. | X | X | | | | | | | | | | 767 |</p>
<table>
<thead>
<tr>
<th>Type</th>
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<th>C</th>
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<th>F</th>
<th>S</th>
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<tr>
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**Nitrogen compounds**

**A. Amino phenol derivatives**

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Benzyltrimethylammonium butoxide - 206
Esters of thioglycolic acid - 451

1056
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<td>Phenylhydrazine</td>
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<td>α-Phenyl-α-methyl-hydrazine</td>
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<td>Resoc. prod. of aliphatic ketone and aminofluorene or aminobenzofuran or aminocarbazole</td>
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Secondary aromatic amine - 751

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<td>p-Phenylenediamine</td>
<td>X X  X Vitamin B complex - 404</td>
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<td>4-Cyclohexyloxydiphenylamine</td>
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<td>N-(4-hexyloxyphenyl)-2-naphthylamine</td>
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<td>m-Diaminoanisole</td>
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<td>(2,2,4-trimethyl-1,2-dihydroquinoline and diphenylamine)</td>
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<td>4-Cyclohexylaminodiphenylamine</td>
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<td>Hydroxy- and amino-subst. diphenylamine</td>
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<td>Metal salt of a monocamide of 2,5-endo-ethylene 3,4 cyclohexene-1,6-dicarboxylic acid</td>
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<td>Quaternary ammonium alkoxide</td>
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<td>(Benzyltrimethylammonium butoxide)</td>
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<td>Anthraquinone diamine derivative</td>
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<td>1,4-Diamino-2,3,5,6-tetramethylbenzene and its alkylated and acetylated derivatives</td>
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<td>2-Thiocyanato-4,6-diamino-2-triazine</td>
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<td>N,N'-dialkyl-ε-phenylenediamine</td>
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<td>RHNC₆H₄NO (R = alkyl or aralkyl)</td>
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<td>1-Salicylamino-guanidine</td>
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<td>Phenylbiguanide hydrochloride</td>
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<td>Reac. prod. of triphenylguanidine, N-dimethylaniline and HCHO</td>
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<td>p-Tolylbiguanide</td>
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<td>Antioxidant for R G C L P E F S M</td>
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<td>D. Amino acids</td>
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<td>Methionine</td>
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<td>Threonine</td>
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<td>Glycine</td>
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<td>H$_3$PO$_4$, and hydroxy-polyoxyalkylene ester of a fatty acid - 564</td>
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### Table 1 (cont.)

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<td>Tryptophan</td>
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<td>Tyrosine</td>
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<td>Butyltyrosine or alanine</td>
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<td>Leucine</td>
<td>Tocopherol, hydroquinone or NDGA</td>
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Table 1 (cont.)

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<td>Alanine</td>
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<td>N-(hydroxyaryl) aspartic acid diesters</td>
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<td>(diethyl-N-(p-hydroxyphenyl) aspartate)</td>
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<tr>
<td></td>
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<td><strong>A. Thiosemicarbazide derivatives</strong></td>
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<td>N-phenyl-N'-p-hydroxyphenylthiourea</td>
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<td>N,N'-bis(p-hydroxyphenyl) thiourea</td>
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<td>N,N-bis(p-hydroxyphenyl) urea</td>
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Table 1 (cont.)

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<td>RR'NCS$_2$NH$_2$RR'</td>
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<td>Sulfonilcarbamate</td>
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<tbody>
<tr>
<td></td>
<td>R</td>
<td>G</td>
<td>C</td>
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<tr>
<td>2,3-Dimercaptopropanol</td>
<td>X</td>
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<tr>
<td>Thioamides (thioacetamide)</td>
<td>X</td>
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<td></td>
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<tr>
<td>Cond. prod. of aldehyde, polyamine and hydroxyaromatic compd. with P2S5, or S2Cl2, or S, or SC12, or PCl3 and S2Cl2</td>
<td>X</td>
<td>X</td>
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</tr>
<tr>
<td>Organic sulfhydryl compd. and a sulfide contg. an unsubst. amino group and 1% water</td>
<td>X</td>
<td></td>
<td></td>
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<tr>
<td>Cyclic mercaptans</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tri-3-thienyltrithio-2-formate</td>
<td>X</td>
<td></td>
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<tr>
<td>Glyoxal-x-butylresol cond. prod. and P2S5 or sulfur chloride</td>
<td>X</td>
<td></td>
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<tr>
<td>Dibenzodimethylthiuram disulfide</td>
<td>X</td>
<td>X</td>
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<tr>
<td>β-Alkylthialkanone</td>
<td>X</td>
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### Table 1 (cont.)

<table>
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<tr>
<td><img src="image" alt="Chemical structure" /></td>
<td></td>
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<tr>
<td>( Z = \text{CH}(R_i)(\text{NR}_2 R_i R_i X) )</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( X = \text{hydroxyl group or an organic or inorganic acid radical} )</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( n = 1 \text{ to } 4 )</td>
<td>X</td>
<td>X</td>
<td>298</td>
</tr>
<tr>
<td>( \text{H}_2\text{S} \text{ and } \text{NH}_2\text{HS} \text{ and oxygen contg. compds.} (ketones, esters, acids, isophorone type fractions) )</td>
<td>X</td>
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<tr>
<td>Bis(ketoalkyl) sulfide</td>
<td>X</td>
<td>X</td>
<td>X</td>
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<tr>
<td>Polymers of thiophene</td>
<td>X</td>
<td></td>
<td>503</td>
</tr>
<tr>
<td>Polymers of thiojene</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Halogenated t-alkyl sulfides and polysulfides</td>
<td>X</td>
<td></td>
<td>299</td>
</tr>
<tr>
<td>Metal salts of aliphatic seleno- or telluromer-captans</td>
<td>X</td>
<td></td>
<td>1006, 1007</td>
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<tr>
<td>Esters of thiophenethiol</td>
<td>X</td>
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<tr>
<td>Bis(isoctylphenol) sulfide</td>
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<tr>
<td>Type</td>
<td>Antioxidant for</td>
<td>A</td>
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</tr>
<tr>
<td>----------------------------------------------------------------------</td>
<td>----------------</td>
<td>---</td>
<td>------</td>
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<tr>
<td>Res. prod. of metal alkyl xanthate with amine salt of organic thiophosphoric or thiophosphorus acid</td>
<td>X</td>
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<tr>
<td>Aliphatic sulfide - sulfur and sulfide - selenium comps.</td>
<td>X</td>
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<td>177</td>
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<tr>
<td>Didodecyl or dioctadeyl esters of thiopropionates</td>
<td>X</td>
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<td>Aliphatic diselenides</td>
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<td>268</td>
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<tr>
<td>2-Hydroxy-3-t-alkyl-5-methylbenzyl-t-alkyl sulfides</td>
<td>X</td>
<td></td>
<td>548</td>
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<tr>
<td>Dithiooxalodiamides and thioacetamides</td>
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<td></td>
<td>588</td>
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<tr>
<td>Aliphatic thioethers of hydroquinone</td>
<td>X</td>
<td></td>
<td>757</td>
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<tr>
<td>Tetraalkyl- (aryl or aralkyl-) thiuramdi- sulfide</td>
<td>X X</td>
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<td>411, 752, 1066, 1067, 1068</td>
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<td>Type</td>
<td>R</td>
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<td>Tetramethylthiuram disulfide</td>
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<td></td>
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<tr>
<td>Sulfurized aliphatic borates</td>
<td>X</td>
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<tr>
<td>Mercaptobenzothioxyd disulfide</td>
<td>X</td>
<td></td>
<td></td>
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<tr>
<td>Secondary and tertiary mercaptans and sulfides</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phenothiazine and derivatives</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
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</tr>
<tr>
<td></td>
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<td></td>
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</tr>
<tr>
<td>Dialkylpolysulfides</td>
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<td></td>
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<tr>
<td>4-(2,2-Dimethylpropyl)-1,2-dithio-4-cyclopentene-3-thione</td>
<td>X</td>
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<tr>
<td>4-Methyl-5-t-butyl-1,2-dithio-4-cyclopentene-3-thione</td>
<td>X</td>
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<tr>
<td>Alkyl derivative of thiophene and thiophene by-product tar</td>
<td>X</td>
<td></td>
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<tr>
<td>Type</td>
<td>Antioxidant for</td>
<td>A</td>
<td>Ref.</td>
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<tr>
<td>----------------------------------------------------------------------</td>
<td>-----------------</td>
<td>---</td>
<td>------</td>
</tr>
<tr>
<td>Reac. prod. of thiophenes or alkylthiophenes with chlorinated paraffin waxes</td>
<td>X</td>
<td></td>
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<tr>
<td>3-Thienylpolysulfides</td>
<td>X</td>
<td></td>
<td>155</td>
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<tr>
<td>Dicarboxylic acid ester selenide or diselenide</td>
<td>X</td>
<td></td>
<td>178</td>
</tr>
<tr>
<td>Subst. sulfur hydrazides</td>
<td>X X</td>
<td></td>
<td>932</td>
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<tr>
<td>(RNH₂NH(X)R')</td>
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<td></td>
<td></td>
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<tr>
<td>X = SO₂, SO₂, SOS or</td>
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<tr>
<td>S₂ or S₃</td>
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<td>1,1-Dicarboxalkoxydiheptadecyl selenides</td>
<td>X</td>
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<td>1005</td>
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<td>H₂S or NH₂HS and unsat. cyclic ketones and</td>
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<td>PCl₅ or POCl₃</td>
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<td>Thiocether esters</td>
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<td>Dialkylatedcresol sulfides</td>
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<td>Mercaptoacetanilide derivatives</td>
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<td>3-Thienylthialcohol</td>
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Table 1 (cont.)

<table>
<thead>
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<th>Type</th>
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<tbody>
<tr>
<td>(Trifluoromethyl) phenothiazine</td>
<td>X</td>
<td>X</td>
<td>976</td>
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<tr>
<td>3-Isopropanylphenothiazine</td>
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<td>X</td>
<td>976</td>
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<tr>
<td>Benzophenothiazine carbonitrile</td>
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<td>X</td>
<td>976</td>
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<tr>
<td>Bis(4-tert-butyl-m-cresol) sulfide</td>
<td>X</td>
<td>X</td>
<td>639</td>
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<tr>
<td>Mixture of phenyl sulfoxide and Sb$_2$O$_3$</td>
<td>X</td>
<td></td>
<td>11</td>
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<tr>
<td>Metal salts of sulfonated ether of hydrogenated cardanol</td>
<td>X</td>
<td></td>
<td>1118</td>
</tr>
<tr>
<td>β-Alkylmercaptoketones, aldehydes or acids</td>
<td>X</td>
<td>X</td>
<td>1060</td>
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<tr>
<td>Di-$t$-alkylpolysulfides</td>
<td>X</td>
<td></td>
<td>300</td>
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<tr>
<td>React. prod. of bicyclic terpenes, P sulfides and alkylated phenols or alcohols and sulfur chlorides</td>
<td>X</td>
<td></td>
<td>1045</td>
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<tr>
<td>Penicillin</td>
<td>X</td>
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Table 1 (cont.)

<table>
<thead>
<tr>
<th>Type</th>
<th>Antioxidant for</th>
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<th>Ref.</th>
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<tbody>
<tr>
<td>β-Alkyl and arylthio-alkanes</td>
<td>X</td>
<td></td>
<td>1051</td>
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<tr>
<td>Cond. prod. of arylsulfonic halide and alkali metal mercaptide</td>
<td>X</td>
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<td>506</td>
</tr>
<tr>
<td>4,4'-Thiobis(6-t-butyl-m-cresol) (Santowhite)</td>
<td>X</td>
<td></td>
<td>87</td>
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<tr>
<td>(Carboxymethylthio) succinic acid</td>
<td>X</td>
<td></td>
<td>318</td>
</tr>
<tr>
<td>Cond. prod. of aldehyde or ketone with polyamine and P sulfide, S halide or S</td>
<td>X</td>
<td></td>
<td>5</td>
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<tr>
<td>1,2-Bis(ethylsulfinyl) ethane</td>
<td>X X X X X X</td>
<td></td>
<td>1058</td>
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<tr>
<td>Sulfides of 3,6-di-alkyl-subst. phenols</td>
<td>X</td>
<td></td>
<td>90</td>
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<tr>
<td>(Thio-bis(3-methyl-6-t-dodecylphenol)</td>
<td>X</td>
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<td></td>
</tr>
<tr>
<td>β,β'-Thiodipropionic acid</td>
<td>X</td>
<td></td>
<td>1163</td>
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<tr>
<td>2,2'-Diaminodialkyl-sulfides</td>
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<tr>
<td>Type</td>
<td>Antioxidant for</td>
<td>Ref.</td>
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<tr>
<td>--------------------------------------------------</td>
<td>-----------------</td>
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<tr>
<td>5-Nitro-2-thiophene-carboxamides</td>
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<tr>
<td>Diamylmercaptodimethyl ether</td>
<td>X</td>
<td>705</td>
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<tr>
<td>Benzothiazole</td>
<td>X</td>
<td>340</td>
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<tr>
<td>Sulfanilamide</td>
<td>X</td>
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<tr>
<td>Phenoselenazine</td>
<td>X</td>
<td>907</td>
<td></td>
</tr>
<tr>
<td>Phenocellurazine</td>
<td>X</td>
<td>907</td>
<td></td>
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<tr>
<td>Barium salts of sulfur-ized alkylphenols</td>
<td>X</td>
<td>853</td>
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<tr>
<td>Mercaptopyrimidine</td>
<td>X</td>
<td>1011, 1046</td>
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<tr>
<td>Unsat. ethers of cyclic sulfides</td>
<td></td>
<td>591</td>
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<tr>
<td>Zinc isopropyl xanthate</td>
<td>X</td>
<td>668</td>
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**C. Sulfoxide and sulfone type derivatives**

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<tr>
<th>Type</th>
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<tr>
<td>Dodecylselenoxide</td>
<td>X</td>
<td>266</td>
</tr>
<tr>
<td>Dodecylselenone</td>
<td>X</td>
<td>266</td>
</tr>
<tr>
<td>Phenylethyl sulfone</td>
<td>X</td>
<td>329</td>
</tr>
<tr>
<td>Type</td>
<td>Antioxidant for</td>
<td>R</td>
</tr>
<tr>
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<td>----------------</td>
<td>---</td>
</tr>
<tr>
<td>Hydrocarbon-subst.</td>
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<td></td>
</tr>
<tr>
<td>thiacyclopentane-1,1-dioxide</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hydrocarbon-subst.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>thiacyclopentene-1,1-dioxide</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Monochloro sulfones</td>
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<td></td>
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<tr>
<td>(3-chloro-2,4-di methylsulfolene)</td>
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<tr>
<td>Stannous bis(p-hydroxyphenyl) sulfoxide</td>
<td>X</td>
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</tr>
<tr>
<td>Subst. ethers of sulfolanes and sulfolenes</td>
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</tr>
<tr>
<td>Ethyl-2-(ethylthio)-ethylsulfoxide</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Ethyl-2-(ethylthio)-ethylsulfone</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Ethyl-2-(ethylsulfonyl)-ethylsulfoxide</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>(\beta)-Mercaptoketone and unsat. sulfoxide or sulfone = (\beta)-thiaketo-sulfoxides or sulfones</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Type</td>
<td>Antioxidant for</td>
<td>A</td>
</tr>
<tr>
<td>----------------------------------------------------------------------</td>
<td>-----------------</td>
<td>---</td>
</tr>
<tr>
<td>Alkaline earth salts of hydrocarbonsulfonic acid and organophosphoric acid</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Arylaminoaryl sulfonylamides (p-anilinobenzenesulfonylpiperidine)</td>
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</tbody>
</table>

**Phosphorous and sulfur compounds**

A. Thiophosphoric acid type compounds

<p>| Reac. prod. of bicyclicterpenes and P₂S₅ mixed with alkylphenol or alcohol and SOCl₂ and CaCO₃ | X               |   | 815  |
| Monocyclic terpenes and sulfur, then P₄S₃ or P₂S₅ or organic-subst. thiophosphoric acid | X               |   | 246, 541 |
| Tetraphenol ester of thiophosphoric acid                                | X               |   | 561  |
| (RO)(R'O)P(S)SCu                                                       | X               |   | 678  |</p>
<table>
<thead>
<tr>
<th>Type</th>
<th>Antioxidant for</th>
<th>A</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heavy metal salt (barium preferable) of decapryl ester of dithiophosphoric acid</td>
<td>X</td>
<td></td>
<td>229</td>
</tr>
<tr>
<td>Zinc salts of diamyl-dithiophosphate</td>
<td>X</td>
<td></td>
<td>229</td>
</tr>
<tr>
<td>Wax-subst. diaryl dithiophosphoric acids or salts</td>
<td>X</td>
<td></td>
<td>231</td>
</tr>
<tr>
<td>Bis(diabietyl) dithiophosphate</td>
<td>X</td>
<td></td>
<td>230</td>
</tr>
<tr>
<td>Bis(tetraabietyl) dithiophosphate</td>
<td>X</td>
<td></td>
<td>230</td>
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<tr>
<td>React. prod. of dioleyl ketone and $P_2S_5$</td>
<td>X</td>
<td></td>
<td>706</td>
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<tr>
<td>Multivalent metal salts of alkyl-subst. thio-phosphoric acid</td>
<td>X</td>
<td></td>
<td>350, 913</td>
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<tr>
<td>React. prod. of terpene with $P_2S_5$</td>
<td>X</td>
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Ester of organic acid and wax-subst. hydroxyaromatic compd. - 354
Long chain alcohols and long chain fatty acids - 28
<table>
<thead>
<tr>
<th>Type</th>
<th>Antioxidant for</th>
<th>A</th>
<th>Ref.</th>
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<tbody>
<tr>
<td>Reac. prod. of sulfurized oleyl ketone with P₂S₅</td>
<td>X</td>
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<tr>
<td>Complex dithiophosphoric acid esters</td>
<td>X</td>
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<td>232</td>
</tr>
<tr>
<td>Alkylated hydroxyaromatic carboxylic acid and P₂S₅</td>
<td>X</td>
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<td>367</td>
</tr>
<tr>
<td>Barium or tin salt of cardanyl thiophosphate</td>
<td>X</td>
<td></td>
<td>713</td>
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<tr>
<td>Cardanyl ester of thiophosphoric acid</td>
<td>X</td>
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<td>713</td>
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<tr>
<td>P₂S₅ turpentine condensate and alkylphenols</td>
<td>X</td>
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<td>655, 656</td>
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<tr>
<td>S₂SP(1S)(OC₆H₄C₈H₁₇-P)₂/₇₂</td>
<td>X</td>
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<td>989</td>
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<tr>
<td>S₂SP(1S)(OC₆H₁₁Me-4)₂/₇₂</td>
<td>X</td>
<td></td>
<td>989</td>
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<td>Metal salt of reac. prod. of phosphorous sulfide and oxygen-contg. wax</td>
<td>X</td>
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<tr>
<td>Cis-9-octadecen-1-ol and P₂S₅</td>
<td>X</td>
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<td>103</td>
</tr>
<tr>
<td>Type</td>
<td>Antioxidant for</td>
<td>A Ref.</td>
<td></td>
</tr>
<tr>
<td>------</td>
<td>----------------</td>
<td>--------</td>
<td></td>
</tr>
<tr>
<td>Pinene and (P_2S_5)</td>
<td>(X)</td>
<td>Ester of organic acid and wax-subst. hydroxyaromatic compd. - 354</td>
<td></td>
</tr>
<tr>
<td>Diaryl sulfides (Se or Te) of alkyl-subst., diaryl dithiophosphoric acid or salt</td>
<td>(X)</td>
<td>841</td>
<td></td>
</tr>
<tr>
<td>Reac. prod. of oxidized petroleum wax and (P_2S_5)</td>
<td>(X)</td>
<td>365</td>
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<td>Metal salts of reac. prod. of olefin and sulfur halide reacted with aromatic compd. followed by phosphorus and sulfur</td>
<td>(X)</td>
<td>Wool fat and sperm oil - 991</td>
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<tr>
<td>(P_2S_5) or (P_2O_5) and unsat. cyclic ketone</td>
<td>(X)</td>
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<td>Reac. prod. of metal alkylxanthate and metal thiophosphite or thiophosphate compds.</td>
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<td>P sulfide and unsat. (t)-amine</td>
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<td>React. prod. of $P_2S_5$ and sulfurized unsat. acyclic ketone</td>
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<td>Calcium salt of di-octyl-di-octylhydroxyphenylmethylidithiophosphate</td>
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<td>Metal salts of phosphosulfurized cond. prod. of a mercaptan and a carbonyl prod.</td>
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<tr>
<td>$(RO)_2PS_n(SS)_nP(OR)_2$</td>
<td>X</td>
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<tr>
<td>(N = 0 or 1)</td>
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<td>Metal salts of react. prod. of P sulfides and organic amines or esters</td>
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<td>Barium salts of react. prod. of $P_2S_5$ and high molecular weight ketone</td>
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<td>Dialkyldithiophosphoric acid-formaldehyde cond. prods.</td>
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<td>C</td>
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<td>Calcium salts of aromatic methylene derivative of thiophosphoric acid</td>
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<td>Reac. prod. of P_2S_5 and olefin</td>
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<td>P_2S_5 and phenolsulfonic acid</td>
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<td>Reac. prod. of aliphatic nitrile and P sulfide</td>
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<td>Triesters of dithiophosphoric acid</td>
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<td>S-alkoxymethyl-O,0'-dialkylidithiophosphates</td>
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<td>S-aminocalkylidithiophosphoric acid triesters</td>
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<tr>
<td>( (RO)_2PS _3P )</td>
<td>X</td>
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<td>X</td>
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<tr>
<td>( (RO)_2PS _2P )</td>
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<td>P₂S₅ and alkenylphosphate, thiophosphate or phosphate esters</td>
<td>X</td>
<td>771</td>
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<td>S-hydroxybensyl-0,0-dialkylthiophosphoric acid triesters</td>
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<td>Ether alcohol of cellulosic type and P₂S₅ and HNO₂ = organodithiophosphoric acid disulfide</td>
<td>X</td>
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<td>P sulfide and hydrocarbon and zinc dithiocarbamate or guanidine carbonate</td>
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<td>Esters of dithiophosphoric acid contg. terpene radicals</td>
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<td>P₂S₅ and ester of unsat. polyhydric alcohols</td>
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<td>Disulfide derivatives of organo-subst. thio phosphoric acids ( \left(\text{ROOC}_n\text{RO}_2\right)_2\text{P}:\text{S} )_₂</td>
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<td>Thialdine salt of organic thiophosphoric acids</td>
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<td>Metal salt of P₂S₅ or P₄S₇ reacting with sat. aliphatic alcohol and sulfurized unsat. aliphatic alcohol</td>
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<td>P sulfide and a polymer of an ester of α,β-unsat. dicarboxylic acid or a co-polymer of the ester with vinyl ester or vinyl aromatic hydrocarbon</td>
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<td>S-(sulfurized terpene) dithiophosphoric acid triesters</td>
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<td>Bicyclic terpene and P₂S₅ and S</td>
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**Phosphorus compounds**

**A. Phosphite esters**

<p>| Cetyltolylphosphite | X | 408 |</p>
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<tr>
<td>Tri-trialkanolamine phosphite</td>
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<td>Tri-triethanolamine phosphite</td>
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<td>Triethanoldiethylamine phosphite</td>
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<tr>
<td>Ditolyl monetriethanolamine phosphite</td>
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<td>271</td>
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<tr>
<td>Reac, prod. of hydroxyester or acid with PCl₃</td>
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<td>Thiodiglycol phosphite</td>
<td>X</td>
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<td>RO(P)OR’(OR”)</td>
<td>X</td>
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<td>Triphenyl phosphite</td>
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<td>473, 639, 888</td>
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<td>Tri-2-tolyl phosphite</td>
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<td>Tri-α-naphthyl phosphite</td>
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<td>473</td>
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<td>Tri-α-phenoxynaphyl phosphite</td>
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<td>Monododecyl phosphite</td>
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<tr>
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<td>Monobutylmonooctylphosphate</td>
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<td>Res. prod. of dialkylphosphites and quinones</td>
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<td>Mixed anhydrides of organo-subst. phosphorus acids and carboxylic acids</td>
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<td>FCl₃ and cyclohexanols</td>
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<td>Fluoroalkanephosphonates</td>
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<td>Mono or dialkyl esters of phosphonic acid or polymers</td>
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<td>Res. prod. of polyhalogenated quinones and trialkyl phosphites</td>
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<td>FCl₃ + CH₂CHOHCO₂H or its esters or salts</td>
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<td>Benzenephosphonous acid</td>
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<td>Aryl polyphosphites</td>
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<td>Type</td>
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<tr>
<td>Organic phosphites and halogen compds.</td>
<td>X</td>
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<tr>
<td>HCHO and heterocyclic amine and hydroxyaromatic and FC13 (wax hydroxybenzylmorpohline and FC13)</td>
<td>X X</td>
<td>768</td>
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B. Phosphate esters

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<td>Chlororesorcinol phosphate</td>
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<td>Biguanide-p-t-amyl-phenyl phosphate</td>
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<td>Sodium-p-t-amyl phosphate</td>
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<tr>
<td>(C₄H₉)₃PO₄</td>
<td>X</td>
<td>340</td>
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<td>Monophenyl phosphate</td>
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<td>Fructose-6 phosphate</td>
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<td>765</td>
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<td>Organic seleno phosphate (triaryl)seleno phosphate)</td>
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<td>Didodecyl phosphate</td>
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<td>Neutral unsat. primary aliphatic amine salt of 3-methylbutyl-2-ethylhexyl-2-phosphoric acid</td>
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<td>Monobutylmonodecyl phosphate</td>
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<td>&quot;Lorol&quot; phosphate</td>
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<td>Phytic acid</td>
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<td>C. Phosphatides</td>
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<td>Lecithin ((C_{42}H_{84}PO_{4}N))</td>
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<td>Phospholipids</td>
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<td>Cephein</td>
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**Table 1 (cont.)**

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Ascorbic monoesters of fatty acids - 861

730, 809
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<th>S</th>
<th>M</th>
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<td>Prod. of cephalin and sulfur</td>
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<td>Lecithin and conc. ammonia or corn oil phosphatide and conc. ammonia</td>
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<td>Phosphatides of soybean, corn and peanut oils</td>
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<td>Phosphatidic acids from phosphatidyl cholines</td>
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- Sucrose - 43
- Quinone
- Tocopherol and hemoglobin, versene - 1130
- Zinc chloride - 71
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| Phenylthioglycol-
  -carboxylic acid | | X | | 1014 |
| Maleic acid | X | X | 383, 437 | 529 |
| Fumaric acid | X | X | | 437, 529 |
| Citric acid and Na
  salt | X | X | X | X | Gum guaiac - 817 | 296, 297 | 555, 556 | 587, 604 | 606, 683 | 723, 730 | 756, 1069 | 1129, 1163 |
| **Alkylcarboxylate (or
dithiocarboxyl or
ether or a keto group
or sulfur, selenium
or tellurium analogue
of either) subst. in
\(\alpha, \beta, \gamma\) alkyl position by Group Vb | | X | | | 322 |
<p>| Vanadyl oleyl phthalate | X | | | | 348 |</p>
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<tr>
<th>Type</th>
<th>Antioxidant for</th>
<th>R</th>
<th>G</th>
<th>C</th>
<th>L</th>
<th>P</th>
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<th>F</th>
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<td>Unsat. aldehydes with 5 conjugated double bonds (11-phenylhendeca-pentaenal, lactaroviolin, lycopersical)</td>
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<td>Cond. prod. of aliphatic nitrile with aromatic amine, phenol or hydrocarbon</td>
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Table 1 (cont.)

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<td>Orange juice, lemon juice, sea buckthorn berry juice, sugar-contg. marmalades</td>
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<td>Di- or tricarboxylic acids – 215</td>
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<td>Ginger starch</td>
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<td>Sesame oil</td>
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<td>Cocomnut oil</td>
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<td>Neomycin</td>
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<td>Cream, skim milk, fermented skim milk</td>
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</tr>
<tr>
<td>Sulphydryl substance in fruit</td>
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<td>Sodium alginate</td>
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<td>Amines and phenols from branch chained olefin and p-cyclic disubst. benzene hydrocarbon (1-p-tolyl-1-(2-methyl-5-cyclohexyl-phenyl) cyclohexane)</td>
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<td>Sustane 3F</td>
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<tr>
<td>2-Furfurylidene malononitrile</td>
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Separation and Identification of Products from the Air Oxidation Experiments

Materials and experimental procedure

Preparation of materials. Tetralin (Kodak practical grade) was washed with concentrated sulfuric acid until the acid layer showed no more red coloration, then washed with water, dried and distilled under 15 to 30 mm. pressure at 91°C.

Diphenylamine (Matheson Co., Inc.) was recrystallized five or six times from Skelly D. M.p. was 54-55°C.

Azo-bis-isobutyronitrile was prepared by the method of Thiele and Heuser, as modified by Dox. The m.p. was 105-106°C.

Chlorobenzene was redistilled, b.p. 130-131°C.

Methacrylonitrile was washed with alkali, dried and distilled.

$\text{p,p}'$-Dihydroxyasobenzene, m.p. 215-216°C, was prepared by the method of Atkinson, et al.

Preparation of phenazine and 9,10-dihydrophenazaine was accomplished by the method of Rie. The 9,10-dihydrophenazaine was recrystallized from chlorobenzene after decolorizing the impure material with carbon black. The m.p. was 305-306°C in a sealed tube under nitrogen, and 270°C was obtained in an open tube. Literature reports 212°C as the melting point. Some of the material was heated in air and a yellow material sublimed which melted at 169°C. Phenazine melts at 169-172°C according to the literature.

$\text{N,N}'$-diphenyl-$p$-phenylenediamine obtained from B.F. Goodrich was purified by five recrystallizations from chlorobenzene after the carbon
black was filtered off. M.p. was 147-149°.

$N,N'$-diphenylquinonediimine (benzoquinone dianil) was prepared by the method of Piccard from $N,N'$-diphenyl-$p$-phenylenediamine by chromic acid oxidation. The product melted at 183.5° (literature 176-180°) after recrystallization from chlorobenzene.

Quinhydrone base from $N,N'$-diphenyl-$p$-phenylenediamine was prepared by the method of Piccard. A light chocolate brown powder, m.p. 123-127° (literature 130-135°), was obtained.

Quinoneazine was prepared by the method of Willstatter and Benz. The crystals were orange-red. On being heated, the crystals darkened at 157-159° and on further heating turned red again at 200-203°. The crystals did not melt when heated to 300°. A flame test gave a sudden, rapid darkening and a burst of flame, then slow oxidation of the residue until it was all gone. On mixing a small amount of material in methylene chloride solution with $p,p'$-dihydroxyazobenzene, the quinhydrone-type material was obtained giving crystals which melted at 205° (pure material m.p. 185°). Since the material had the dark appearance of the complex and no careful preparation of a 1:1 ratio had been made, it was concluded that the quinoneazine had been prepared. (Other authors give the quinoneazine m.p. as 158° (see Hunter and Barnes 117), while Bielstein states that there are two crystalline forms, one dark red prisms, the other dark yellow needles. The latter mentions a darkening on heating and explosion at 158°.)

Preparation of phenylazotriphenylmethane was carried out by the method of Gomberg and Berger. In the first preparation the hydrazo compound did not dissolve readily in alcohol as the literature indicates, so methylene chloride was used to dissolve it. On cooling, beautiful yellow crystals
precipitated which had a m.p. of 114-116°. The literature warns that considerable contamination with the hydrazo compound had little effect on the m.p. as was later discovered in using this material. The yield was 55.72 g. The oxidation with N₂O₃ as Gomberg and Berger recommend was thus not performed.

A second preparation was performed using the same method to prepare the hydrazo compound. The oxidation which followed was according to the method of Cohen and Wang²¹ using hydrogen peroxide and bicarbonate solution to obtain the azo compound. The ether solution of the azo compound was separated, and the ether evaporated and a mixture of alcohol and methylene chloride was used as a recrystallization solvent. The crystals melted at 116-118°. It was noted that some were yellow and some tended to be white and that the yellow crystals melted first. Another recrystallization gave large clear yellow crystals and also small lighter colored ones, m.p. 115-118°. The yield was 59.97 g. or 48 percent. This material proved to be contaminated with hydrazo compound, as was the first, even though the reaction was carried on overnight, instead of five hours as recommended. The hydrogen peroxide may have deteriorated. Further purification was later commenced.

An attempt was made to use the method of Gomberg and Berger³⁸ to prepare p-nitrophenylazotriphenylmethane from tritylchloride and p-nitrophenylhydrazine. The material which precipitated was not the hydrochloride of the hydrazine as anticipated, but was not very soluble in water, gave no precipitate with silver nitrate and melted at 175-180°. Two other attempts were made but met with no success.
Apparatus and procedures. A three-necked standard taper flask was equipped with a stopper, a tube with small holes on the end through which air could bubble, and a Friedrick condenser. Suction was applied to the top of the condenser causing air to bubble from the gas dispersing tube through the solution in the flask and out of the condenser. A drying tube was placed on the end of the air inlet bubbler tube to keep moisture out of the system. The three-necked flask was placed in a constant temperature bath and after the reaction mixture was homogeneous, the suction applied so that air bubbled slowly up through the solution.

Peroxide determinations were by the method of Hammond.\(^419\)

**Attempted separation and identification of products from air oxidation using diphenylamine as inhibitor**

Air oxidations with tetralin as substrate. A mixture of 200 g. of tetralin (1.51 moles), 16.7 g. of diphenylamine (0.0987 mole) and 15 g. (0.0987 mole) of ase-bis-isobutyronitrile was placed in a 500 ml. round bottom flask and placed in the constant temperature bath as described above. The bath was maintained at approximately 75°C. The reaction mixture turned brown within an hour and was black within four hours of reaction time. A great deal of work and time was spent in an effort to separate and identify the reaction products. The original reaction mixture was extract-ed with 2N sodium hydroxide, then with hydrochloric acid. Then followed various methods of separation. Steam distillation, distillation under reduced pressure, recrystallization, sublimation and chromatography were employed. Infrared spectra were used in an effort to establish something of the identity of many of the chromatographic fractions. Most of the
compounds were oily materials and no clean-cut separation was achieved by chromatography or the other methods, with the exception of recovering the starting materials, tetralin and diphenylamine, and the dimer, tetramethylsuccinonitrile, from the decomposition of the initiator. Work involving isolation of products from air oxidations with diphenylamine as an inhibitor was later abandoned in favor of inhibitors which exhibit the property of reacting with two peroxy radicals per molecule. From kinetic studies\(^{136}\) it appears that many inhibitors exhibit small integral numbers (often two) as stoichiometric factors of peroxy radicals reacting with inhibitors. However, diphenylamine and its methoxy derivative prove to be anomalous in this respect showing a nonintegral stoichiometric factor, indicating that at least two reaction paths are probably being followed simultaneously. This being the case, inhibitors were subsequently chosen which gave the usual stoichiometric factor of two for the studies performed on reaction products of inhibited air oxidized systems.

A second oxidation with tetralin as substrate follows. A mixture of 100 g. (.755 mole) of tetralin, 8.35 g. (.04935 mole) of diphenylamine and 7.5 g. (.04935 mole) of azo-bis-isobutyronitrile was placed in a 500 ml. round bottom three neck flask and after the mixture was homogeneous the flask was placed in the water bath which was held at 62.2°. The experimental set up had been modified from the previous run in order to try to follow the uptake of oxygen. A stopper was placed in the top of the Friedrich condenser, a stirrer was placed in the middle neck and an oxygen delivery tube in the third neck. The oxygen delivery tube was connected to a tube reaching to the top of an inverted two liter graduate cylinder which was filled with oxygen. A slight pressure was maintained on the oxygen in
the cylinder by raising the leveling bulb, containing mineral oil, a small amount above the level in the cylinder. The whole system was previously flushed with oxygen before the reaction was started. The effort to determine oxygen uptake was not successful because of a leak around the stirrer. The accuracy of the method is also limited by the necessity of calculating the amount of nitrogen evolved during the run. This is almost equal to the oxygen absorbed, as later discovered. After five days, another 7.5 g (0.04935 mole) of azo-bis-isobutyronitrile was added. The reaction was allowed to proceed for 24 days. The tetralin and tetramethylsuccinonitrile were removed from the reaction mixture by distillation under reduced pressure of 1 to 5 mm, through a four foot Oldershaw column. Because of the difficulty previously encountered in separating tetralin and tetramethylsuccinonitrile, it was decided to estimate the amount of the dinitrile in the tetralin by making standard solutions of a known amount of the solid material dissolved in a known volume of tetralin. This was done, but the infrared spectrum of the solutions did not lend itself to any quantitative results. The refractive index was also disappointing. However, it was possible to obtain a good estimate of the amount of the dinitrile present in tetralin saturated at 25° by placing the volumetric flasks in a 25° water bath overnight and weighing the excess crystals.

Since only a very small crystal was obtained from Sample #2, this was taken as being most accurate since there would be less tetralin adsorbed on this crystal than on the others.
Table 2

Ten ml. of tetralin and tetramethyloxycinnonitrile mixture at 25°

<table>
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<tr>
<th>Sample #</th>
<th>Beginning wt. of dinitrile</th>
<th>Wt. of dinitrile recovered</th>
<th>Wt. of dinitrile dissolved</th>
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<tbody>
<tr>
<td>1</td>
<td>0.9998 g.</td>
<td>0.5241 g.</td>
<td>0.4787 g.</td>
</tr>
<tr>
<td>2</td>
<td>0.5006 g.</td>
<td>0.00017 g.</td>
<td>0.5004 g.</td>
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<td>3</td>
<td>0.2493 g.</td>
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<tr>
<td>4</td>
<td>0.1251 g.</td>
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</table>

By this means the total amount of dinitrile accounted for in saturated tetralin solution and from excess crystals amounted to 5.66 g. (0.0435 mole) which is 44.1 percent of the total charge of the initiator azo-bis-isobutyronitrile. This is in substantial agreement with the findings of Hammond, Sen and Booser concerning the efficiency of radicals produced which initiate chains instead of dimerizing.

The residue from the distillation under reduced pressure was titrated by the method of Hammond giving 0.0199 milliequivalents of peroxide per ml. With approximately 78 ml. of solution this gives a total of 0.78 millimoles of peroxide as compared to 108 millimoles of hydroperoxide theoretically possible, assuming 44 percent dimerization of the initiator radicals. This low result raised the possibility that the peroxide was so unstable it was being destroyed during removal of the solvent.

After the peroxide determination, the residue was dissolved in Skelly B and adsorbed on a chromatographic column of Al₂O₃. Numerous fractions were eluted but no products were isolated and identified other than diphenylamine. Infrared spectra were taken in numerous cases as an aid in
trying to differentiate and identify components in the varicolored oily fractions. The work on diphenylamine products was subsequently abandoned for the reasons given in the early part of this section.

Air oxidations with chlorobenzene as solvent. A mixture was made of 84.98 g. (.755 mole) (76.8 ml.) of chlorobenzene, 8.35 g. (.04935 mole) of diphenylamine and 7.5 g. (.04935 mole) of azo-bis-isobutyronitrile. The homogeneous mixture was placed in the water bath which was at 62.5°. Oxygen under slight pressure was placed over the stirred reaction mixture as in the previous run. After 128 hours (approximately 8 half lives of the initiator) the spent gases, mostly nitrogen, from the initiator were flushed out of the system and new oxygen was added along with another charge of 7.5 g. (.04935 mole) of azo-bis-isobutyronitrile.

A one ml. sample of the reaction mixture was titrated for peroxide by the method of Hammond giving .02985 milliequivalents of peroxide per ml. of reaction mixture.

After a total time of 284 hours (156 hours and 9.8 half lives since the last charge of initiator) the run was stopped. Since the odor of hydrogen cyanide had been detected previously, some of the gas above the reaction mixture was passed through slightly acidified silver nitrate solution. A copious precipitate of silver cyanide was obtained.

Four one ml. samples were titrated for peroxide. The concentration was not over .0542 milliequivalents per ml. of reaction mixture. Assuming no loss of solvent (76.8 ml.), this corresponds to a total of 4 milliequivalents or 2 millimoles of hydroperoxide in the solution, compared to a total of approximately 108 millimoles of hydroperoxide theoretically possible,
assuming 44 percent of the initiator radicals formed dimers as shown in the previous experiment. This indicated that the low peroxide content was not primarily a result of separation procedures. After considerable effort to isolate products, the work on diphenylamine products was ceased for the reasons previously given.

**Air oxidation of azo-bis-isobutyronitrile**

A mixture of 15 g. (.0987 mole) of azo-bis-isobutyronitrile and 85 g. (.755 mole) (76.8 ml.) of chlorobenzene was placed in a three-necked round bottom flask, which was, upon dissolving the initiator, immersed in a water bath at 62.2°. Oxygen under slight pressure was maintained over the stirred reaction mixture as in the previous experiment. The increase in the peroxide content was followed at intervals during the run (144.5 hours). It was noted that the determination of peroxide content gave results approximately twice as great when hydrogen chloride was used along with acetic acid to catalyse the formation of iodine from iodide ion as when no strong acid was used. This is assumed to be a possible indication of the presence of peroxide and a more unstable hydroperoxide. The final reading with hydrogen chloride catalyst was .0873 milliequivalents of peroxide per ml. or 6.74 total milliequivalents based on starting volume assuming no change in concentration during the run (only 3.8 ml. of solvent were unaccounted for after the solvent was removed under vacuum). Taking into account the efficiency of initiation and assuming all that was formed was hydroperoxide, only 2.62 percent of the material formed stable hydroperoxide. Assuming all the material formed was peroxide, the percentage would be 5.24. At the end of the run the gas above the reaction mixture was drawn into
aqueous silver nitrate, from which silver cyanide precipitated copiously. Attempts to isolate other products by fractional recrystallizations and solvent extractions using petroleum ether, methylene chloride, alcohol and various solvent mixtures led to the isolation of only tetramethylsuccinonitrile and a white amorphous solid. The latter showed strong infrared absorption at 2240, 1740 and 1680 cm\(^{-1}\) and appeared to have a spectrum similar to polymethaerylonitrile.

**Air oxidation of methacrylonitrile**

A mixture of 33.55 g. (.5 mole) of methacrylonitrile, 423 g. of chlorobenzene and 15 g. (.0987 mole) of azo-bis-isobutyronitrile was placed in a one liter, three-necked round bottom flask. After dissolution of the initiator, the flask was placed in a water bath at 62.2\(^\circ\). Oxygen was slowly bubbled through the solution. After 74 hours the oxygen was turned off. During the run the concentration of hydroperoxide steadily increased to about .217 milliequivalents per ml. Since about 400 ml. of solvent was recovered on distillation under reduced pressure, the total concentration of hydroperoxide was 46.8 millimoles. This concentration fell to .169 milliequivalents per ml. as the reaction mixture was allowed to stand at room temperature for a week before distillation. After distillation at 41\(^\circ\) and 45 mm. a viscous orange residue remained which weighed 34.9 g. Hydrogen cyanide was again detected in the gases above the reaction mixture.
Separation and identification of products from air oxidations with \( \text{N,N}'-\text{diphenyl-p-phenylenediamine as inhibitor} \)

**Isolation of \( \text{N,N}'-\text{diphenylquinonedimine} \).** A mixture of 3.75 g. (.02467 mole) of \( \text{azo-bis-isobutyronitrile} \), 2.485 g. (.00957 mole) of \( \text{N,N}'-\text{diphenyl-p-phenylenediamine} \) and 200 ml. of chlorobenzene in a round bottom three-necked flask equipped as in the first oxidation run was placed in a water bath maintained at 62.2\(^\circ\). The light yellow solution turned brown in five minutes and was red at the end of the run. Oxygen was bubbled through the solution for 8 half lives of the initiator (96 hours). Cooling overnight in the refrigerator produced no solid crystals so the chlorobenzene was removed from the reaction mixture by distillation at 50 mm. and 40\(^\circ\). A small amount of polymeric solid (polymethacrylonitrile) was observed on the side of the reaction vessel.

After the distillation the solid material in the residue was filtered off and the remaining chlorobenzene removed by evaporation with a stream of air. The solid material which was filtered off was extracted with one part methylene chloride and 40 parts Skelly D to remove the dinitrile. After the extraction, the remaining solid was recrystallized from methylene chloride. A fraction weighing 0.35 g. gave a m.p. of 183-185\(^\circ\) and a mixed melting point with previously prepared \( \text{N,N}'-\text{diphenylquinonedimine} \) (m.p. 183.5) gave a melting point of 184.5-188\(^\circ\). Other fractions, not quite so pure, brought the total weight to 1.14 g., a yield of approximately 46 percent. The product, which one could observe in other fractions along with a dark viscous material, was never separated, but seemed to decompose on repeated attempts at purification. This may not be surprising since
Tulagin \(^{1078}\) remarks that quinonimine dyes are unstable to light, acids and alkalis. The spectra of two of the worst fractions were taken in the ultraviolet and visible regions with a Beckman Model DU spectrophotometer. One gave a minimum at 265 m\(_\mu\) and a maximum at 293 m\(_\mu\), the other gave a minimum at 250 m\(_\mu\) and a maximum at 295 m\(_\mu\). These have little resemblance to the spectra of \(N,N'\)-diphenyl-\(p\)-phenylenediamine. Another spectrum was taken of a grey amorphous product which precipitated from all the fractions with addition of Skelly B to the methylene chloride solutions. Minima at 300 and 340 m\(_\mu\) and maxima at 305 and 350 m\(_\mu\) were obtained.

**Spectra of \(N,N'\)-diphenyl-\(p\)-phenylenediamine and its derivatives in methanol.** The following solutions were made:

- 0.000030 g./ml. of \(N,N'\)-diphenyl-\(p\)-phenylenediamine,
- 0.00002436 g./ml. of quinhydrone,
- 0.0000180 g./ml. of \(N,N'\)-diphenyl-\(p\)-phenylenediamine.

The spectra in the ultraviolet and visible were observed with a Beckman Model DU spectrophotometer. Sharp maxima were observed at 290 and 310 m\(_\mu\) for \(N,N'\)-diphenyl-\(p\)-phenylenediamine (see Fig. 1). Minima were at 295 and 380 m\(_\mu\). For the quinhydrone (Fig. 2) there was a sharp maximum at 300 m\(_\mu\) and a low broad one at 440 m\(_\mu\). A minimum was at 370 m\(_\mu\). Since the first peak is nearly in the same position as for the pure amine, the optical density of this peak gives the total concentration of the quinhydrone base while the optical density at 440 m\(_\mu\), which is due to dissociated \(N,N'\)-diphenyl-\(p\)-phenylenediamine (Fig. 3), is not as large as it should be (see Table 3), indicating that the quinhydrone is not completely dissociated in the solution. The maxima for the \(N,N'\)-diphenyl-\(p\)-phenylenediamine was at
Fig. 1 Ultraviolet and visible spectrum of \( N,N' \)-diphenyl-\( p \)-phenylenediamine in methanol

Fig. 2 Ultraviolet and visible spectrum of the quinhydrone of \( N,N' \)-diphenyl-\( p \)-phenylenediamine in methanol
N, N'-diphenyl-β-phenylenediamine
3 X 10⁻⁵ g/ml. in methanol

Quinhydrone of N, N'-diphenyl-β-phenylenediamine
2.436 X 10⁻⁵ g/ml. in methanol
Fig. 3 Ultraviolet and visible spectrum of N,N'-diphenyl-π-phenylene-
dimine in methanol

Fig. 4 Ultraviolet and visible spectrum of oxidized N,N'-diphenyl-π-
phenylenediamine in chlorobenzene and tetralin solution
N, N'-diphenyl-\(\beta\)-phenylenediamine
1.8 \times 10^{-5} \text{ g/ml. of methanol}

oxidized N, N'-diphenyl-\(\beta\)-phenylenediamine
5.2 \times 10^{-5} \text{ g/ml. in 4:1 chlorobenzene and tetralin solution}
Table 3

Optical density from the spectrum of a known quantity of \(N,N'\)-diphenyl-\(p\)-phenylenediamine and quinhydrone in methanol

<table>
<thead>
<tr>
<th></th>
<th>Optical density</th>
<th>Concentration Actual</th>
<th>Calculated</th>
</tr>
</thead>
<tbody>
<tr>
<td>(N,N')-diphenyl-(p)-phenylenediamine</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>First peak (300 m(\mu))</td>
<td>1.66</td>
<td>1.8 (\times) (10^{-5}) g./ml.</td>
<td>----</td>
</tr>
<tr>
<td>Second peak (440 m(\mu))</td>
<td>.51</td>
<td>1.8 (\times) (10^{-5}) g./ml.</td>
<td>----</td>
</tr>
<tr>
<td>Quinhydrone</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>First peak (300 m(\mu))</td>
<td>2.33</td>
<td>2.436 (\times) (10^{-5}) g./ml.</td>
<td>2.525 (\times) (10^{-5}) g./ml.</td>
</tr>
<tr>
<td>Second peak (440 m(\mu))</td>
<td>.27</td>
<td>1.218 (\times) (10^{-5}) g./ml.</td>
<td>.952 (\times) (10^{-5}) g./ml.</td>
</tr>
</tbody>
</table>

305 m\(\mu\) and 440 m\(\mu\), the latter a broad flat maximum (see Fig. 3). The minimum is at 360 m\(\mu\).

Oxidation of \(N,N'\)-diphenyl-\(p\)-phenylenediamine in the kinetics apparatus and comparison of the spectrum with that of \(N,N'\)-diphenyl-\(p\)-phenylenediamine. A mixture of two ml. of chlorobenzene, one ml. of tetralin, two ml. of azo-bis-isobutyronitrile solution (500 g./10 ml. or 100 mg. of initiator) and two ml. of the inhibitor (.13 g./10 ml. or .0026 g. (10\(^{-5}\) moles) total inhibitor) \(\sqrt{MV} = 260.33\) g\(\text{ml}\) was placed in the reaction vessel. The rate was observed until the inhibition period was over and then the solution was taken from the reaction vessel. The reaction mixture was diluted by ten times with a 4:1 mixture of chlorobenzene and tetralin, making a solution of 2 \(\times\) \(10^{-7}\) moles per ml. = .000052 g. per ml. When comparing the spectrum of \(N,N'\)-diphenyl-\(p\)-phenylenediamine in
methanol (Fig. 3) and that of the oxidation products in tetralin and chloro-
benzene (Fig. 4), optical density reading on the first peak on the lower
wave lengths does not give reliable results in determining the concentra-
tions of solute in solution since the maximum shifts from 300 mµ to 325 mµ.
The lower, broader peak at 440 mµ gives more reliable results (see Table
3). Comparing the spectra of N,N'-diphenyl-p-phenylenediamine (Fig. 3)
and its quinhydrone base (Fig. 2) which was obtained in the same solvent
(methanol), results are more consistent (see Table 3). Since N,N'-di-
phenyl-p-phenylenediamine (Fig. 3) and N,N'-diphenyl-p-phenylenediamine
(Fig. 1) both have maxima at 300 mµ, if the quinhydrone were completely
dissociated the concentration for the second peak (440 mµ), which does
not appear in the spectra of the unoxidized amine (Fig. 1), should be half
that obtained at the first peak (300 mµ). The first peak gives concen-
trations very close to that of the quinhydrone base actually present (Fig.
2), while the results on the lower peak are 22 percent lower than the con-
centration of the N,N'-diphenyl-p-phenylenediamine present in the quinhy-
drone (compare Fig. 2 with Fig. 3), indicating that part of the oxidized
form is complexed as the quinhydrone even at these dilutions (see Table
3). These results are consistent with what one might expect.

The calculations in Table 4 were obtained by using the optical density
obtained for the quinoneimine in methanol (Fig. 3) to calculate the concen-
trations of this substance formed by air oxidation of N,N'-diphenyl-p-
phenylenediamine in the presence of initiator radicals (Fig. 4). As men-
tioned previously, the shifted peak does not give reliable results. How-
ever, the second peak gives results very close to the theoretical amount
possible if only quinoneimine were formed in the reaction.
Table 4

\( N,N' \)-diphenyl-p-phenylenediamine oxidized in tetralin and chlorobenzene

<table>
<thead>
<tr>
<th>Concentration</th>
<th>Optical density</th>
<th>Actual if 100% quinoneimine formed</th>
<th>Calculated g./ml.</th>
</tr>
</thead>
<tbody>
<tr>
<td>First peak (325 ( \mu ))</td>
<td>2.65</td>
<td>( 5.2 \times 10^{-5} ) g./ml.</td>
<td>( 2.855 \times 10^{-5} ) g./ml.</td>
</tr>
<tr>
<td>Second peak (440 ( \mu ))</td>
<td>1.41</td>
<td>( 5.2 \times 10^{-5} ) g./ml.</td>
<td>( 4.97 \times 10^{-5} ) g./ml.</td>
</tr>
</tbody>
</table>

Separation and identification of products from the air oxidations of \( p,p' \)-dihydroxyazobenzene

First oxidation run. A mixture of 2.05 g. (0.00957 mole) of \( p,p' \)-dihydroxyazobenzene, 3.75 g. (0.02467 mole) of azo-bis-isobutyronitrile and 200 ml. of chlorobenzene were placed in a round bottom, three-necked flask equipped as described in the section on apparatus and procedures. The material did not dissolve at room temperature so it was placed in the water bath at 62.2°. After dissolution had appeared to take place, air was slowly bubbled through the solution. The color changed a light brown to a dark brown color. This color remained until the mixture was taken from the bath after 8 half lives for the initiator had passed (96 hours). The reaction mixture was decanted from the flask. About one g. of a dark residue was left in the flask. The dark colored solution was refrigerated overnight and filtered the next morning. A red-brown solid and a red solution were obtained. After removing most of the chlorobenzene by distillation at 50 mm. and 40°, the remainder was removed by evaporation. A dark colored solid material weighing 4.85 g. was recovered. The dark material,
which was obtained by decanting the original reaction mixture, proved to be interesting. The small, dark lumps proved to have a light brown center. The melting point of the dark outer material was 185-190°. The quinhydronazine melts at 184°. Inside the larger particles were found light brown, unreacted \( \text{p,p}'\text{-dihydroxyazobenzene} \) as indicated by the m.p. of 212-220°. The brown material changes to bright red on heating, as does the original starting material. This would indicate that among the products formed was the quinoneazine which formed a molecular complex with the unreacted starting material.

The solid material filtered from the cooled reaction mixture and the residue after removal of chlorobenzene both gave a white solid covered with small spherical deposits of orange-red oil on extraction with Skelly A and methylene chloride. This material was assumed to be tetramethylsuccinonitrile, but was never purified and identified. The residue from one (the material filtered from the original reaction mixture and then extracted with solvent) was a brown solid with some orange material. The residue from the other (the material from the chlorobenzene solution) was dark orange or reddish-brown oil. Numerous solvent extractions and recrystallizations were attempted, but no other product was isolated and identified.

**Second oxidation run.** A sample of finely divided \( \text{p,p}'\text{-dihydroxyazobenzene} \) was weighed out 1.538 g. (0.00718 mole) and placed in 200 ml. of chlorobenzene which was heated to boiling to try to dissolve the material. Not all of the material dissolved, but the residue appeared to be a fine powder so the run was continued. After the temperature had dropped to 50°, 2.813 g. (0.0185 mole) of \( \text{azo-bis-isobutyronitrile} \) was added. The three-necked flask with the mixture was placed in the constant temperature bath
at 62.2°. Air was to be bubbled through as in the first oxidation. By mistake, suction was applied to the bubbler and part of the solution was lost. The run was continued, however, even when it was noted that some water had collected in the flask from the condenser due to the high humidity. At the end of 100 hours the run was stopped. There was quite a bit of solid material in the flask. This was filtered off and the solution was refrigerated. The solid material weighed 1.03 g. and consisted of a mixture of black and brown particles. Some of the black particles were picked out and were found to have a small amount of brown material in the center as in the previous run. The m.p. of the black material was 185-187°, which corresponds to the m.p. of the quinhydroneazine. The brown material of the mixture was not unreacted starting material, but behaved on the melting point block as did the pure quinoneazine. No melting point was observed even on heating to 310°. Assuming the solid to be mostly reaction products from the inhibitor, the amount unaccounted for is only .508 g. or .00217 mole. Part of this probably was lost at the beginning of the run.

The reaction mixture filtrate volume was 139 ml. By freezing in an acetone-dry ice mixture, a small amount of material was obtained which gave a peroxide test. It was a light yellow oil which turned red on standing in air. The filtrate was refrigerated for three weeks and then titrated for peroxide. A value of .00193 mole was obtained for the solution. By coincidence, this is close to the value of unaccounted-for inhibitor. However, this probably included peroxides from the initiator as well. The filtrate was extracted with 25 percent aqueous sodium hydroxide. After neutralization, neither fraction gave a test for peroxide. No further work was done with this mixture.
Spectrum of \( p,p' \)-dihydroxyazobenzene in methanol. The solution was 0.0000204 g. per ml. of \( p,p' \)-dihydroxyazobenzene. The spectrum (see Fig. 5) in the ultraviolet and visible was followed on a Beckman Model DU spectrophotometer. Sharp maxima were obtained at 246 m\( \mu \) and 360 m\( \mu \). A minimum was observed at 270 m\( \mu \).

Spectrum of \( N,N' \)-diphenylquinoneazine in tetralin and chlorobenzene. The solution was \( 2.118 \times 10^{-5} \) g. per ml. of quinoneazine in four parts of chlorobenzene and one part tetralin. Maxima were at 335 m\( \mu \) and 380 m\( \mu \) and a minimum was observed at 370 m\( \mu \) (see Fig. 6).

Oxidation of \( p,p' \)-dihydroxyazobenzene in the kinetics apparatus and the spectrum of the reaction mixture compared to that of \( N,N' \)-diphenylquinoneazine. A mixture of two ml. of chlorobenzene, one ml. of tetralin, two ml. of azo-bis-isobutyronitrile solution (0.5004 g./10 ml. or 100 mg. total) and two ml. of inhibitor slurry (0.107 g./10 ml. or \( 10^{-5} \) moles total inhibitor \( \text{MW} 214.22 \) g.) was placed in the reaction vessel. The rate was observed until the inhibition period was over, then the solution was taken from the reaction vessel. The mixture was diluted to ten times the volume by a mixture in the ratio of four ml. of chlorobenzene to one ml. of tetralin. The concentration of the oxidized inhibitor thus became \( 2 \times 10^{-7} \) moles = .0000428 g. per ml. The ultraviolet and visible spectra were obtained (see Fig. 7). They corresponded to the spectrum of the known quinoneazine (Fig. 6), except there was a slight shift of the maxima to 345 m\( \mu \) and 390 m\( \mu \) with a minimum at 385 m\( \mu \). The conversion of \( p,p' \)-dihydroxyazobenzene to quinoneazine occurs in about 50 percent yield under the conditions of a kinetic run since the optical
Fig. 5 Ultraviolet and visible spectrum of $p,p'$-dihydroxyazobenzene in methanol

Fig. 6 Ultraviolet and visible spectrum of $N,N'$-diphenylquinoneazine in chlorobenzene and tetralin solution

Fig. 7 Ultraviolet and visible spectrum of oxidized $p,p'$-dihydroxyazo-benzene in chlorobenzene and tetralin solution
J2 ' dihydroxyozobenzene
2.04 x 10^{-5} g/ml in methanol

N,N-diphenylquinone
2.116 x 10^{-5} g/ml in 4:1 chlorobenzene and tetralin solution

oxidized J2 ' dihydroxyozobenzene
4.20 x 10^{-5} g/ml in 4:1 chlorobenzene and tetralin solution
density is approximately the same for the two spectra, yet the concentra-
tion of inhibitor in the oxidation run is twice that of the known sample of 
quinoneazine (see Tables 5 and 6).

**Attempted preparation of diphenylperoxide by the air oxidation of phenyl-
azotriphenylmethane**

**First oxidation run (at 25°C).** A mixture of 100 ml. of benzene and 
17.4 g. (0.05 mole) of phenylazotriphenylmethane was placed in a round 
bottom, three-necked flask and air was bubbled through the solution as 
described in the section on apparatus and procedures. The half life was 
calculated as 47 hours$^{581b}$ at 25°C and it was decided to run the reaction 
for 5 half lives. A white precipitate was observed on dissolving the com-
 pound and was probably hydrazo contaminant. It soon dissolved. Soon a 
white precipitate appeared. During the run the benzene had to be replen­
ish ed several times and the flask went dry several times overnight. At 
the end of the run the flask was dry, so the residue was extracted with 
Skelly B and the extract was chromatographed on alumina. A number of 
fractions gave a peroxide test, but most of these were solids. The residue 
which remained after filtering off the benzene reaction mixture weighed 
4.62 g. and melted at 181-184°C, indicating that it was ditritylperoxide. 
One of the better fractions from the column was recrystallized. The oily 
material in it was found to give a peroxide test while the solid did not. 
After considerable recrystallization from dry ice and acetone, an infrared 
spectra of the material was taken. An OH frequency was observed so the 
material was dissolved in Skelly A and placed over solid sodium hydroxide. 
A white finely divided material was filtered off and the Skelly A was
Table 5

Optical density from the spectrum of a known quantity of quinoneazine in methanol

<table>
<thead>
<tr>
<th>Optical density</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Actual</td>
</tr>
<tr>
<td>First peak (335 m(\mu))</td>
<td>2.84</td>
</tr>
<tr>
<td>Second peak (380 m(\mu))</td>
<td>2.39</td>
</tr>
</tbody>
</table>

Table 6

\(\text{N,N'}\)-Dihydroxyazobenzene oxidized in tetralin and chlorobenzene

<table>
<thead>
<tr>
<th>Optical density</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Actual</td>
</tr>
<tr>
<td>First peak (335 m(\mu))</td>
<td>2.9</td>
</tr>
<tr>
<td>Second peak (380 m(\mu))</td>
<td>2.27</td>
</tr>
</tbody>
</table>

evaporated off. Another infrared spectrum was taken. The OH frequency was gone. A white crystalline substance began to precipitate which gave a negligible peroxide test. The oil was not as good as that obtained in the next run, so work was concentrated on that run.

Second oxidation run (at 62.3°). The procedure was the same as in the first run, except that through a miscalculation the run went for 45 half lives (12.6 minutes at 62.3°). The white solid filtered out of the reaction mixture weighed 3.68 g. and melted at 184-186°, indicating that it was dityritylperoxide. The solution was titrated giving a total of .05557 mole
of peroxide in the solution. The benzene solution was distilled at reduced pressure and the oily residue extracted with n-pentane, followed by Skelly A and B, methylene chloride, acetone, ether and hot benzene. The material in n-pentane and the Skelly solvents was a light yellow oil. All the other residues proved to be ditritylperoxide. The oily material from the n-pentane and Skelly solvents was placed on the alumina column and chromatographed. The weight of material was about 6.75 g. The second fraction gave a good peroxide test so it was recrystallized repeatedly from a freezing mixture of dry ice and acetone. Infrared spectra indicated O-H present, so solid sodium hydroxide was used to precipitate the hydroperoxide from the Skelly B solution of the material. The infrared spectrum indicated that all hydroperoxide had been removed. A white crystalline substance began to precipitate slowly. This gave a negligible peroxide test. The oil gave a strong test which, however, developed slowly. However, after more crystallizations, the oil gave no more than 25 percent of the theoretical peroxide titer for diphenylperoxide.

Third oxidation run (at 62.2°C). The oxidation was carried out as before for 2 hours (10 half lives). Most of the benzene was gone by the end of the run. The viscous yellow oil was extracted with pentane, which caused the separation of a white precipitate. The Skelly B solution of the oil was placed over sodium hydroxide for three days. After removal of hydroperoxide the material was chromatographed. None of the fractions gave a good test for peroxide. The solid residue obtained on extraction of the original reaction mixture weighed 9.28 g. and the m.p. was 109-113°C. Since this material does not correspond to the m.p. of ditritylperoxide, which
should be produced in the reaction, it was assumed that lack of oxygen caused the production of the low melting material, so the run was discarded.

**Fourth oxidation run (at 62.2°C).** A fourth run was started and handled much as before. No product was obtained.
A study of the mechanism of the air oxidation of unsaturated compounds in the presence of inhibitors was recently instituted in these laboratories. A typical oxidizable hydrocarbon, tetralin, was chosen for a standard substrate since its oxidation has been studied and the rate of decomposition of the derived peroxide is known. The thermal decomposition of azo-bis-isobutyronitrile in the presence of oxygen was used as a chain initiator for the oxidation reaction. The decomposition of the initiator is not sensitive to the presence of radicals.\textsuperscript{589} The generally accepted mechanism of initiated air oxidation is the following:\textsuperscript{72}

\begin{align*}
(1) \quad & (\text{CH}_3)_2\text{C} = \text{N} - \text{N} - \text{C} - (\text{CH}_3)_2 \overset{k_d}{\longrightarrow} 2 (\text{CH}_3)_2\text{C} = \text{N} + \text{N}_2 \\
(2) \quad & (\text{CH}_3)_2\text{CCN} + \text{O}_2 \overset{k_1}{\longrightarrow} (\text{CH}_3)_2\text{CO}_2 \cdot \\
(3) \quad & \text{CH}_3\text{CO}_2 \cdot + \text{RH} \overset{k_2}{\longrightarrow} (\text{CH}_3)_2\text{CO}_2\text{H} + \text{R} \cdot \\
(4) \quad & \text{R} \cdot + \text{O}_2 \overset{k_3}{\longrightarrow} \text{RO}_2 \cdot \\
(5) \quad & \text{RO}_2 \cdot + \text{RH} \overset{k_4}{\longrightarrow} \text{RO}_2\text{H} + \text{R} \cdot \\
(6) \quad & 2 \text{RO}_2 \cdot \overset{k_t}{\longrightarrow} \text{unknown products.}
\end{align*}

It cannot be assumed that the rate of initiation is equal to twice the rate of decomposition. Therefore, rate of initiation equals rate at which (2) occurs or
(7) \[ k_1 \left[ \text{(CH}_3\text{)}_2\text{CN} \right] \frac{\dot{O}_2}{\text{I}} = 2ak_3 \left[ \text{AlBN} \right] \]

where \( a \) is a factor which is one or less than one. By making the steady state approximation the rate law required by the above mechanism can be derived as follows:

(8) \[ \frac{d\sqrt[3]{R^\text{I}^+}}{dt} = 2ak_3\sqrt[3]{\text{I}^+} - k_2\sqrt[3]{O_2 \cdot R^\text{I}^+} = 0 \]

\( \sqrt[3]{\text{I}^+} \) is initiator concentration.

\( \sqrt[3]{R^\text{I}^+} \) is \( (\text{CH}_3\text{)}_2\text{CN} \) concentration.

(9) \[ \frac{d\sqrt[3]{\text{RO}_2 \cdot I}}{dt} = k_1\sqrt[3]{\text{RO}_2 \cdot I} - k_2\sqrt[3]{\text{RH} / \text{RO}_2 \cdot I} + k_3\sqrt[3]{\text{R}^\text{I}^+} \sqrt[3]{\text{RO}_2 \cdot I} - k_4\sqrt[3]{\text{RO}_2 \cdot I} \sqrt[3]{\text{RH}} \]

\[ - 2k_4\sqrt[3]{\text{RO}_2 \cdot I^2} = 0 \]

(10) \[ \frac{d\sqrt[3]{R^\text{II}^+}}{dt} = k_2\sqrt[3]{\text{RH} / \text{RO}_2 \cdot I} - k_3\sqrt[3]{\text{R}^\text{I}^+} \sqrt[3]{O_2 \cdot I} + k_4\sqrt[3]{\text{RH} / \text{RO}_2 \cdot I} = 0 \]

If one assumes \( R^\text{I}^+ \) and \( \text{RO}_2 \cdot I \) have equal reactivities, then \( k_2 = k_4 \). This is probably approximately true. Then the following is true.

(11) \[ \sqrt[3]{R^\text{I}^+} = \frac{2ak_3\sqrt[3]{\text{I}^+}}{k_1\sqrt[3]{O_2 \cdot I}} \]

(12) \[ \sqrt[3]{\text{RO}_2 \cdot I} = \frac{ak_3\sqrt[3]{\text{I}^+}}{k_1} \]

(13) \[ \sqrt[3]{R^\text{II}^+} = \frac{k_2\sqrt[3]{\text{RH} / (ak_3\sqrt[3]{\text{I}^+})}}{k_3\sqrt[3]{O_2 \cdot I} / k_1} \]
This proposed mechanism then requires that the rate of the reaction shows (1) no dependence upon oxygen pressure, (2) half order dependence of the rate on initiator concentration, and (3) first order dependence of the rate on the concentration of the substrate (RH). If one assumes the other chain termination steps as being in effect, then the rate becomes dependent on oxygen pressure. It was demonstrated that the three conditions demanded by the proposed mechanism were met by the systems studied. In the presence of a good inhibitor the chain terminating step (equation 6) would be replaced by (16).

\[ \text{(16)} \quad \text{In} + n\text{RO}_2^\cdot \rightarrow \text{products.} \]

This equation may possibly represent several steps for some inhibitors, however, several possibilities that suggest themselves, which would lead to different values for the stoichiometric factor \( n \), are as follows:

1. In the case that In is a free radical and adds to \( \text{RO}_2^\cdot \) \( n \) should equal one.

2. If the reaction of In with \( \text{RO}_2^\cdot \) gave stable free radicals which destroyed each other by dimerization or other reactions \( n \) should equal one.

3. If the inhibitor reacted with two \( \text{RO}_2^\cdot \) to give products which are stable in the presence of the other constituents of the reaction
mixture the \( n \) value would be two.

(4) If the products produced by the reaction of two \( \text{RO}_2^- \) with \( \text{In} \) are unstable and react with more \( \text{In} \) to give stable products, \( n \) would equal one if a 1:1 ratio of product and inhibitor reacted.

(5) If the primary reaction of \( \text{RO}_2^- \) with inhibitor gave a product which was itself an inhibitor, then higher \( n \) values would result, which vary according to the nature of the succeeding reactions.

In order to determine the stoichiometric factor \( n \), two methods of approach are applicable. If one accurately determines the rate of initiation of the reaction, then measures with a like accuracy the lifetime of an inhibitor which is present in known concentration, one may calculate the number of chains (\( \text{RO}_2^- \)) which each inhibitor has stopped. This is relatively simple and a very good method for those inhibitors with well defined inhibition periods. The difficulty in the method lies in determining the efficiency of chain initiation which is obtained when an initiator undergoes thermal decomposition. Most of the commonly used initiators do not give a quantitative yield of two chain initiating radicals per molecule when they undergo decomposition as implied by equation (1). Therefore, the rate of initiation of chains is some fractional value of twice the decomposition rate (equation 7). From equation (15), it would appear that by measuring the rate of nitrogen evolution from the initiator in the absence of oxygen and then by measuring the rate of uptake of oxygen by the initiator as it decomposed thermally in the presence of oxygen, one could determine the efficiency factor \( \alpha \). However, this did not prove to be the case, probably due to the production of unstable products, since approximately as much oxygen was used as there was nitrogen released.
would give an efficiency factor of one which does not correlate with other facts obtained by trapping the chain initiating radicals with various scavengers such as mercaptans, iodine and inhibitors, all of which correlate well with each other.

Another independent method of determining the stoichiometry of the reaction of peroxy radicals with an inhibitor is that of product analysis. If one carries out a careful study of the products from the inhibition reaction, one may infer from the products the number of chains which the inhibitor has terminated. An important feature of the product study is the necessity of obtaining practically a quantitative recovery, along with the separation and identification of products. Small amounts of a substance isolated from a reaction mixture in small yield is no guarantee that one will be able to infer the principle course of the reaction from it. Although a thorough product study is slow and time consuming, the information gained from it can be very useful and can help a great deal in determining the stoichiometry of the inhibitor in question, and may be applied to others as well. Since one successful product analysis and assignment of stoichiometry is also a measure of initiator efficiency, the lifetime method previously mentioned may be used to determine the stoichiometry of other inhibitors under the same reaction conditions. It is also possible to check assignments of stoichiometry for various inhibitors for reasonableness and consistency by comparing the results obtained with the various inhibitors.

The attack reported in this thesis upon the problem of the stoichiometry of the reaction of peroxy radicals with inhibitors is based upon product identification. To be successful the method requires:
(1) Quantitative material balance.

(2) The products isolated must be related to a particular stoichiometry of reaction of $\text{RO}_2^\cdot$ with inhibitor in a reasonable manner.

The inhibitor first chosen for study was diphenylamine, which has long been known as an antioxidant. The substrate chosen was tetralin, a compound whose air oxidation has been extensively studied. The initiator chosen was azo-bis-isobutyronitrile, a well-known initiator whose decomposition rate is not sensitive to the presence of radicals as previously mentioned. A great deal of time was spent in trying to obtain products from the air oxidation of diphenylamine inhibited systems without success. The only products separated and identified were tetralin, diphenylamine and tetramethylsuccinonitrile, a coupling product of the radicals produced by the decomposition of azo-bis-isobutyronitrile. By this time, kinetic evidence was sufficient to establish the fact that diphenylamine exhibited a nonintegral stoichiometry with respect to reaction with peroxy radicals, whereas, most other good inhibitors exhibit a stoichiometry of two. For this reason further work with diphenylamine was not undertaken. During this part of the investigation, however, a fact was established which coincided with the results of the work of Hammond, Sen and Boozer concerning the efficiency of radical production from azo-bis-isobutyronitrile. By means of radical traps (butylmercaptan, iodine and oxygen) it was established that only 60 percent of the radicals produced in chlorobenzene solution could be trapped. (The efficiency varies with the solvent.) The remainder formed the dimer which was produced after release of nitrogen from the initiator with formation of radicals and before the radicals could diffuse away from each other in the solvent and react with the trapping
compounds. In one of the experiments in which the oxidation of the di-
phenylamine was initiated by peroxy radicals produced from oxygen reacting
with the available radicals from the thermal decomposition of aso-bis-
isobutyronitrile, there was recovered 44 percent of the total charge of
initiator in the form of tetramethylsuccinonitrile. This indicates that
the efficiency of radical production was 56 percent and agrees well with
the 60 percent obtained by kinetic measurements. The difference may be due
do poorer distribution of oxygen in the large scale run. As previously
mentioned, a knowledge of the efficiency of peroxy radical production is
necessary in establishing the stoichiometry of reaction of peroxy radicals
with inhibitors by the lifetime of inhibitor method. For this reason, the
above independent confirmation by obtaining the actual product formed by
the radicals which did not initiate chains is of importance in establishing
the mechanism of reaction. The isolation of the dinitrile is semiquantita-
tive confirmation of the efficiency factor ultimately assigned.

Another inhibitor chosen for study was N,N'-diphenyl-\(\pi\)-phenylenedi-
amine. From a large scale oxidation reaction a 46 percent yield of N,N'
diphenylquinonediimine was isolated from the reaction mixture, with proper-
ties which identified it as the same as the compound prepared by independ-
ent synthesis. With this excellent yield, it was decided to obtain the
ultraviolet spectrum of a known sample of N,N'-diphenylquinonediimine and
compare it with the spectrum of the reaction mixture obtained under the
conditions used in the kinetics apparatus when N,N'-diphenyl-\(\pi\)-phenylene-
diamine was used as the inhibitor. It was thus established that 90-96
percent of the inhibitor was converted to the product previously isolated.
With this excellent yield, the course of the reaction must certainly be
since the need of two RO\textsubscript{2}· to abstract the hydrogen is quite apparent. In this way, the stoichiometric factor was assigned and the efficiency of radical initiation determined, which agreed with the results previously obtained in the work on diphenylamine and with other workers in the laboratory.\textsuperscript{136}

It has commonly been assumed that the rate determining step in the reaction of peroxo radicals with inhibitors is the abstraction of hydrogen from the inhibitor. However, kinetic isotope studies did not bear this out.\textsuperscript{420} On the basis of the termolecular order of the reaction and the lack of kinetic isotope effect, it was proposed that reversible complexing first took place between a peroxo radical and the inhibitor followed by the attack of the second peroxo radical.

\begin{align*}
\text{Equation 17) } & \quad 2 \text{RO}_2\cdot + \text{ArN}^\text{H} \rightleftharpoons 2 \text{RO}_2\cdot + \text{ArN}^\text{H} \\
\text{Equation 18) } & \quad \text{RO}_2\cdot + \text{In} \rightleftharpoons \text{RO}_2\cdot + \text{In}^\text{7} \\
\text{Equation 19) } & \quad \text{RO}_2\cdot \rightleftharpoons \text{In}^\text{7} + \text{RO}_2\cdot \rightleftharpoons \text{products.}
\end{align*}

If only a small part of the RO\textsubscript{2}· radicals are complexed, and the following reaction is very rapid, no isotope effect would be observed. Thus dehydrogenation can be the overall change despite the indication that abstraction is not the first step.

Another assumption which has often been made is that a good inhibitor must have a labile hydrogen, since it was assumed ease of hydrogen removal was important in the rate determining step. However, a case of a good
inhibitor has recently been reported\textsuperscript{135} which had no labile hydrogen and other cases have been reported in which products were obtained in which one peroxo radical abstracted hydrogen and the second gave substitution on the benzene ring.\textsuperscript{110a,136,704} However, it might still be assumed that a compound with two labile hydrogens would be expected to lose them quite easily and thus form a dehydrogenated product exclusively. Such did not prove to be the case upon the oxidation of $2,2'$-dihydroxyazobenzene. The expected product, quinoneazine, was synthesized and its spectrum and the spectrum of oxidized $2,2'$-dihydroxyazobenzene were compared. In this case, only 50 percent of the starting material lost two hydrogens to form the quinoneazine. The identity of the other product is not known. In all probability a peroxo radical has been substituted at some position. In so doing, it must deactivate the rest of the molecule with respect to further reaction with peroxo radicals. This would be necessary or the stoichiometric factor would not be two, as determined by the lifetime method. The inactivation of the ring may come from the peroxo substituent preventing further complexing with peroxo radicals.

Kinetics demands that in the chain termination of uninhibited air oxidation of olefins two peroxo radicals destroy themselves or react with solvent and go to stable products (equation 6). With a tertiary $R^*$ this reaction is hard to formulate. Since (CH\textsubscript{3})\textsubscript{2}C-CN is a radical with a tertiary carbon it was felt that the study of the oxidation of the initiator might prove to be rewarding. Kinetic studies were made on the volume of gas consumed during the oxidation of initiator in order to determine if the following reactions were probable.
Since the efficiency of initiation is only 0.65 in chlorobenzene it would be expected the total gas volume should expand from the N₂ released by the initiator if either reaction occurred. It was found that the oxygen uptake exceeds by a slight amount the rate of nitrogen evolution. A large scale oxidation was carried out. It was noted that a very much lower peroxide content was obtained than was theoretically to be expected if the peroxides were stable. It was also noted that there was hydrogen cyanide gas in the vapors over the reaction mixture. These same results were noted in other runs in which inhibitors or other substrates were used, indicating that the (CH₃)₂COOH which one might assume to be formed at least to some extent was not stable. Perhaps the decomposition of the peroxide derived from the initiator may have proceeded along a path such as the following:

\[
\begin{align*}
(22) \quad \text{CH}_3 \text{C-N-C-N} & \rightarrow \text{CH}_3 \text{C-O} + \text{X} \cdot \\
\text{CN} & \quad \text{CN}
\end{align*}
\]

An attempt was made to utilize air oxidation for the preparation of diphenylperoxide or phenylhydroperoxide. These represent a class of compounds which have not yet been prepared. It would be desirable to obtain them since they might have interesting properties. The diphenylperoxide
might be similar to hydrazobenzene and give the benzidine rearrangement. Also it would probably be a good source of the phenoxy radical, producing only one kind of radical in decomposition, and thus being of interest as a chain initiator.

It was hoped that the following reaction would take place in aromatic solvent:

(23) \( \varphi_3\text{C}N=\varphi \longrightarrow \varphi_3\text{C}^\cdot + \varphi^\cdot + \text{N}_2 \)

(24) \( \varphi^\cdot + \text{O}_2 \longrightarrow \varphi\text{O}_2^\cdot \)

(25) \( \varphi\text{O}_2^\cdot + \text{ArH} + \text{R'}^+ \longrightarrow \varphi\text{O}_2\text{Ar} + \text{R'H} \)

If \( \text{R'} = \varphi\text{O}_2^\cdot \) this gives \( \varphi\text{O}_2\text{H} \).

Results indicate that phenylperoxy compounds may have been produced. However, if they were formed they decomposed at low temperature with formation of \( \varphi^\cdot \) which underwent coupling or aromatic substitution reactions.
SUMMARY

1. The determination of the stoichiometry of the reaction of peroxy radicals with inhibitors was attacked by an analysis of reaction products. An attempt was made to obtain a quantitative material balance and, from the products isolated, establish the stoichiometry of reaction as well as the efficiency of initiation by the initiator of the reaction. This was accomplished. An excellent yield of the oxidation product of N,N'-diphenyl-p-phenylenediamine was obtained and through ultraviolet spectra it was determined that N,N'-diphenyl-p-phenylenediamine was obtained in 90-96 percent yield. From this, the stoichiometry was assigned as two and the efficiency of initiation was determined. Previous work leading to the isolation of tetramethylsuccinonitrile from radicals which did not start chains also was in agreement in assigning the efficiency of chain initiation. Other work with diphenylamine was without result.

2. It was shown that an inhibitor with labile hydrogen does not always lose hydrogen as a principle course of reaction when attacked by peroxy radicals. It was shown that p,p'-dihydroxyazobenzene gave the quinoneazine in only 50 percent yield.

3. Studies of termination reactions of the uninhibited air oxidation of olefins proved unlikely to be rewarding because of the instability of the primary products.

4. An attempt was made to utilize air oxidation as a means of preparing diphenylperoxide or phenylhydroperoxide. This proved unsuccessful.
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PART II. REDUCTION OF NITRO COMPOUNDS
WITH TITANIUM III
INTRODUCTION

Quantitative determination of certain organic compounds by reduction of various functional groups with standard titanous chloride or sulfate solutions has long been practiced. This method has been especially useful in determining organic nitro compounds.

Titanous ion (titanium III) is able to lose only one electron to an oxidizing agent, forming titanium IV in the process. It has been discovered that certain oxidizing agents are able to react only if two electrons at a time are obtained from the reducing agent, while other oxidizing agents are able to react only if one electron is obtained from the reducing agent. There are some oxidizing agents which can react both ways at the same time.

Most commonly used reducing agents for the nitro group, such as zinc, iron, tin, stannous chloride, or hydrogen sulfide, are capable of reducing the nitro group with two electrons at a time. Since the oxygen requires two electrons to reach its normal reduced state, a nitroso compound, the question arises whether two electrons are necessary before a reaction will occur. Since titanium III, which can lose only one electron per ion, is a good reducing agent for nitro compounds, the manner in which the oxygen is reduced becomes of interest. By observing the kinetics of the reaction between titanium III and a nitro compound, the order of the reaction can be obtained. If it is first order in titanium III ion and nitro compound, it is implied that oxygen of the nitro group will react by accepting one electron at a time in the slow step of the reaction. If the order is first in nitro compound and second order in titanium III, then the oxygen of the
nitro group will react only if two electrons are available in the slow step of the reaction. It was the purpose of this work to find which occurred.
HISTORICAL REVIEW

Use of titanous chloride and titanous sulfate as analytical reagents in the determination of organic compounds containing functional groups which are subject to reduction has long been known and applied. A monograph on the subject appeared as early as 1918. Titanous chloride reduction is often applied in the determination of organic compounds containing azo, hydrazo or nitro groups.

It has been observed that certain oxidizing agents will react only if two electrons are obtained from the reducing agent at the same time, others will react only if one electron is obtained at a time, and still others will react both ways at once. These conclusions were in opposition to those of Michaelis and Weiss who had proposed "the principle of compulsory univalent oxidation" about the same time the previous observations were made. Remick was able to extend a further explanation of the observed facts and showed that the principle of compulsory univalent oxidation did not hold true in all cases.

Most commonly used reducing agents for the nitro group are such substances as zinc, iron, tin, stannous chloride, or hydrogen sulfide. These materials are capable of supplying two electrons at once to the oxygen of the nitro group. On the other hand, titanium III can only give up one electron per ion on reducing a nitro group, forming titanium IV in the process. Since each oxygen of the nitro group requires two electrons, the question arises as to whether or not the oxygen attacked will accept only one electron in the slow step of the reaction with titanium III or if two are necessary. After the first oxygen is removed, the resulting nitroso
compound is reduced more rapidly than the nitro compound, so it will not affect the kinetic order of the reactants. If two electrons are essential before the oxygen will react in the slow step, then the reaction would be second order in titanium III; if only one electron is needed, it should be first order with respect to the reductant.

This work was commenced in the fall of 1950, in order to determine whether a one electron transfer occurred or two electron transfer was essential in the slow step of the reduction of organic nitro compounds. For ten years prior to this time, interest in titanous reactions had been largely confined to analytical studies. However, since this time Johnson and Weinstein\(^1\) have reported a kinetic study of the reduction of sodium anthraquinone-2-sulfonate by titanous ion and Hinshelwood and co-workers\(^5\) have published a paper on the reduction by titanous chloride of nitrobenzenes and its derivatives which bears directly on the work reported here.
Preparation of Equipment and Standard Solutions

Preparation of a nitrogen purification train

A nitrogen purification train was constructed in order to maintain a nitrogen atmosphere over the titanous sulfate solution. The nitrogen from the tank was led through two bottles of alkaline pyrogallol to absorb any oxygen which might contaminate the nitrogen. The gas was then washed with concentrated sulfuric acid to remove water vapor and then passed through Drierite and sodium calcium hydrate in a drying tower. Appropriate traps were used to prevent mixing or loss of the solutions in the train. After passing through the drying tower, the nitrogen was led into the storage bottle for the titanous sulfate solution and also into the top of an automatic burette, so that the nitrogen atmosphere could be maintained while the burette filled with titanous sulfate solution from the storage bottle. In order to maintain a constant nitrogen pressure on the system, the gas was also led from the top of the drying tower into the bottom of a bottle with a small amount of mercury in it. When the pressure became too high, the nitrogen escaped by bubbling out through the mercury and a small positive pressure was maintained on the rest of the system. Arrangements were also made so that it purified nitrogen to bubble through solutions which were being titrated, thus excluding the air.

Preparation of titanous sulfate solution by the method of Vogel

Sixty ml. of commercial titanous sulfate solution (20 percent solution obtained from La Motte Chem. Products Co., Baltimore, Md.) was placed in
200 ml. of 1:3 sulfuric acid solution and boiled for two to three minutes. After cooling, the solution was diluted to two liters with carbon dioxide free water. The air in the storage bottle was displaced by nitrogen and the solution, which was approximately .0156 mole per liter, was then placed in the tank.

Preparation of 0.1 M ceric sulfate solution

To 28 ml. of concentrated sulfuric acid in 500 ml. of water was added 52.8 g. of ceric bisulfate (G. Fred. Smith, Columbus, Ohio). On solution, the mixture was diluted to one liter.

Standardization of ceric sulfate

Standardization of ceric sulfate solution was accomplished by using arsenious oxide as a primary standard with ferroin indicator. The iodine monochloride catalyst was prepared according to the method of Swift and Gregory. The ceric sulfate solution was .0775 N. Later a more dilute solution was also prepared, which was .0153 N, by taking a volume of the above solution and diluting with four volumes of water.

Standardization of titanous sulfate

Standardization of titanous sulfate solution was performed by adding 9 ml. of ferric chloride solution (approximately .577 M) to a flask which was then flushed out with nitrogen. The titanous sulfate was added rapidly and the ferroin indicator was added. The reduced iron (ferrous) was then titrated with the standard ceric sulfate solution, the end point changing sharply from a pink to a greenish-yellow color. The normality of the
titanous sulfate was 0.0138 N.

Preparation of o-nitrobenzoic acid solution

A solution of o-nitrobenzoic acid (m.p. 147.5°, observed 145-147°) was prepared by dissolving 3.3416 g. in two liters of water. The solution was approximately 0.01 M (or 0.06 N) in reference to reduction by titanous sulfate.

A second solution was prepared which was 0.0023 M (0.0138 N) in o-nitrobenzoic acid and 0.025 M in sodium sulfate. This solution was prepared by taking 38.33 ml. of the 0.01 M o-nitrobenzoic acid solution prepared above, adding 3.55 g. of sodium sulfate and diluting to one liter.

Preparation of the reaction flask

Two round bottom, standard taper flasks of 200 ml. capacity were joined by two tubes. One tube of 15 mm. size was sealed to the bottoms of the flasks and was curved upward between the flasks, having the form of an inverted "U". In this way a solution could be placed in either or both flasks without mixing, until the flask was tipped to pour it in. Just below the joint another smaller tube was run between the two flasks, so the air pressure in each would be the same during mixing. Each flask was stoppered with a stopper which had a stopcock on the end, so that air could be evacuated or flushed out with nitrogen or carbon dioxide and then the atmosphere could be excluded. Samples could be withdrawn from either flask by syringe without contamination with oxygen. A capillary tube was sealed in the flask near the top and led to the bottom of each flask. A short piece of 6/16 inch tubing was sealed to the top of the capillary and was
stopped with small sample bottle caps. The syringe was pushed through the rubber cap, filled, taken out and the sample adjusted to 10 ml. capacity. The sample was then delivered into ferric chloride solution to quench the reaction and the analysis was begun.

Preliminary Kinetic Measurements

Run #1 (investigation of concentrations of reactants needed for easy kinetic measurement)

Fifty ml. of .0023 M (.0138 N) o-nitrobenzoic acid and .025 M sodium sulfate mixture were added to 50 ml. of .0138 N titanous sulfate solution. Both solutions were previously cooled to 20.9°. After stirring well in the volumetric flask, the mixture was again placed in the constant temperature bath at 20.9°. Ten ml. samples were pipetted out at 10 minute intervals and added to an excess of ferric chloride solution. The amount of titanous ion still present was determined by titrating the ferrous iron formed when the reaction mixture was added to the ferric chloride, thus quenching the titanous sulfate reduction of o-nitrobenzoic acid. The ferrous chloride was titrated with .0775 N ceric sulfate using ferroin as indicator. The color change was from pink to greenish-yellow.

Since this run appeared to have an initial induction period and the reaction was only about 30 percent complete in 80 minutes, it was decided to try a higher temperature. On plotting the data, it appeared that the reaction was nearer second order than any other. However, there was considerable scatter in the points and the line curved off from that for a true second order plot.
Run #2 (determination of a temperature at which to measure rates)

The same procedure was used as in Run #1 with the exception that the water bath was maintained at 24.8° and the normality of titanicus sulfate solution had now fallen to .01208 N. During the latter part of the run samples were taken every 20 minutes instead of every 10 minutes. It was noted that the end point faded rapidly in the titrations with ceric sulfate using ferrocen as indicator, the change being a rapid change from greenish-yellow to yellow. Titration was to the first change noted.

On plotting the data as a second order plot a curve was obtained quite similar to that of Run #1, with the exception that there appeared to have been an extremely rapid reaction between the time of mixing and the first sample, where previously an induction period was observed. Contamination by air was considered probable with oxygen reacting with titanicus ion.

Run #3 (preliminary investigations of reaction order)

The same procedure was used as before, except the normality of the titanicus sulfate was .0051 N. Mixing of the solution took one minute and a sample was taken after two minutes.

Runs #4, 5 and 6 (preliminary investigations of reaction order)

The same procedures were used. The normality of the titanicus sulfate solution was .01206 N. Upon plotting the data from Runs 3 to 6, the same general results were obtained. The reaction between time of mixing and the taking of the first sample (usually 2 minutes from the beginning of the reaction) was much faster than the reaction which followed. The order
approached that of a bimolecular reaction. However, scattering of points on the curve was quite noticeable.

Runs #7-10 (investigation of the effects of oxygen in the air)

The normality of titanous sulfate solution was the same as in Runs 4 to 6. However, the concentration of solution of p-nitrobenzoic acid was decreased to .000767 M in the acid and .00833 M in sodium sulfate. The last two runs were commenced by putting dry ice in the reaction flasks so that carbon dioxide was generated and air excluded. Upon plotting the data of these runs, the same general results were observed as before. The dry ice eliminated some of the initial fast reaction and the points on the curves still scattered. It was decided to construct a new reaction flask from which it would be possible to exclude air during the run, even while extracting samples for titration. (See preparation of reaction flask.)

Run #11 (No reaction occurred.)

A solution (100 ml.) of .0023 M in p-nitrobenzoic acid and .025 M in sodium sulfate was placed in one side of the reaction flask and 100 ml. of .0138 N titanous sulfate was placed in the other. Evacuation was attempted but the pressure equalizing tube between the flasks was not yet in place so the solution in the second flask began to be pulled over into the first flask. Evacuation was ceased and dry ice was dropped in each flask instead. After the carbon dioxide had swept the air out of the flasks the stopcocks were closed. After mixing, the flask was placed in the 24.6° constant temperature bath. Sampling occurred at intervals during the day. No reaction to speak of occurred during the day. After six more days at room
temperature, no reaction had yet occurred, so the reaction mixture was placed in a 55° constant temperature bath for two days with the same results.

These results were disturbing, so an attempt was made to isolate the products of the reaction of the same mixture as used above in the presence of air. No definite results were obtained.

Run #12 (further investigation of concentration of reactants, temperature and catalysis)

With the surprising results of Run #11, it was decided to increase the concentration of reactants, to use the higher constant temperature bath (55°) and to add chloride ion to the mixture to see if this would catalyze the reaction, since titanous chloride is normally used in analytical procedures and the chloride ion often enters into the mechanisms of reaction when present.

In one side of the reaction vessel was placed 100 ml. of a solution .0059 M (.0356 N) in o-nitrobenzoic acid and .064 M in sodium sulfate. In the other side was placed 1.17 g. of sodium chloride and 100 ml. of .0356 N titanous sulfate. The flasks had been flushed out previously with carbon dioxide generated from dry ice and, after the solutions were added, more dry ice was dropped in to maintain the carbon dioxide atmosphere. After the carbon dioxide had been generated, the stopcocks were closed to exclude the air and the reaction vessel was placed in the 55° constant temperature bath. After the solutions had reached 55°, they were mixed together and a 10 ml. sample was removed and analyzed for titanous ion. The reaction was quite rapid. The purple color due to titanous ion was not discernible within the
first hour. Another difficulty was now encountered in the end point in the 
titrations to determine titanous ion. Where previous end points were 
changes from a clear greenish-yellow to a clear yellow color, the end point 
now appeared to be a dark yellow as if a brown or black or some opaque sub-
stance was also produced. For this reason, the end point was hard to read 
and the titrations were in error.

Runs #13 and #14 (investigation of the effect of the absence of chloride ion 
catalyst and lowering of the temperature of reaction)

Run #13 was conducted in the same way as Run #12, with the exception 
that no sodium chloride was added. Again the reaction was quite rapid with 
the purple color of the titanous ion disappearing in about 10 minutes. The 
end point of the titration to determine titanous ion gave the same diffi-
culty as in Run #12.

Run #14 was conducted in the same way as Run #13 with the exception 
that the temperature was maintained at 24.6° instead of 55°. The purple 
color of the titanous ion disappeared after the first half-hour. After the 
first titration of the first sample, difficulty was encountered in detect-
ing the end point as in the last two runs.

Runs #15 and #16 (investigation of the use of sodium diphenylamine sulfonate 
as an indicator in the volumetric determination of titanous ion)

Run #15 was carried out with the same concentration of reactants and 
the same temperature as Run #14 except the 100 ml. of titanous sulfate had 
decreased in concentration to .0261 N. The first 10 ml. sample was taken 
by syringe one minute after mixing and was added to 5 ml. of .577 M ferrie
chloride. Following this, 10 ml. of concentrated orthophosphoric acid was added, and then 2 drops of sodium diphenylamine sulfonate indicator was dropped in. A very sharp end point was obtained with this and all other samples taken during the run. The color change was from colorless to purple. A blank was run to determine the error due to the added indicator.

Run #16 was carried out in the same manner and under the same conditions as Run #15 and results were similar except that practice in the titration gave more accurate data.

Upon plotting the data from Runs #14, 15 and 16, it was noted that there was a steady improvement in the accuracy of the data, in that the scattering of points became much less. The data for Run #16 gave almost a smooth curve. Similar results were obtained as previously. The curves indicated that the overall reaction was not truly second order. A first order plot for titanous ion gave a slightly curved line, while with the data from Run #16 a second order plot appeared to be possibly a straight line.

Run #17 (a check of the absence of side reactions of titanous ion)

In one side of the reaction flask was placed 100 ml. of a solution .00078 M in 2-nitrobenzoic acid and .0083 M in sodium sulfate. In the other side was placed 100 ml. of .0261 N titanous sulfate, the air in the reaction flask having been expelled by carbon dioxide from dry ice. After the solutions had reached the temperature (24.6°) of the constant temperature bath, they were mixed and samples were taken at intervals for titration. If the 2-nitrobenzoic acid were completely reduced, the normality of the reaction mixture should be .01075 N in titanous ion. After 114 hours
the normality was not less than .01125 N, indicating that no side reaction of any consequence was occurring. The reaction was essentially complete within 6 hours.

Since the details of the way in which the experiments were run has been presented previously, only a compilation of the essential data from the various runs which followed the solving of experimental difficulties is here presented. A sample of the data from one of the runs is included as follows:

Beginning concentration of the reaction mixture for run #21.

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanous sulfate</td>
<td>.0178 N</td>
</tr>
<tr>
<td>Sulfuric acid</td>
<td>.696 N</td>
</tr>
<tr>
<td>p-Nitrobenzoic acid</td>
<td>.0356 N (.0059 M)</td>
</tr>
<tr>
<td>Sodium sulfate</td>
<td>.032 M</td>
</tr>
</tbody>
</table>

Table 7
Data from run #21

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Time after mixing</th>
<th>N of titanus sulfur in reaction mixture</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1 min. 30 sec.</td>
<td>.0115</td>
</tr>
<tr>
<td>2</td>
<td>6 &quot; 20 &quot;</td>
<td>.0089</td>
</tr>
<tr>
<td>3</td>
<td>15 &quot; 20 &quot;</td>
<td>.0046</td>
</tr>
<tr>
<td>4</td>
<td>20 &quot; 05 &quot;</td>
<td>.0037</td>
</tr>
<tr>
<td>5</td>
<td>26 &quot;</td>
<td>.0025</td>
</tr>
<tr>
<td>6</td>
<td>32 &quot; 50 &quot;</td>
<td>.0019</td>
</tr>
<tr>
<td>7</td>
<td>37 &quot;</td>
<td>.0014</td>
</tr>
<tr>
<td>8</td>
<td>40 &quot; 10 &quot;</td>
<td>.0013</td>
</tr>
<tr>
<td>9</td>
<td>49 &quot; 55 &quot;</td>
<td>.0008</td>
</tr>
<tr>
<td>10</td>
<td>60 &quot; 10 &quot;</td>
<td>.0005</td>
</tr>
<tr>
<td>11</td>
<td>70 &quot;</td>
<td>.0003</td>
</tr>
</tbody>
</table>
Table 8 which follows gives the data concerning the initial concentration of reactants in the mixture for each run. Constant ionic strength was maintained below Run #27 with the exception of Runs #32 and 33, except for changes due to change in concentration of titanous ion. Those runs marked with an asterisk proved to be too fast for accurate measurement, the reaction being 35 percent or more completed when the first sample was taken a minute to a minute and one-half after mixing.

Table 8

Initial concentration of the reaction mixtures

<table>
<thead>
<tr>
<th>Run #</th>
<th>N of titanous ion</th>
<th>N of hydrogen ion</th>
<th>N of p-nitrobenzoic acid</th>
<th>M of sodium sulfate</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>.0131</td>
<td>1.54</td>
<td>.0178</td>
<td>.032</td>
</tr>
<tr>
<td>16</td>
<td>.0131</td>
<td>1.54</td>
<td>.0178</td>
<td>.032</td>
</tr>
<tr>
<td>17</td>
<td>.0131</td>
<td>1.54</td>
<td>.0023</td>
<td>.004</td>
</tr>
<tr>
<td>18*</td>
<td>.0178</td>
<td>.696</td>
<td>.0178</td>
<td>.032</td>
</tr>
<tr>
<td>19*</td>
<td>.0178</td>
<td>.696</td>
<td>.089</td>
<td>.032</td>
</tr>
<tr>
<td>20*</td>
<td>.0178</td>
<td>.696</td>
<td>.0445</td>
<td>.032</td>
</tr>
<tr>
<td>21</td>
<td>.0178</td>
<td>.696</td>
<td>.0356</td>
<td>.032</td>
</tr>
<tr>
<td>22</td>
<td>.0232</td>
<td>.905</td>
<td>.0356</td>
<td>.032</td>
</tr>
<tr>
<td>23*</td>
<td>.0089</td>
<td>.348</td>
<td>.0356</td>
<td>.032</td>
</tr>
<tr>
<td>24</td>
<td>.0178</td>
<td>.696</td>
<td>.0356</td>
<td>.032</td>
</tr>
<tr>
<td>25</td>
<td>.0089</td>
<td>.348</td>
<td>.0178</td>
<td>.032</td>
</tr>
<tr>
<td>26</td>
<td>.0154</td>
<td>.603</td>
<td>.0237</td>
<td>.032</td>
</tr>
<tr>
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<td>.0232</td>
<td>.905</td>
<td>.0178</td>
<td>.032</td>
</tr>
<tr>
<td>28</td>
<td>.0226</td>
<td>.905</td>
<td>.0356</td>
<td>.032</td>
</tr>
<tr>
<td>29*</td>
<td>.0226</td>
<td>.452</td>
<td>.0356</td>
<td>.258</td>
</tr>
<tr>
<td>30*</td>
<td>.0226</td>
<td>.226</td>
<td>.0356</td>
<td>.371</td>
</tr>
<tr>
<td>31</td>
<td>.0178</td>
<td>.905</td>
<td>.0356</td>
<td>.032</td>
</tr>
<tr>
<td>32*</td>
<td>.0178</td>
<td>.452</td>
<td>.0356</td>
<td>.159</td>
</tr>
<tr>
<td>33*</td>
<td>.0178</td>
<td>.218</td>
<td>.0356</td>
<td>.277</td>
</tr>
<tr>
<td>34</td>
<td>.0089</td>
<td>.905</td>
<td>.0356</td>
<td>.032</td>
</tr>
<tr>
<td>35*</td>
<td>.0089</td>
<td>.452</td>
<td>.0356</td>
<td>.258</td>
</tr>
<tr>
<td>36*</td>
<td>.0089</td>
<td>.226</td>
<td>.0356</td>
<td>.371</td>
</tr>
<tr>
<td>Repeat 32*</td>
<td>.0178</td>
<td>.452</td>
<td>.0356</td>
<td>.258</td>
</tr>
<tr>
<td>33*</td>
<td>.0178</td>
<td>.226</td>
<td>.0356</td>
<td>.371</td>
</tr>
<tr>
<td>Run #</td>
<td>N of titan-</td>
<td>N of hydro-</td>
<td>N of 2-nitro-</td>
<td>M of sodium</td>
</tr>
<tr>
<td>-------</td>
<td>------------</td>
<td>-------------</td>
<td>---------------</td>
<td>-------------</td>
</tr>
<tr>
<td></td>
<td>ous ion</td>
<td>gen ion</td>
<td>benzene acid</td>
<td>sulfate</td>
</tr>
<tr>
<td>37</td>
<td>.0189</td>
<td>.226</td>
<td>.0178</td>
<td>.371</td>
</tr>
<tr>
<td>38</td>
<td>.0189</td>
<td>.905</td>
<td>.0178</td>
<td>.032</td>
</tr>
<tr>
<td>39</td>
<td>.0189</td>
<td>.452</td>
<td>.0178</td>
<td>.258</td>
</tr>
<tr>
<td>40</td>
<td>.0169</td>
<td>.905</td>
<td>.0178</td>
<td>.032</td>
</tr>
</tbody>
</table>

The concentration of various reactants in the above table were varied in a systematic way in an endeavor to learn the kinetic order of the various reactants.
DISCUSSION

Upon the compilation of the data, efforts were made to interpret the results so that the essentiality of a one or two electron transfer in the slow step from titanous to nitro compound might be determined. Since many of the reactions were quite rapid, it was decided to plot the concentrations against time on very large graph paper and determine the initial slopes. Results from this effort were very unsatisfactory until it was realized that many reactions were over 35 percent completed by the time the first sample was taken. The data from these runs were rejected and the initial slopes of the remaining runs compared. However, they were not as accurate as could be desired. It was again determined to try some first order plots of log titanous ion versus time.

If one has two reactants (one in large excess) and the rate of disappearance is followed, then the rate equation

\[
(1) \quad \frac{dA}{dt} = k\sqrt{A} \sqrt{B}^m \text{ becomes}
\]

\[
(2) \quad \frac{dA}{dt} = k'\sqrt{A} \sqrt{B}^m \text{ (since } \sqrt{B}^m \text{ is essentially constant).}
\]

Integrating, if \( n = 1 \) (first order in \( A \)), one obtains

\[
(3) \quad -2.303 \log A = k't + \text{constant.}
\]

If \( \log A \) is plotted against time and a straight line is obtained, the reactant \( A \) is first order in the slow step of the reaction. If one ignores the calculated starting concentration of titanous ion (which is always higher than the line through the other points, thus indicating the titer of titanous ion is reduced, probably by oxygen which is not completely flushed
out of the flask), on plotting the data from four different runs good straight lines were obtained for each run, indicating that the order for titanous ion was first order. In other words, that the slow step of the reaction involves only one titanous ion, thus establishing the fact that the nitro group will accept only one electron at a time.

From the first order curves for titanous ion the following was obtained.

Table 9

<table>
<thead>
<tr>
<th>Sub</th>
<th>Slope</th>
<th>First order constant (k')</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>-0.0146</td>
<td>0.0337</td>
</tr>
<tr>
<td>28</td>
<td>-0.0158</td>
<td>0.0364</td>
</tr>
<tr>
<td>31</td>
<td>-0.0188</td>
<td>0.0433</td>
</tr>
<tr>
<td>34</td>
<td>-0.0218</td>
<td>0.0503</td>
</tr>
</tbody>
</table>

Similar first order plots could not be made for the $\beta$-nitrobenzoic acid since every series in which the concentration had been varied in the presence of such an excess of titanous ion that the titanous concentration was essentially constant the speed of the reaction had been such as to make the data obtained of no value.

However, upon establishing the fact that the reaction was first order in titanous ion, it now became possible to check whether $\beta$-nitrobenzoic acid was also first order. If both reactants are near the same concentration, so that both vary during the reaction, the following equation applies:
(4) \[ t = \frac{2.303}{k(a - b) \log a} + \frac{2.303}{k(a - b) \log b} \left( \frac{a - x}{b - x} \right) \]

where \( a \) and \( b \) are initial concentrations and \( x \) is the decrease in concentration after time \( t \). If one plots \( \frac{a - x}{b - x} \) against \( t \), then a straight line will be obtained if the reaction is bimolecular, being first order in \( a \) and \( b \). The slope of the curve will be \( \frac{2.303}{k(a - b)} \) from which \( k \) can be calculated.

On plotting the data from 14 runs as suggested above, good straight lines were obtained with good fits to the points in every case except one. Thus the postulate that \( p \)-nitrobenzoic acid is also first order in the reaction is confirmed. The slope of each line was taken and the bimolecular constant calculated from

(5) \[ \text{slope} = \frac{2.303}{k(a - b)} \text{ or } k = \frac{2.303}{\text{slope} (a - b)} \]

The data from these calculations are compiled in Table 10.

Another point of interest is the role which hydrogen ion plays in the reaction. It is easily observable that an increase in hydrogen ion concentration slows down the rate of reaction. However, of a number of runs in which hydrogen ion was varied only two were slow enough so that the data was good enough to compare. Since the rate of reaction decreases with increase of hydrogen ion concentration, the rate must be inversely proportional to some power of the hydrogen ion concentration. Since the bimolecular rate constant, as previously determined, varies with hydrogen ion concentration, it should be able to be corrected to a true specific rate constant by dividing the bimolecular rate constant by the hydrogen ion concentration raised to the appropriate power. For the results, see Table 11.
Table 10
Bimolecular constants and necessary data for their calculation

<table>
<thead>
<tr>
<th>Run #</th>
<th>Slope</th>
<th>(a - b)</th>
<th>Bimolecular constant (k)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>-0.0149</td>
<td>-0.0047</td>
<td>3.29 x 10^4</td>
</tr>
<tr>
<td>16</td>
<td>-0.0194</td>
<td>-0.0047</td>
<td>2.53 x 10^4</td>
</tr>
<tr>
<td>21</td>
<td>-0.0203</td>
<td>-0.0178</td>
<td>6.38 x 10^3</td>
</tr>
<tr>
<td>22</td>
<td>-0.01206</td>
<td>-0.0124</td>
<td>1.542 x 10^4</td>
</tr>
<tr>
<td>24</td>
<td>-0.0183</td>
<td>-0.0178</td>
<td>7.08 x 10^3</td>
</tr>
<tr>
<td>25</td>
<td>-0.0246</td>
<td>-0.0089</td>
<td>1.052 x 10^4</td>
</tr>
<tr>
<td>26</td>
<td>-0.01206</td>
<td>-0.0083</td>
<td>2.31 x 10^4</td>
</tr>
<tr>
<td>27</td>
<td>+0.0052</td>
<td>+0.0054</td>
<td>8.21 x 10^4</td>
</tr>
<tr>
<td>28</td>
<td>+0.0110</td>
<td>-0.0130</td>
<td>1.615 x 10^4</td>
</tr>
<tr>
<td>31</td>
<td>-0.0150</td>
<td>-0.0178</td>
<td>8.63 x 10^3</td>
</tr>
<tr>
<td>34</td>
<td>-0.02025</td>
<td>-0.0267</td>
<td>4.27 x 10^3</td>
</tr>
<tr>
<td>38</td>
<td>+0.00146</td>
<td>+0.0011</td>
<td>1.432 x 10^6</td>
</tr>
<tr>
<td>39</td>
<td>+0.002125</td>
<td>+0.0011</td>
<td>9.85 x 10^5</td>
</tr>
<tr>
<td>40</td>
<td>-0.00079</td>
<td>-0.0009</td>
<td>3.24 x 10^6</td>
</tr>
</tbody>
</table>

*In this case, the concentrations have been left in terms of normality so the bimolecular constant contains these dimensions.*
Table 11

Calculations pertaining to the order of hydrogen ion

<table>
<thead>
<tr>
<th></th>
<th>Run #38</th>
<th>Run #39</th>
</tr>
</thead>
<tbody>
<tr>
<td>$k$</td>
<td>$1.432 \times 10^6$</td>
<td>$9.85 \times 10^5$</td>
</tr>
<tr>
<td>$k/k_{H^+}$</td>
<td>.905 N</td>
<td>.452 N</td>
</tr>
<tr>
<td>$k/k_{H^+}^2$</td>
<td>$1.583 \times 10^6$</td>
<td>$2.18 \times 10^6$</td>
</tr>
<tr>
<td>$k/k_{H^+}^2$</td>
<td>.82</td>
<td>.206</td>
</tr>
<tr>
<td>$k/k_{H^+}^2$</td>
<td>$1.746 \times 10^6$</td>
<td>$4.78 \times 10^6$</td>
</tr>
</tbody>
</table>

Table 12

Variation of the bimolecular constant with ionic strength

<table>
<thead>
<tr>
<th>Run #</th>
<th>$k_{H^+}$</th>
<th>$k$</th>
<th>$k/k_{H^+}^2$</th>
<th>$\mu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>28</td>
<td>.905</td>
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</table>
The results shown in Table 11 are disappointing, since no assignment can be made as to the order for hydrogen ion.

An effort was also made to determine what effect varying the ionic strength had on the reaction rate (see Table 12 above), but results were not enlightening. The reaction constant $k$ was divided by the square of the hydrogen ion concentration (since Hinshelwood and co-workers\textsuperscript{5} found the rate inversely proportional to hydrogen ion concentration) and the ionic strength was calculated for the mixture. Examination for any trends proved unfruitful.
SUMMARY

1. An investigation was made concerning whether a nitro compound can be reduced by a one electron step or whether it must have two electrons in the rate determining step to be reduced. It has been shown in this investigation that titanous ion, which can release only one electron per ion exhibits first order kinetics in the reduction of nitro compounds. Therefore, a nitro compound may be reduced by a one electron transfer in the rate determining step.

2. It has further been shown that the reaction is bimolecular with respect to titanous ion and nitro compound.

3. Although it is easily observed that the rate of the reaction decreases with increased hydrogen ion concentration, the exact dependence of the rate on hydrogen ion was not determined.

4. An unsuccessful attempt was made to discover the effect of changes in ionic strength upon the rate of the reaction.
BIBLIOGRAPHY

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