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TECHNICAL REPORT ARCSL-TR-77080

OXIDATION OF PHOSSY WATER BY OZONE

by

Donald L. Campbell

Munitions Division

12

September 1977

SEP 30 1977

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
Chemical Systems Laboratory
Aberdeen Proving Ground, Maryland 21010

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This work was undertaken to establish the feasibility of using ozone to lower the elemental phosphorus level of phosphorus contaminated water at Edgewood Arsenal. Separate samples of this phossy water were treated with ozone at various mass rates in a sparged stirred reactor. The ozone repeatedly reduced the elemental phosphorus concentration below the limits of detection. Simple aeration also oxidized the white phosphorus but not at the rate nor to the extent that ozone showed. No correlation could be developed between elemental phosphorus lowering and

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20. ABSTRACT (Contd)

its extent of conversion between elemental phosphorus decreases and its extent of conversion to the phosphate form. A white precipitate comprised mainly of calcium phosphate formed as the phossy water was oxidized. Thus, preliminary data resulted which prove the feasibility of using ozone to treat phossy water and which will lead the way for further studies in this area.

PREFACE

The work described in this report was authorized by Project 5761336, Production Filling Equipment Technology. This work was started in October 1976 and completed in March 1977.

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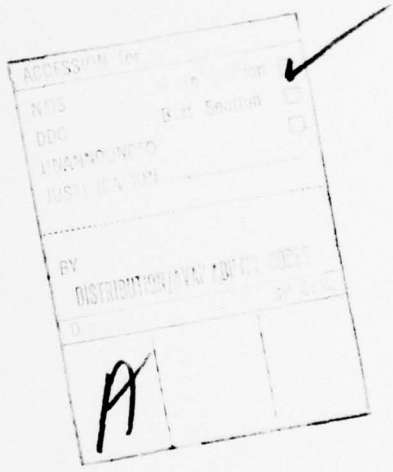

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OXIDATION OF PHOSSY WATER BY OZONE

I. INTRODUCTION.

A. Objective.

To evaluate the feasibility of using ozone to decrease the elemental phosphorus concentration of phossy water at Edgewood Arsenal.

B. Background.

White phosphorus (WP) fill operations have occurred on an intermittent basis for the past several years at Edgewood Arsenal. Phosphorus contaminated water (phossy water) from the WP plant is associated with WP transfer from melt tanks, the preparation of chemical mixtures, the filling of munitions, demilitarization work and cleanup operations. Approximately 20,000 gallons of this water is presently being stored in two 10,000-gallon tanks in building E5188. Further dry fill operations are expected to generate up to 40,000 gallons of additional phossy water per year.[1] This pending action necessitates the development of a treatment process for the disposal of the phossy water in an environmentally acceptable manner.

The Chemical and Plants Division in conjunction with the Mechanical Process Technology Division initiated the development of a tentative scheme for the treatment of phossy water and WP slag at Edgewood Arsenal. Dr. Henry Luh and Mr. John Bane, both with the Chemical Process Technology Branch, reported on 1 November 1976 that treatment of this phosphorus contaminated water with ozone should significantly lessen its elemental phosphorus level.[2] They further stated that there is a general lack of data available on ozonation of phosphorus in phossy water and recommended that a bench-scale study be performed to determine more exacting treatability information. No results were available at the time this report was prepared concerning ozone experiments at Pine Bluff Arsenal.

The purpose of this study was to develop the preliminary data needed to evaluate ozone as a practical means to reduce the elemental phosphorus level of phossy water. Several tests have been conducted in conjunction with this objective to determine the rate and extent of elemental phosphorus conversion to orthophosphate.

II. INVESTIGATION.

A. Equipment.

A batch reactor, support instrumentation, and associated piping were set up adjacent to analytical support in building E3640. The laboratory-scale reaction unit used in this study was a liquid-batch, continuously gas sparged five-liter vessel. This type of reactor permitted many data points to be obtained at intermittent times during a run. A schematic diagram of the apparatus is shown in figures 1 and 2. A pneumatic mixer was used which resulted in complete agitation at a

[1] Memorandum For Chief, Mech Proc Tech Div, 30 Nov 76, Subject: Expected Amount and Concentration of Phosphorus Contaminated Water to be Generated in Building E5188. Richard B. Belmonte.

[2] Bane, John M., and Luh, Ming D. Tentative Scheme for the Treatment of Phossy Water and White Phosphorus Slag. Manufacturing Technology Directorate. November 1976.

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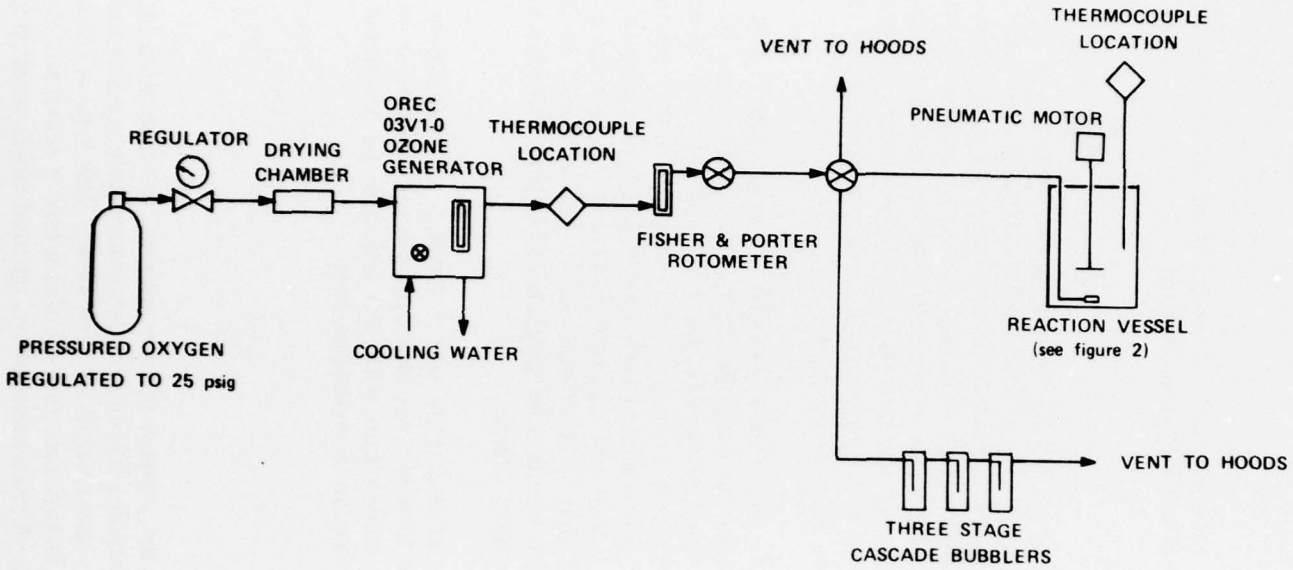

Figure 1. Flow Diagram of Apparatus for Ozonation Runs

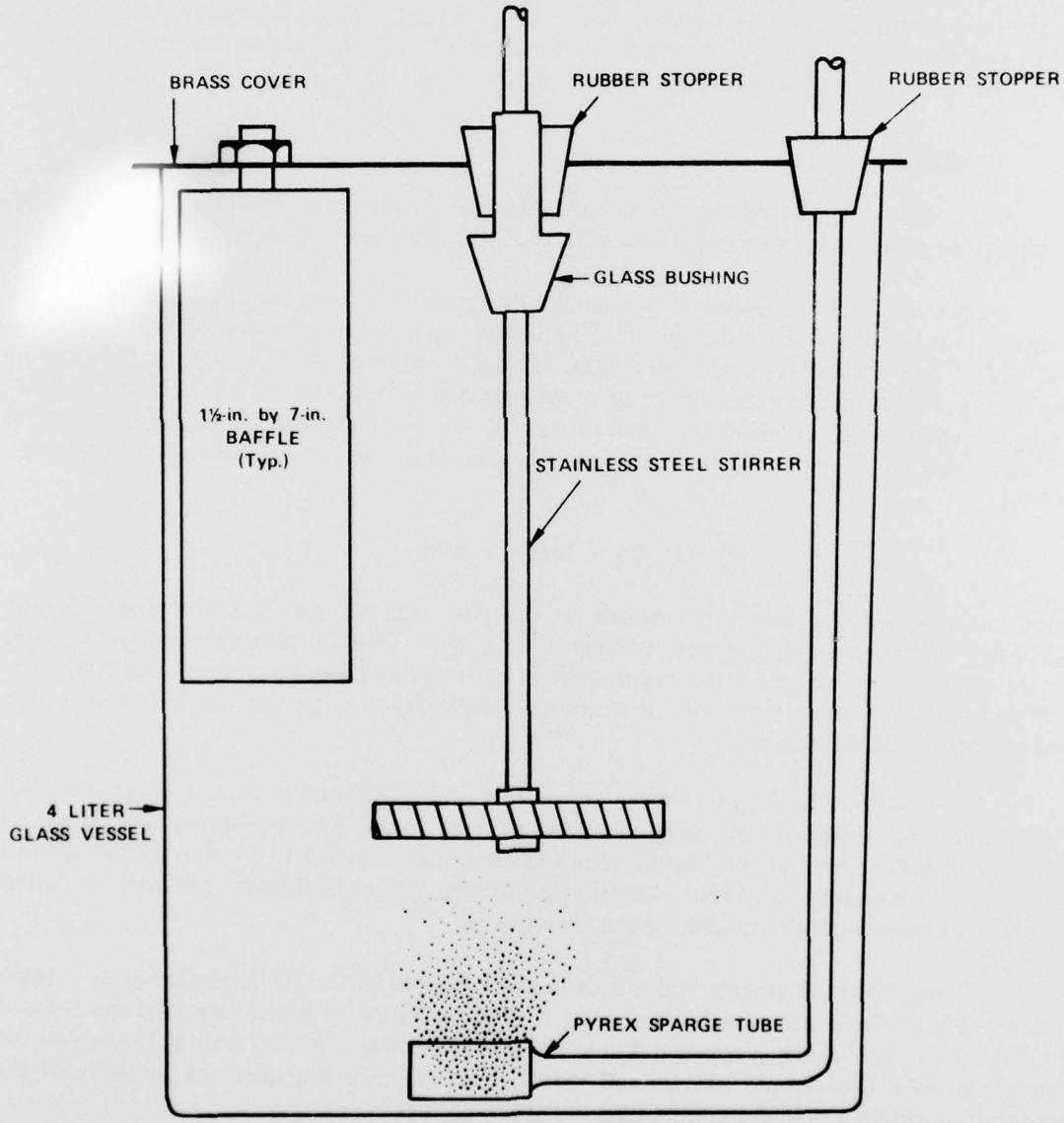

Figure 2. Reactor Configuration

rotational speed of 400 rpm. Two baffles were strategically placed to eliminate any vortexing action caused by rapid agitation. Automatic temperature monitoring was accomplished with thermocouples in combination with a multipoint recorder.

Ozone was produced from an Ozone Research & Equipment Corporation (OREC) Model 03V1-0 water-cooled generator. The rated capacity of the ozonator was 200 milligrams of ozone per hour using an oxygen feed. Output was adjustable by varying the primary current to the ultraviolet lamp. The unit was designed for operation at gas pressures from 0 to 5 pounds per square inch (gage). Ozone concentrations could be varied over the range of 0.2% to 0.8% by weight by varying the ratio of power input to feed gas flow rate. Upon calibration, the ozone dosage to the reactor was determined by the gas flow rate. The gas supplied to the ozone generator was dry commercial grade oxygen with a dew point around -50°F.

B. Experimentation.

The analytical methods used to detect elemental phosphorus, orthophosphate and total inorganic phosphorus are described in the appendix and will, therefore, not be repeated here.

Once an ozone generator is chosen and its capacity known, reactor sizing and piping needs can be identified. An ozone dosage of 170 milligrams per hours was assumed to be adequate for this study based on the typical output curve included with the OREC ozonator. This should have resulted in 0.46 weight percent ozone at an oxygen/ozone flow rate of 0.4 liters per minute. Assuming a 10-ppm level of elemental phosphorus in any one test sample and 60 minutes of reaction time needed to react all of the ozone in association with the stoichiometric limiting equation:

$$P_4 + 10\ O_3 \rightarrow 2P_2O_5 + 10\ O_2, \qquad (1)$$

a 4.4-liter reaction volume would be needed. A reaction such as this does not progress along stoichiometric lines in most circumstances because the ozone/elemental phosphorus contact is not adequately intimate. By designing the experiment to allow 60 minutes for a 1-to-1 stoichiometric ratio, up to twice the amount of ozone that is needed is allowed to enter the reaction vessel within the time constraints of the test run.

The actual output curve of the ozonator varied substantially from the data supplied with the unit. At 0.4 liters of oxygen/ozone mixture only 110 milligrams per hour of 0.30-weight-percent ozone were produced. This variation was assumed to be due to the age and condition of the ozonator itself. The calibration procedure, however, showed a consistent output that would not compromise the validity of the experiment.

Twenty liters of phossy water were taken from one of the 10,000-gallon storage tanks and isolated in a nitrogen atmosphere to prevent further oxidation of WP. Analysis of this isolated sample yielded 350 ppb of WP, a considerably lower value than was expected from past analyses. So as to not lose time, this sample was treated with ozone to check not only the integrity of the apparatus but to obtain a basis for comparison of further studies.

Procedure.

1. Four liters of phossy water were purged from the sealed container into the glass reactor.

2. An initial sample of 250 milliliters was taken for analysis.

3. A precalibrated ozone stream was sparged into the reactor, the pneumatic stirrer adjusted, and the reaction time started.

4. Analytical test samples of 100 milliliters each were taken at 5, 15, 30, 60, and 120 minutes into the reaction and analyzed for elemental phosphorus, orthophosphate and total inorganic phosphorus. The intermediate phosphorus oxide concentration level was determined by subtracting the following phosphorus equation:

$$\text{Total} - \text{ortho} - \text{elemental} = \text{intermediates}. \qquad (2)$$

5. At the end of the run, the ozone stream and pneumatic stirrer were stopped and the vessel was emptied into a 4-liter storage flask.

A second 20-liter sample of phossy water was taken from the other 10,000-gallon storage tank in hopes of obtaining a higher concentration of elemental phosphorus. The sample was drawn with minimal turbulence and at a level closer to the white phosphorus slag in the bottom of the tank. As many precautions as possible were taken to obtain an elemental phosphours concentration closer to the expected level of 10 ppm. Again the analytical tests yielded a considerably smaller value, this time only 250 ppb. The funds and time that remained for this study were running low so it was decided that the experiments would have to be concluded with the latest sample.

The same procedure was used that was outlined above but this time two runs were executed. The first run of this last series used ozone at a higher mass rate than before. The final run tested simple aeration, no ozone, as a practical means to treat phossy water. A basis for comparison of ozone versus aeration was thus obtained.

C. Results.

The data obtained from the series of phossy water runs are summarized in the following three tables. A discussion of these results is provided in section III of this report.

Table 1. Phossy Water Run 1

Sample number	Contact time	Elemental phosphours	Ortho-phosphate	Total phosphorus	Temperature of gas	Temperature of liquid
	minute	ppb as P	ppm as P	ppm as P	°C	°C
1a	0	1392	380	NA	35	26
1b	5	868	380	NA	35	27.5
1c	15	488	400	NA	35	27
1d	30	188	400	NA	35	28.5
1e	60	46	410	NA	35	31
1f	120	<22	400	NA	35	35

NOTE: NA = Not available; oxygen/ozone feed rate = 0.4 liters/min; ozone concentration = 110 mg/hr; ozone weight percent = 0.30; stirrer RPM = 370.

Table 2. Phossy Water Run 2

Sample number	Contact time	Elemental phosphorus	Ortho-phosphate	Total phosphorus	Temperature of gas	Temperature of liquid
	minute	ppb as P	ppm as P	ppm as P	°C	°C
2a	0	836	290	280	35	26
2b	5	600	290	280	35	27
2c	15	280	280	290	35	28
2d	30	60	290	280	35	29
2e	60	<22	290	280	35	32
2f	120	<22	280	290	35	35

NOTE: oxygen/ozone feed rate = 0.6 liters/min; ozone concentration = 150 mg/hr; ozone weight percent = 0.26; stirrer RPM = 400.

Table 3. Phossy Water Run 3

Sample number	Contact time	Elemental phosphorus	Ortho-phosphate	Total phosphorus	Temperature of gas	Temperature of liquid
	minute	ppb as P	ppm as P	ppm as P	°C	°C
3a	0	904	NA	NA	34	27
3b	5	824	NA	NA	34	27
3c	15	708	NA	NA	34	29
3d	30	540	NA	NA	34	30
3e	60	268.8	NA	NA	34	32
3f	120	82.8	NA	NA	34	34

NOTE: NA = Not available; oxygen/ozone feed rate = 0.4 liters/min; ozone concentration = 0.0 mg/hr (aeration); ozone weight percent = 0.0; stirrer RPM = 360.

III. DISCUSSION.

A. Elemental Phosphorus Oxidation.

Ozone appears to be a quite effective means to reduce the elemental phosphorus concentration of phossy water. As can be seen from figures 3 and 4 which represent the results of the two ozone runs, the white phosphorus concentration is repeatedly reduced to below detectable limits. There seems to be a correlation between ozone input rate and contact time needed to bring the reaction mixture below the detection limit. A tradeoff of these two parameters to determine optimal ozone dosages should be accomplished in further studies.

Table 3 shows that aeration also reduces the elemental phosphorus concentration of phossy water. However, the effectiveness of this treatment did not equal that of ozone. Figure 5 has been developed to vividly depict this difference. The vertical scale has been recomputed to "percent removal" due to the variations of the initial elemental phosphorus concentrations. The figure shows that aeration will reduce the WP level up to 90 percent. The efficiency of this treatment seems to level off at this point. On the other hand, ozonation removes the white phosphorus quicker and will essentially take out all the elemental phosphorus within 120 minutes of contact time.

Neither twenty-liter sample of phossy water taken from the two 10,000-gallon storage tanks showed a concentration of elemental phosphorus close to the expected level of 10 ppm. It is assumed that enough agitation of the samples occurred during withdrawal, isolation, and transportation to have partially oxidized the white phosphorus. Improved sampling techniques will have to be employed to evaluate further the impact of ozone on phossy water. It is assumed that increased levels of white phosphorus will just lengthen the contact time necessary to bring the concentrations below detectable limits.

B. Reaction Products.

The conversion of elemental phosphorus to its phosphate form, PO_4^{-3}, progresses with many intermediate oxide byproducts being formed along the way:

$$P_4 \rightarrow H_2PO_2^{-1} \rightarrow HPO_3^{-2} \rightarrow PO_4^{-3} \qquad (3)$$

This study not only attempted to quantify the oxidation of white phosphorus by ozone but also correlate the extent of its conversion by observing the reaction products. The first phase of this objective was accomplished satisfactorily and discussed in the preceding section. However, the analytical test procedures for the second phase were not sensitive enough to detect any appreciable changes in the orthophosphate concentration levels throughout the experiment. Expressed as P, the elemental phosphorus was being reduced by approximately 1400 ppb or 1.4 ppm. If this conversion was taken completely to PO_4^{-3}, there theoretically should be an increase in PO_4^{-3} levels of 1.4 ppm. The precision of the analytical procedures used was only plus or minus 5 ppm. Therefore, no correlation was possible.

It is noteworthy to look at table 2 and notice that the orthophosphate and total inorganic phosphorus levels remain essentially constant throughout the run. This reinforces the belief that conversion of elemental phosphorus to phosphate is essentially complete and that the phosphate compound comprises the majority of the mixture.

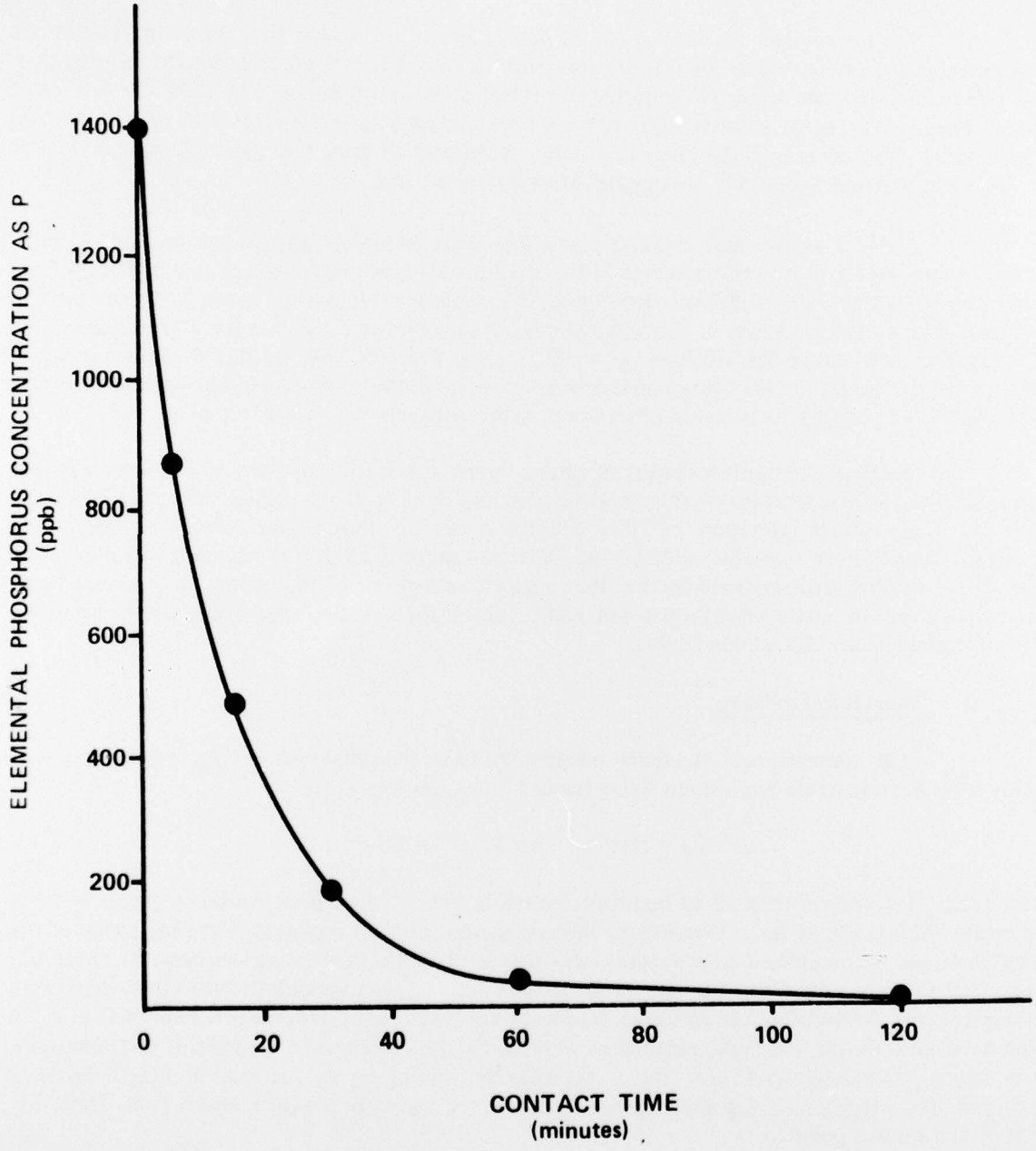

Figure 3. Elemental Phosphorus Decrease in Run 1

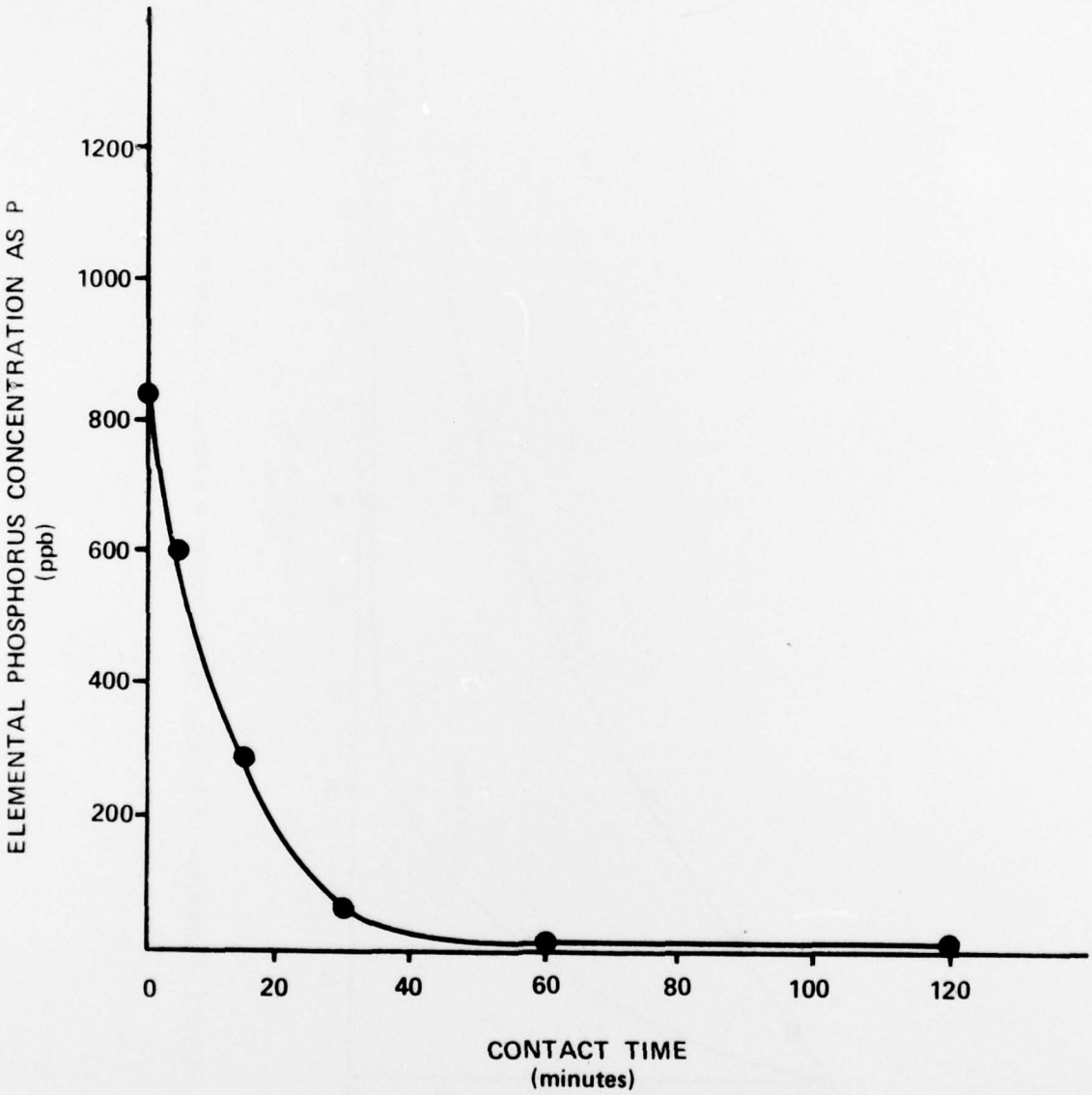

Figure 4. Elemental Phosphorus Decrease in Run 2

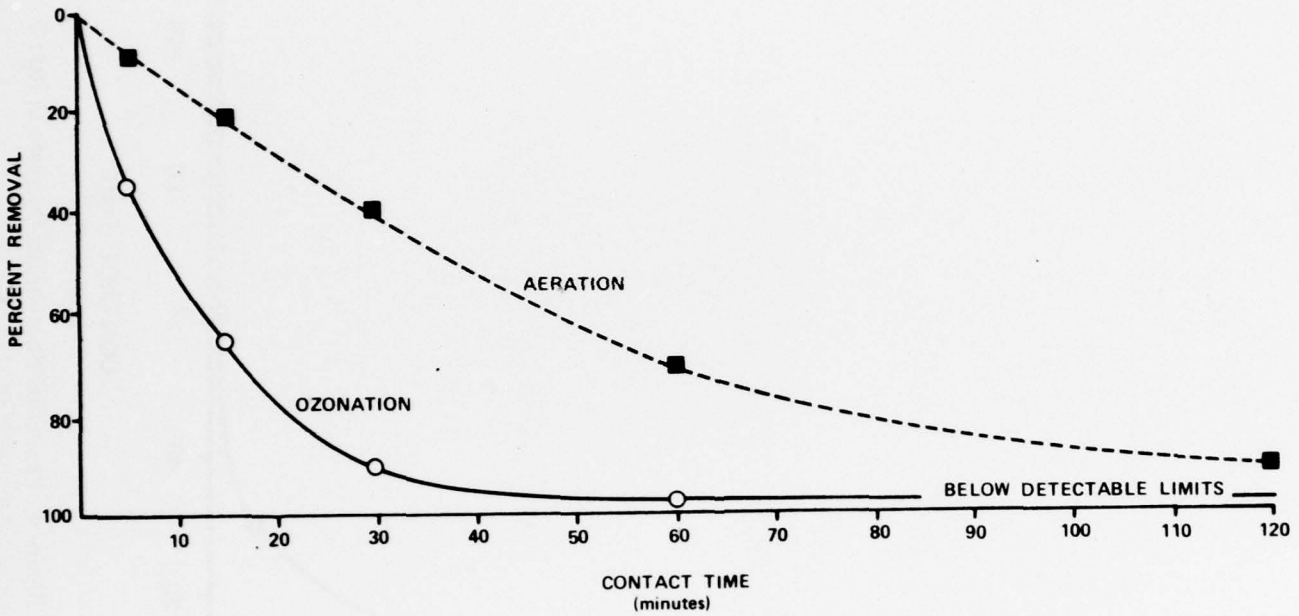

Figure 5. Comparison of Aeration with Ozonation as a Means to Oxidize the Elemental Phosphorus

An unexpected fine white precipitate formed during the three runs of this study. This white solid formed in the reaction batch immediately upon introduction of the ozone feed and became more predominate as the reaction progressed. It tended to cause the mixture to become more viscous as noted by the gradual decrease of the agitator speed. Due to its fine amorphous structure, a large settling time was needed to decant the mixture to a clear solution. This white solid was found to be made up primarily of calcium phosphate, $Ca_3(PO_4)2$. Concentrations ranged anywhere from 0.38 to 0.41 milligrams of solid per milliliter of solution for the aerated and ozonated runs, respectively. The same white solid has been shown to be present at the bottom of the two 10,000-gallon phossy water storage tanks. A closer analysis is necessary to assess the quantity of this material and to determine if its presence will inhibit disposal of the WP slag.

IV. CONCLUSIONS.

1. Ozone has been shown to be a practical means to reduce the elemental phosphorus concentration of phossy water. Reductions of WP to below detectable limits are possible with this method. Optimal dosages of ozone and length of time needed for contact will have to be determined in later studies.

2. Aeration has shown limited effectiveness in reducing the elemental phosphorus concentration of phossy water. Reductions up to 90 percent were noted. Ozone appeared to be much quicker and more effective in oxidizing the white phosphorus.

3. No correlation could be developed between white phosphorus lowering and its extent of conversion to the phosphate form. Either greater initial concentrations of elemental phosphorus or improved analytical procedures must be developed to make this correlation.

4. A white amorphous precipitate formed as the elemental phosphorus was oxidized. A phosphate complex was formed with the calcium ions of the phossy water. Concentrations as great as 410 milligrams per liter were found. The majority of the slag in the bottom of the two 10,000-gallon phossy water storage tanks may be comprised of this substance.

5. Sampling techniques limited the maximum concentration of elemental phosphorus that could be tested to 350 ppb (1400 ppb expressed as P). It is foreseen that considerably greater concentrations will not affect the validity of this report's conclusions.

V. RECOMMENDATIONS.

1. Further research of advanced techniques to increase the reactivity of phosphorus towards ozone should be investigated. Substantial quantities of photochemical energy have been shown to be imparted to a reaction mixture with the addition of ultraviolet (UV) irradiation. Because of the interaction between the UV light and the electronically excited molecules, reactions occur much quicker and to a greater extent than nonirradiated samples.

2. Improved analytic procedures for the determination of total inorganic phosphorus and orthophosphate concentrations in water should be investigated. Precision in the range of plus or minus 0.5 ppm would be desirable.

3. A Cubic Corporation Hydrocube Wastewater Treatment system available through MERADCOM, Ft. Belvoir, Virginia, should be transferred to Edgewood Arsenal. This unit

which includes a 6-gram-per-hour ozone generator, would be set up and used to test the pilot-scale feasibility of using ozone to treat the phossy water being stored in the two 10,000-gallon storage tanks. Near optimal operating conditions would be developed through an associated study.

4. The two 10,000-gallon storage tanks in Building E5188 at Edgewood Arsenal should be sampled more completely to determine their contents. The disposal techniques to be used for the phossy water and WP slag would then be better defined.

APPENDIX

ANALYTICAL PROCEDURES

1. Elemental Phosphorus Determination.

Several practical methods exist to determine the concentration of elemental phosphorus in water.[1] A highly sensitive method was needed for this study to evaluate fully the impact of ozone on the phosphorus in the phossy water. Neither the molybdenum blue nor the phosphovanadomolybdate methods detect any appreciable changes in the elemental phosphorus levels throughout the ozonation runs. Hence, the vapor phase chromatographic (Vpc) method was used on apparatus set up in the Product Assurance Directorate. Extremely accurate and reproducible data resulted throughout the study by using this analytical procedure.

Method. An aliquot of the phossy water was shaken with 5 milliliters of isooctane for one minute immediately upon withdrawal from the reaction vessel. The layers were allowed to settle, and a portion of the isooctane layer was drawn off for analysis on a gas chromatograph with a flame ionization phosphorus detector. Aliquot ratios of 5:1 or 10:1 were used to produce a detection limit of 1 to 5 ppb.

2. Orthophosphate Determination.

The orthophosphate phosphorus concentration in phossy water was determined by the phosphovanadomolybdate method.[2] This analysis is a colorimetric method in which the phosphorus concentration is estimated from the intensity of yellow color which develops with treatment of the samples with color-forming reagents. Only step I is used from the inclosure to make this determination.

3. Total Inorganic Phosphorus Determination.

The total inorganic phosphorus concentration in the phossy water was determined by the phosphovanadomolybdate method.[2] This analysis is a colorimetric method in which the phosphorus concentration is estimated from the intensity of yellow color which develops with treatment of the sample with dilute acid and color-forming reagents. Steps I and II were used from the inclosure to make this determination.

---

[1] Ash Stevens, Inc. Chemical Process Studies for Commercially Unavailable Compounds. Final Report (Comprehensive). Contract DAAA15-69-0-0584, page 32. 1973.

[2] Valis, Robert, Vigus, Ellen. ARCSL-TR-77023 (in printing). Pyrolysis of Detoxified Agent Waste: Part I, Spray Dried GB Salts. Appendix B. September 1977.

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