

Selected Bibliography on Ozone, its Biological Effects and Technical Applications

by H. Rosenthal

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TECHNICAL REPORT NO. 456

SELECTED BIBLIOGRAPHY ON OZONE, ITS BIOLOGICAL
EFFECTS AND TECHNICAL APPLICATIONS

by

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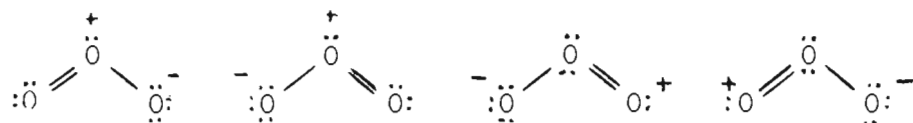
TABLE OF CONTENTS

	Page
INTRODUCTION	1
Nature of ozone and its properties	1
Determination of ozone	2
Biological effects of ozone	4
Applications to human activities	5
State of the literature	9
Current bibliography	10
BIBLIOGRAPHY	11
ADDENDA	137
SUBJECT INDEX	143

INTRODUCTION

Nature of ozone and its properties

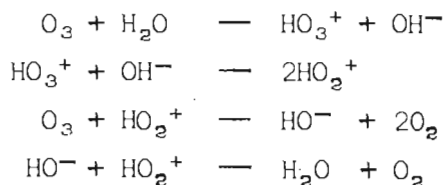
Ozone is nothing more than a particular molecular state of oxygen. The ozone molecule contains three oxygen atoms, has a molecular weight of 48.00 grams per mole, and possesses a very low dipole moment of 0.49-0.58 debye. Some work has been done to identify the molecular structure of this very unstable compound, but its structural determination remains controversial (Smyth and Lewis 1939). The first complete structural determination was made by Shand and Spurr (1943). According to Riebel et al. (1960) the ozone molecular bonds form an obtuse angle ($116^{\circ}45'$), there being two oxygen-oxygen bonds of equal length. The molecule can be described as a resonance hybrid of the four possible structures:



Two basic mechanisms may be considered as responsible for the oxidizing effect of ozone:

- (1) electrophilic addition of the total ozone molecule to C = C double bonds and to lone electron pairs, and
- (2) self decomposition of ozone via short living radicals, such as HO° and HO_2° , which initiate oxidizing reactions (Forchheimer and Taube 1952, 1954; Abel 1955).

The second mechanism may be considered to occur predominantly in aqueous solutions. The formation of free radicals in water is suggested by the following reaction scheme:



Although, ozone is relatively stable in organic solvents, decomposition in aqueous solutions is rather rapid. Rate of decomposition depends on the composition and properties of the solution, especially the organic load and pH value (Sulzer 1958) (Fig. 1). In the presence of hydroxyl ions decomposition is accelerated (Weiss 1935; Stumm 1956). Therefore in sea water, where pH values normally are higher than in fresh water, the breakdown of ozone is more rapid. On the other hand, in strong alkaline solutions ($> 10^{-4}$ N NaOH)

the stability of ozone again increases (Heidt and Landi 1964). According to Stumm (1956) solubility of ozone in water depends on temperature, salt concentration and pH value. Hoather (1948) investigated the solubility and stability of ozone in water in relation to temperature and turbidity. Alder and Hill (1950), and Stumm (1954) showed that ozone in solution is relatively stable at temperatures near the freezing point. Shechter (1973) found that decomposition of ozone was correlated with turbulence and initial concentration of ozone.

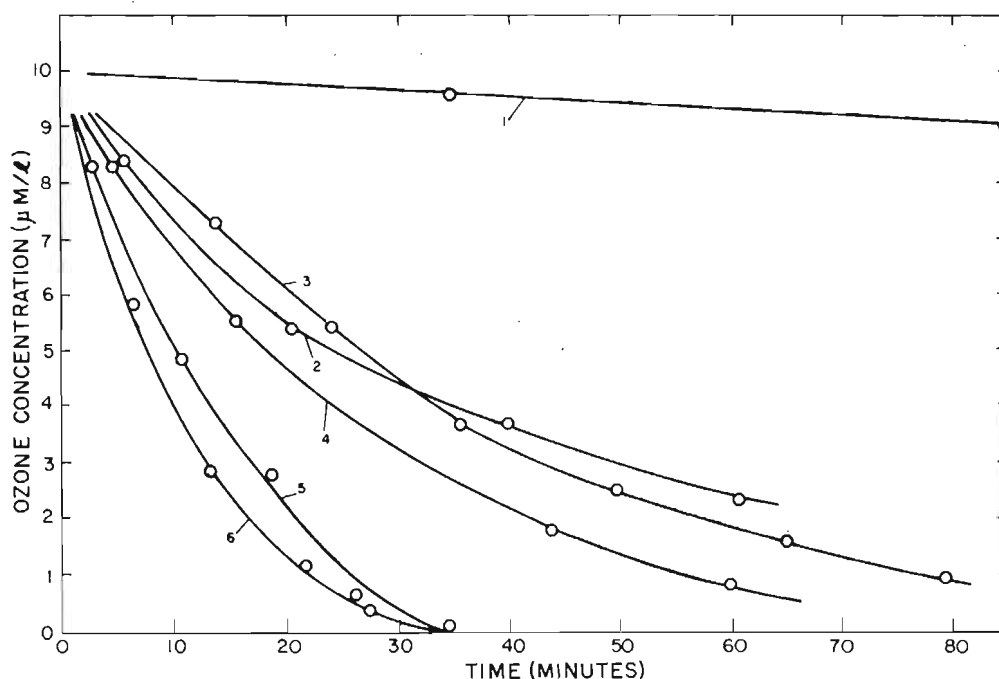


Fig. 1. Ozone decomposition in different types of water at 20°C. 1 = double distilled water, 2 = distilled water, 3 = tap water, 4 = ground water of low hardness, 5 = filtered water from Lake Zurich, 6 = filtered water from the Bodensee (modified after Sulzer 1958).

Determination of ozone

Quantitative determinations of ozone have been troublesome, both in air and in aqueous solutions. Among the most important methods for low-level determination of ozone in air are the starch-iodine method (Thorp 1940), the phenolphthalein oxidation reaction (Haagen-Smit 1958), the long-path ultraviolet absorption method (Renzetti 1957), the nitrogen dioxide equivalent method (Salzman and Gilbert 1959), the sodium diphenylaminesulphonate reaction (Bovee and Robinson 1961), the fluorophotometric determination method (Watanabe and Nakadoi 1966), and the peroxy-isocyanate method (Layton et al. 1970). More recently modified methods for continuous ozone measurement have been developed, using automatic read-out devices (Komhyr 1969; Luebke 1970; Warren and Babcock, 1970; Vigroux 1971; Steinberger and Goldwater 1972; Hosaka and Arai 1973).

Problems of determination of ozone in water arise from its rapid decomposition and its interactions with other ions in solution, e.g. chlorine (Ingols et al. 1956; Sulzer 1958; Koppe and Muehle 1965; Hofman and Stern 1969). The classical procedure (Am. Public Health Assoc. 1971) for the determination of residual ozone in water requires large sample volumes and gives uncertain results at low concentrations. A highly sensitive spectrophotometric method for ozone determination in water was developed recently by Shechter (1973). This method will detect residual concentrations of ozone down to 0.01 ppm.

The most important methods of ozone determination in water are summarized in the following table:

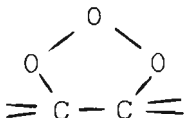
Method	References	Remarks
Manganese oxidation and color reaction with orthotolidine	Zehender and Stumm (1953) Sulzer (1958)	$Mn^{++} \rightarrow Mn^{+++}$ (spectrophotometric determination at 440 mμ, 5-20 μM/l)
Oxidation of leuco crystal violet	Layton and Kinman (1970)	Redox indicator, colorimetric procedure at a wave length of 592 mμ in acidic solution (sub-ppm levels)
Spectrophotometry (visible region)	Alder and Hill (1950) Kilpatrick et al. (1956)	typical absorption band at 260 mμ (< 0.4 μM/l)
reaction with potassium iodide and colorimetric determination of iodine	Shechter (1973)	two modified applications for low level (0.01-0.30 ppm) and high level determination (0.30-2.00 ppm)
reaction with potassium iodide and colorimetric determination of iodine	Parry and Hern (1973)	stoichiometry
oxidative decoloration of indigo-sulfonate	Dorta-Schaeppi and Treadwell (1949)	
reaction of ozone with potassium iodide	Wanner (1971)	$2 KI \rightarrow I_2$
reaction of ozone with potassium iodide	Rawson (1953)	colorimetric determination of the liberated iodine with starch
automatic reading device	Littman and Benoliel (1953)	

Biological effects of ozone

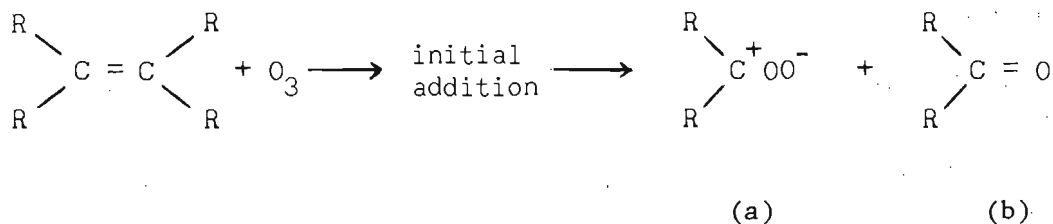
The acute toxicity of ozone to laboratory animals and to man is diverse and varies considerably from species to species.

Giese and Christensen (1954) reported that freshwater ciliates exposed to ozone (3.5×10^{-4} moles per liter gas bubbled continuously through the water column) died within 3 to 4 minutes, whereas rotifers became inactivated after 25 minutes of exposure. In sea urchin larvae a gradual decrease of swimming speed could be observed with increasing exposure time. After 20 minutes of exposure they became completely inactive, but cytolysis did not occur. Bacteria and viruses are destroyed by ozone rapidly. For example, *Escherichia coli* (MPN 60,000 per ml) is killed within 5 seconds in an ozone concentration of 0.1 ppm, whereas 15,000 seconds are required to destroy these bacteria, if chlorine is applied in the same concentration. PM-virus is inactivated in 2 minutes in an ozone concentration of 0.05 to 0.45 ppm (Klein 1969). The poor insecticidal power of ozone is demonstrated by the work reported by Toul (1930). Recently, Blogoslawski et al. (1973) determined the feasibility of inactivating toxic red tide metabolites by ozone treatment.

The mode of action of ozone is not yet fully understood. It has been suggested that all damage may occur through the activity of free radicals, which may be generated by the reaction of ozone with sulfhydryl groups. According to Bailey (1958) it is possible that free radicals may be produced through lipid oxidation. Criegee's mechanism of ozonolysis (Criegee 1953) suggests that ozone may unite with carbon-carbon double bonds to give a very unstable intermediate primary ozonide



which readily decomposes to give (a) a zwitter ion and (b) a carbonyl fragment, according to the following equation (after Loan et al. 1965; Druett and Packman 1972):



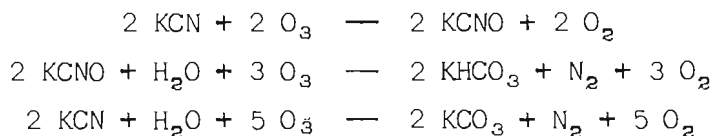
When man is exposed to 4-5 ppm ozone, lung oedema develops within a few hours. Lower concentrations primarily affect respiration by irritation, and also cause headaches and nausea. The literature shows quite conclusively that long term exposure of humans to ozone concentrations of more than 1.5 ppm by weight in air is definitely detrimental (Hill 1942). It should also be emphasized that ozone is a highly odorous compound. A concentration of 0.1 ppm by weight in air is easily detected by the sense of smell. This level may be considered non-symptomatic in short term exposures, according to Goldsmith and Nagel (1969). Therefore ozone is, in a sense, self-policing. In addition, equipment used to generate ozone is protected by fail-safe devices. On the other hand, ozone loses its characteristic odour in the presence of other odours, especially at low concentrations. About 1960, the ACGIH (American Conference of Governmental Industrial Hygienists) reduced the threshold limit value (TLV) for ozone from 1 ppm to 0.1 ppm, and again in 1963 from 0.1 to 0.05 ppm. Those working with ozone should be trained in its use and detection.

Applications to human activities

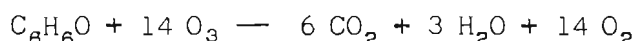
Ozone has been found useful in a number of applications related to human activities since its discovery by Schönbein in 1840. The first commercial use of ozone in a water sterilization plant occurred in 1893, when Schneller, Van der Sleen and Tindal constructed an industrial apparatus in Holland for the sterilization of Rhine water. The city of Paris first used ozonation in 1898; in 1936 there were more than 100 ozone installations in France. Extensive investigations on ozone production methods and applications subsequently were carried out by Siemens and Halske (1926a, 1926b). Ozone has been used commercially for more than 80 years for sterilization of drinking water (von Ermengen 1895). It is being used in Japan, the United States and Canada, but more particularly in Europe (Hann 1956; Whitson 1948) where some of the world's largest ozone treatment units are installed: London, U.K. was treating 15 mgd (million gallons per day) in 1933; Düsseldorf, Germany has processed 28 mgd since 1952; Paris, France has treated 176 mgd since 1969 [the total volume of ozonated water supplied each year in France is more than 1 billion m³ (265 billion gallons) serving approximately 8,000,000 people]; and the Midlands Michigan works, U.S.A. has treated 6.5 mgd since 1963. In Canada, 20 smaller facilities are in operation, and in the United States the number of ozone treatment units is increasing rapidly. In recent years the Soviet Union has increased its activities in ozone research for different applications. In cooperation with the Compagnie Generale des Eaux (France) the USSR plans to install the world's largest ozone generator in a plant in Moscow. Today, more than 500 municipalities in 50 countries use ozone as a disinfectant (Evans 1972).

In Europe, treatment of swimming pool water with ozone is quite common (Klein 1966, 1969; Havenmeister and Jentsch 1971; Overfield 1943; Flick 1969; Kurzman 1973). In the preparation of soft drinks (Schweppes Ltd.) water is disinfected using ozone, and then softened by ion exchange (Anon. 1956).

Ozone also has been proposed as a means of oxidizing cyanide wastes (Bauch and Burchard 1970; Walker and Zabban 1953; Sondak and Dodge 1961; Gabovich and Kurennoi 1966; Eiring 1969; Eisenhauer 1968, 1971). Cyanides are oxidized rapidly by ozone (Tyler et al. 1951; Niegowski 1956). The reaction can be summarized as follows:

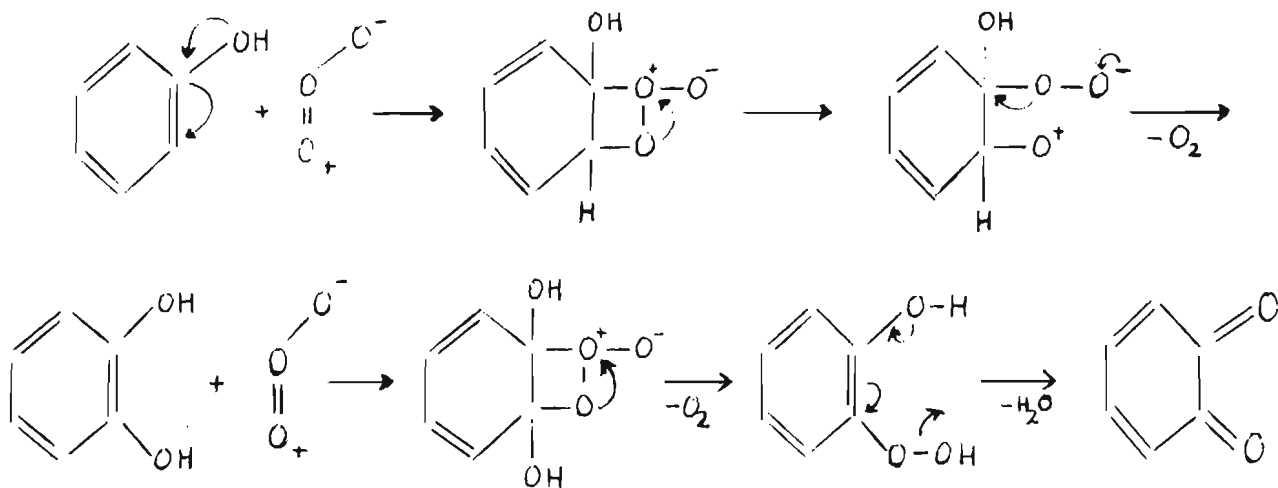


Niegowski (1956) and Eisenhauer (1971) have shown that phenols in petroleum waste waters from refineries are rapidly destroyed by ozone oxidation to form carbon dioxide, water and oxygen (see also Kandzas and Mokina 1973; Kandzas et al. 1971). The overall equation for complete breakdown of phenols can be considered as

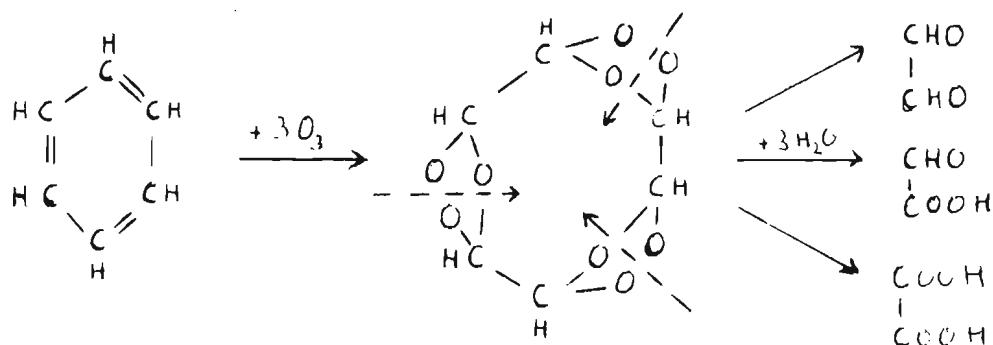


According to Eisenhauer (1971) the complete ozonization of phenol to water and carbon dioxide cannot be achieved economically, because of its high O_3 consumption.

The first step of phenol oxidation gives catechol, requiring 1.7 moles ozone per mole phenol. O-Quinone has been proposed as the second oxidation product. Eisenhauer (1971) suggests that 98% of the phenol content can be removed from waste water by using only 5 moles of ozone per mole phenol, if the second intermediate oxidation product is sufficient to satisfy pollution control requirements. The initial attack of ozone on phenols is illustrated by the following reaction scheme (after Eisenhauer 1971):

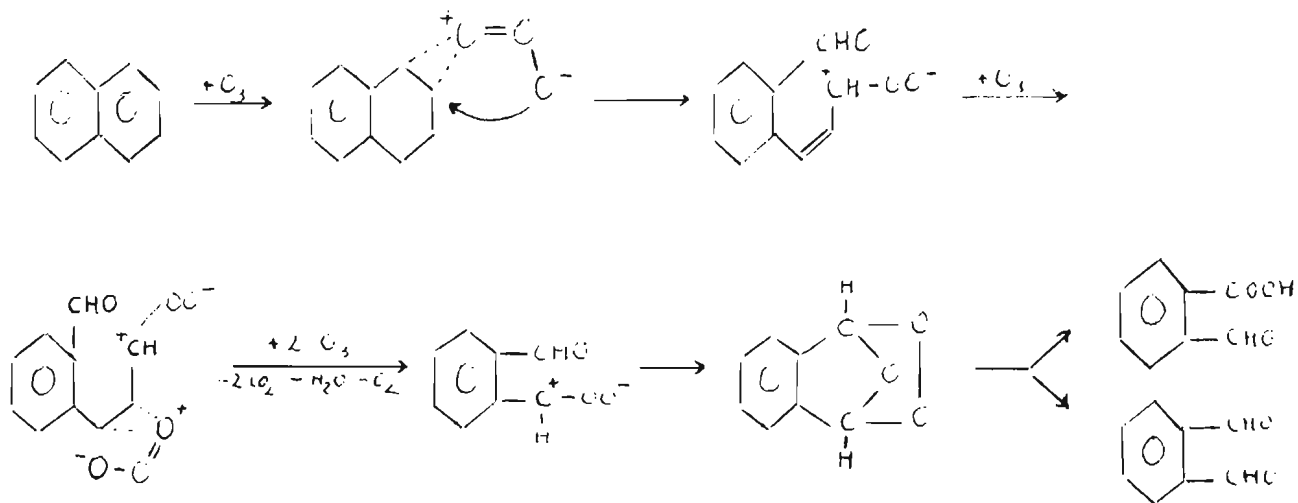


Ozonization was used to confirm the structural formula of benzol, predicted by Kékulé. The total breakdown of the ring structure, in principle, is comparable to the oxidation process occurring with phenols and can be summarized by the following scheme:



Hydrolytic cleavage produces the final reaction products glyoxal, glyoxylic acid and oxalic acid.

The reaction of ozone with higher aromatic compounds, e.g. Naphtalin-derivatives, results in separation of the ring systems. According to Bailey (1957) and Wibaut and Kampschmidt (1955), Naphtalin will be transferred into Phtalic acid:



In heterocyclic compounds (e.g. Indol) the initial attack of ozone occurs nearly almost in the ring system with the hetero atom.

Evans and Ryckman (1963) reported that ozonation of ABS (anionic surfactant) destroyed the biological inertness of the surfactant. The ozonide of ABS in solution probably serves as an oxidizable substrate for a mixed microbial population. In laboratory experiments ozone was found to be a highly efficient oxidant for the removal of certain chlorinated pesticides from waters (Buescher et al. 1964). The reactions of ozone with water-soluble inorganic compounds of certain divalent heavy metals are rapid and quantitative: Fe (II) and Mn (II) are oxidized stoichiometrically. Nitrite is quantitatively oxidized to nitrate, but ammonia remains unchanged (Gad and Columbus 1956). Ozone also can be used for destroying organics upstream of ion exchange processes, an important application since increasing pollution has caused operational problems in ion exchange systems.

In water sterilization and tertiary sewage treatment, ozone virtually was replaced by chlorine during the first half of this century because of higher production costs of ozone. An exception occurs in France, where ozonization was well established prior to the first World War and has continued to be an important means of sterilizing drinking water.

In more recent years interest is returning to ozone as an effective agent in water pollution control. New and more efficient production methods have been developed that reduce operational costs. Concurrently, the cost of chlorine has been increasing. Furthermore, the development of more efficient contacting procedures in water treatment has increased the efficiency of ozone utilization (Diaper 1965; Stopka 1970). Investigations by O'Donovan (1965) in Ireland indicate that ozonation has a definite place in future water treatment applications. As Rosen (1973) has pointed out, there are some advantages to be gained in using ozone instead of chlorine in wastewater treatment when the problems of proper gas-liquid contacting are solved. The chemical oxidation potential of ozone is twice that of the hypochlorite ion. Most of the reactions are rather rapid. Furthermore, in contrast to chlorine, ozone does not always have to be in solution before reaction is initiated (Hewes 1971; Rosen 1973). According to Bringman (1954) destruction of bacteria by ozone under test conditions was between 600 and 3,000 times more rapid than by chlorine. Wuhrmann and Meyrath (1955) found a concentration of 1 μg ozone/l to be sufficient for destruction of Escherichia coli. Recent investigations also have shown that ozone as a virtual control agent acts so rapidly that it is difficult to follow these reactions analytically. Finally, ozone leaves a beneficial oxygen residue as a product of the ozonation process; generally its reaction products are less toxic than those formed by chlorination. Very recently, Esvelt et al. (1973, J. Water Pollut. Control Fed. 45: 1558) have completed a study demonstrating that chloramines, which are produced by chlorination of municipal waste waters, are toxic to aquatic life.

Although ozonization techniques are very effective, it is surprising that as yet there is little rational and scientific basis for its practical application.

Findings of Hubbs (1930) and Doudoroff and Katz (1950) tended to discourage the use of ozone as a means of water treatment for aquaculture purposes. Benoit and Matlin (1964) were the first to state that a

reexamination of ozone could be useful as a means for disease control in American fish hatcheries. It can be assumed that ultraviolet lamps, commonly used in sea water sterilization for oyster purification units and supplies of sea water to public aquaria, produce some ozone directly in the water. As a matter of fact, ultraviolet radiation in air produces ozone, particularly in the very short ultraviolet range.

Furthermore, the earlier results of Salmon (1937), who used ozone successfully for sea water treatment in oyster purification, seem largely to have been overlooked. While aquaculturists showed no interest in ozone during the last decade, ozonization has become a most beneficial standard method for water treatment of home aquaria. Furthermore, several public sea water aquaria are utilizing ozone in combined recycling systems (Sander 1970). In Canada and in some other countries, ozone is used in sea water sterilization for control of shellfish disease in aquaculture pilot plants and in shellfish purification units. The author of this bibliography has used ozone successfully in sea water recycling systems for several years for rearing various marine fishes from larval stages to adults. Fisheries biologists increasingly are considering the application of ozone in water treatment and re-use systems. The current bibliography was assembled primarily with these applications in mind.

State of the literature

The literature on ozone is widespread. Thorp (1954) has given an excellent first summary of earlier literature on ozone to about 1954. The first part of his review lists some 266 references dealing with analytical methods for gaseous ozone, and covers a period of approximately 100 years. The second part of his review lists 980 patents related to ozone. These are separated into 52 groups dealing with such subjects as air conditioning systems, ozone generators, and ozone-resistant materials. Thorp believed that up to 1954 his lists were at least 90 per cent complete. Whereas Thorp's (1954) bibliography dealt primarily with engineering applications and patents, his 1955 review considers mainly uses in industrial medicine and surgery. Those seeking information on special applications of ozone in organic chemistry should see the review by Bailey (1958). Those seeking information on short or long term exposures to ozone, and their effects on human health, should see the reviews and reports by Thorp (1950, 1955), Stokinger (1965), Kelly and Gill (1965), Jaffe (1967), and Goldschmidt and Nagel (1969). Recently, several toxicological investigations were made in connection with ozone problems in high-altitude aircraft cabins during supersonic transport (Johnson, 1972).

After compiling this bibliography, the author was forced to conclude that the literature is rather unbalanced. For example, there are more than 100 publications available on determination of ozone in air, yet only a few are concerned with ozone determination in water. In addition the majority of studies of biological effects consider ozone as an air pollutant, and relate either to injury or metabolism in plants or to lung function in mammals. Only a few papers deal with biological effects of ozone on aquatic organisms.

With respect to water treatment, the majority of publications are concerned with ozone as a sterilizing agent for drinking water; only a small minority describe trials using ozone in advanced water re-use systems. From his own experience the author believes that ozone can play an important role in water treatment re-use systems. In this regard there is an urgent need for further research and information on the proper application of ozone in water and wastewater treatment problems.

Current bibliography

This bibliography attempts to marshal the more important available literature on the biological effects and, to some extent, the chemical reactions of ozone. The current bibliography also includes selected references in related fields of interest, including the general chemistry of ozone, effects of ozone on materials, and toxicity to humans.

Some arbitrary decisions were made with respect to those references which would be excluded. The bibliography excludes texts on water treatment practice in which ozone application is mentioned only briefly. Most of the literature dealing with ozone in air, its distribution and its occurrence in air pollution, has been omitted. Where retained, such references contain useful information on methods of determination of ozone or data on its biological effects. Furthermore, the more recent literature was given the major attention, considering the fact that Thorp's earlier (1954, 1955) bibliographies are available. The current bibliography is estimated to cover about 85 per cent of the literature available on the biological effects of ozone and its application in water treatment, excluding patent information, up to October-December, 1973. Out of about 1350 references included, only 280 were published earlier than 1954. About 30 per cent of the titles included were seen by the author as originals. Those references checked personally by the author include, as far as possible, reference numbers to the various abstraction services in which abstracts may be found.

The following bibliography is in two parts. The first part is an Author Index in alphabetical order, with each entry assigned a serial number. The second part is a Subject Index. Under the key words in the Subject Index will be found the serial numbers referring to the full references in the Author Index.

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SUBJECT INDEX

1. Preparation and generation of ozone (ozonizers)

44, 88, 96, 101, 114, 139, 167, 171, 212, 237, 238, 239, 240, 243, 257, 269, 286, 358, 390, 397, 399, 400, 444, 452, 474, 481, 499, 531, 575, 586, 610, 620, 623, 624, 632, 648, 652, 653, 739, 740, 742, 763, 772, 787, 820, 866, 867, 884, 885, 903, 935, 936, 941, 942, 945, 981, 991, 1011, 1014, 1015, 1034, 1044, 1058, 1059, 1065, 1075, 1076, 1107, 1116, 1130, 1131, 1138, 1144, 1173, 1175, 1176, 1214, 1221, 1229, 1247, 1251, 1278, A13, A27, A60

2. Determination of ozone

2.1. in air

14, 16, 20, 21, 39, 42, 62, 69, 75, 100, 105, 109, 116, 122, 123, 124, 125, 127, 129, 137, 144, 158, 159, 160, 161, 162, 166, 206, 207, 231, 278, 285, 287, 295, 318, 319, 320, 329, 339, 364, 398, 407, 423, 468, 469, 470, 471, 477, 478, 479, 502, 515, 534, 549, 550, 551, 568, 638, 659, 660, 661, 700, 701, 702, 721, 722, 732, 738, 747, 775, 789, 857, 858, 922, 924, 932, 959, 960, 968, 1000, 1005, 1007, 1008, 1009, 1040, 1063, 1065, 1081, 1096, 1097, 1098, 1099, 1100, 1106, 1170, 1173, 1201, 1230, 1242, 1249, 1253, 1320, A7, A19, A25, A32a, A37, A59

2.2. in the presence of interfering gases

14, 206, 232, 478, 556, 611, 1031, 1057

2.3. in water

126, 251, 409, 535, 557, 583, 664, 667, 897, 1045, 1243, 1277, 1314, 1315, A34

3. Physical-chemical properties

3.1. properties in general

25, 46, 68, 70, 91, 95, 101, 141, 185, 209, 288, 311, 383, 384, 525, 526, 536, 627, 744, 772, 872, 904, 1079, 1125, 1173, 1252, 1287

3.2. chemical structure and molecular weight

421, 620, 717, 1041, 1129, A68

3.3. solubility in aqueous solutions

548, 859, 893, 949, 954, 985

3.4. decomposition

3.41. in air

97, 180, 316, 328, 457, 458, 527, 544, 545, 588, 606, 851,
918, 982, 998, 1032, 1215, 1262, 1289, 1291, 1292, 1293,
1313, A9

3.42. in aqueous solutions

2, 3, 12, 250, 386, 443, 625, 640, 757, 818, 1038, 1132,
1139

3.5. interactions with elementary oxygen

5, 65, 89, 411, 417, 418, 430, 627, 716, 729, 847, 848, 849,
850, 851, 1258, 1261, 1263, 1288

3.6. interactions with other gases

91, 143, 168, 169, 222, 232, 256, 269, 527, 759, 798, 918, 1101,
1127, 1128, 1282, A1, A20, A52

4. Chemical reactions of ozone

4.1. chemical reactions in general

50, 62, 111, 140, 142, 143, 241, 294, 336, 337, 338, 569, 765, 1077,
1093, 1103, 1166, 1174, 1248, 1284, A55, A57, A62

4.2. ozonolysis and ozonolysis mechanisms

59, 61, 110, 210, 235, 314, 374, 376, 377, 378, 379, 380, 381, 453,
454, 595, 621, 626, 725, 730, 733, 836, 846, 854, 871, 905, 952,
964, 967, 1068, 1069, 1118, 1119, 1120, 1122, 1177, 1267, 1273, A57

4.3. ozonides

38, 84, 215, 236, 244, 245, 280, 513, 528, 725, 727, 734, 735, 852,
853, 939, 943, 1082, 1083, 1095, 1121, 1222

4.4. organic compounds

4.41. organic compounds in general

60, 67, 72, 80, 138, 197, 448, 500, 506, 512, 532, 567, 713,
838, 839, 842, 846, 875, 927, 928, 940, 955, 956, 957, 958,
1092, 1104, 1149, 1167, 1276, A10, A18, A53, A71

4.42. aliphatic compounds

4, 56, 57, 58, 61, 66, 170, 198, 223, 234, 302, 380, 431,
501, 521, 553, 584, 684, 745, 746, 794, 887, 907, 953, 983,
1028, 1029, 1030, 1074, 1102, 1219, 1259, 1267, 1279, 1280,
1281, 1297, A12, A17, A42

4.43. cyclic and semicyclic compounds

1, 49, 51, 52, 54, 55, 59, 140, 142, 203, 291, 314, 513,
635, 882, 1002, 1004, 1054, 1266, 1272, 1273, 1274, 1275,
A11, A63

4.44. oils

192, 279, 280, 296, 297, 769

4.5. inorganic compounds

4.51. inorganic compounds in general

741, 1095, 1216, 1227, 1233, 1235, 1262, 1265, 1290

4.52. heavy metals

207, 597, 731, 784, 837, 995, 1027, 1039, 1073, 1234, 1244

4.6. in aqueous solutions

3, 130, 250, 289, 290, 340, 384, 385, 408, 459, 460, 528, 558,
612, 666, 697, 933, 1135, 1139, 1151, 1152, 1153, 1260, 1303,
A41, A42, A47

5. Applications of ozone

5.1. water treatment

5.11. water treatment in general

27, 33, 34, 43, 81, 112, 113, 119, 120, 194, 242, 252, 281,
282, 341, 406, 426, 445, 449, 480, 494, 495, 558, 559, 564,
582, 619, 630, 650, 656, 665, 666, 671, 672, 674, 676, 689,
708, 762, 764, 776, 819, 834, 863, 883, 938, 948, 950, 987,
989, 990, 991, 992, 996, 1010, 1025, 1049, 1053, 1123, 1133,
1154, 1158, 1165, 1232, 1239, 1241, 1268, 1269, 1300, 1301,
A4, A28, A33, A35, A44, A48, A61, A70

5.12. drinking water (sterilization)

27, 28, 82, 85, 98, 99, 153, 173, 174, 193, 194, 208, 300, 313, 321, 396, 401, 403, 404, 405, 406, 424, 440, 441, 456, 466,, 467, 472, 473, 475, 476, 491, 496, 498, 507, 554, 555, 565, 581, 584, 587, 589, 609, 639, 641, 658, 680, 682, 683, 690, 691, 714, 719, 758, 774, 874, 879, 880, 881, 896, 908, 909, 921, 923, 944, 947, 965, 966, 969, 970, 971, 1017, 1018, 1020, 1021, 1022, 1046, 1047, 1060, 1061, 1062, 1072, 1084, 1085, 1091, 1134, 1159, 1178, 1203, 1223, 1226, 1236, 1237, 1245, 1271, 1296, A15, A26, A46, A51, A54, A64, A65, A66

5.13. swimming pools

151, 322, 375, 517, 646, 647, 692, 892, 1080, 1246

5.14. aquarium techniques

31, A72

5.15. tertiary waste treatment

30, 86, 120, 121, 157, 252, 267, 342, 387, 405, 410, 462, 463, 465, 485, 537, 538, 574, 580, 642, 643, 645, 675, 681, 724, 766, 770, 786, 822, 826, 827, 864, 910, 986, 999, 1052, 1134, 1159, 1179, 1294, 1299, 1318, A3, A16, A45, A49, A69

5.16. industrial wastes (general)

81, 90, 107, 419, 613, 614, 668, 695, 723, 736, 771, 797, 1105, 1117, 1124, 1142, 1150, 1224, 1295, 1302, A2

5.161. phenols

83, 325, 326, 402, 447, 487, 488, 497, 547, 617, 726, 870, 908, 915, 1048, A14, A29, A39

5.162. cyanides

29, 32, 45, 324, 579, 636, 637, 1090, 1218, A5, A6, A43, A58

5.163. coke work effluents

486, 844, 994

5.17. degradation of pesticides, detergents, and detoxication of heavy metals

149, 156, 157, 323, 342, 402, 615, 678, 694, 773, 784, 788, 878, 993, 1027, 1039, 1225, 1244, 1270, A21

5.18. treatment of sea water

113, 517, 892, 946, 1003, A73

5.19. reactions with activated charcoal

213, 655, 687, 1010

5.2. non-water treatment applications

5.21. food storage

26, 176, 181, 246, 348, 349, 350, 425, 503, 508, 687, 688, 877, 1026, 1255

5.22. medical science and surgery (including sterilization of air)

40, 41, 70, 78, 177, 179, 199, 201, 204, 244, 245, 284, 302, 327, 330, 367, 372, 389, 390, 429, 446, 451, 590, 607, 608, 663, 728, 833, 1071

5.23. bleaching of materials (textiles, pulp)

150, 293, 294, 510, 511, 790, 1036, 1088, 1089, A24

5.24. others

13, 106, 154, 172, 216, 303, 331, 509, 698, 831, 919, 946, 1250

6. Biological effects

6.1. general references on toxicity of ozone

197, 298, 317, 351, 365, 392, 427, 437, 450, 546, 573, 596, 785, 799, 861, 894, 1012, 1013, 1023, 1110, 1111, 1113, 1171, 1172, 1174, 1204, 1256, A56

6.2. antioxidants; resistance to and protection against ozone

200, 226, 260, 332, 374, 437, 570, 631, 749, 808, 832, 895, 912, 913, 974, 1056, 1111, 1181, 1197

6.3. cell structures and cell membranes (permeability)

305, 346, 347, 357, 368, 369, 370, 391, 673, 699, 756, 873, 889,
1147, 1168

6.4. phytotoxicity (plant injury)

6.41. plant injury in general

6, 9, 10, 37, 38, 102, 117, 128, 131, 132, 134, 135, 136,
175, 182, 183, 211, 224, 225, 226, 228, 229, 230, 253, 254,
260, 263, 266, 334, 361, 362, 363, 368, 369, 420, 518, 519,
520, 523, 539, 542, 543, 563, 572, 603, 631, 670, 703, 710,
778, 779, 780, 781, 782, 806, 821, 823, 843, 890, 891, 925,
931, 962, 963, 972, 973, 975, 976, 978, 1035, 1043, 1115, 1155,
1156, 1160, 1162, 1169, 1180, 1182, 1184, 1185, 1188, 1193,
1195, 1208, 1257, 1283

6.42. beans

8, 186, 248, 308, 333, 343, 394, 522, 552, 571, 761, 777,
911, 912, 913, 925, 976, 977, 1037, 1168, 1183, 1187, 1200,
1205, 1257

6.43. pinos

73, 74, 103, 104, 117, 118, 217, 218, 219, 220, 221, 264,
265, 292, 344, 345, 720, 824, 825, 979, 1064

6.44. tobacco

7, 133, 268, 360, 413, 522, 524, 704, 705, 706, 707, 711,
712, 749, 750, 751, 752, 753, 754, 802, 803, 804, 805, 807, 808,
809, 810, 811, 812, 813, 814, 815, 841, 961, 1094, 1181,
1198, 1217

6.45. plant metabolism (growth, respiration)

74, 93, 187, 188, 229, 307, 309, 335, 343, 750, 911, 1156,
1186, 1190, 1192, 1217

6.46. photochemistry (photosynthesis)

117, 118, 262, 271, 335, 541, 1192, 1194

6.47. biochemistry

73, 186, 187, 225, 227, 248, 261, 306, 312, 394, 704, 706,
815, 840, 888, 1183, 1191, 1196, 1199, 1200

6.5. viruses, bacteria, fungi, lichens, Protozoa

41, 94, 112, 145, 146, 177, 178, 181, 189, 204, 259, 267, 271, 272, 273, 274, 282, 302, 350, 371, 424, 482, 504, 519, 540, 560, 562, 585, 607, 608, 618, 628, 633, 634, 649, 685, 686, 693, 709, 770, 783, 801, 855, 868, 910, 923, 926, 937, 938, 1038, 1078, 1086, 1140, 1150, 1157, 1209, 1231, 1238, 1250, 1254, 1255, 1286, 1296, 1322, A23, A40, A73

6.6. aquatic organisms (other than 6.5)

94, 113, 146, 271, 272, 273, 274, 282, 298, 604, A8

6.7. insects

87, 216, 422, 715, A67

6.8. fish hatcheries and oyster culture

94, 1003, A72

6.9. vertebrates and man

6.91. general

165, 356, 357, 435, 438, 561, 662, 679, 748, 791, 793, 835, 898, 899, 900, 901, 902, 1001, 1019, 1066, 1067, 1136, 1141, 1146, 1148, 1207, 1210, 1285, 1298, 1316, 1317, A30

6.92. blood

115, 164, 433, 434, 439, 743, 1137, 1202

6.93. lung physiology

17, 18, 19, 36, 80, 108, 152, 155, 195, 196, 205, 270, 276, 283, 299, 315, 352, 353, 356, 388, 393, 395, 412, 436, 455, 461, 489, 576, 577, 605, 792, 795, 796, 800, 829, 860, 862, 914, 920, 934, 984, 1056, 1087, 1112, 1145, 1164, 1206, 1211, 1212, 1213, 1228, 1264, 1304, 1305, 1306, 1307, 1308, 1309, 1311, 1321, A50

6.94. human health hazard

154, 179, 255, 370, 416, 429, 432, 450, 461, 489, 566, 578, 591, 592, 593, 594, 629, 696, 755, 816, 832, 833, 886, 1050, 1051, 1070, 1108, 1109, 1146, 1204, 1247, 1306, 1310, 1312, A30

7. Effects on materials

15, 23, 24, 71, 77, 163, 184, 191, 233, 249, 275, 442, 464, 516, 657,
845, 856, 997, 1016, 1163, 1220, 1297, 1319, A22, A32, A36, A38

8. Miscellaneous

39, 42, 69, 75, 92, 122, 162, 247, 366, 416, 514, 598, 677, 718, 1143,
1161, A31