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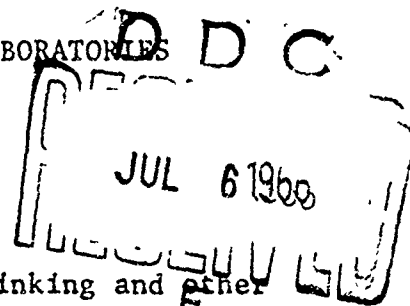
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CLARIFICATION OF POTOMAC RIVER WATER WITH CATIONIC POLYELECTROLYTES

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I. INTRODUCTION

In the United States Army, water for drinking and other purposes is supplied to the troops in the field by means of mobile water purification units. These units are capable of producing from highly contaminated sources, under combat conditions, water equal in clarity and potability to the product of the most advanced municipal water treatment plants. The purification process consists of coagulation and disinfection, in an upflow-solids-contact coagulation basin, by simultaneous addition of ferric chloride, limestone, and calcium hypochlorite, and filtration through pressure diatomite filters. Recent studies have shown that certain water-soluble, long-chain, positively-charged, organic polymers have the ability of producing excellent flocculation, when added to clay suspensions in quantities of only a small fraction of that required for flocculation with conventional coagulants. Because of these low dosage requirements and other possible advantages, this study was undertaken to determine the feasibility of using these new polymers, called cationic polyelectrolytes, or cationics, for coagulation, in place of the ferric chloride and limestone, presently used in the standard Army mobile water purification units. It is the purpose of this paper to report significant results obtained in this study.

The turbidity of present-day natural waters is made up of a complex mixture of particles, consisting, principally, of various colloidal clays and to a lesser extent--depending on the degree of contamination--of colloids from domestic and industrial wastes, live and decaying algae and their decomposition products, bacterial cells, decaying organic matter, and color colloids. Some colloidal particles, such as clay, may be hydrophobic; others, like certain sewage colloids, hydrophilic. These turbidity particles exhibit a wide distribution in size. Color colloids have been shown by Black (1) to be mainly in the 4 to 10 millimicron range. Clay particles generally fall into the 0.1 - 2.0 micron range. Some filamentous algae exceed

300 microns in length. Figure 1 is a photomicrograph of a sample of Potomac River water, showing filamentous algae mixed with clay turbidity.

Turbid natural water exhibits many of the characteristics of a stable colloidal system. If subjected to electrophoresis, the colloidal particles migrate to the positive electrode, indicating a negative charge. To clarify such a water by direct filtration would not be practical. A filter, fine enough to give an effluent of acceptable clarity, would clog in a very short time. Use of a more open filter to attain any practical flow would permit the passage of intolerable amounts of turbidity. Thus, it is clear that the destabilization of this colloidal system by the agglomeration of these particles into settleable or filterable aggregates (Figure 2), prior to filtration, is a necessary step in the clarification process.

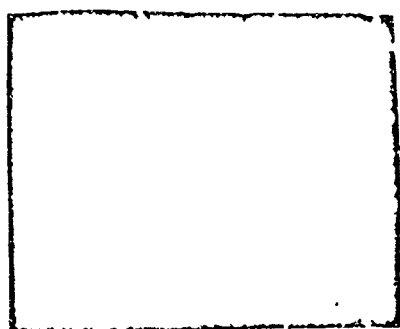


Figure 1. Photomicrograph of sample of Potomac River water, showing algae mixed with clay (X55).

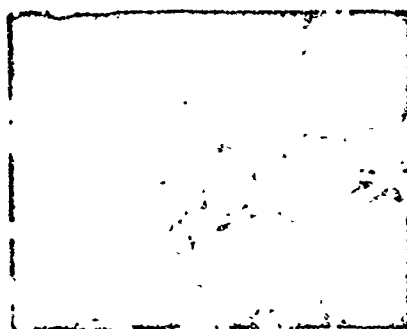


Figure 2. Photomicrograph of floc particle produced by addition of cationic polyelectrolyte to Potomac River water (X55).

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The numerous factors involved in the destabilization of various colloidal systems have been set forth in detail (2) - (5). The colloidal particles in untreated water are subject to two opposing groups of forces--one producing destabilization (or flocculation) and the other stabilization (or dispersion). Those factors contributing to destabilization are Brownian movement and the van der Waals forces of attraction. Opposing factors, producing stabilization, are hydration and electrostatic repulsion. Hydrophobic colloidal systems are stabilized by the latter; hydrophilic systems are stabilized by both hydration and electrostatic repulsion (6). A measure of this repulsion is the magnitude of the "zeta potential" of the colloid particle.

The concept of the zeta potential is incorporated in the Stern-Gouy diffuse double layer theory. Negatively-charged colloids attract to their surface by electrostatic attraction, a stationary, surrounding layer of positive ions, referred to as the Stern layer. Surrounding this fixed layer is a moveable, diffuse layer wherein the concentration of positive ions falls off as it extends to the area of

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electroneutrality in the bulk of the suspending medium. The overall potential drop between the surface of the particle and the bulk of the solution is the Nernst potential. The potential drop in the diffuse layer is called the zeta potential. It is the repulsive interaction of charged double layers which prevents the joining of colloidal particles into settleable aggregates as they approach each other as the result of Brownian movement. The "thickness" of the double layer is a function of the ionic content of the solution, decreasing with increasing concentration of electrolyte. The van der Waal forces of attraction are, however, practically unaffected by changes of ionic content.

In present day municipal and Army water treatment practice, the process of coagulation is accomplished through the use of aluminum or iron salts. When added to water, these salts hydrolyze and produce positively-charged polymeric hydroxides which adsorb on the turbidity particles and neutralize the repulsive negative charges residing on the particle surfaces, permitting them to agglomerate. The metal hydroxides produce a gelatinous floc which acts as a binder, trapping the agglomerated particles and settling them in a coagulation basin. The supernatant water is then filtered through sand filters, generally, in municipal practice, or through diatomite filters in Army equipment.

Recently there have become available a small, but increasing number of water-soluble, synthetic or modified natural polymers, cationic in nature, capable of neutralizing the negative surface charges on colloids and binding them into readily settleable and filterable agglomerates. These polymers, almost exclusively nitrogen compounds, have been shown to be effective in the flocculation of clay suspensions (200-250 ppm) in dosages well below 1 ppm. Because of these low dosage requirements and other advantages, these polymers appear attractive--especially for military water treatment. Several of these polymers are presently being used in wastewater treatment and in sludge conditioning. Although, to date, only one has been approved by the U. S. Public Health Service for use with potable waters, many others, it is expected, will be approved.

An understanding of the action of these polyelectrolytes has evolved from a number of investigations of the interaction of various polymeric flocculants with pure clay suspensions (7) - (10) and slimes (11). First proposed by Ruhrwein (2), this theory holds that in a system of negatively-charged colloids, for example, the positively-charged polymer is adsorbed on the surface of the particle. The particle surface charge is reduced or neutralized sufficiently to permit cohesion and aggregation on collision, via residual valence forces. The polymer may, if long enough, adsorb on more than one particle and thus be responsible, by a bridging action, for inter-particle bonding. LaMer (11) has distinguished between two destabilization mechanisms, designating the one resulting from charge neutralization alone, as coagulation, and the other, involving bridging, with

or without charge neutralization, as flocculation. Water treatment technologists generally use the term coagulation to describe any process producing floc regardless of mechanism.

Very little has appeared in the literature regarding the use of cationic polyelectrolytes as sole coagulants in the treatment of natural waters. Cohen (13) has reported that, with some waters, good coagulation was obtained with carefully controlled dosages of a cationic polyelectrolyte. Heavy algae (*Chlorella*) suspensions were completely coagulated, although high (100 ppm) polyelectrolyte dosages were required. Anionic polyelectrolytes were ineffective, indicating that charge neutralization was involved in the agglomeration of the negatively-charged algae cells. Crook (14) has reported on the removal of significant quantities of turbidity from Delaware River water, using a cationic polyamine as sole coagulant, with dosages of 1 - 3 ppm.

II. INVESTIGATION

The investigation was divided into two parts. The first part consisted of a series of laboratory experiments, using eight cationic polyelectrolytes (Table I), Potomac River water, and, for comparative purposes, natural water from four other area sources (Table II). The laboratory studies, which evaluated the effects of pH, hypochlorination, mixing rate, and color removal, were followed by full-scale plant studies using a 3,000-gph standard Army mobile water purification unit, Potomac River water, and the most promising polyelectrolyte, as determined by the laboratory study.

A. Laboratory Experiments

1. Procedures. The laboratory study consisted of a series of experiments, commonly referred to as "jar tests," using a multiple-stirring apparatus, equipped with six, 2" x 3" stirrers and a variable mixing control. Five-hundred ml quantities of water in 1-liter beakers were placed on the mixing stand. Mixing was started at 80 rpm. After the addition, by pipette, of the desired dosages of polymer, or calcium hypochlorite and polymer, in hypochlorination studies, the mixing was continued for 20 minutes. The rate was then reduced to 20 rpm and mixing continued for 20 minutes more. At the end of the mixing period, the beakers were removed from the stand and the floc was allowed to settle for 15 minutes. One-hundred ml samples were then withdrawn for turbidity and zeta potential measurements by constant, gentle suction from a fixed point, at the center of the bulk of liquid, with a special device, fabricated from a 100-ml pipette. The water remaining in the beakers was used for other determinations, such as pH, residual chlorine, and color. Analytical methods used were those given in Standard Methods (15). Turbidity was measured with a Lumetron Model 450N Photovolt colorimeter, calibrated with Fuller's earth suspensions against a Jackson candle turbidimeter. Residual chlorine was determined with orthotolidine and a

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TABLE I

OPTIMUM DOSAGE REQUIREMENTS AND EFFECTS OF HYPOCHLORINATION
IN COAGULATION OF POTOMAC RIVER WATER

<u>Polyelectrolyte</u>	<u>Type</u>	<u>Optimum Dosage* (ppm)</u>	<u>Hypochlorination Effect (11 ppm Avail. Chlorine)</u>
Polymer A	Poly-quaternary Ammonium	4	Unaffected
Polymer B	Polyamine	3	Adverse
Polymer C	Polyamine	2	Adverse
Polymer D	Polyamine	3	Adverse
Polymer E	Polyamine	2	Adverse
Polymer F	Poly-quaternary Ammonium	3	Unaffected
Polymer G	Polyamine	20**	Adverse
Polymer H	Polyamine	20**	Adverse

*Based on active ingredient

**Based on total weight of polymer solution - percent active ingredient unknown.

TABLE II

ANALYSES OF WATERS USED IN LABORATORY STUDIES

<u>Characteristic</u>	<u>Water</u>				
	<u>Potomac River</u>	<u>Allen's Fresh</u>	<u>Miller's Pond</u>	<u>Occoquan Creek</u>	<u>Rappahannock River</u>
pH	7.3	6.2	6.4	6.6	6.6
Turbidity (units)	67	54	39	300	135
Color (units)	5	30	25	50	45
Conductivity (micromhos/cm)	180	136	47	-	60
Total Hardness (ppm CaCO ₃)	72	24	14	28	23
Alakalinity (ppm CaCO ₃)	50	4	6	16	12
Chloride (ppm Cl)	7	26	4	5	4
Sulfate (ppm SO ₄)	14	11	6	17	9
Chemical Oxygen Demand (ppm)	5	13	11	-	11
Zeta Potential (mv)	-24	-22	-25	-23	-21

standard chlorine disk in an Army comparator. Zeta potential values were determined using a ZETA-METER apparatus which has been described by Riddick. (5).

2. Results

a. Typical Curves. The results obtained upon adding increasing dosages of Polymer A to Potomac River water are shown in Figure 3. The same general type of curve was obtained in all jar tests conducted without hypochlorination, irrespective of polymer or water used. The negative charge on the turbidity particles was progressively reduced; increasing flocculation took place and reached its optimum as the zeta potential was reduced to a value near zero (± 5 mv). With increasing dosages beyond the optimum, a charge reversal took place. The original negative charges were now replaced by positive charges and redispersion of turbidity particles became operative, with the residual turbidity and zeta potential curves approaching a maximum, limited by polymer adsorption. This same mechanism has been observed in the flocculation of biocolloids with a cationic polyelectrolyte (16) and is in accordance with presently accepted theory.

Figure 4 shows the results obtained in tests with Polymer A and five natural waters. Optimum flocculation dosages of polymer fall into the range of 2 to 5 ppm with waters of less than 120 units of settled turbidity. With Occoquan Creek water of 300 units of settled turbidity, the optimum dosage of polymer was 15 ppm. The waters of low pH required the lowest dosages and gave the best settled water. The dependence of dosage requirement on turbidity, or better, on the amount of surface to be covered by the adsorbing polymer, has been demonstrated by other investigators (10), (11), (16). The broad optimum dosage range indicated for Allen's fresh water was reproduced in tests with Polymer C, indicating that the nature of the water, rather than the specific polymer used, is the predominant factor determining the optimum dosage range. Tests with pure kaolinite clay indicated that tap water suspensions of comparable turbidity could be flocculated with optimum dosages of Polymer A in the 0.3 to 0.6 ppm range. This indicates that natural waters contain stabilizing elements which can increase the flocculation dosage requirements over those which would be predicted by tests with well characterized clay suspensions of comparable turbidity.

b. Effects of pH. Figure 5 shows the effect of pH variations on Polymer A in Potomac River water whose pH was adjusted by additions of sodium hydroxide or hydrochloric acid. As the pH was increased, a settled water of poorer quality was obtained. Zeta potential measurements during this study of pH effects indicated that charge reversal took place at increasingly higher dosages of polymer as the pH was raised. These data suggest a lowering of the cationic character of the polymer at higher pH values by deprotonization as proposed by Crook (14).

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c. Effects of Chlorine. Because standard Army coagulation procedures employ simultaneous hypochlorination and coagulation, the effects of normal amounts of chlorine on the various polymers was of particular importance in this study. The data of Table I and Figures 6 and 7 indicate that those polymers of the polyamine type were adversely affected by normal calcium hypochlorite dosages (11 ppm available chlorine). The polymers of the poly-quaternary ammonium type were unaffected. Figure 7 shows a comparison between these two types of polymers in Rappahannock River water to which calcium hypochlorite (11 ppm chlorine) had been added. With Polymer A, a poly-quaternary ammonium compound, the turbidity and zeta potential curves exhibit the same shapes as observed with unchlorinated water. Residual chlorine values were constant after the initial chlorine demand was satisfied. With Polymer C, a polyamine, however, even with polymer doses of 15 ppm, negative zeta potential values were observed and a steady decline in residual chlorine values, probably due to chloramine interactions, was noted as polymer dosage increased. The data shown in Figure 6 indicate that a good quality settled water can be obtained with Polymer C, in the presence of chlorine, but at the expense of relatively high polymer dosage. Other polyamines were practically destroyed with normal calcium hypochlorite dosages.

d. Color Removal. Figure 8 illustrates the data obtained in tests of color removal from Rappahannock River water with Polymer A. The mechanism involved appears to be one of chemical interaction, in contrast to the mechanism of charge neutralization and particle bridging illustrated by the turbidity and zeta potential curves. Similar sets of curves were obtained in tests with Polymer A and Miller's Pond water and in tests with Polymer C and waters from the Rappahannock River, Allen's Fresh Creek, and Miller's Pond.

e. Mixing Effects. The importance of the mixing rate, as a variable in the flocculation process, is indicated by the results of mixing tests with Potomac River water and Polymer A, plotted in Figures 9 and 10. Figure 9 shows that gentle mixing, even at the low rate of 5 rpm, was sufficient to produce good flocculation. Figure 10 indicates that beyond a certain mixing period (15 mins), a decidedly better settled water was produced by mixing at 20 rpm, as contrasted with mixing at 80 rpm. These results, which correspond to those of other investigators (10), (16), (17), indicate that too vigorous agitation can produce redispersion, as a result of a "break-up" of already-formed floc, and, perhaps, even increasing positive charge on the sheared particles, resulting from increased polymer adsorption.

B. Studies with Standard Army Unit

1. Description of Test Unit. The 3,000-gph standard Army mobile water purification unit, used in these studies, is shown in Figure 11. Basically, it consists of an upflow type solids-contact coagulation basin and two pressure diatomite filters. A cross-section

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of the coagulation basin is shown in Figure 12. The unit is a complete water treatment plant on wheels. All necessary equipment such as pumps, feeders, and filters, required for coagulation, disinfection, and filtration are assembled in a special van-type body, mounted on a standard military 2 1/2-ton truck chassis. Water flowing through the coagulation basin at the rate of 3,000 gph has a rise rate of 1.1 gal/min/sq ft and a detention time of 20 minutes. Each filter housing contains six elements with a total filtering area of 10 sq ft. Lindsten has described this unit in more detail (18).

2. Procedure. Potomac River water was pumped to the unit at two operating rates (3,000 gph and 1,500 gph). Polymer A, selected on the basis of its performance in the laboratory studies, was used as the sole coagulant. Chlorine residuals between 1 and 2 ppm were maintained by addition of a calcium hypochlorite solution. The mixing disks were rotated at 68 rpm, the lowest rate attainable.

For filtration, only one of the two 1,500-gph filters was used. In operations at rated capacity (3,000 gph), half of the coagulated water was run to waste. The filter was operated with a diatomite precoat of 0.1 lb/sq ft and a body feed of 25 ppm. The filter cycle was ended when a pressure differential of 40 psi was reached. From the filter cycle duration, a filtrability index was calculated to indicate the gallons of filtered water produced per pound of diatomite used for precoating and body feed.

The dosage of polymer was regulated through the use of jar tests and zeta potential measurements. Samples of raw, coagulated, and filtered water were taken at regular intervals, usually hourly, for chemical analysis and zeta potential measurements. Routine determinations were made of pH, turbidity, hardness, alkalinity, chloride, conductivity, chlorine residual, and temperature.

3. Results. The floc produced upon addition of the polymer was a loose, readily settleable mud, in contrast to the much lighter, gelatinous, ferric hydroxide floc produced in standard operation with ferric chloride and limestone. Whereas, with the standard coagulants, a floc bed is usually built up in the coagulation basin to the draw-off level, four feet from the bottom, within a few hours, the floc produced with the polymer reached a level of only about 2 feet (measured during operation), even after several days of operation without sludge draw-off. Several methods of continuous sludge draw-off were tested, but results were not significantly better than when intermittent draw-offs were used. Turbidity measurements of the coagulated water, indicative of floc carryover to the filter, indicated considerably higher values than normally encountered with ferric chloride operation. The nature of the two flocs is, of course, decidedly different. The floc produced by the polymer is considerably more filterable than the gelatinous ferric hydroxide floc produced with ferric chloride.

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No direct comparison was made between standard ferric chloride-limestone operation and polymer operation. This comparison is planned for future studies. Nevertheless, it is of value, in assessing the efficiency of coagulation with the polymer, to compare the volume of water filtered per pound of diatomite used during a filter cycle with the average figure of 2,000 gal/lb diatomite, a figure for standard ferric chloride-limestone operation, established from the results of many field tests on many different types of water, throughout the United States.

During the course of these studies, over 300 filter cycles were run during two distinct operating periods--one (Period A) extending from February through June, and the other (Period B) from September through December. Table III is a compilation of average values of the various feedwater characteristics during the testing periods indicated. Table IV is a summary of results obtained during each operating period.

TABLE III

AVERAGES OF CHARACTERISTICS OF POTOMAC RIVER WATER
DURING RUNS WITH 3,000 GPH ARMY UNIT

<u>Characteristic</u>	<u>Period A (Feb thru Jun)</u>	<u>Period B (Sep thru Dec)</u>
pH	7.3	7.9
Turbidity (units)	60	65
Total Hardness (ppm CaCO ₃)	84	167
Alkalinity (ppm CaCO ₃)	61	73
Chloride (ppm Cl)	10	210
Conductivity (micromhos/cm)	186	650
Chemical Oxygen Demand (ppm)	5	30
Zeta Potential (mv)	-18	-4

TABLE IV

SUMMARY OF RESULTS OF RUNS WITH 3,000 GPH ARMY UNIT

<u>Testing Period</u>	<u>Feedwater Rate (gph)</u>	<u>No. of Filter Cycles</u>	<u>Filter Cycle Time (Avg) (min)</u>	<u>Filtrability Index (gal filtered water (per lb diatomite)</u>
A - (Feb thru Jun 65)	3,000	31	84	1,460
	1,500	58	170	2,170
B - (Sep thru Dec 65)	3,000	157	50	1,000
	1,500	35	68	1,250

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The results obtained depended on the characteristics of the water being treated. During Period A, the filtrability index was 1,460 gal/lb diatomite, at a feedwater rate of 3,000 gph. At half this rate, the filtrability index increased to 2,170 gal/lb diatomite. During Period B, the filtrability indices dropped to 1,000 and 1,250 gal/lb diatomite for the corresponding feedwater rates. Additions to the feedwater of 25 ppm carbon or 50 ppm Ft. Belvoir clay failed to improve these Period B indices. The turbidity of the filtered water during Period A was very low, in most runs less than 0.2 unit. During Period B, filtered water turbidity averaged 1.6 units, with a range of 0.1 to 6 units.

The drop in filtrability indices and filtrate quality during Period B can be correlated with the changing character of the feedwater, as shown in Table III. The values for Period A are "typical" for Potomac River water, during periods of normal flow in the winter and spring of the year. The values for Period B reflect a significant change in the character of the water from that of Period A. The marked increase in chlorides, conductivity, and hardness can be attributed to salt water intrusion, due to tidal effects, which resulted from drought conditions and record low flows in the Potomac River, during the last six months of 1965. The increase in chemical oxygen demand is indicative of a marked increase in the organic content, in all probability, resulting from the concentration of sewage and other organic pollutants, due to these same drought conditions. The sewage treatment plant, serving the District of Columbia, is located about fourteen miles upstream from the test location. Other smaller sewage treatment plants are located within a few miles of the test-water intake. Filamentous green algae (Ulothrix, Zygnema, and Spirogyra) grew abundantly during this period. Counts as high as 2600 filaments per ml were recorded. Increases in pH, to values as high as 9.5, correlating with algae growth, were also noted.

Perhaps even more important than many other factors, to an understanding of how the nature of the raw water affected the efficiency of the clarification process, is the factor of zeta potential. The marked change in the zeta potential, from -18 mv of Period A to the much lower value of -4 mv in Period B, is indicative of a definite change in the nature of the raw water turbidity. This change can be attributed, not only to increased ionic content, but to other causes as well. It has been generally reported that, on the average, the zeta potential of surface water turbidity falls into the range of -15 mv to -25 mv. That zeta potential values can be observed considerably above and below this range, due to the effects of sewage, industrial wastes, and other pollutants, has been indicated by other investigators (19). The clay particles, which constitute the bulk of surface water turbidity, are hydrophobic. Sewage and other organic pollutants contain colloids which are mostly hydrophilic. As noted above, there is a marked difference in the nature of these two types of colloids. Whereas the stability of a

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hydrophobic dispersion depends mainly on electrostatic repulsion, the hydrophilic colloids are stabilized by both electrostatic forces and the element of hydration. It has long been known that, in a colloidal system of both hydrophobic and hydrophilic colloids, the latter can be adsorbed on the surface of the former and impart to the hydrophobic particles the characteristics of surface charge and electrophoretic mobility of the hydrophilic colloid. Consequently, in the treatment of a water polluted with an appreciable quantity of hydrophilic colloids, the coagulation process can be made more difficult by the "protective" action of these colloids, which are known to be far less sensitive to flocculation than hydrophobic colloids.

This protective action is reflected in the data on polymer dosage requirements during the two operating periods. During Period A, the optimum dosage, as determined by jar tests and zeta potential measurements, fluctuated between 3 and 4 ppm. During Period B, the dosage ranged between 6 and 8 ppm with an average of 7 ppm. In numerous jar tests, there was a good correlation between optimum dosage and very low or zero zeta potential values. Since it required a higher dosage of polymer to reach the electroneutrality point from -4 mv, during Period B, than from -18 mv, during Period A, it can be concluded that in the treatment of a natural water, subject pollution, the nature of the turbidity, as well as other factors, rather than the zeta potential, per se, determines dosage requirements. It appears then that in the treatment of such a water with a cationic polyelectrolyte, the success of the coagulation process may be more dependent on interparticle bridging than on surface charge neutralization.

Based on past experience with the standard Army unit, it is estimated that, if a comparative study were made, the dosage of polymer would have been about 1/20 of the combined ferric chloride-limestone dosage.

III. CONCLUSION

From the results of this study, it can be concluded that, for the clarification of a natural water, a cationic polyelectrolyte of the poly-quaternary ammonium type can, even in the presence of normal amounts of chlorine, act effectively as a sole coagulant. It can replace a conventional metal salt in a system comprised of a solids-contact upflow type coagulation basin and a pressure-diatomite filter.

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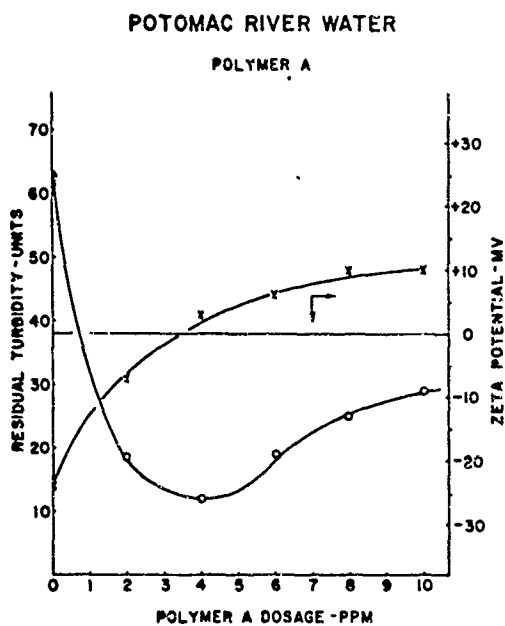


FIG 3

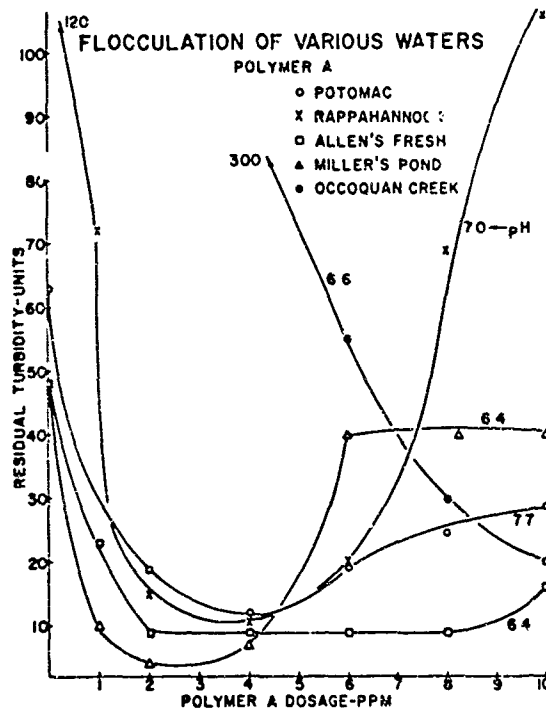


FIG 4

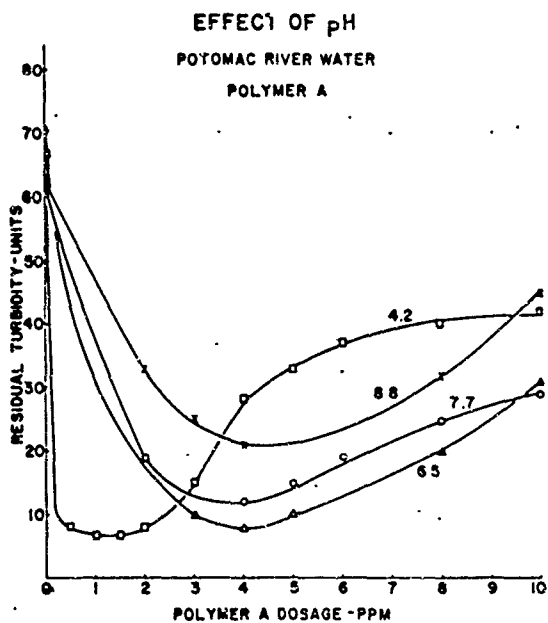


FIG 5

EFFECT OF CHLORINE ON VARIOUS POLYMERS
POTOMAC RIVER WATER
11 PPM CHLORINE ADDED

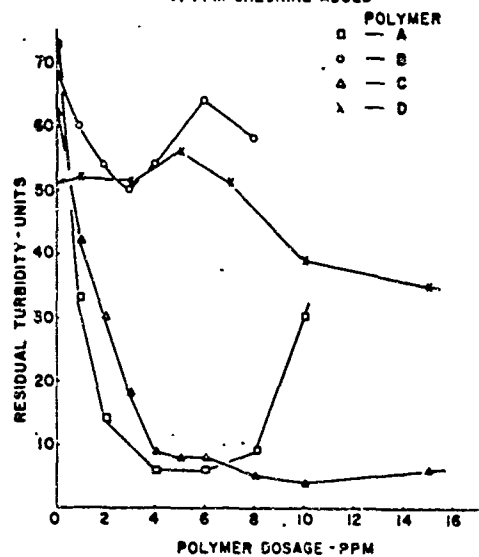


FIG 6

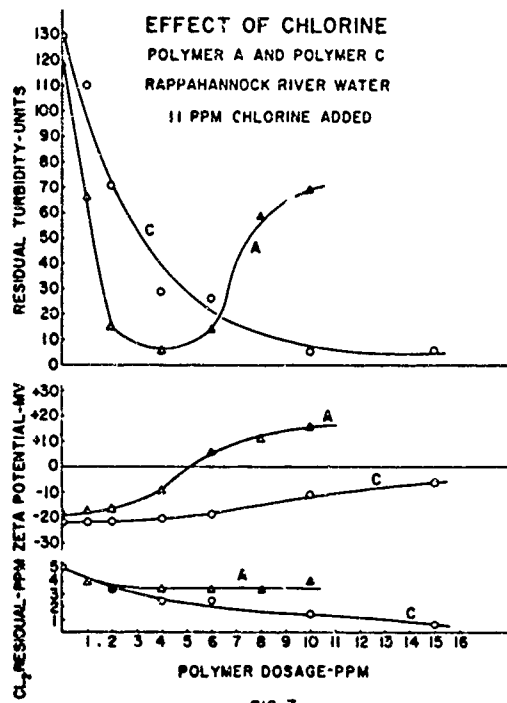


FIG 7

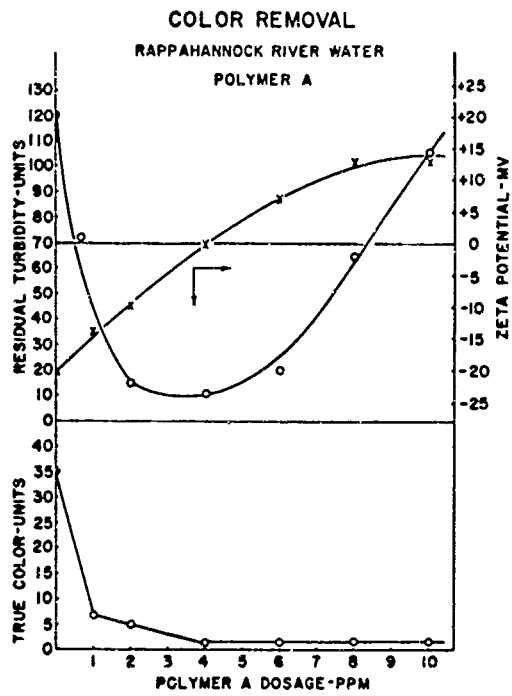


FIG 8

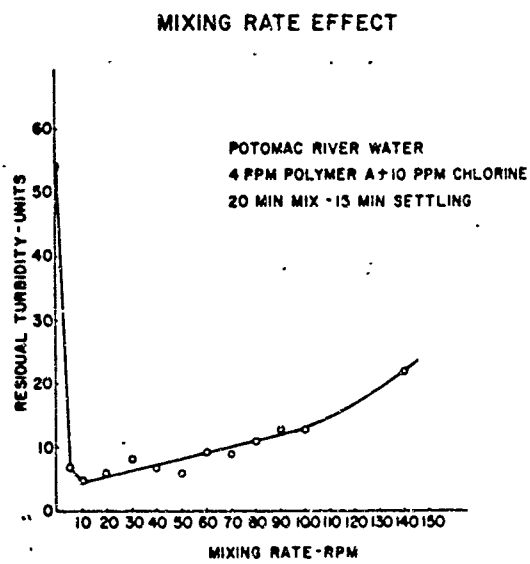


FIG 9

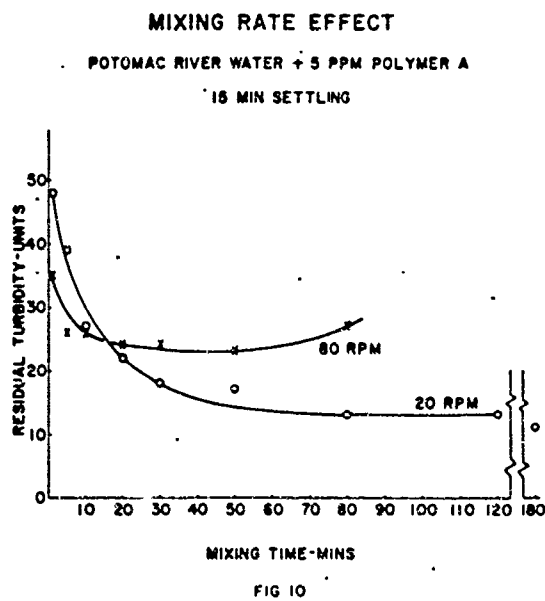


FIG 10

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GRATING NOT DIMENSIONABLE

Figure 11. U. S. Army Mobile Water Purification Unit in Operation

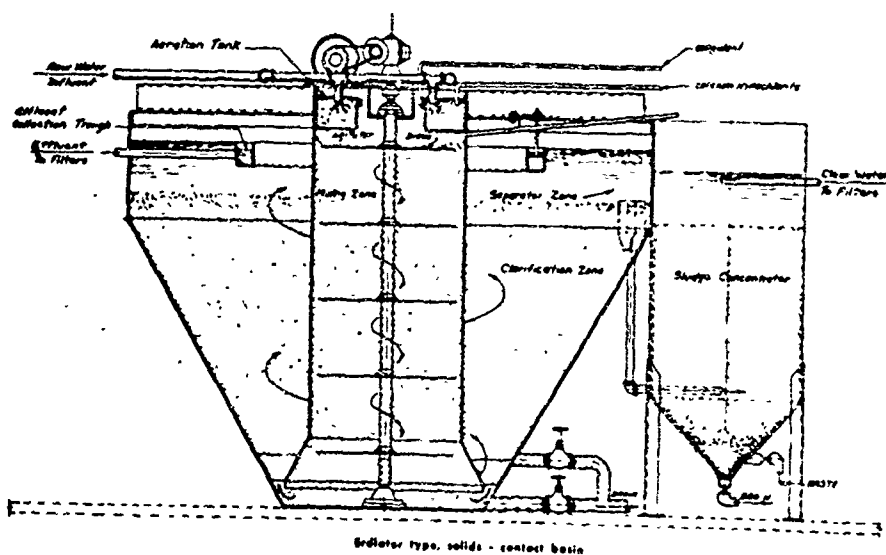


Figure 12. Cross-section of Coagulation Basin of U. S. Army Mobile Water Purification Unit