

DEFLUORIDATION OF TYPICAL EASTERN VIRGINIA GROUNDWATER

by

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Chapter 1 - INTRODUCTION

In 1901, a Public Health Service Physician in Italy found that the black teeth of emigrants from a nearby region could be attributed to water charged with volcanic fumes. In 1916, Dr. Frederick S. McKay, a Colorado Springs dentist, reported the same defect in his patients. It was not until 1926 that a chemist in a water treatment plant suggested that fluoride was the cause of the black or mottled teeth. Mottling is characterized by a slight opaque whitish area on some of the back teeth. More severe, it becomes more wide spread and changes in color from gray to black. Most severe, pitting of the enamel may occur and the teeth may wear down to the gums, and dentures may be required (1).

In the late 1920's, Dr. H. T. Dean of the United States Public Health Service conducted studies which indicated:

1. When fluoride concentrations exceed 1.5 milligrams per liter (mg/l) there is no further decrease in the decayed, missing and filled tooth incidence, but there is an increase in the occurrence and severity of mottling.
2. At fluoride concentrations of 1.0 mg/l the optimum occurs - maximum reduction in caries with no significant mottling.
3. At fluoride levels below 1.0 mg/l some benefits, but not as great are evident.

The benefits reduce to zero as the fluoride level decreases to zero (1).

Figure 1 presents a listing of the various reported effects of fluoride in drinking water as noted by different investigators and reported in the literature (2).

Fluoride is found in every water supply used by man for drinking water, although in varying concentrations. The element fluorine ranks 13th in abundance in the earth crust and 12th in oceans. It is also 13th in the human body (3). Fluoride in groundwater may be derived from:

1. fluorite, the principal fluoride mineral of igneous rocks.
2. other fluoride bearing minerals, or
3. volcanic or fumarolic gases.

In the State of Virginia, groundwaters high in fluoride concentration are found along the east coast as indicated on the map in Figure 2. The fluoride concentrations may range from approximately 1.0 to greater than 7.0 mg/l. In the past, the Commonwealth of Virginia accepted the natural waters in the State with higher than desirable fluoride concentration where no better water of comparable drinking quality and quantity was available or until a practicable method of removing the fluoride was developed and proven (4).

With the passage of the Safe Drinking Water Act (P.L.

Concentration of Fluorides, mg/l	Reported Effect
0.2	Mottled teeth in 1 percent of children
0.6	No effects at this concentration or lower
0.7	Mild dental fluorosis in 8.5% of children
0.8	No effects at this concentration or lower
0.8 to 0.9	Mild mottling of teeth
0.8 to 1.5	Threshold for mottling of teeth
0.9	Mild mottling of teeth
0.9	Mottling occurred as a result of high water use
0.9	Critical concentration for mottling
1.0	Threshold for mottling of teeth
1.0	10% of children had mottled teeth
1.0	90% of children had mottled teeth
1.0 to 2.0	Mild to moderate mottling
1.2	No effect at this concentration
1.4	No skeletal sclerosis found
1.7 to 1.8	50% of children had mottled teeth
2.0	Gave mottling and weakening of tooth structure
2.0 to 3.0	Retained in system
2.0 to 3.0	Moderate to severe mottling
2.5	75 to 80% children had mottled teeth
2.5	No evidence of skeletal fluorosis
3 to 4	Not likely to cause endemic cumulative toxic fluorosis in adults
3 to 6	Gave severe mottling
3.5 to 6.2	No adverse effects on carpal bones of children
4.0	90% of children had mottled teeth
4.0	No disorders other than dental mottling
4.4 to 12	Caused chronic fluorosis and affected skeletal system
5.0	No effect on height, weight or bones
6.0	Threshold for appreciable effect on bones
6.0	100% of children had mottled teeth
6.0	Gave pitting and chipping of tooth enamel
8.0	No deleterious bone changes except dental mottling
10	Some cases of skeletal fluorosis
11.8	Gave chronic fluorine intoxication to adults
12	Affects deciduous teeth
13.7	100% of children had mottled teeth
115	Sub-lethal in drinking water
180	Toxic to man in drinking water
2000	Lethal dose in drinking water

Fig. 1 - REPORTED EFFECTS OF FLUORIDES IN DRINKING WATER (2)

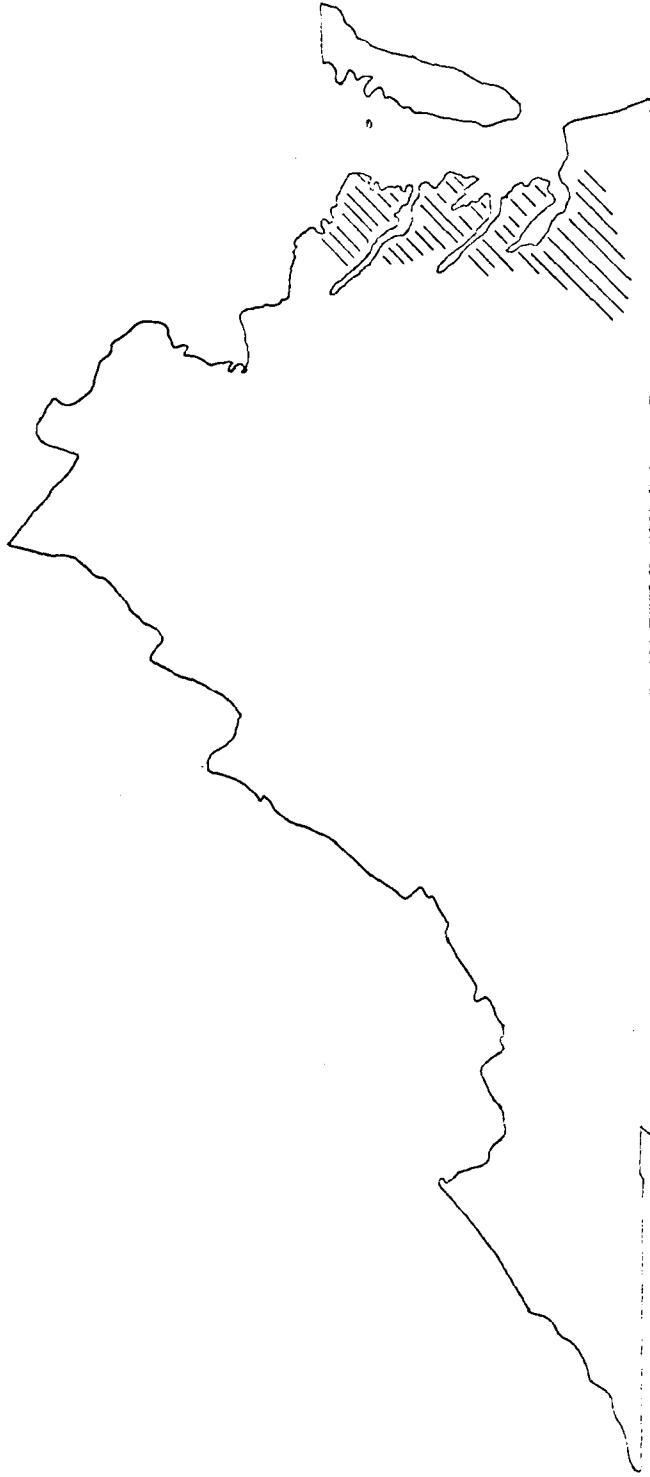


Fig 2 Location of granitoid with high fluoride concentration in the State of Virginia.

93-523), the Environmental Protection Agency issued the National Interim Primary Drinking Water Regulations on December 24, 1975. These regulations took effect on June 24, 1977. In these regulations, the Maximum Contaminant Level (MCL) for fluoride was established as follows (5):

When the annual average of the maximum daily air temperature for the location in which the community water system is situated is the following, the maximum contaminant levels for fluoride are:

Temperature Degrees Fahrenheit	Degrees Celsius	Level, milligrams per liter
53.7 and below	- 12.0 and below	- 2.4
53.8 to 58.3	- 12.1 to 14.6	- 2.2
58.4 to 63.8	- 14.7 to 17.6	- 2.0
63.9 to 70.6	- 17.7 to 21.4	- 1.8
70.7 to 79.2	- 21.5 to 26.2	- 1.6
79.3 to 90.5	- 26.3 to 32.5	- 1.4

In reviewing the last three years' data on the maximum daily air temperatures for the City of Suffolk, it was determined that the annual average was 69.1 degrees Fahrenheit. Based on this, the fluoride MCL for the Suffolk area is 1.8 mg/l. The recommended optimum fluoride level based on this temperature is 0.9 mg/l.

The Safe Drinking Water Act does allow for the granting of variances or exemptions to the MCL set forth in the regulations: however, this is only a temporary relief be-

cause the owners of a public water system must set up a compliance schedule to meet the MCL's. Therefore, water systems with high fluoride which in the past may not have required treatment may find that they have to provide treatment or find an alternate source to meet the Safe Drinking Water Act.

The purpose of this research was to explore the use of alum or alum and lime as coagulants for the reduction of fluoride in groundwater along the east coast of Virginia to the MCL set by the Environmental Protection Agency.

Chapter 2 - LITERATURE REVIEW

This literature review was divided into two parts. The first was an investigation of defluoridation techniques reported in the literature. The second was an investigation of sludge dewater techniques used in the laboratory.

Defluoridation Methods

The literature reflects many proposals for fluoride reduction or removal, some of which have been proposed and dropped and some of which have been in use for many years.

In a publication prepared for the Environmental Protection Agency, Water Supply Branch, David Volkert and Associates (6) listed four treatment methods for fluoride reduction. These methods included reverse osmosis (90 to 97% removal efficiency with raw water fluoride concentration of 11 to 60 mg/l), electrodialysis (approximately 80% removal efficiency with raw water fluoride concentration of 5.5 to 9 mg/l), distillation (approximately 99.9% removal efficiency with raw water fluoride concentration of 1000 mg/l), and ion exchange (approximately 95% removal efficiency with raw water fluoride concentration of 22 to 37 mg/l). Other methods listed by Volkert were adsorption, using bone char or activated alumina; alum coagulation; and lime-soda softening, using crushed dolomite limestone.

Other authors (7, 8, 9, 10, 11, 12) have explored and reported on the use of lime-soda softening to reduce fluoride. It has been reported that up to 3.3 mg/l fluoride can be

reduced to 1.0 mg/l in the presence of 100 mg/l magnesium.

Scott, et al (8) reported that the following equation can be used to express the reduction of fluoride in terms of the magnesium removed:

$$Y = F - 0.07 F X^{\frac{1}{2}}$$

where

Y = Residual fluoride (mg/l)

F = Initial fluoride (mg/l)

X = Magnesium removed (mg/l)

Lime-soda softening for fluoride reduction appears to be feasible when the water has sufficient magnesium hardness associated with the fluoride to make adsorption possible. Otherwise, a magnesium salt (such as magnesium sulfate) must be added to the raw water.

The use of activated carbon has been reported by McKee and Johnson (13). Fluoride concentrations as high as 7.5 mg/l have been reduced using activated carbon but the drawback is that the pH must be reduced to 3.0 or below.

According to Maier (10), the affinity of bone for fluoride is based on the anion exchange capacity of apatites. He stated that "the carbonate radical in the apatite comprising bone, $n\text{Ca}_3(\text{PO}_4)_2 \cdot \text{CaCO}_3$ is replaced by the fluorides in the water, forming an insoluble fluorapatite. In the regeneration of the material with sodium hydroxide the fluorapatite probably becomes an hydroxy-apatite and the

fluorides are removed in the form of soluble sodium fluoride. The hydroxy-apatite subsequently becomes available as an exchange material by the replacement of its hydroxy radical with fluoride". In 1960, Maier (14) reported that a defluoridation plant located in Britton, South Dakota, had been using bone char media since 1953.

Another plant in operation in 1960, as reported by Maier, (14) was located in Bartlett, Texas. This plant used activated alumina for the media through which the water passes. The raw water contains 8.0 mg/l and the finished water contains 1.0 mg/l. The media is regenerated by the use of a one per cent caustic solution made up of sodium hydroxide followed by a 0.05 N sulfuric acid rinse. This plant was placed on line in 1951. Maier also reported that a new well was developed with a lower fluoride concentration, 3.0 mg/l instead of 8.0 mg/l. Due to this change, the capacity of the media dropped from 700 grains of fluoride per cubic foot of media to 400 grains.

The formation of aluminum fluoride compounds has been explored by many authors. The first was Boruff (7) in 1934. Soon after fluoride was associated with the mottling of teeth, investigators began to explore the different methods of water treatment to determine which method could best be modified to remove or reduce fluoride. Boruff chose to study

aluminum sulfate, sodium aluminate, zeolite, activated alumina and bauxite. Of these only the aluminum sulfate produced sufficient fluoride reduction.

Boruff (7) worked with water to which sodium fluoride was added in the laboratory. The method of treatment consisted of adding 8.5 to 171 mg/l aluminum sulfate to the fluoridated water, stirring for 30 minutes and allowing to settle for 18 to 24 hours before filtering of the floc. From these tests, Boruff concluded that in order to reduce the fluoride concentration from 3.0 mg/l to 1.0 mg/l, it would require 34 mg/l aluminum sulfate. Waters up to 5.0 mg/l fluoride would require 170 mg/l aluminum sulfate in a single dose, or 85.5 mg/l and 51.3 mg/l aluminum sulfate in a double dose to reduce the fluoride to 1.0 mg/l. Boruff also stated that the optimum pH range was between 6.25 and 7.5 with a slight advantage at pH 6.7.

Kempf, et al (15) used Boruff's work as a basis to do further work with aluminum sulfate. They collected a sample of water with a pH of 8.4 and a fluoride concentration of 7.5 mg/l. After adjusting the pH to 7.15, 342 mg/l aluminum sulfate was added and stirred for 30 minutes. The solution was allowed to settle and samples were collected. Two hours after the start of treatment, the fluoride concentration had dropped to 1.25 mg/l; 6 hours later, 0.85 mg/l; and 24 hours later, 0.40 mg/l.

Boruff, Buswell, and Upton (16) performed a study in which calcium fluoride, magnesium fluoride, aluminum fluoride, sodium fluoride and sodium fluorosilicate were dissolved in stock solutions and the fluoride concentration adjusted to approximately 4.0 mg/l. These waters were then treated with 166 mg/l aluminum sulfate, stirred for one hour, and allowed to settle for one hour. The water with the sodium fluoride appeared to have the best reduction of fluoride. The authors stated that "these experiments indicate: (1) The cation associated with the fluoride ion in water greatly affects the completeness of its removal by alum floc, and hence this method is not applicable to defluorination of many natural waters."

Culp and Stoltenberg (17) performed tests on a soft highly mineralized water using aluminum sulfate. From these tests, they reported that if lime is required for stabilization then it should be added near the end of the flocculation period because calcium appears to interfere with fluoride removal. Also they reported that a 10% decrease in alum requirements could be realized by incremental feeding to the rapid mix rather than a single point application.

Work performed in other countries has indicated that high fluorides are not just a concern of the United States. Gabovich (18) reported on fluoride concentrations in the USSR which ranged from 4.3 to 5.1 mg/l. He studies several methods

of treatment and concluded that treatment with aluminum sulfate was the most effective.

In India, the Nalgona technique was developed and reported on by Nawlakhe, et al. (19, 20). They determined that the removal of fluoride by aluminum sulfate is a function of both the initial fluoride concentration and the initial alkalinity. In order to reduce the fluoride, high doses of aluminum sulfate are required followed by lime, if needed to keep the alkalinity from dropping too low. The authors attempted to determine the mechanism of the fluoride reduction but could not. They stated that it did not appear to be due to adsorption or coprecipitation.

Fatkillin, Berezyuk, and Pushkarev (21) performed tests on removal of fluoride sorbed on aluminum hydroxide precipitates. From these tests, the authors found that an equation associated with the fluoride sorption isotherms took the form of:

$$C_s = \frac{a}{n} C_e^{1/n}$$

where

C_s = the amount of fluoride sorbed
(mg per unit weight of precipitate)
 C_e = Equilibrium concentration in
solution (mg/l)
 a and n = parameters of the sorption
process

The authors found that the plot of $\log C_s$ vs. $\log C_e$ was linear and consisted of two regions. The first region follows

the Freundlich isotherm, corresponding to sorption of fluoride in the form of complexes. The second region has "n" less than 1 and corresponds to aggregation of the sorbed substance on the solid surface.

Sludge Dewatering

In chemical precipitation of contaminants from water, sludge is produced and its disposal is required. Laboratory tests have been developed which assist the design engineer in the sludge disposal process selection.

Specific resistance is a measure of the dewaterability of a sludge. The method employed is to filter a known volume of sludge and to measure the volume of filtrate and the time of filtration. A plot of time divided by volume vs. volume is made and the slope determined. The specific resistance can be calculated from the equation:

$$r = \frac{2 b p A^2}{uw}$$

- Where
- r = specific resistance (M/kg)
 - b = slope of plot of time divided by volume vs. volume (s/m⁶)
 - p = pressure across filter (N/m²)
 - A = area of filter (m²)
 - u = viscosity of filtrate (Ns/m²)
 - w = weight of dry cake solids per unit volume of filtrate (kg/m³)

The specific resistance test can be used to determine the optimum method and type of sludge conditioning necessary for sludge dewatering and disposal. The lower the specific resistance value, the better the sludge dewaterability (22, 23).

As the literature indicates, many methods of defluoridation have been explored. The groundwaters of eastern Virginia are very soft, high in fluoride and high in alkalinity and should be suited for reduction of fluoride by the addition of alum or alum and lime in accordance with the Nalgonda technique.

Chapter 3 - EXPERIMENTAL PROCEDURES

This chapter will be divided into eight sections to explain the methods used for performing each test. These sections include: Samples, Fluoride Detection, Jar Tests, Hardness, Alkalinity, pH, Aluminum, and Sludge Volume, Total Solids and Filterability.

Samples

On the average, the groundwaters of the east coast of the State of Virginia have the following chemical constituents:

<u>Parameter</u>	<u>Concentration</u>
Fluoride	4.5 mg/l
Alkalinity	400 mg/l as CaCO ₃
Hardness	6.1 mg/l as CaCO ₃
pH	8.5

Three waters were chosen for this study. The first was from Chippokes Plantation State Park located in Surry County. The fluoride concentration of this water was approximately 2.1 mg/l. Next a sample from the Wilroy Industrial Park located in the City of Suffolk was obtained. This water had a fluoride content of approximately 3.0 mg/l but it was "spiked" in the laboratory with sodium fluoride to approximately 8.0 mg/l. Finally a sample was obtained from a well owned by the City of Portsmouth but located at their water treatment plant in the City of Suffolk. (Groundwater is

pumped from this well, in addition to another nearby well, directly into the clear well to provide fluoridation of the water.) The fluoride content of this water was approximately 5.6 mg/l. A copy of a complete chemical analysis for each of these waters is attached in the Appendix.

Fluoride Detection

The specific ion electrode (Orion Model 94-09, Orion Research Incorporated, Cambridge, Mass.) was selected as the method for determining fluoride concentration in the raw and treated waters. The matching reference electrode (Orion Model 90-01-00, filled with equitransferent filling solution, Orion Catalog Number 90-00-01, Orion Research Incorporated, Cambridge, Mass.) was also used. These electrodes were connected to a Fisher Accumet Model 230 pH/Ion Meter (Fisher Scientific Co., Pittsburgh, Pennsylvania). Due to the long time period between fluoride measurements, a standard curve was not utilized, but instead the method of Known Addition, as described in the Orion Instruction Manual (24), was used. A problem associated with a linear scale read by a needle soon became apparent when using the pH/Ion Meter. The accuracy of the readout was only about 0.5 millivolt which could make a difference of 0.2 to 0.5 mg/l fluoride. To help overcome this problem, it is recommended that a digital millivolt readout be used in any future work.

The standard fluoride solutions used in the known additions were diluted from a stock solution prepared in accordance with Standard Methods (25) section 414 B.

Jar Tests

All jar tests were performed on the Phipps and Bird Six Paddle Stirrer (Phipps and Bird, Inc., Richmond, Va.). Six square battery jars were filled with 1 liter of the raw water to which chemicals were added to five, the sixth jar was kept as a control to determine if any fluoride reacted with the glass during the time the water was in the glass jars. While the chemicals were added, the jars were mixed at the highest speed and then the speed was reduced to approximately 20 rpm. After flocculation for approximately 30 minutes, the paddles were cut off and the water allowed to settle for 3 hours. At the end of the settling period, the liquid was siphoned out of each jar and placed in plastic bottles until the necessary tests had been performed. This procedure was employed to prevent any reaction between the glass and the fluoride in solution.

The chemicals used in the jar tests were either aluminum sulfate (alum) or alum and lime. The alum was mixed in a stock solution such that 1 ml added to 1 liter of raw water gave a dosage of 100 mg/l. The lime stock solution was prepared such that 1 ml added to 1 liter of raw water gave a dosage of 10 mg/l.

Hardness

Total hardness was run on each raw water sample but not on the treated water. The method used was the EDTA titrimetric method listed in Standard Methods (25).

Alkalinity

Total alkalinity was performed on every sample both raw and treated. The method used was the methyl orange end point titration with 0.02 N sulfuric acid.

pH

Raw and treated water pH measurements were made using the Corning pH meter Model 7 (Corning Scientific Instruments, Corning, N. Y.).

Aluminum

After settling, some of the treated water was collected and placed in 125 ml plastic bottles. Aluminum determination was performed on these samples in accordance with the Atomic Absorption Spectrophotometric Method listed in Standard Methods (25).

Sludge Volume, Total Solids and Filterability

An investigation of the maximum production of sludge from each of the raw waters was made. After completing the testing to determine chemical dosages to reduce the fluoride, the remaining raw water was placed into the jars and sufficient chemicals added to each to produce the maximum fluoride

reduction. The sludge produced was concentrated by drawing off as much of the supernatant as possible and then combining the sludge and allowing it to resettle and then drawing off more supernatant. The sludge volume was determined by placing the sludge into a graduated cylinder and recording the volume.

The total solids of the sludge were determined by the method outlined in Standard Methods (25) as Total Residue Dried at 103 - 105 C.

The filterability of the sludge was determined by filtering 100 ml of the sludge through a Whatman No. 42 filter in a Buchner funnel using 20 inches of mercury vacuum. The method of measuring the filtrate was to stand a 100 ml graduated cylinder in the bottom of a vacuum flask and to put a Buchner funnel into the flask so that the filtrate drained into the cylinder. Readings were made every 15 seconds until the flow of filtrate stopped. The area of the filter was determined. The weight of the dry cake solids was determined by weighing the oven dried filter paper before filtering, drying the sludge and filter paper and re-weighing. The increase in weight was due to the dry solids.

The supernatant from the sludge production was checked for residual fluorides, total alkalinity, total hardness and pH. The methods used were as previously discussed.

Chapter 4 - EXPERIMENTAL RESULTS

Each of the waters used in the experimental work was analyzed for fluoride reduction using alum alone or in combination with lime. In the following sections, the results of this work are presented.

Series "A" - Chippokes Plantation State Park

Figure 3 indicated the fluoride reduction with increasing alum dose for the Chippokes Park Water. Run 1 was made without lime addition, while Runs 2 and 3 had 100 and 200 mg/l lime added, respectively. Figure 4 indicates the "fine tuning" to determine the alum dose required, in the absence of lime, to meet the MCL for the area of 1.8 mg/l fluoride. In addition, an attempt was made to reduce fluoride concentration to the recommended optimum level of 0.9 mg/l. The results of this phase of the study are also indicated in Figure 4. Figure 5 indicates the pH variance with alum dose. Runs 1, 2 and 3 were conducted under the conditions noted above. Table I indicates the change in alkalinity with alum dose and Table II presents the residual aluminum found in the unfiltered treated water.

The sludge studies for this water indicated a sludge volume of 7 per cent of total volume tested, with total solids content of 1443 mg/l and a specific resistance of 2.17×10^{14} m/kg. This result is based on treating the raw water with 800 mg/l alum and 200 mg/l lime. The treated water had a fluoride residual of 0.17 mg/l, an alkalinity of 100 mg/l as CaCO_3 , a hardness of 190 mg/l as CaCO_3 , and

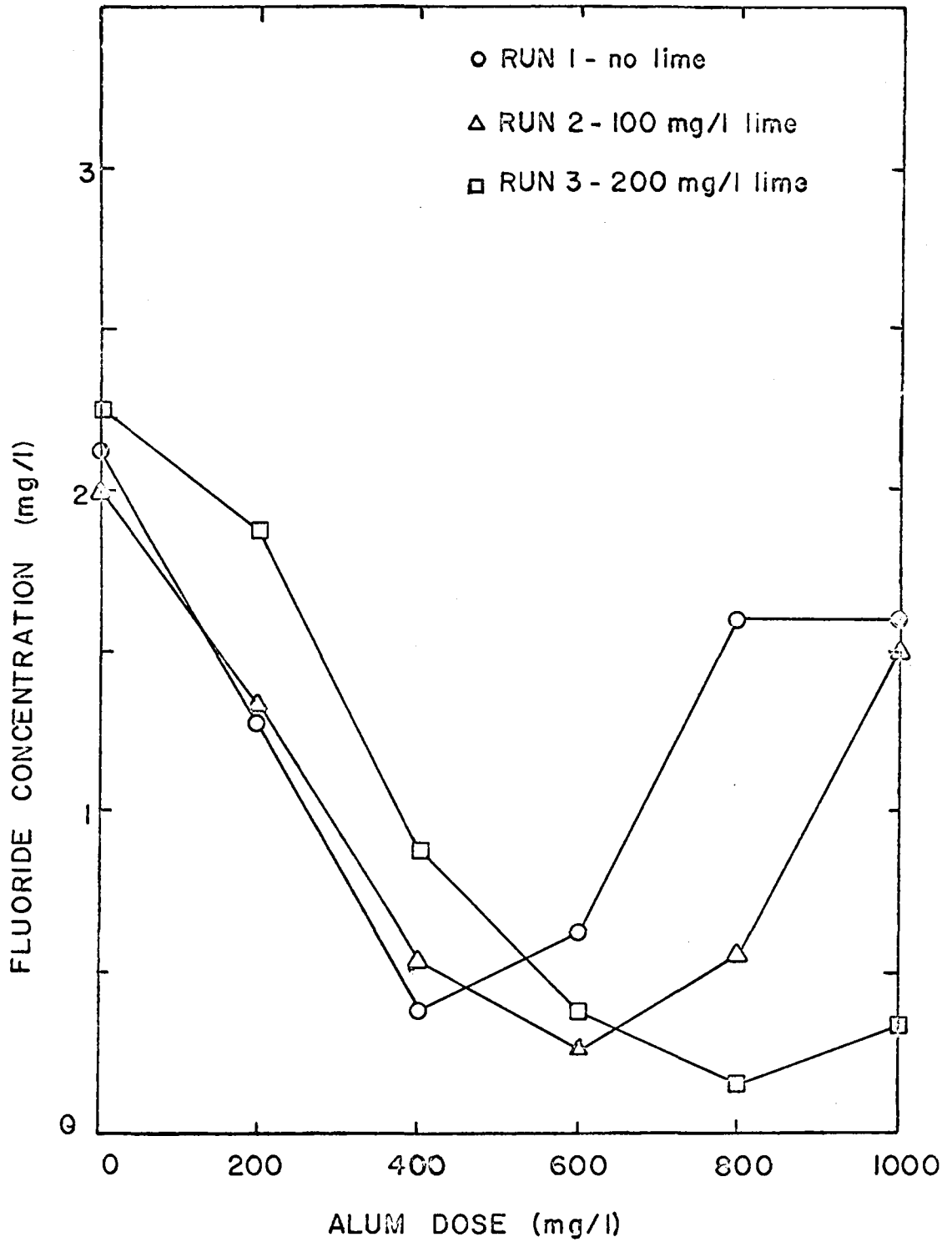


Fig. 3 SERIES 'A' ALUM DOSE vs. FLUORIDE CONCENTRATION

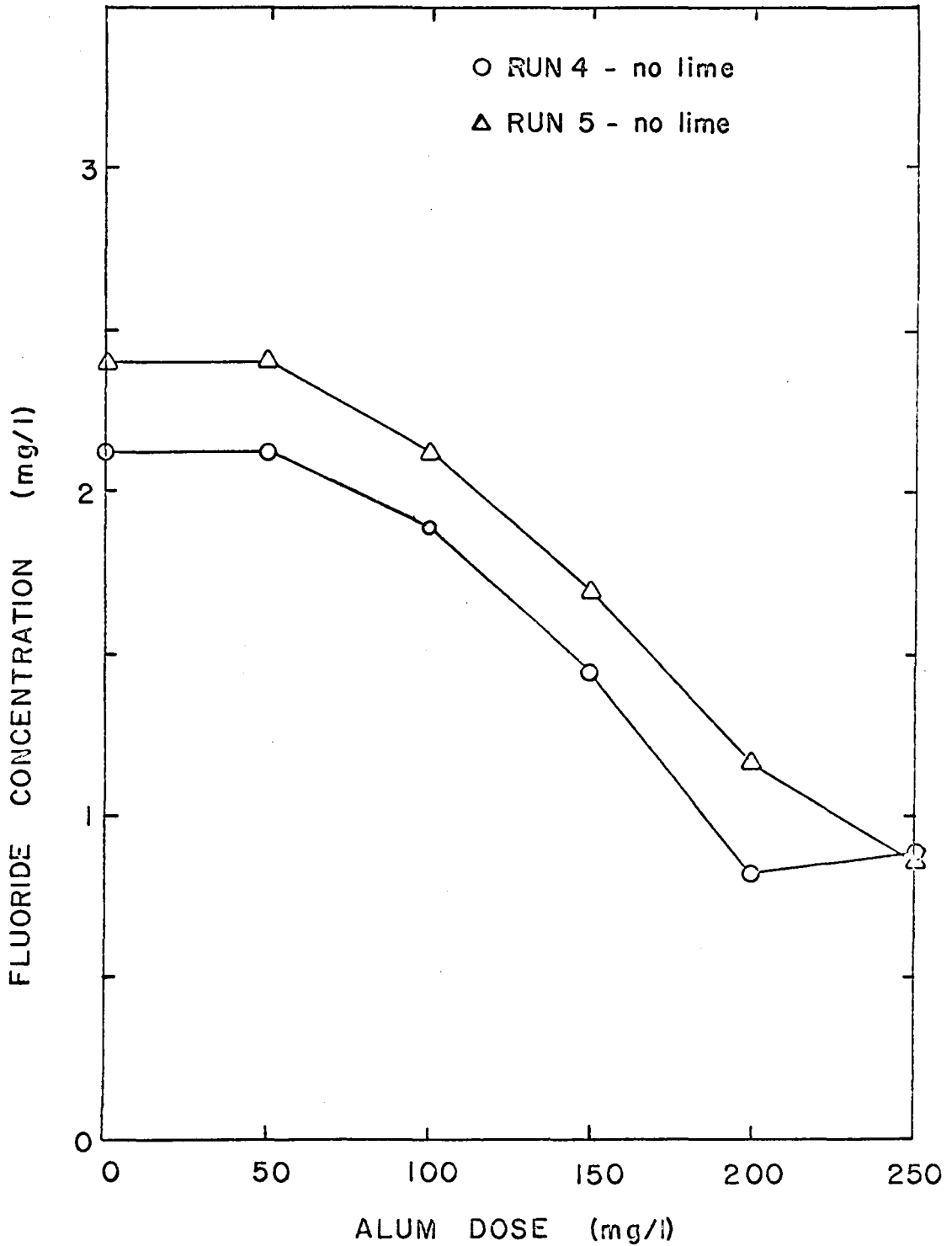


Fig. 4 SERIES 'A' ALUM DOSE vs. FLUORIDE CONCENTRATION

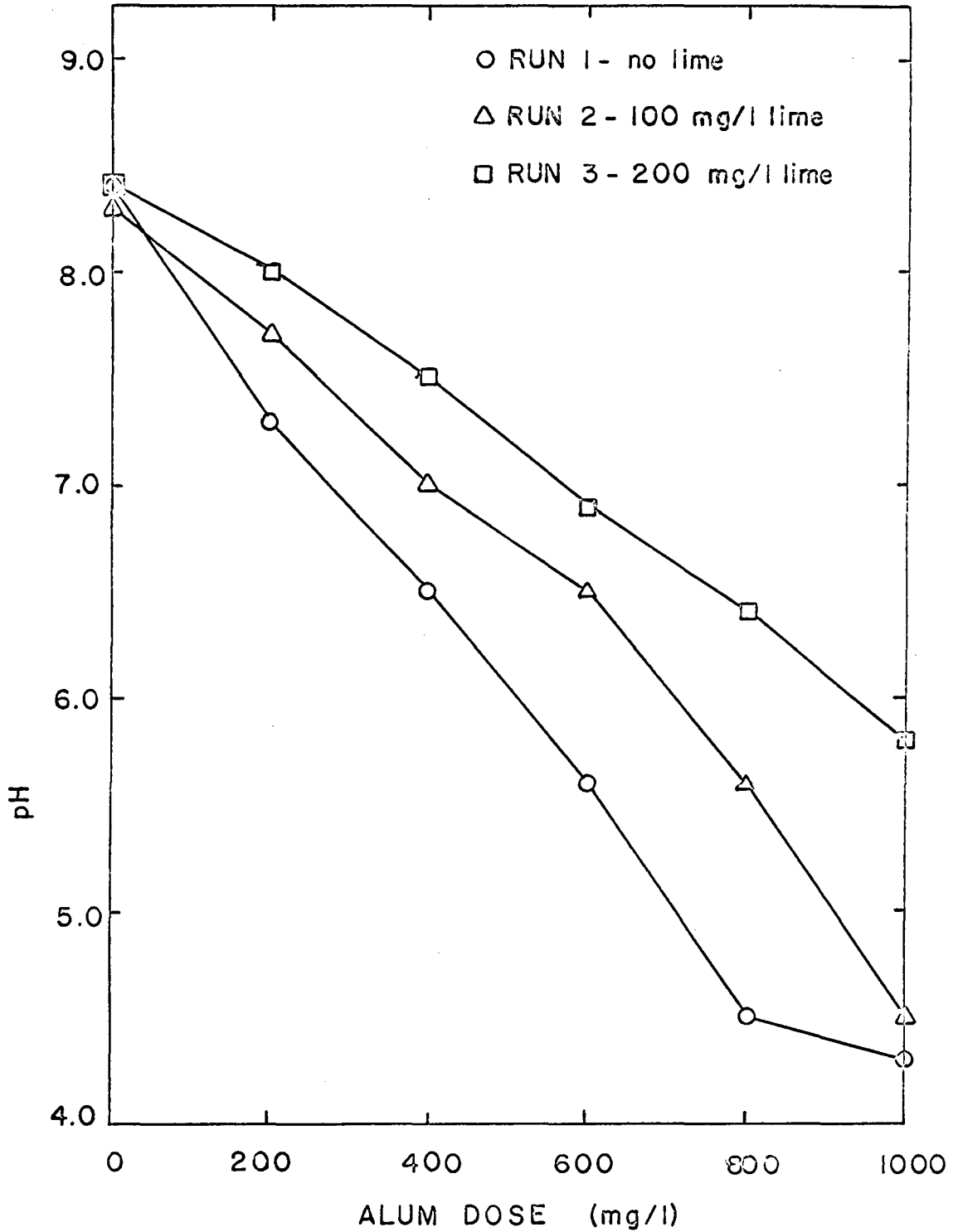


Fig. 5 SERIES 'A' ALUM DOSE vs. pH

TABLE I: ALKALINITY DATA FOR SERIES "A"
Alkalinity mg/l as CaCO₃

Alum Dose mg/l	No Lime	100 mg/l Lime	200 mg/l Lime
0	250	-	-
200	166	220	228
400	86	144	186
600	26	78	130
800	8	26	76
1000	8	16	34

TABLE II: ALUMINUM DATA FOR SERIES "A"

Alum Dose mg/l	Aluminum Residual mg/l		
	No Lime	100 mg/l Lime	200 mg/l Lime
0	Too low to read in all samples		
200	0.1	0.4	0.7
400	0.2	0.4	0.3
600	1.2	0.6	0.1
800	10.9	1.4	0.4
1000	26.0	11.2	0.9

a pH of 6.5.

Series "B" - Wilroy Industrial Park

Figure 6 indicates the fluoride reduction with increasing alum dose for the Wilroy Industrial Park water. The same additions of lime were maintained for the different runs as noted in Series "A". Figure 7 indicated the "fine tuning" to reduce the fluoride to the MCL of 1.8 mg/l for the area. Figure 8 describes further refining to try and reach the recommended optimum level of 0.9 mg/l fluoride. Figure 9 indicates the pH variance with alum dose for the runs as noted above. Table III indicates the change in alkalinity with alum dose and Table IV points the residual aluminum in the unfiltered treated water.

Sludge studies were conducted by treating the raw water with 1100 mg/l alum and 100 mg/l lime. The sludge volume was 12 per cent of the total treated water with total solids of 4276 mg/l and a specific resistance of 9.8×10^{14} m/kg. The treated water had a fluoride residual of 0.8 mg/l, an alkalinity of 144 mg/l as CaCO_3 , a hardness of approximately 250 mg/l as CaCO_3 , and a pH of 6.3.

Series "C" - City of Portsmouth Water Treatment Plant

Figure 10 indicates the reduction in fluoride with increasing alum dose for the Portsmouth water. Again the runs reflected the different lime doses as noted in Series "A".

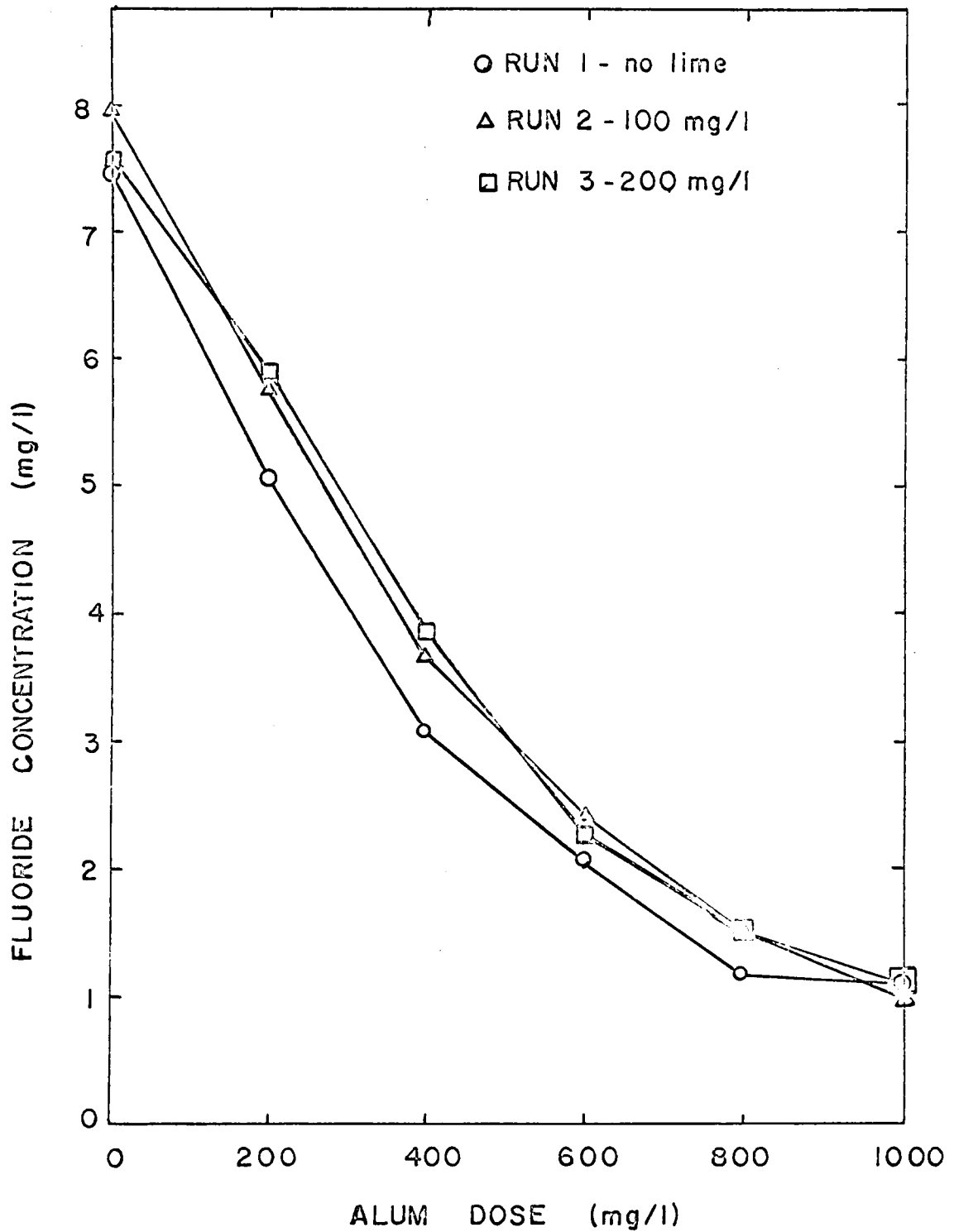


Fig. 6 SERIES 'B' ALUM DOSE vs. FLUORIDE CONCENTRATION

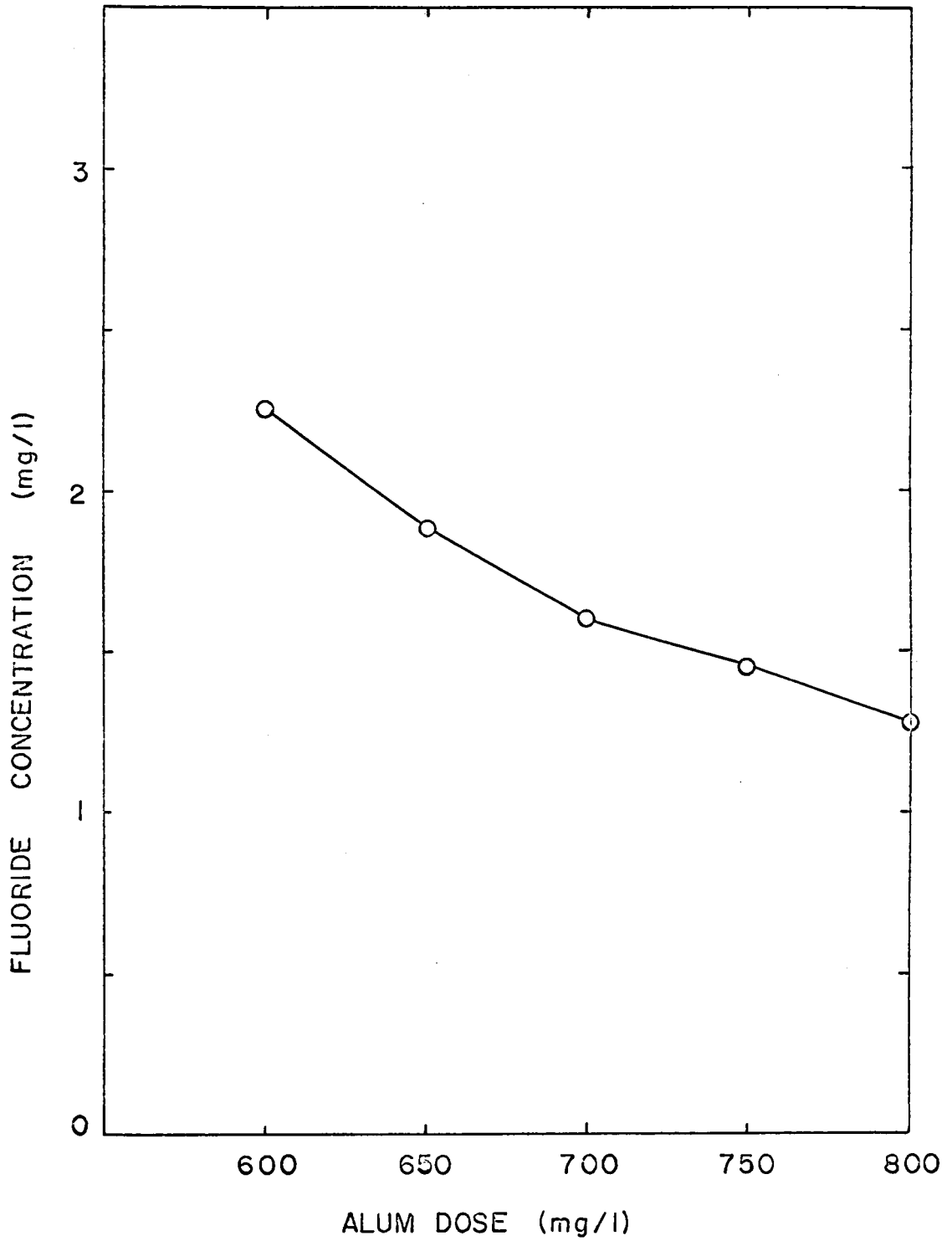


Fig.7 SERIES 'B' RUN 4 (no lime) ALUM DOSE vs.
FLUORIDE CONCENTRATION

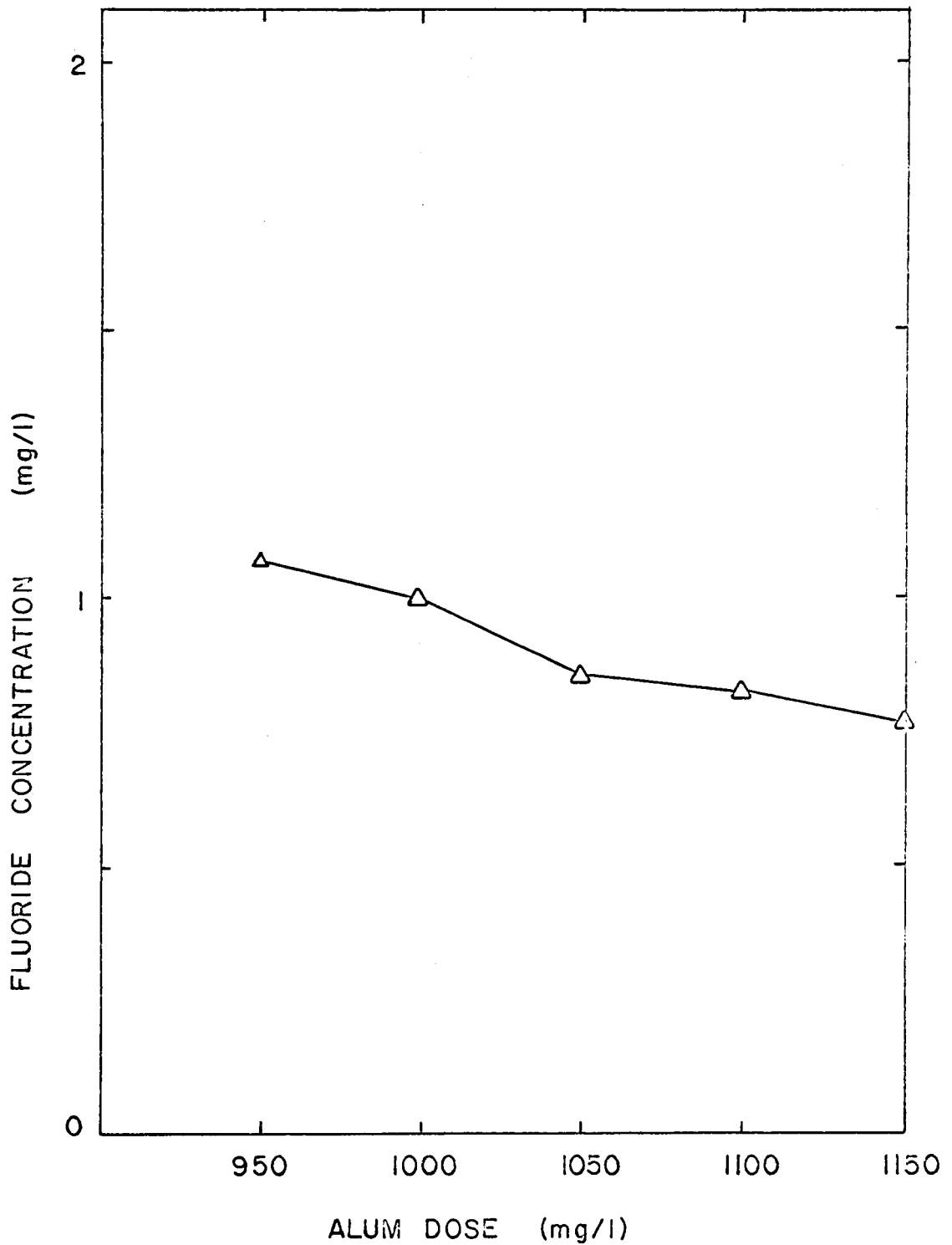


Fig. 8 SERIES 'B' RUN 5 (100 mg/l lime) ALUM DOSE vs. FLUORIDE CONCENTRATION

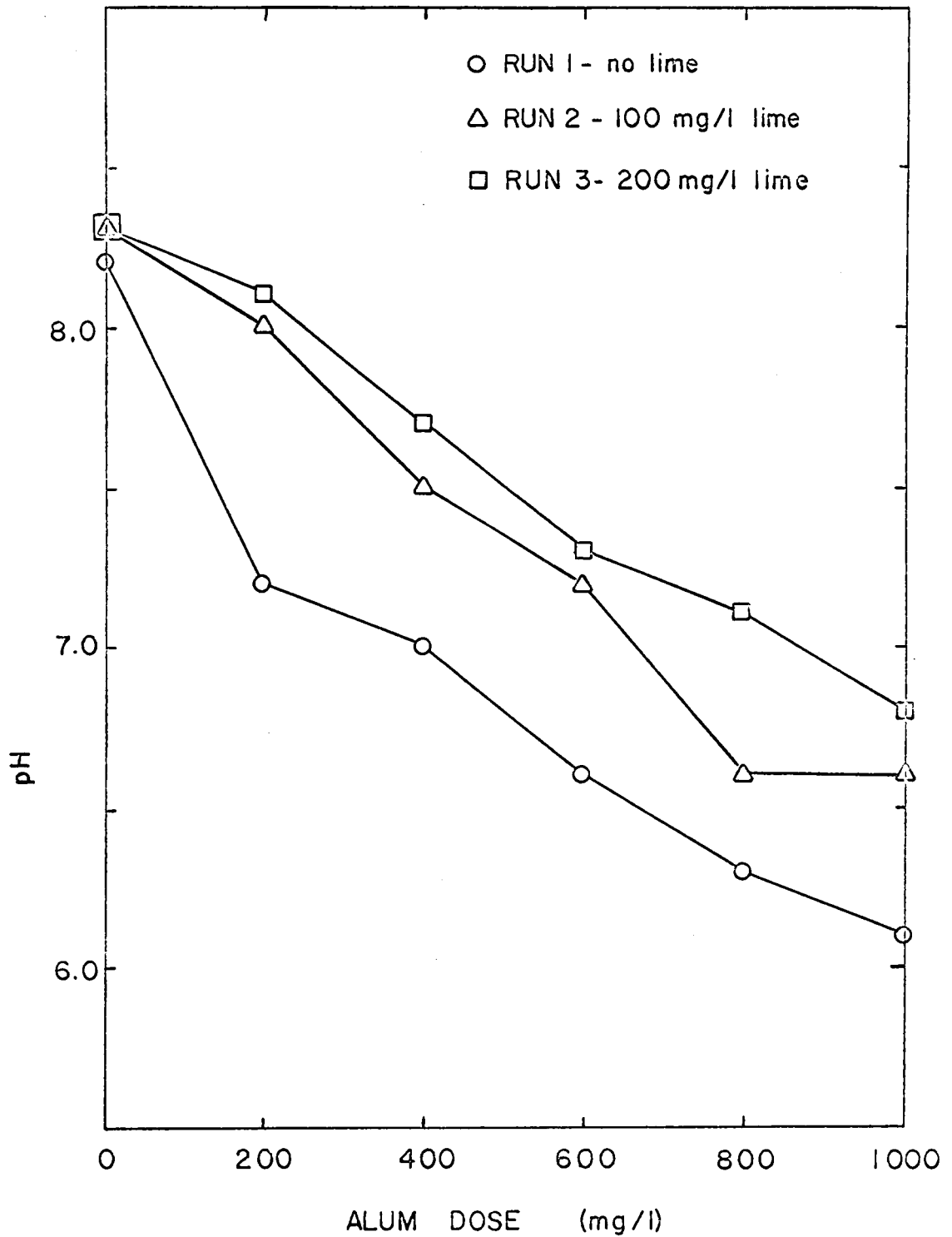


Fig. 9 SERIES 'B' ALUM DOSE vs. pH

TABLE III: ALKALINITY DATA FOR SERIES "B"

Alum Dose mg/l	Alkalinity mg/l as CaCO ₃		
	No Lime	100 mg/l Lime	200 mg/l Lime
0	550		
200	460	482	482
400	358	430	440
600	280	354	390
800	194	280	332
1000	126	196	278

TABLE IV: ALUMINUM DATA FOR SERIES "B"

Alum Dose mg/l	Aluminum Residual mg/l		
	No Lime	100 mg/l Lime	200 mg/l Lime
0	Too low to read in all samples		
200	0.5	0.6	0.7
400	0.3	0.3	0.3
600	1.0	0.6	0.4
800	0.8	0.5	0.3
1000	1.1	1.0	0.3

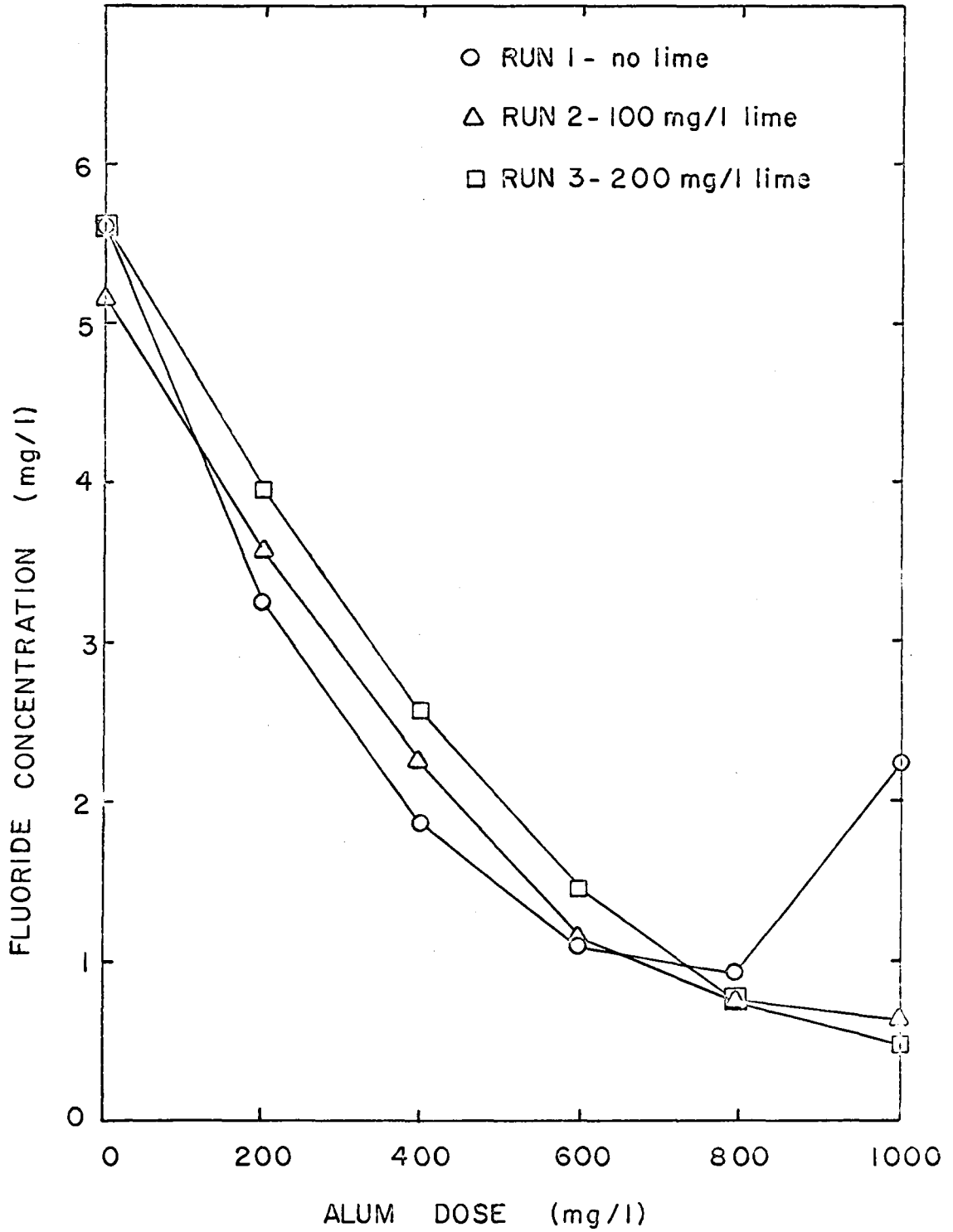


Fig. 10 SERIES 'C' ALUM DOSE vs. FLUORIDE CONCENTRATION

Figure 11 indicates the attempt to reach the MCL of 1.8 mg/l for the area and Figure 12 illustrates the attempt to achieve the recommended optimum level of 0.9 mg/l fluoride. Figure 13 indicates the pH variance with alum dose for the runs as noted above. Table V lists the change in alkalinity with alum dose and Table VI lists the residual aluminum in the unfiltered treated water.

Sludge studies were performed by treating the raw water with 1000 mg/l alum and 200 mg/l lime. The sludge volume was 9 percent of the tested water with total solids of 3685 mg/l and a specific resistance of 1.9×10^{14} m/kg. The treated water had a fluoride residual of 0.6 mg/l, an alkalinity of 150 mg/l as CaCO_3 , a hardness of 192 mg/l as CaCO_3 , and a pH of 6.6.

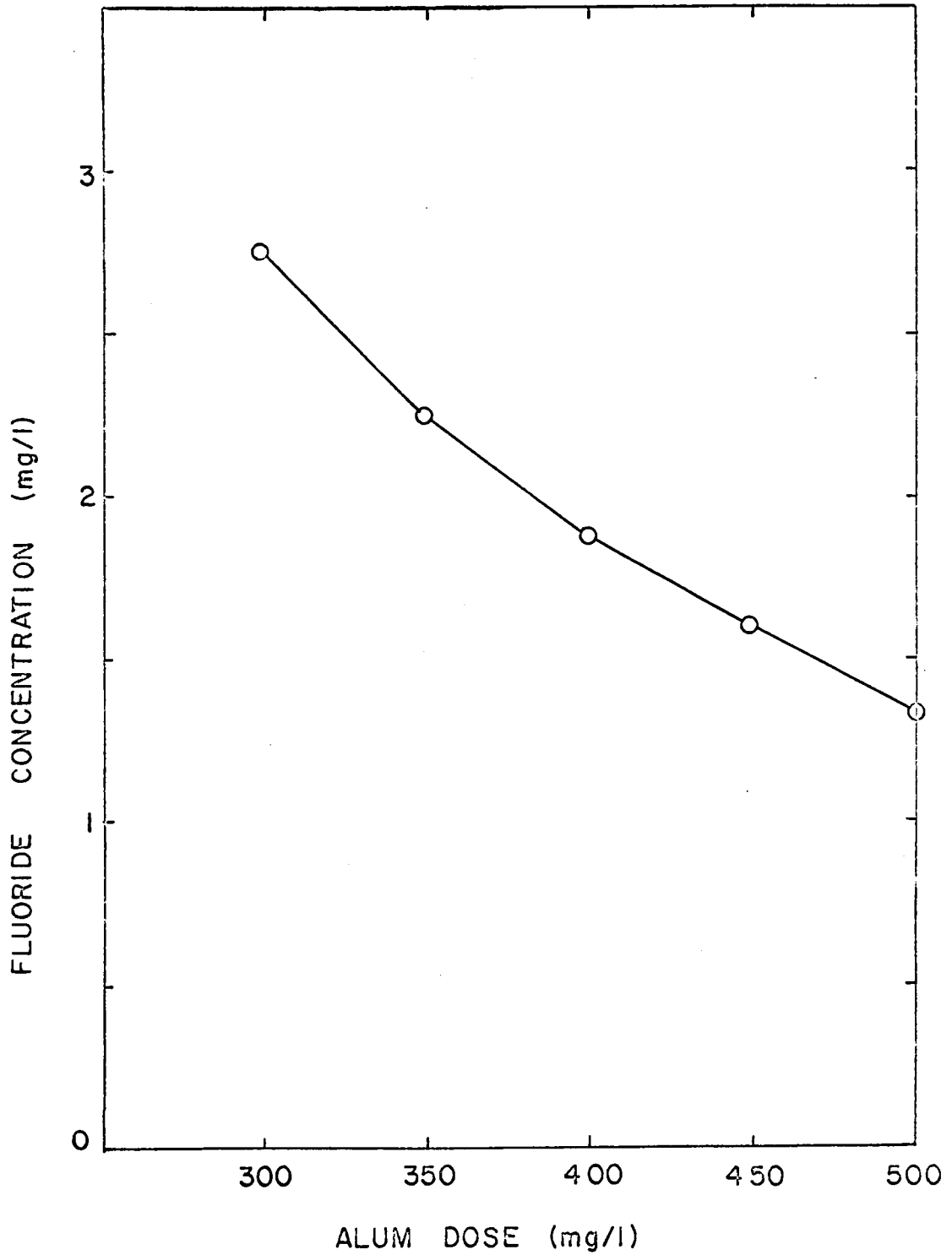


Fig. II SERIES 'C' RUN 4 (no lime) ALUM DOSE vs.
FLUORIDE CONCENTRATION

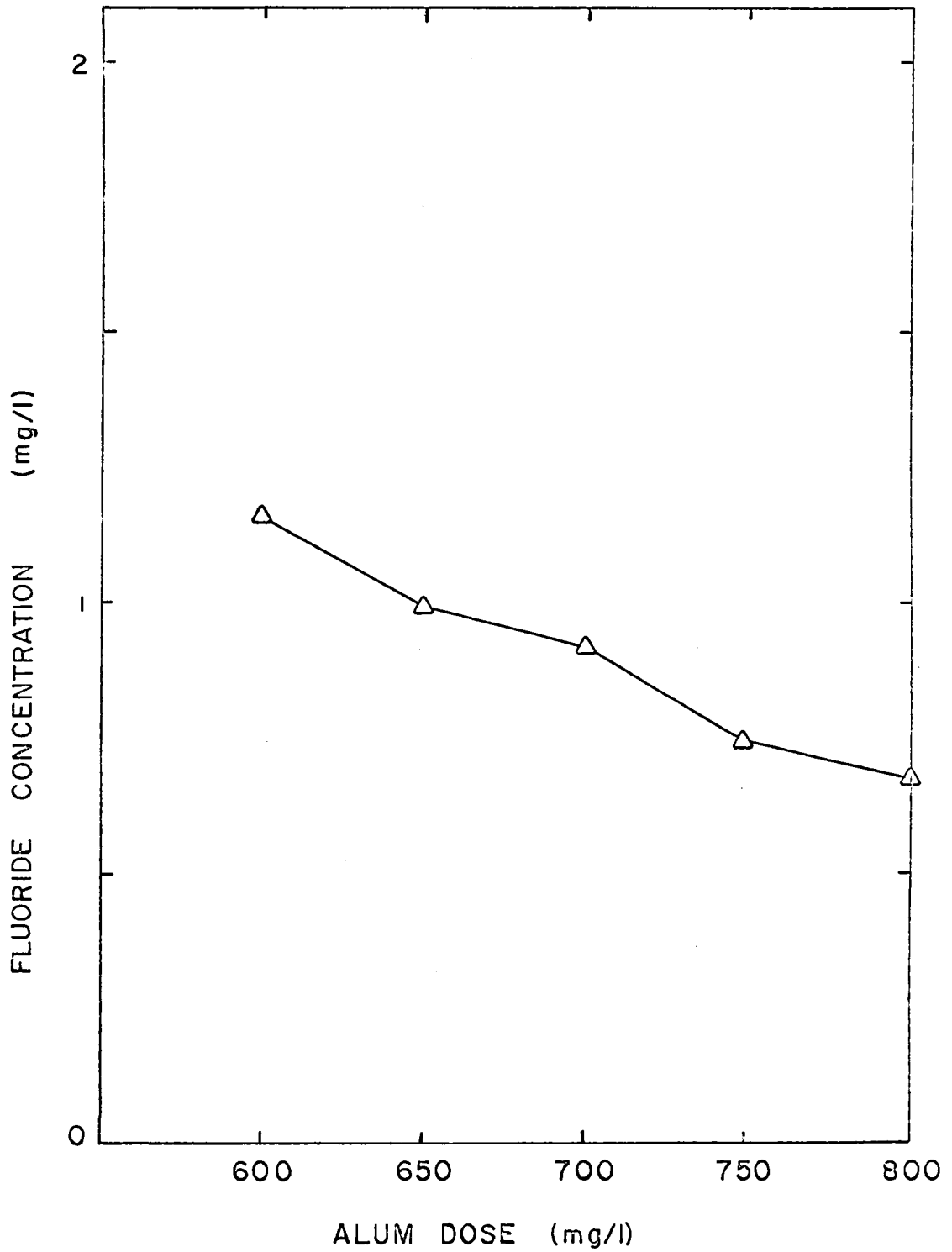


Fig. 12 SERIES 'C' RUN 5 (100 mg/l lime) ALUM DOSE vs. FLUORIDE CONCENTRATION

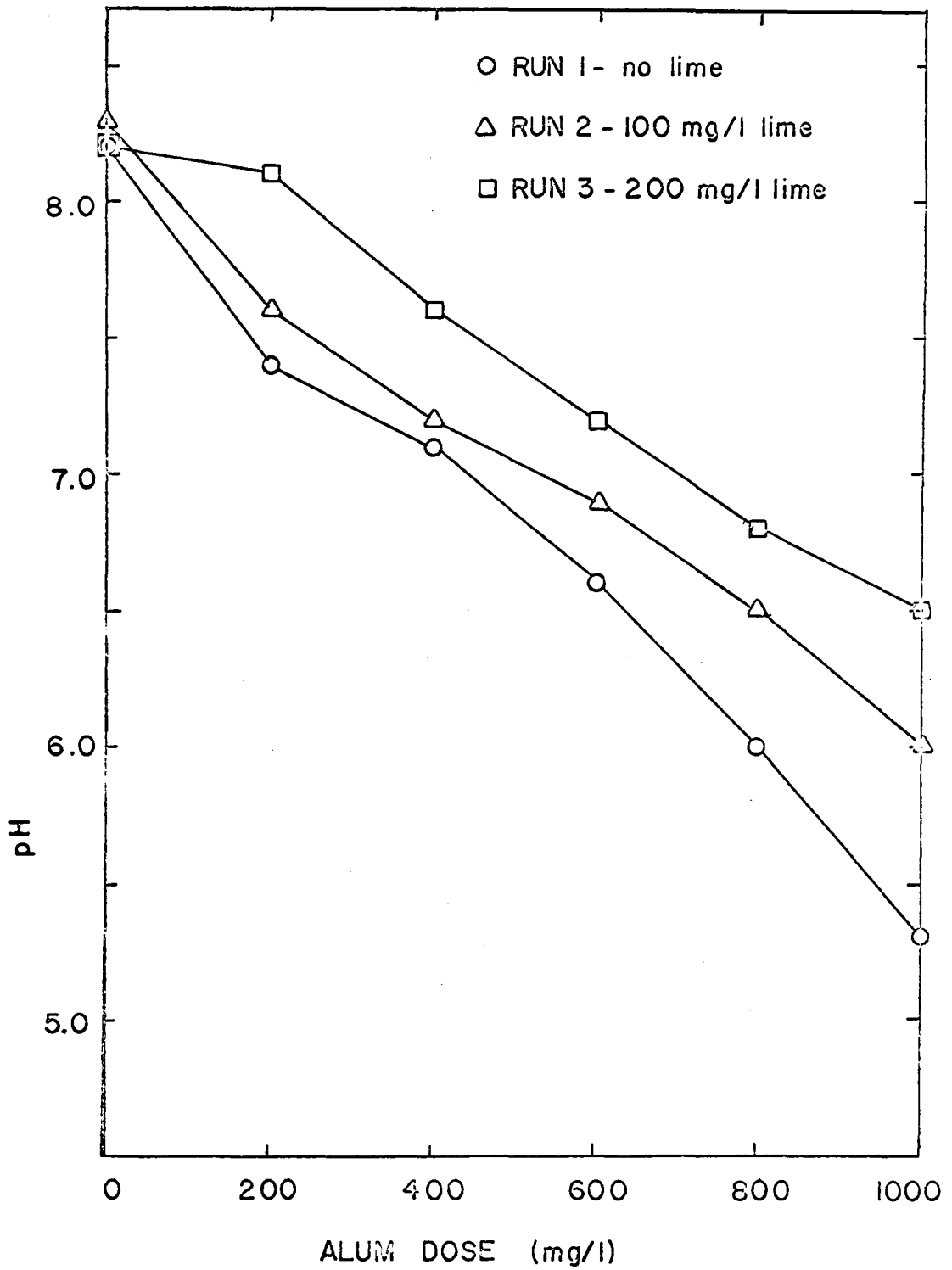


Fig. 13 SERIES 'C' ALUM DOSE vs. pH

TABLE V: ALKALINITY DATA FOR SERIES "C"

Alkalinity mg/l as CaCO₃

<u>Alum Dose</u> <u>mg/l</u>	<u>No Lime</u>	<u>100 mg/l</u> <u>Lime</u>	<u>200 mg/l</u> <u>Lime</u>
0	406		
200	316	376	362
400	224	298	330
600	148	218	268
800	80	160	208
1000	36	84	150

TABLE VI: ALUMINUM DATA FOR SERIES "C"

Alum Dose mg/l	Aluminum Residual mg/l		
	No Lime	100 mg/l Lime	200 mg/l Lime
0	Too low to read in all samples		
200	0.2	0.4	0.6
400	0.3	0.2	0.1
600	0.9	0.3	0.2
800	1.1	0.4	0.2
1000	3.0	0.6	0.4

Chapter 5 - DISCUSSION OF RESULTS

The results given in Chapter 4 are discussed in this chapter under the following headings: Fluoride Reduction, pH Changes, Hardness Changes, Aluminum and Sulfate Changes, Sludge Dewatering, and Costs of Treatment. The raw waters tested will be covered under these headings.

Fluoride Reduction

Figure 4 indicates that in order to reduce the fluoride concentration from approximately 2.1 mg/l found in the Chippokes Plantation State Park raw water to the MCL, an average of 125 mg/l alum without lime was required. By increasing the alum feed to an average of 220 mg/l, the recommended optimum level of 0.9 mg/l was obtained. Figure 3 discloses that the minimum value to which the fluorides were reduced from the initial value of 2.1 mg/l without lime addition was 0.4 mg/l while with 100 mg/l lime the lowest figure was 0.25 mg/l and with 200 mg/l lime, 0.15 mg/l.

The amount of alum necessary to reduce the fluoride concentration from approximately 8.0 mg/l found in the "spiked" Wilroy Industrial Park raw water to the MCL was 670 mg/l without lime (see Figure 7). In order to reach the recommended optimum level, it was necessary to increase

the alum dose to 1035 mg/l with 100 mg/l lime, as indicated in Figure 8. There did not appear to be any appreciable difference between the minimum fluoride value obtained with or without lime addition. Figure 6 shows that, within the range of alum concentration studies, the minimum resulting fluoride concentration was approximately 1.0 mg/l. It is evident in Figure 8 that a reduction to 0.8 mg/l can be obtained, with a 1150 mg/l alum dose and 100 mg/l lime.

When the fluoride concentration was approximately 5.5 mg/l, as found in the City of Portsmouth well water, 415 mg/l alum without lime was required to reach the MCL (Figure 11). To further reduce the fluoride level to the recommended optimum concentration 710 mg/l alum with 100 mg/l lime was needed, (Figure 12). The minimum fluoride value obtained with the 5.5 mg/l initial fluoride without lime addition was 0.9 mg/l, with 100 mg/l lime, the minimum value was 0.6 mg/l, and with 200 mg/l lime the minimum value was 0.4 mg/l (Figure 10).

These figures and values indicate that fluoride in the groundwaters of eastern Virginia can be reduced to or below the MCL by using alum. The level of reduction depends on the amount of alum employed. In some cases, further reduction requires the addition of lime to keep the pH at a level appropriate to provide adequate coagulation of the alum.

pH Changes

Figures 5, 9, and 13, all show the decrease in pH with increasing alum dose. It appeared that good fluoride reduction was achieved down to pH 6.0 at which time the concentration of fluoride and aluminum in the treated water was increased. The addition of the lime provided the increase in pH necessary to allow the alum to hydrolyze and ultimately led to the removing of the fluoride.

Hardness Changes

Due to the low hardness found in the raw waters, 3 mg/l as CaCO_3 in Series "A", 26 mg/ as CaCO_3 in Series "B", and 3 mg/l as CaCO_3 in Series "C", hardness tests were not performed on the treated water. In the course of the sludge studies, hardness tests were made on the treated waters. These tests indicated that the hardness increased to 190 mg/l as CaCO_3 for Series "A" approximately 250 mg/l as CaCO_3 for Series "B" and 192 mg/l as CaCO_3 for Series "C". Series "A" and "C" had 200 mg/l lime added to the raw water while Series "B" had 100 mg/l lime added. The increase can be attributed to the addition of lime to the raw water. In order to keep the hardness from increasing, the use of soda ash should be considered.

Aluminum and Sulfate Changes

Standard Methods (25) states that filtered water should have an aluminum concentration of not greater than 0.05

mg/l. Culp and Culp (26) state that a level exceeding 0.05 mg/l aluminum may precipitate upon standing or in the distribution system. This study indicated that the unfiltered aluminum concentration ranged from 0.1 to 0.4 mg/l at the higher pH (above 6.0). No data was obtained as to the expected aluminum reduction as the treated water passes through a standard or high rate rapid sand filtration process.

The Commonwealth of Virginia Waterworks Regulations state that the esthetic limit for sulfates is 250 mg/l. No sulfate tests were made on the treated waters. However, the chemical analysis of the raw water indicate low sulfate readings (See Appendix). For each 100 mg/l alum added to the raw water, there will be an increase in sulfate concentration of 43.24 mg/l. Any future investigations of fluoride reductions using alum should include an analysis of sulfates. Table VII indicates the increase in sulfate with alum addition to meet the MCL and the recommended optimum level for fluoride concentration.

Sludge Dewatering

The sludge volume of each of the treated waters ranged from 7 to 12 per cent of the amount treated. The higher the chemical dose, the higher was the volume of sludge. This result indicates that approximately 10 per cent of the settling basin must be set aside for sludge storage

TABLE VII - INCREASE IN SULFATES WITH ALUM ADDITION
TO MEET THE MCL AND THE RECOMMENDED
OPTIMUM LEVEL FOR FLUORIDE

Raw Water Source	Alum Dose to meet MCL mg/l	Sulfate Increase mg/l	Alum Dose to Meet Recommended Optimum Level mg/l	Sulfate Increase mg/l
Series A	125	54	220	95
Series B	670	290	1035	447
Series C	415	180	710	307

when fluorides are to be reduced to the lower values.

The total sludge solids after settling ranged from 0.15 to 0.4 per cent (1500 to 4000 mg/l). This result indicates a sludge with a very high water content was produced and must be dewatered before disposal.

The specific resistance ranged from 1.8×10^{14} to 9.8×10^{14} m/kg (to convert to sec^2/g divide by 9.81×10^3). This test indicates a sludge which is difficult to dewater without the aid of a polymer to concentrate the sludge and to change its physical-chemical properties.

Costs of Treatment

The July, 1977, cost of alum and lime in the City of Suffolk was \$3.77 and \$2.59 per 100 pounds, respectively. Tables VIII and IX gives the predicted chemical costs to treat the waters under investigation to the MCL and the recommended optimum level. As would be expected, the higher the fluoride concentration, the higher the cost of removal. Likewise, the further the reduction desired, the higher the cost. These costs do not reflect any other costs other than the chemicals; other costs such as ultimate sludge disposal, salaries, construction, etc. are not included.

TABLE VIII - CHEMICAL COSTS TO MEET THE MCL
FOR FLUORIDE

Raw Water Source	Alum Dose mg/l	Lime Dose mg/l	Chemical Costs \$/1000 gallons
Series A	125	0	0.04
Series B	670	0	0.21
Series C	415	0	0.13

TABLE IX - CHEMICAL COSTS TO MEE THE RECOMMENDED
OPTIMUM LEVEL FOR FLUORIDE

Raw Water Source	Alum Dose mg/l	Lime Dose mg/l	Chemical Costs \$/1000 gallons
Series A	220	0	0.07
Series B	1035	100	0.35
Series C	710	100	0.25

Chapter 6 - CONCLUSIONS & RECOMMENDATIONS

The following conclusions were reached based upon this study:

1. Alum alone or in combination with lime will reduce the fluoride concentration of the groundwaters of eastern Virginia to or below the MCL.

Based on the waters in this investigation, it required 125 mg/l alum to reduce the fluoride concentration from 2.1 mg/l to 1.8 mg/l, 415 mg/l alum was used to reduce the fluoride concentration from 5.5 mg/l to 1.8 mg/l, and 670 mg/l alum was utilized to reduce the fluoride concentration from 8.0 mg/l to 1.8 mg/l.

2. The pH of the water being treated must be kept above 6.0 in order to obtain proper fluoride reduction without adding excessive dissolved aluminum to the system.

3. The chemical costs for the method are very high, but could be reduced in problem areas by a dual system where all drinking water is treated and water for other purposes is not.

4. The sludge volume produced will require approximately 10% additional basin volume in the settling tank for sludge storage, if continuous sludge removal equipment is not provided.

5. The sludge produced will have a high water content and a high specific resistance which may require polymer addition to change its physical-chemical properties.

This study indicated that the following should be considered for further investigation:

1. In order to keep the hardness low and still maintain the pH, another chemical, in lieu of lime, such as soda ash, should be explored.

2. In order to reduce the sulfate buildup, other aluminum compounds, such as aluminum chloride should be tested.

3. Studies with polymers should be made to determine the most appropriate method of sludge dewatering.

4. Studies using polymers in the treatment process with alum and alum and lime to determine if the flocculation and settling times can be adjusted to optimum conditions should be made.

5. Any future work using the fluoride specific ion electrode should be with a digital readout pH/Ion meter.

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New York, N. Y., 1974.

APPENDIX

Chemical Analysis

Chippokes Plantation State Park	54
Wilroy Industrial Park	55
City of Portsmouth Deep Well # 1	56

April 1974
 DIVISION OF CONSOLIDATED LABORATORY SERVICES
 BUREAU OF ENVIRONMENTAL SCIENCE

Out: 2 June 1974

Laboratory #: _____ B.S.E. # : 18522
 City/County: Surry Collected by: J.I. Capito
 Date Collected: 12 February 1974 Place: Chipokes Plantation State Park

RECEIVED
 JUN 10 1974

Source: _____
 Other Information: _____

FIELD TESTS: Color: _____ Odor: _____ Turbidity: _____
 Temp.: _____ Cl₂: _____ pH: _____ Alkalinity (8.3): _____
 Hardness: _____ Ca Hardness: _____ Other: _____

Acidity (OC.)	
Alkalinity (8.3)	0.0
(Total-4.5)	241.0
Aluminum	0.5-
Arsenic (0.01)* (0.05)**	0.003-
Barium (1.0)*	0.5-
C.O.D.	
Boron	
Cadmium (0.01)**	0.001-
Calcium	0.41
Ca Hardness	1.02
Carbon - Total Inorganic	
Total Organic	
CO ₂ (free)	
C.O.D.	
Chloride (250)*	1.0
Pesticides 1.	
Chlorine	
Chromium (+6) (0.05)**	0.01
(Total)	0.01
Color (pH: 8.3)	5- CU
Copper (1.0)*	0.01-
Cyanide (0.01)* (0.2)**	
Fluoride	2.1
Hardness (TDTA)	3.0
(by calculation) 2.	2.25
Iron (Total) (0.5)*	0.22
Lead (0.05)**	0.01-
Lithium	
Magnesium	0.30
Mg Hardness	1.25
Manganese (0.05)*	0.01
Mercury (0.001)**	0.0005-
Nickel	0.10-
Nitrogen-Ammonia	0.10-
NH ₃	0.12-
NH ₄	0.15-
Nitrogen-Nitrate	0.01-
NO ₃ (45)*	0.03-

Concentrations in Mg/l unless otherwise noted

Nitrogen-Nitrite	0.01-
NO ₂	0.03-
Total Kjeldahl	0.10-
Organic Contam. (CAB)	
(CCB) (0.2)*	
Oxygen (dissolved)	
Petroleum Contaminants	
pH	8.3
Phenols (0.001)*	
Phosphate (total)	0.92
(total-soluble)	0.92
(ortho-total)	0.92
(ortho-soluble)	0.92
(condensed-total)	
(condensed-soluble)	
Potassium	4.75
Residue/Total (185°C)	201.0
Volatiles	40.0
Fixed (550°C)	161.0
Residue/Filtrable (185°C) (550)*	201.0
Volatiles	40.0
Fixed (550°C)	161.0
Residue/Nonfiltrable (185°C)	0.0
Volatiles	0.0
Fixed (550°C)	0.0
Selenium (0.01)**	
Silica (as SiO ₂)	30.0
Silver (0.05)**	
Sodium	115.0
Specific Conductance (µ mho/cm)	462.584
Strontium	0.01
Sulfate (250)*	3.8
Sulfide	0.001-
Sulfite	0.01-
Surfactant (0.5)*	0.01-
Tannin & Lignin	
Turbidity (N.T.U.)	0.02
Zinc (5.0)*	0.01-

* Recommended Maximum
 ** Maximum Chemical Limit

- (Chlorinated & thiophosphate Pesticides)
- (From Ca, Mg, Sr, Fe, Al, Zn & Mn)

Chemist: ROBERT D. POTTS

LABORATORY REPORT

Date: APR 21 1977

DIVISION OF CONSOLIDATED LABORATORY SERVICES
BUREAU OF ENVIRONMENTAL SCIENCE

Laboratory #: _____ B.S.E. #: H-946
 City/County: Suffolk Collected by: _____
 Date Collected: 11-3-25-77 Place: Wilson Memorial Park
 Source: Well - sample tap
 Other Information: System owned by City of Suffolk

FIELD TESTS: Color: _____ Odor: _____ Turbidity: _____
 Temp.: _____ Cl₂: _____ pH: _____ Alkalinity (8.3): _____ (4.5): _____
 Hardness: _____ Ca Hardness: _____ CO₂: _____ Other: JUN 6 1977

RECEIVED
 JUN 6 1977

Alkalinity (8.3)	4
(Total-4.5)	460
Aluminum	<0.20
Arsenic (0.05)*	<0.002
Barium (1.0)*	<0.5
Cadmium (0.01)*	<0.01
Calcium	2.86
Ca Hardness	7.14
Carbon - Total Inorganic	9.8
Total Organic	20
Chloride (250)*	150
Chromium-Total (0.05)*	<0.01
Color (pH:8.4) (15CU)*	5 cu
Copper (1.0)*	<0.01
Cyanide (0.2)*	<0.01
Fluoride	5.19
Hardness (EDTA)	26.0
(by calculation) 1.	19
Iron-Total (0.3)*	0.22
Lead (0.05)*	<0.003
Magnesium	2.88
Mg Hardness	11.85
Manganese (0.05)*	<0.01
Mercury (0.002)*	<0.0005
Nickel	<0.10
Nitrogen-Ammonia	<0.10
NH ₃	<0.12
NH ₄	<0.13
Nitrogen-Nitrate	0.31
NO ₃ (45)*	1.37
Nitrogen-Nitrite	<0.01
NO ₂	<0.03
Total Kjeldahl	<0.10
Organic Contam. - CCE (0.7)*	

(Concentration in Mg/l unless otherwise noted)

pH	
Phenols	
Phosphate-Total	0.92
Total/soluble	0.92
Ortho/total	0.86
Ortho/soluble	0.86
Condensed/total	0.06
Condensed/soluble	0.06
Potassium	12.6
Residue/Total (185°C)	8.5
Volatile	1.73
Fixed (550°C)	6.81
Residue/Filterable (185°C)	
Volatile	
Fixed (550°C)	
Residue/Nonfilterable (185°C)	
Volatile	
Fixed (550°C)	
Selenium (0.01)*	<0.001
Silica (as SiO ₂)	22.7
Silver (0.05)*	<0.01
Sodium	288
Specific Conductance (µ mho/cm)	17.36
Strontium	0.21
Sulfate (250)*	7.7
Sulfide	<0.01
Sulfite	<0.01
Surfactant (0.5)*	<0.01
Turbidity (J.T.U.) (1)*	0.85
Zinc (5.0)*	0.04

STATS DEPT. OF HEALTH
 BUREAU OF SANITARY ENGINEERING
 NORFOLK REGIONAL OFFICE

1. (From Ca, Mg, Sr, Fe, Al, Zn & Mn)

* Approval

RECEIVED
 APR 27 1977
 DIV. OF ENGINEERING
 STATE DEPT. OF HEALTH
 RICHMOND, VA.

Chemist:

Laboratory: _____
 City/County: Portsmouth Date Collected: 12-22-76 Place: Deep Well #1

Source: Discharge of Deep well #1

Other Information: Owned by City of Portsmouth - used for fluoridation

FIELD TESTS: Color: _____ Odor: _____ Turbidity: _____
 Temp.: _____ Cl₂: _____ pH: _____ Alkalinity (8.3): _____ (4.5): _____
 Hardness: _____ Ca Hardness: _____ CO₂: _____ Other: _____

Alkalinity (8.3)	0.0	pH	7.3
(Total-4.5)	3.6	Phenols	
Aluminum	<0.10	Phosphate-Total	1.84
Arsenic (0.05)*	<0.002	Total/soluble	1.84
Barium (1.0)*	<0.50	Ortho/total	1.05
Cadmium (0.01)*	<0.01	Ortho/soluble	1.05
Calcium	0.60	Condensed/total	<0.01
Ca Hardness	1.65	Condensed/soluble	<0.01
Carbon - Total Inorganic	9	Potassium	2.4
Total Organic	9	Residue/Total (185°C)	5.23
Chloride (250)*	10.5	Volatile	1.72
Chromium-Total (0.05)*	<0.01	Fixed (550°C)	3.51
Color (pH: 7.3) (150U)*	5 u	Residue/Filterable (185°C)	
Copper (1.0)	<0.01	Volatile	
Cyanide (0.2)*	<0.01	Fixed (550°C)	
Fluoride	5.85	Residue/Nonfilterable (185°C)	
Hardness (EDTA)	5.0	Volatile	
(by calculation) 1.	3.67	Fixed (550°C)	
Iron-Total (0.3)*	0.18	Selenium (0.01)*	<0.001
Lead (0.05)*	0.002	Silica (as SiO ₂)	16.1
Magnesium	0.39	Silver (0.05)*	<0.01
Mg Hardness	1.61	Sodium	156.7
Manganese (0.05)*	<0.01	Specific Conductance (µ mho/cm)	221
Mercury (0.002)*	<0.0005	Strontium	0.05
Nickel	<0.10	Sulfate (250)*	1.5
Nitrogen-Ammonia	0.20	Sulfide	<0.01
NH ₃	0.01	Sulfite	<0.01
NH ₄	0.26	Surfactant (0.5)*	<0.01
Nitrogen-Nitrate	<0.05	Turbidity (J.T.U.) (1)*	0.18
NO ₃ (45)*	<0.22	Zinc (5.0)*	<0.01
Nitrogen-Nitrite	0.07		
NO ₂	0.07		
Total Kjeldahl	0.20		
Organic Contam. - CCE (0.7)*			

Concentration in Mg/l unless otherwise noted

1. (From Ca, Mg, Sr, Fe, Al, Zn & Mn)

* Approval Limit

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the scanned document**

DEFLUORIDATION OF TYPICAL EASTERN VIRGINIA GROUNDWATERS

By

JOHN I. CAPITO

(ABSTRACT)

Groundwater on the eastern coast of the State of Virginia exceeds the Environmental Protection Agency Maximum Contaminate Level (MCL) for fluoride. This thesis explores the use of alum and alum and lime for the reduction of fluoride to the MCL or lower. In order to reduce an initial fluoride concentration of 2.1 mg/l to 1.8 mg/l 125 mg/l alum was required, for reduction from 5.5 mg/l to 1.8 mg/l, 415 mg/l alum was needed, and for reduction from 8.0 mg/l to 1.8 mg/l, 670 mg/l alum was utilized. If the system pH was lowered below 6.0 the fluoride reduction was insufficient and thus lime was required to maintain the pH above 6.0.

However, hardness increased with the lime dose and other methods of pH adjustment such as employing soda ash should be considered.

Aluminum was detected in the unfiltered treated water, perhaps in sufficient concentrations to cause problems in the distribution system, but further testing of filtered samples is needed. Sulfates were low in the raw water, but buildup with the addition of alum. No tests were made for sulfates in the treated water.

Tests of the sludge produced by the addition of alum and lime indicated high volumes, low solids and very poor dewatering characteristics. Polymers should be considered as a possible means to achieve better sludge dewatering.