



STUDIES ON THE CHEMISTRY OF ORTHOPHOSPHATE AND  
POLYPHOSPHATE REMOVAL WITH FERRIC CHLORIDE

A Preliminary Report

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### Abstract

The chemistry of inorganic phosphorus precipitation with ferric chloride has been investigated. The effects of several variables, such as pH, coagulant dosage, and initial phosphorus concentration on phosphorus removal with ferric chloride have been determined.

Orthophosphate and the inorganic polyphosphates, pyrophosphate and tripolyphosphate, are removed in proportion to the amount of ferric chloride added. The removal of phosphates with Fe(III) is highly pH dependent and appears to occur via the formation of insoluble ferric phosphate complexes. The point of minimum solubility, hence maximum phosphorus removal, occurs in the region of pH 5.3. However, it has also been shown that the addition of calcium to the system extends the useful pH range for phosphorus removal into the mildly alkaline region. Simultaneously, calcium enhances the removal of phosphorus with Fe(III) by aiding in the formation of a more settleable floc.

## Studies on the Chemistry of Orthophosphate and Polyphosphate Removal with Ferric Chloride

### Introduction

In the past decade, the problem of accelerated eutrophication of our natural waters has been the subject of intensive discussion and investigation. The increased biological proliferation in lakes, ponds, and other surface waters has been manifested mainly by vast growths of algae and aquatic vegetation, and is often accompanied by the production of obnoxious odors, tastes, colors, and other attendant problems. The resultant deterioration of the water source can render it unfit for domestic, industrial, or recreational use.

Although eutrophication occurs naturally, it has been well documented (U. S. Dept. of H. E. & W., 1961) that an increased input of nutrients can accelerate this process and produce nuisance conditions in previously unblemished lakes. The major nutrients that have been associated with this accelerated eutrophication are nitrogen and phosphorus (Sawyer, 1952).

As a step in preventing or controlling accelerated eutrophication, it is thought that the removal of these nutrient materials from domestic wastewater effluents would be advantageous. Attention has been focused chiefly on phosphorus removal since nitrogen may become available to natural waters through the process of nitrogen fixation by plants and microorganisms, including several species of blue-green algae

(Jackson, 1967).

A variety of techniques have been investigated and proposed for removing phosphates from wastewaters. Biological methods, based on the control of aeration parameters in secondary treatment, have been shown to be capable of effecting limited phosphorus reductions (Connell, et al., 1967). Chemical methods, including ion exchange, lime precipitation, and coagulation with aluminum or ferric salts have been successful in removing up to 99<sup>+</sup> percent of the phosphates from sewage effluents (Martin and Weinberger, 1966). It is generally thought that some form of chemical coagulation, either as a separate tertiary treatment step, or in combination with secondary biological treatment, would be the most effective and economical means of phosphorus reduction (Nesbitt, 1966).

Although chemical methods have been shown to be efficient, there is considerable variability among the results of the investigators. Inconsistencies are apparent in such data as optimum pH ranges, coagulant requirements, residual phosphorus concentrations, and removal efficiencies. It is thought that this lack of agreement is largely due to the wide disparity of conditions under which these tests were performed, and the failure to recognize the influences of dissimilar chemical environments.

This research was undertaken in an attempt to describe the effects of several variables affecting the removal of inorganic phosphates with ferric chloride. Among those considered were pH, coagulant dosage,

initial phosphorus concentration and calcium ion concentration. All studies were performed using pure solutions in order to minimize certain potential interferences associated with natural waters and wastewaters, such as alkalinity, hardness, suspended solids, etc. It was the aim of this investigation to provide fundamental information regarding the chemistry of phosphorus removal with trivalent metal ions in general, and Fe (III) in particular.

#### Experimental Procedures

All Jar Test coagulation studies were performed using a six-paddle laboratory stirring apparatus, manufactured by Phipps and Bird, Inc., Richmond, Va. The device was equipped with an illuminated base which facilitated observation of floc formation.

Each Jar Test was conducted on 1 liter volumes of freshly prepared phosphate solutions of known initial concentration. Following adjustment of the mixing speed to about 200 RPM, appropriate quantities of coagulation chemicals were dispensed into each jar. The pH of each solution was then adjusted to the desired value using 0.1 N or 1.0 N NaOH. Rapid-mixing at 200 RPM was continued for at least 10 minutes after chemical addition, followed by slow mixing at 20 RPM for 15 minutes. (These time requirements were established during a series of preliminary experiments designed to show the effects of mixing speeds and times on

phosphorus removal in this particular system.) After two hours' settling, samples were withdrawn for filtered and unfiltered phosphate analysis. Filtration was accomplished using Whatman No. 5 filter paper in 60° filter funnels. The analysis for orthophosphate was performed using the stannous chloride - molybdate method, as described in Standard Methods (APHA, 1965). Total phosphate was determined by the same technique after the recommended acid hydrolysis step.

All solutions were formulated using reagent grade chemicals and distilled water. The sole exception was the sodium tripolyphosphate solution, which was prepared from a commercial grade of this chemical (Warren 1004; 98 percent sodium tripolyphosphate, 1.9 percent sodium pyrophosphate).

### Experimental Studies and Results

#### A. The Effect of pH and Calcium on Phosphorus Removal with Ferric Chloride.

These experiments were undertaken to determine the region of pH values in which phosphorus is optimally removed by coagulation with ferric chloride. In addition, information was sought regarding the effect of calcium on coagulative phosphorus removal.

A series of Jar Test coagulation studies was initiated by preparing solutions containing 8.15 mg P/l orthophosphate, to which 30.9 mg Fe(III)/l in the form of ferric chloride was added. The molar ratio of

Fe(III) to initial phosphorus ( $Fe/P_i$ ) in these solutions was approximately 2.1 and the pH, measured after coagulant addition was about 3.0. No flocculation was observed under these conditions. The pH of the solutions was then adjusted to a range of values between pH 3.75 - 9.75. The study was repeated with calcium chloride added at varying concentrations (5.4 - 54 mg Ca/l).

The variation of residual phosphorus concentration as a function of pH is illustrated in Figure 1 and Figure 2. Examination of Figure 1, a plot of unfiltered residual phosphorus versus pH, shows that orthophosphate is most effectively removed from solution with ferric chloride alone in the region of pH 5.1 - 5.5. At these values, the unfiltered residual phosphorus concentration is reduced to about 0.01 mg P/l.

From Figure 2, the plot of filtered residual phosphorus concentration versus pH, it will be noted that orthophosphate is substantially removed between pH 4.3 - 6.5 with only Fe(III) added. Although no flocculation was observed below pH 4.7, apparently some of the phosphorus is incorporated into a colloidal complex which can be removed by filtration through Whatman No. 5 paper.

A comparison of the filtered and unfiltered phosphate removal curves (Figures 1 and 2) above pH 6 also suggests that some suspended colloidal complex is formed which resists settling but may be removed by filtration. For example, at pH 6.75, with only ferric chloride added, an unfiltered residual of 4.73 mg P/l was obtained, compared with a filtered residual

concentration of 0.55 mg P/l.

The addition of calcium chloride to the system has the effect of increasing phosphorus removal with ferric chloride at neutral and mildly alkaline pH values. It will be noted by examination of Figures 1 and 2, that residual phosphorus concentrations at pH values above the optimum (pH 5.1 - 5.5) may be decreased in proportion to the amount of calcium chloride added. For example, at pH 8.6, where virtually no reduction in phosphorus concentration was achieved with ferric chloride alone, filtered residual phosphorus concentrations of 0.7 mg P/l, 0.5 mg P/l, and 0.2 mg P/l were obtained for added calcium concentrations of 5.4, 12.0, and 27.0 mg Ca/l, respectively. Increasing calcium dosage also appears to improve settling of the bound phosphate, since filtered and unfiltered phosphate residuals become nearly identical for calcium concentrations between 27 - 54 mg Ca/l. Under the experimental conditions, the addition of calcium apparently had no significant effect on phosphorus removal below pH 6.0.

#### B. The Effect of Coagulant Concentration on Phosphorus Removal with Initial Phosphorus Concentration as a Parameter.

These investigations were undertaken to determine the effect of ferric chloride concentration on phosphorus removal. Jar Tests were performed on solutions containing several different initial orthophosphate concentrations (1.63 - 11.4 mg P/l). For each set of initial phosphorus



concentrations, the dosage of ferric chloride was varied to cover a range of molar ratio ( $Fe/P_i$ ) values. In all tests, the concentration of calcium was held constant (54 mg Ca/l ) and the pH was adjusted to 7.0 after coagulant addition.

Throughout this study, generally good agreement was obtained between filtered and unfiltered residual phosphorus concentrations. Figure 3 depicts filtered residual phosphorus concentrations as a function of the molar ratio ( $Fe/P_i$ ) for each set of initial orthophosphate concentrations. For initial phosphorus concentrations of 1.63 mg P/l and 3.26 mg P/l, the phosphate residuals were observed to decrease almost linearly for molar ratio values less than unity. For higher initial phosphorus concentrations (4.89 - 11.4 mg P/l), the respective curves were extrapolated linearly to their initial phosphorus values at  $Fe/P_i=0$ .

It will be noted that, for  $Fe/P_i < 1$ , the residual phosphorus concentration is a function of the initial phosphorus concentration as well as the molar ratio,  $Fe/P_i$ . However, for molar ratio values greater than unity, the residual phosphorus concentration is only dependent on the molar ratio. Phosphate residuals were observed to decrease asymptotically to about 0.05 mg P/l at  $Fe/P_i \geq 2$ . In effect, this is indicative of the limit of phosphorus under the experimental conditions of pH and ionic composition of the solutions.

### C. The Removal of Polyphosphates.

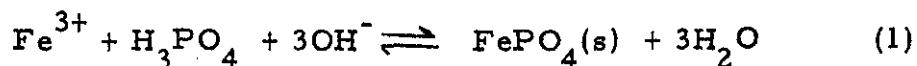
Since many modern synthetic detergent builders are largely composed of the complex phosphates, pyrophosphate and tripolyphosphate, a study was undertaken to determine the removal characteristics of these species in an iron-calcium system. Following the procedure used in previous experiments, molar ratio studies were conducted on 1 liter solutions of tetrasodium pyrophosphate and sodium tripolyphosphate, having concentrations of approximately 7.5 mg P/l. Sufficient calcium chloride was added to produce a concentration of 54 mg Ca/l, followed by varying quantities of ferric chloride. The pH was adjusted to 7.0 in all tests.

The results of these experiments are illustrated in Figure 4, which shows the residual phosphorus concentration as a function of the molar ratio from  $\text{Fe}/\text{P}_i = 1.0$  to  $\text{Fe}/\text{P}_i = 2.5$ . The removal of orthophosphate in this region of molar ratio values at pH 7.0 is included for comparison, and is represented by the dashed curve. As can be seen from the lower solid curve, the removal of pyrophosphate closely follows the removal of orthophosphate under the same experimental conditions. Tripolyphosphate is less effectively removed up to a molar ratio of about 2, as by the upper solid curve. Above  $\text{Fe}/\text{P}_i = 2$ , the residual concentrations of all phosphate species investigated approach the same minimum limit of approximately 0.05 mg P/l.

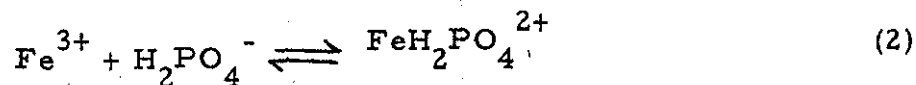
### Discussion

Orthophosphate was found to be optimally removed with ferric chloride

in pure solution in the range of pH 5.1 - 5.5. This result compares favorably with the point of minimum solubility of ferric phosphate, which was reported by Stumm (1964) to occur at pH 5.3. Galal-Gorchev and Stumm (1963) have given the following reaction to represent this chemical precipitation:



Although the unfiltered residual phosphorus concentration attained a minimum value of about 0.01 mg P/l between pH 5.1 - 5.5, comparatively good removals were obtained at lower pH values (pH 4.3 - 5.1) if the coagulated solutions were filtered prior to analysis. This difference between filtered and unfiltered phosphate removals in the range of pH 4.3 - 5.1 suggests that orthophosphate is complexed by Fe(III) at these pH values, but is not effectively precipitated. This phenomenon may be visualized by a consideration of the following reactions (Stumm, 1964):



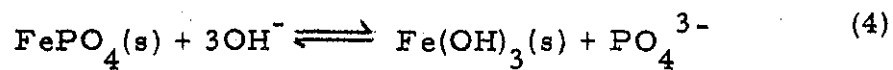
The positively-charged products in the above reactions are complexes which conceivably could undergo polymerization, forming charged colloids which resist agglomeration and settling due to charged-particle repulsion. Consequently, although the filtered removal data indicate that orthophosphate has reacted with Fe(III) even at lower pH values, it is not efficiently precipitated until the apparent isoelectric point (pH 5.3) has been reached.

At pH values above 6, both unfiltered and filtered phosphate residuals were observed to increase with increasing pH. At these pH values, it is postulated that two important phenomena act to reduce the extent of phosphorus removal with Fe(III). The first of these is replacement of the coordinated phosphate ligand by hydroxyl ion, for which Fe(III) has a greater affinity, as evidenced by the solubility product constants for the two compounds (Stumm, 1964):

$$K_{sp}[\text{Fe}(\text{OH})_3] = 10^{-36}$$

$$K_{sp}[\text{FePO}_4] = 10^{-23}$$

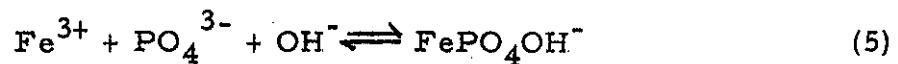
This replacement reaction may be visualized hypothetically as follows:



Since it has already been shown that orthophosphate is complexed

between pH 4.3 - 5.5, the appearance of increasing quantities of soluble phosphorus in the filtrate as the pH is raised above 6.0 seems to substantiate the contention of hydroxyl displacement of the bound phosphate.

The second effect, which may be noted by a comparison of filtered and unfiltered phosphate residuals above pH 6.0, is suggested to be the formation of negatively-charged ferric hydroxo phosphate complexes. The following hypothetical reaction may be written to illustrate this effect:



These complexes may again form colloidal polymers, such that charge repulsion would act to inhibit particle agglomeration and settling.

This phenomenon was observed primarily in the region of pH 6.0 - 8.0, where unfiltered residual phosphorus concentrations were significantly greater than filtered residual concentrations. Above pH 8.6, where both filtered and unfiltered residuals approach the initial phosphorus concentration, hydroxyl substitution would appear to be the dominant effect.

As indicated in Figures 1 and 2, the addition of calcium enhances the removal of phosphorus with ferric chloride at neutral and mildly alkaline pH values. The addition of only 5.4 mg Ca/l facilitates the

removal of some orthophosphate even above pH 9.0.

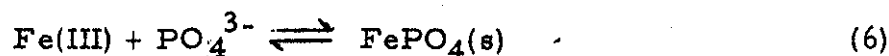
The occurrence of decreased phosphorus removals with Fe(III) alone at pH > 6 has been postulated to result primarily from two phenomena. Consequently, in promoting better phosphate removals in the mildly alkaline pH region, it is reasonable to expect that calcium influences each of these mechanisms. In the first instance, calcium may coordinate directly with negatively-charged ferric hydroxo phosphate complexes. Charge repulsion would thus be minimized and good flocculation promoted. Evidence for this role of calcium is represented by the good agreement between filtered and unfiltered phosphate residuals at calcium concentrations above 10 mg Ca/l. This indicates that essentially all of the phosphorus that had been complexed was effectively flocculated and settled.

Secondly, calcium may compete with Fe(III) for hydroxyl ion (Stumm, 1967), thereby preventing hydroxyl displacement of the phosphate ligand from the ferric phosphate complex. This effect is illustrated by decreasing filtered residual phosphorus concentrations at alkaline pH values with increasing calcium dosages, as shown in Figure 2. With added calcium, the phosphorus apparently remains complexed with Fe(III) despite the presence of excess hydroxyl ion.

It has been assumed throughout this work that phosphorus removal

with trivalent metal ions, specifically Fe(III), occurs by complexation and precipitation. In studies of a similar nature, Henriksen (1963) concluded that phosphorus removal with iron and aluminum occurred by these mechanisms. Stumm and Morgan (1962) and Galal-Gorchev and Stumm (1963) have also presented rather convincing evidence for the complex-precipitation mechanism, using UV absorption techniques, redox potential measurements, and alkalimetric titration studies.

In any chemical precipitation process, an equilibrium may be established between the "insoluble" precipitated compound and its soluble constituents. If it is assumed that the precipitation of orthophosphate with Fe(III) proceeds according to the reaction,



an expression governing the quantity of Fe(III) or  $\text{PO}_4^{3-}$  which remains in solution may be written in terms of a solubility product constant,  $K_{\text{sp}}$ . For the above reaction:

$$K_{\text{sp}} = \left[ \text{Fe(III)} \right] \left[ \text{PO}_4^{3-} \right] \quad (7)$$

The quantities in the brackets represent the equilibrium concentrations of Fe(III) and  $\text{PO}_4^{3-}$ , expressed in moles per liter, in equilibrium with solid  $\text{FePO}_4$ .

At this point, it should be stressed that reaction (6) and equation (7) are not intended to accurately represent the behavior of ferric iron

and orthophosphate in solution. Indeed, depending on pH and other conditions, Fe(III) and  $\text{PO}_4^{3-}$  may not be the predominant ionic forms in solution. Moreover, it is probable that several different reactions may take place between Fe(III) and orthophosphate in solution. These equations are presented as an example in order to illustrate the mathematical behavior of the iron-orthophosphate reaction.

As Fe(III) is added to a solution containing orthophosphate, some of the phosphorus will react with the iron to form insoluble  $\text{FePO}_4$ . An expression based on reaction (6) and equation (7), which governs the concentration of phosphate remaining in solution as a function of the initial concentrations of phosphorus ( $P_i$ ) and iron ( $Fe_i$ ), is given below:

$$P = 1/2 \left[ P_i - Fe_i + \sqrt{(P_i + Fe_i)^2 - 4(P_i Fe_i - K_{sp})} \right] \quad (8)$$

At the stoichiometric amount, corresponding to a molar ratio,  $Fe/P_i$  equal to unity ( $Fe_i = P_i$ ), the molar concentration of phosphorus and iron in solution is given by:

$$[P] = [Fe] = \sqrt{K_{sp}} \quad (9)$$

This indicates that, at a molar ratio  $Fe/P_i = 1$ , the residual concentration of phosphorus is fixed, regardless of the initial phosphorus concentration. At molar ratios less than unity, equation (8) may be written:

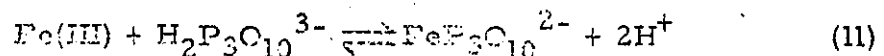
$$[P] = fP_i + \sqrt{(fP_i)^2 + K_{sp}} \quad (10)$$



where  $f$  is a function of the molar ratio,  $\text{Fe}/\text{P}_i$ . Equation (10) demonstrates that, for  $\text{Fe}/\text{P}_i < 1$ , the residual phosphorus concentration is dependent on both the molar ratio (related to  $f$ ) and the initial phosphorus concentration. This behavior is qualitatively indicated in Figure 3.

As  $\text{Fe(III)}$  is added to the system in excess of the stoichiometric quantity, the equilibrium is shifted to the right, resulting in the precipitation of more phosphorus from solution. Theoretically, if equation (6) accurately represented the only equilibrium involved in the precipitation of phosphorus by  $\text{Fe(III)}$ , it would be expected that the residual phosphorus concentration would approach zero at very high  $\text{Fe(III)}$  concentrations. In practice, this probably does not occur, since it has been reported that for molar ratios in excess of unity, a mixed ferric hydroxo phosphate is formed (Stumm, 1964).

The results of other workers (Stumm, 1964) have indicated that a considerable molar excess of coagulant was necessary to precipitate the complex phosphates in pure solution. Stumm (1964) has suggested that polyphosphate "interference" with orthophosphate coagulation occurs by soluble complex formation, such as:



Phenomena such as this would tend to diminish the importance of the adsorption theory of coagulative phosphorus removal. Solely on the

basis of the Schultze - Hardy Rule, it would have been expected that the more highly negatively-charged polyphosphates would be removed even more effectively than orthophosphate. The fact that polyphosphates have been reported to be less substantially removed than orthophosphate in pure solution by Fe(III) coagulation indicates that specific chemical equilibria, such as complex formation equilibria, are frequently more important than double layer compaction by counter ion adsorption.

In the present study, the observation that pyrophosphate is as effectively removed as orthophosphate may again be due to the effect of calcium. Calcium may act to inhibit soluble complex formation by associating with negatively-charged species, as previously described. In support of this contention are the results of others (Henriksen, 1962), who studied the removal of phosphorus in wastewaters (presumably containing some calcium) with trivalent metal ions, which indicated that the polyphosphates were as effectively removed as orthophosphate.

#### Conclusions

The need for the control of accelerated eutrophication of our natural waters has been well established. Chemical methods of phosphate removal appear to offer considerable promise in accomplishing this end. Sound engineering design can be effectively applied only with a knowledge of the basic chemistry of phosphate removal. It is hoped that this study will help provide a basis for

further work in this area, ultimately leading to the practical extraction of phosphorus from domestic wastewaters.

Based upon the results obtained in this present work, it may be concluded:

(1) The removal of orthophosphate, pyrophosphate, and tripolyphosphate increases with increasing ferric chloride dosage. The predominant mode of removal appears to be the formation of insoluble ferric phosphate complexes.

(2) The efficiency of phosphorus removal with ferric chloride is highly pH-dependent. Minimum residual phosphorus concentrations were observed near pH 5.3. With ferric chloride alone, severely reduced removal efficiencies were obtained below pH 4.8 and above pH 6.5.

(3) Calcium ion enhances the removal of phosphorus above pH 5.3, thereby allowing effective removals with ferric chloride over a wider range of pH values. Calcium probably acts as a competitor for hydroxyl ion, partially preventing hydroxyl displacement of phosphate from ferric phosphate. Calcium also appears to promote the formation of a more settleable floc at neutral and alkaline pH values. It is anticipated that the presence of calcium (and perhaps other ions) in natural waters and wastewaters would aid in the removal of phosphorus with trivalent metal ions. However, if the calcium concentration in these waters is inadequate,

or if interfering ionic species are present in significant amounts, supplementary addition of calcium may be necessary. Further studies are required in this area.

(4) Residual phosphorus concentrations decrease with increasing molar ratio of iron to initial phosphorus,  $Fe/P_i$ . Below  $Fe/P_i = 1$ , the reduction is approximately linear and related to the initial phosphorus concentration. For  $Fe/P_i$  values greater than unity, the residual phosphorus concentration is constant for a specified  $Fe/P_i$ , and approaches a minimum limit.

(5) Under the experimental conditions (pH 7.0; 54 mg Ca/l added) the minimum residual phosphorus concentration approaches a limit of about 0.05 mg P/l in the region of  $Fe/P_i = 2$ . Lower residuals are attainable for comparable coagulant dosages at pH values nearer to the optimum (pH 5.3).

(6) Pyrophosphate is removed as effectively as orthophosphate at molar ratios ( $Fe/P_i$ ) greater than unity. Tripolyphosphate is somewhat less efficiently removed in an iron-calcium system at pH 7 than pyrophosphate or orthophosphate for  $Fe/P_i = 2$ . For  $Fe/P_i = 2$ , tripolyphosphate residuals (expressed as P) are comparable to those of orthophosphate and pyrophosphate, coagulated under the same conditions. The relatively good removals of the complex phosphates obtained in this study, compared with those predicted in the literature, have been

attributed to the enhancing effect of calcium, which suppresses soluble complex formation.

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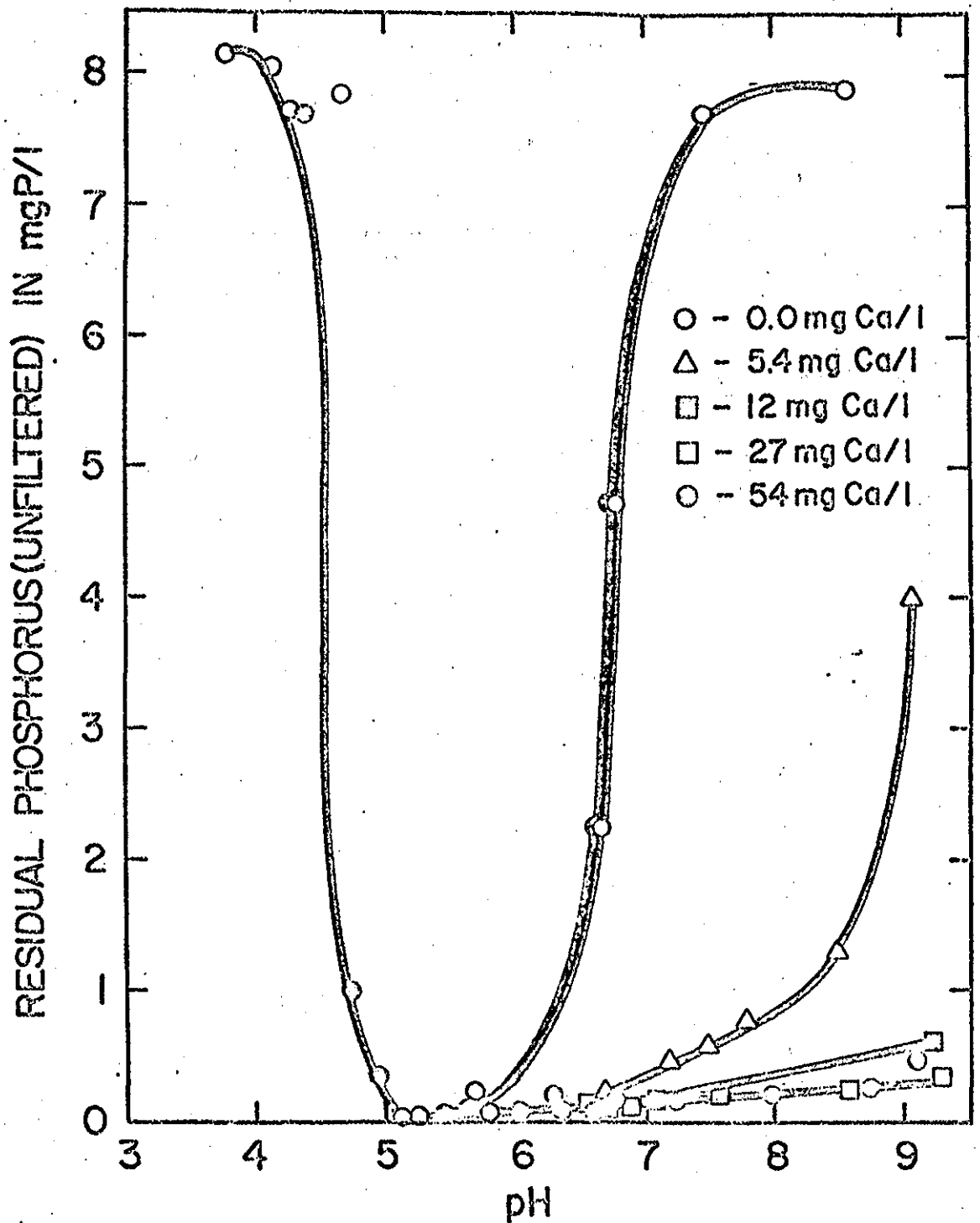
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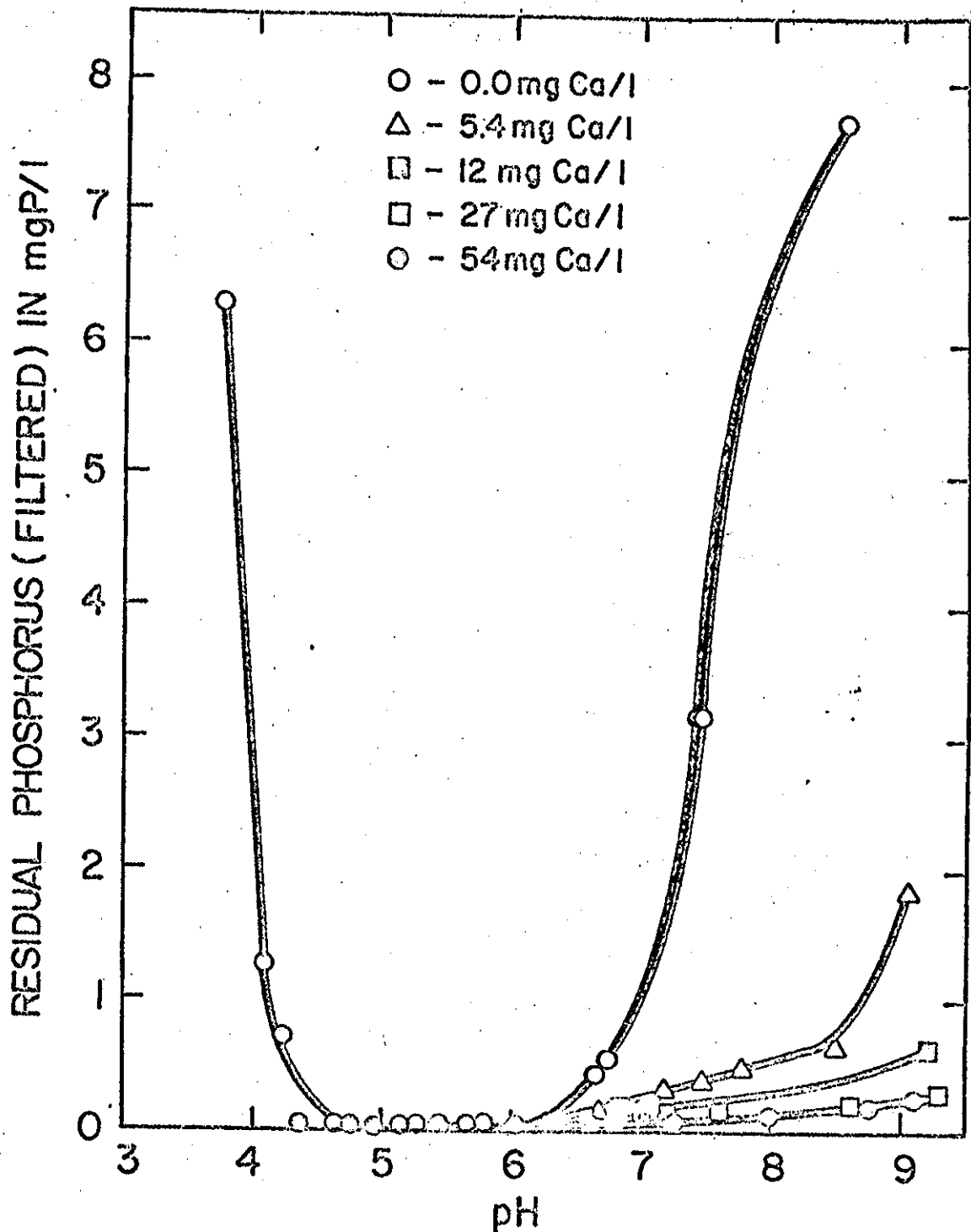
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INITIAL ORTHOPHOSPHATE: 8.15 mgP/l

COAGULANT CONCENTRATION: 30.9 mg Fe(III)/l

FIGURE 1. THE EFFECT OF pH ON UNFILTERED RESIDUAL PHOSPHORUS CONCENTRATION WITH CALCIUM AS A PARAMETER



INITIAL ORTHOPHOSPHATE: 8.15 mg P/l  
 COAGULANT CONCENTRATION: 30.9 mg Fe(III)/l

FIGURE 2. THE EFFECT OF pH ON FILTERED RESIDUAL PHOSPHORUS CONCENTRATION WITH CALCIUM AS A PARAMETER



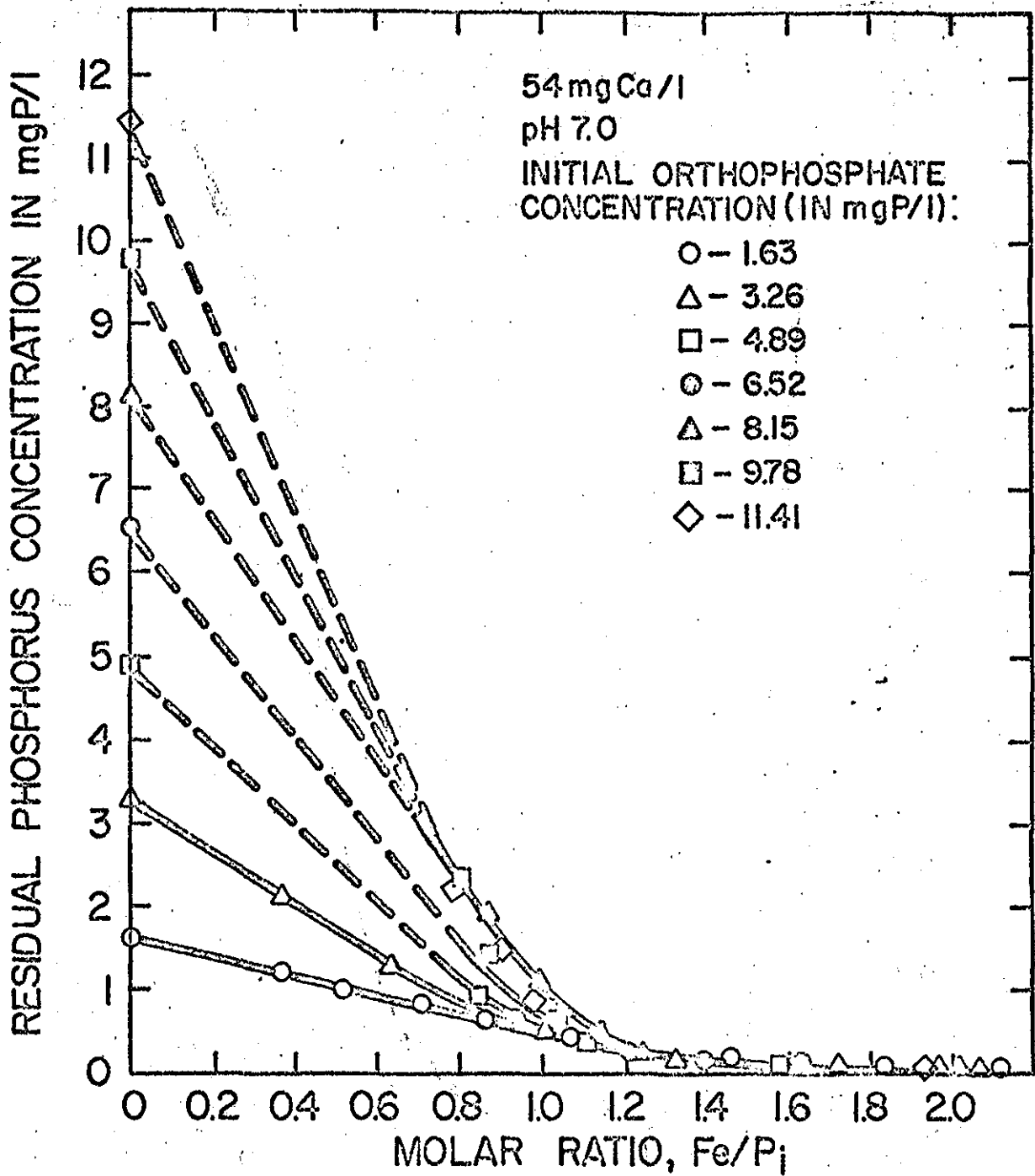
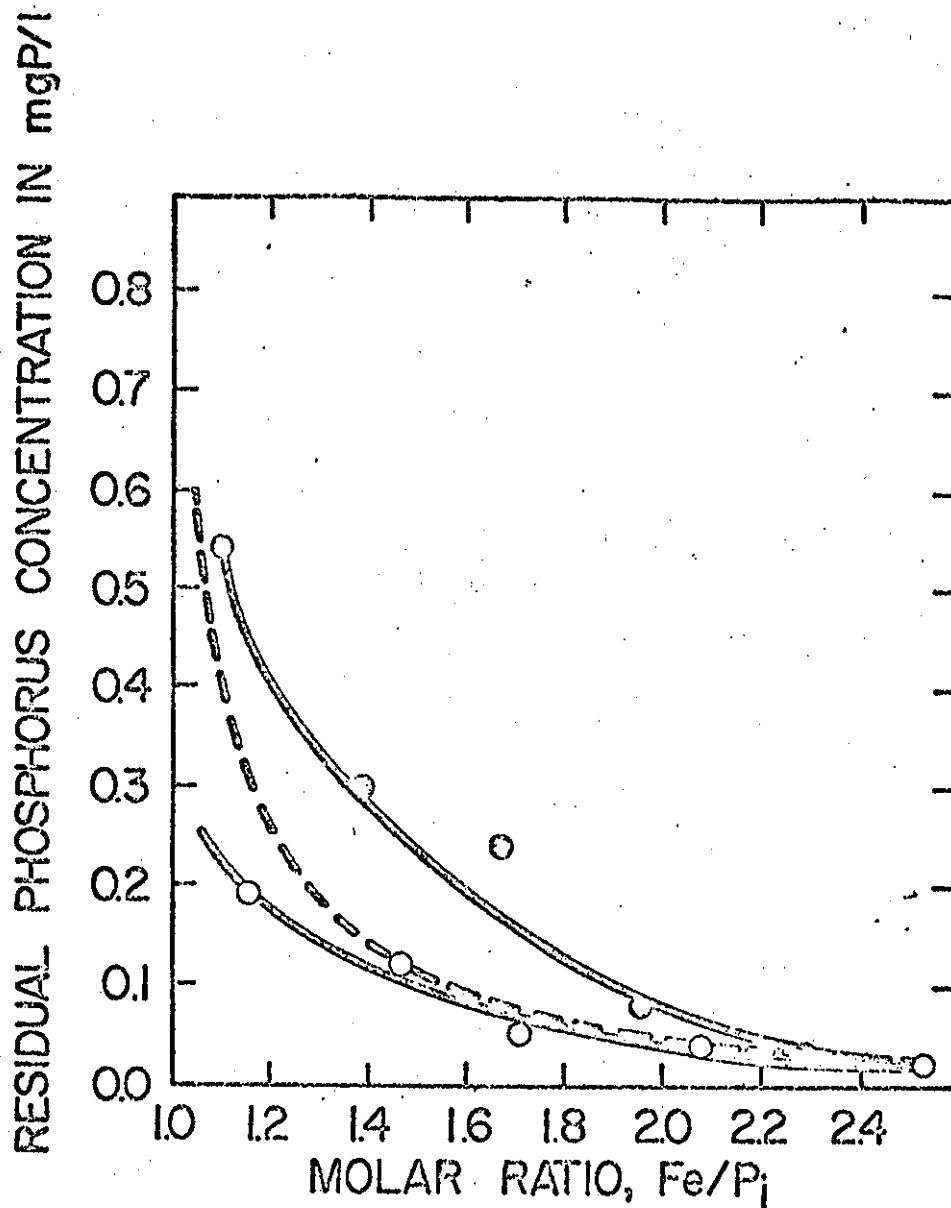


FIGURE 3. THE EFFECT OF COAGULANT CONCENTRATION (MOLAR RATIO, Fe/P<sub>i</sub>) ON THE CONCENTRATION OF RESIDUAL PHOSPHORUS



INITIAL CONCENTRATIONS:

- - PYROPHOSPHATE: 7.16 mgP/l
- - TRIPOLYPHOSPHATE: 7.53 mgP/l
- ORTHOPHOSPHATE

54 mg Ca/l

pH 7.0

FIGURE 4. THE EFFECT OF COAGULANT CONCENTRATION ON THE RESIDUAL CONCENTRATION OF PYROPHOSPHATE AND TRIPOLYPHOSPHATE