

THE DEVELOPMENT OF A FIELD PROCEDURE FOR DETERMINING THE  
CHLORIDE CONTENT OF CONCRETE AND AN ANALYSIS IN THE  
VARIABILITY OF THE EFFECTIVE DIFFUSION CONSTANT

by

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(ABSTRACT)

During the initial phase of the investigation, four methods were selected for investigation from a review of literature sources, the specific ion probe, spectrophotometer, digital titrator, and Quantab titrator strips.

The initial results from the laboratory testing procedure and evaluation based on cost, speed, accuracy, and level of expertise required indicated the specific ion probe was more suitable for use in the field when compared to the remaining methods selected.

Effects of cement content and reaction temperature on the results obtained for the specific ion probe were also investigated. Results of the tests for the effects of cement content were somewhat inconclusive, but indicated more variability in the results as the amount of chloride present in the specimens increased. However, correlation between the increase in variability and cement content was not indicated. The specific ion probe

is affected by differences in temperature and the appropriate correction factor for the variation was determined.

Field validation of the procedure was undertaken to substantiate the findings from the laboratory investigation. This was accomplished by subjecting the specific ion probe to testing specimens from bridges located in different exposure groups within the United States. Four bridges were tested for chloride content in Pennsylvania. Following this initial phase, three bridges were tested in Virginia, Florida, and Wisconsin respectively.

An analysis of the variability in the effective diffusion constant for the bridges tested was also performed to determine any relationships which exist between different exposure groups and to determine effects of time.

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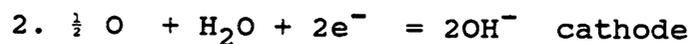
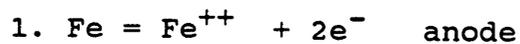
## INTRODUCTION

The deterioration of concrete bridges in the United States has developed into a monumental problem. The seriousness of the decadence was first noted in the early 1960's [1,2] and has grown in magnitude to its present state. The financial consequence of the problem associated with the corrosion of reinforced concrete bridges was recognized in the early 1970's. The Federal Highway Administration estimated the cost of bridge deck repairs at \$70 million per year [3] in 1973. By 1975, the estimate for bridge deck repairs was increased to \$200 million per year [4]. A more recent projection presented in the final report SHRP Research Plans [5] estimates the liability of corrosion induced deterioration in bridges at \$20 billion increasing by a rate of \$.5 billion annually.

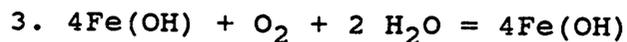
Many of the studies pertaining to concrete bridge deterioration address the corrosion of the reinforcement steel in concrete. The corrosion of reinforcing steel in concrete is influenced by many factors. Corrosion is considered a function of concrete permeability (W/C ratio), consolidation of the concrete during placement, the type of cement, cover depth above the reinforcement steel, relative humidity, and the ions present at the steel surface [6].

In reinforced concrete, a protective layer is provided in the alkaline environment and passivity (resistance to corrosion) exists so long as a lime layer remains in contact

with the surface of the reinforcement steel. Two mechanisms are considered to cause the passive layer to be destroyed in a high alkaline environment.[7] The first mechanism is the reduction of alkalinity by leaching of water or a reaction with carbon dioxide or some other acidic material to cause a partial neutralization of the passive layer. The second mechanism involves the chloride ions in an electrochemical reaction in the presence of oxygen. The reactions at the anode and cathode were reported by Hausmann [8] by:



After combining these two reactions ferrous hydroxide may react with water and oxygen in the following manner:



which is much larger than the volume of steel. Other reactions involving Fe and H<sub>2</sub>O are also reported [9], but ultimately, the pressures induced cause a rupture in the concrete above the surface of the reinforcing steel. Once the concrete surface has cracked, the chloride penetration rate increases which results in an increase in the corrosion rate of the steel.

The primary sources of the chloride ion detected in concrete bridges are deicing salts, sodium and/or calcium chloride, and sea water spray which infiltrates into the concrete through a series of cracks or by diffusion [10].

Chloride may also be present in the mixing water, admixtures, or aggregates used in the concrete mixture.

Considering the amplitude of concrete bridge deterioration in the United States, procedures have been developed to ascertain the physical condition of concrete bridges. One procedure involves the determination of the chloride content of concrete for the different bridge components. The methods presently being used are tedious, expensive, or may be lacking in accuracy.

Thus, the purpose of this project was to seek out and investigate alternative methods for determining the chloride content of concrete. Four methods were evaluated for cost, speed, accuracy, equipment limitations, and level of expertise required for operation. Ultimately, a field method for determining the chloride content in bridge decks, abutments, beams, and piers was developed and validated in the field.

In addition, the relationships and variability of the effective diffusion constant throughout the United States was analyzed according to various bridge components, different exposure groups, and various combined groups to determine what relationships are present.

With the development of effective diffusion constants and a rapid field method for determining the chloride content of concrete, the chloride contamination level of the various concrete components can be assessed and the most

cost-effective protection and/or rehabilitation method can be identified.

The objective of this research, assessment of the chloride contamination level of concrete bridge components, was accomplished through the following independent but interrelated tasks:

- o Literature Review,
- o Laboratory Evaluation of Test Methods of Chlorides in Concrete,
- o Field Validation of Selected Test Method of Chloride in Concrete, and
- o Analyzing the Variability of Chloride Effective Diffusion Constants.

The following presents the results of the above research tasks.

## LITERATURE REVIEW

### GENERAL PROCEDURE

Manual and computer data base searches were performed to identify test methods used to determine the chloride content of concrete. In addition, sources of information containing the chloride content for various bridges within the United States were identified. The Engineering Indexes Annual from 1988 to 1955, HRIS Abstracts from 1987 to 1979, Chemical Abstracts, and Government Documents were searched.

### Test Methods for the Chloride Content of Concrete

Methods to use to determine the chloride ion content of concrete may be classified as non-destructive and destructive techniques. Non destructive techniques include the dual neutron-gamma ray and the neutron-gamma ray spectroscope. Destructive methods, requiring a drilled powdered sample, include the AASHTO Standard Method, T260-84, potentiometric titration method, X-ray fluorescence, gas chromatograph, quantab chloride titrator strips, specific ion electrode, spectrophotometer, and argentometric digital titrator. The following presents the initial evaluation of the chloride ion content of concrete test methods used to select the four test methods to be included in the laboratory evaluation study.

The dual neutron-gamma ray technique was developed for the Federal Highway Administration (FHWA) by Columbia

Scientific Industries [11,12]. The instrument is capable of measuring chloride content with respect to depth having detection limits of 0.04 to 0.08 lbs/c.y and depth resolutions of 0.40 to 8 inches, depending on the calibration model. The choice of the wrong calibration model ( $\text{Cl}^-$  depth distribution model) will result in significant measurement errors. Calibration models would have to be developed for specific types of bridges (e.g., concrete or steel beams, varying deck thickness, varying overlay thickness, coastal or inland bridges) [12]. The method is fast, 2 to 3 measurements per hour with a 10 minute data acquisition time. Instrument operation is straightforward and requires little training. However, instrument calibration and setup must be done by qualified scientists. Although operational costs are relatively low, capital costs are very high. Also, the equipment is very large and is mounted on a self-contained vehicle and thus would be limited to use on bridge decks. However, the instrument can be used to measure the chloride content of 300 to 400 gram powder concrete samples with a 0.25 inch maximum particle size, taken from other instrument-inaccessible bridge members.

Further investigation of the dual neutron-gamma ray technique is not warranted for reasons of cost, number of required chloride distribution calibration models,

portability, limitations, and technical qualifications of setup and calibration personnel.

Neutron-gamma ray spectroscopy also uses a californium-252 neutron source for composition measurements, but uses a high-resolution, high-purity germanium detector rather than a NaI (Tl) crystal gamma-ray detector [13,14,15]. The instrument has been used to measure the relative difference with regard to surface position of the chloride content of a masonry wall by normalizing the chloride intensities to the value of silicon ( $\text{Cl}^-/\text{Si}$  ratio). The procedure assumes the value of silicon remains relatively constant throughout the material. The following presents some of the characteristics of the present neutron-gamma ray spectroscope.

1. The apparatus for building walls consists of a neutron source, liquid nitrogen cooled Ge(HP) gamma-ray detector, amplifier, multichannel analyzer, tape and chart recorder, and readout unit. The apparatus was found to be awkward to transport and set up on building walls. However, smaller portable versions of the multichannel analyzer, amplifiers, and tape deck are commercially available. Similarly, small-sized liquid nitrogen flasks are available. These improvements would increase the portability of the apparatus.
2. For measurements of building walls, the neutron source is placed on one side of the wall and the gamma-ray detector, directly opposite the source on the other side of the wall. This measurement setup cannot be used on most bridge components. However, measurements can be made with the detector and source on the same flat surface if a shield is provided for the source and detector. The increased weight would significantly reduce the portability of the apparatus.

3. The instrument measures the resulting gamma rays from a hemispherical volume with a radius of 6 to 8 inches around the detector and thus measures the average gamma-ray response for the volume of about 460 to 1100 in<sup>3</sup>. The instrument detects relative differences between average chloride contents because no general linear relation exists between the intensity and the concentration of an element [14]. To measure absolute values, the development of calibration standards would be required for various normalized chloride contents (normalized to silicon). Thus, calibration standards would have to be developed for varying amounts of silicon and chloride contents. In addition, the presence of iron and water also influences intensity, and calibration standards regarding the influence of these elements must be developed.
4. The cost of the equipment is on the order of \$100,000, and skilled technicians are required to maintain the equipment [15]. In addition, because each bridge or group of bridges may require some adaptation, the apparatus may be most productive when operated by specialists with a knowledge of nuclear physics. It has been suggested that the technique may best be used through the services of specialized consulting firms [15].
5. The technique, in measuring the average chloride content of a volume of concrete, may be of limited value to the application of measuring the chloride content of reinforced concrete bridges because it is capable of measuring only the magnitude of chloride content, but not changes of 30 - 40 percent [16]. Thus, one may be unable to distinguish between the various levels of chloride distribution, particularly since the measured volume is hemispherical.

The Goddard Space Flight Center is under contract with the National Science Foundation (NSF) to do basic research in the development of calibration models for neutron-gamma ray spectroscopy, and initial results seem promising [16].

Given the current state of development of the technique, the apparent portability problems, calibration

requirements and cost, further investigation of the technique is not warranted.

X-ray fluorescence and gas chromatography analysis methods of powder samples offer no advantage over other powder analysis methods because of the related power requirements (X-ray fluorescence requires approximately a 100-kv source), costs (between \$25,000 and \$100,000 for equipment only), and level of expertise required. These limitations would restrict the use of the instruments to laboratory use. Therefore, continued evaluation of the instruments is not warranted.

Quantab chloride titrator strips are simple and fast to use and are applicable to field measurement techniques of powder samples. However, some problems have been encountered in interpreting the results. The National Ready Mixed Concrete Association has developed a method for measuring the chloride ion content of freshly mixed concrete using Quantab titrators. The method has not been standardized, nor has it been subjected to multilab cooperative testing to derive its precision. However, some additional investigation is warranted because of the relatively low cost and simplicity of the method [17].

Simplified procedures for the 'Quantab' method were also developed by the Building Research Station, Garston, Wartford for analyzing the chloride content in portland cement concrete. The analysis procedure developed for the

Quantab titrator strips consists of an acid digestion of a 5 gram sample of powdered concrete. Fifty milliliters of 1 N nitric acid solution were used to digest the chloride followed by neutralization of the acid using 5 grams of anhydrous sodium carbonate. Chloride concentration of the sample was then determined using the Quantab titrator strips [18].

The Building Research Station, Garston, Watford, also developed a simplified procedure for the chloride determination in portland cement concrete using the 'Hach' (argentometric) test method. In this procedure, the 5 gram powdered concrete sample is as acid digested with 50 milliliters of 1 N nitric acid. While stirring the sample mixture, 5 grams of sodium bicarbonate are added to the sample. Once completely dissolved, the sample is allowed to stand for 1 to 2 minutes. Finally, the sample is filtered into a beaker using Whatman 41 or an equivalent filter paper. The filtered solution is then titrated by pouring 5.75 milliliters of the filtrate into a mixing bottle. One capsule of potassium chromate powder indicator is added to the sample. The sample is swirled to mix. A silver nitrate solution is added drop by drop, swirling the sample after each drop. The sample is titrated from a bright yellow to faint reddish brown color and the number of digits required to complete the titration is recorded [18].

The techniques developed by the Building Research Station were proposed methods to obtain preliminary results for the chloride content in portland cement concrete. Little expertise were required in obtaining reproducible results at the site or other 'convenient places'. Both procedures were noted for having less accuracy than standard laboratory procedures. Consequently, use of the 'Quantab' method and the 'Hach' test procedures were recommended as a screening process to identify chloride contaminated samples which require a more detailed analysis. The results could also be used as a justification for the additional expenditure associated with the laboratory procedure [18].

The most promising methods based on initial equipment cost, level of expertise required, and portability which may be adapted to the rapid field measurement of the chloride content of reinforced concrete using powder samples are the specific ion electrode (James Instruments), spectrophotometric (Hach Instruments), argentometric digital titration (Hach Instruments) methods, and Quantab titrator strips. The direct measurement of chloride content in a drill hole using a specific ion electrode was excluded because it is applicable only to horizontal surfaces [19], and, if a hole is to be drilled, little advantage can be realized over the powder sample measurement method.

Based on the initial equipment costs, level of expertise required to use the equipment, ruggedness for

field use, portability, and accuracy the specific ion probe, spectrophotometric, digital titrator and the Quantab titrator strips were selected for further laboratory analysis for accuracy of results compared to the standard potentiometric titration method. The standard method is too costly and complex and not sufficiently rugged for field use.

#### Chloride Effective Diffusion Constant

The identified literature on the chloride content data for bridges within the United States was compiled and reviewed. All the chloride content data identified was for bridge decks. Various characteristics of the data were summarized to identify the variations in data collection and presentation.

The chloride content data, as presented in the literature for bridge decks, is not standardized. The chloride data for bridge decks are presented in the form of individual cores of bridge decks, average values for bridge decks, and average values for several bridges within a region. Most of the literature contained chloride content data with regard to depth of the sample which is essential, but also represented bridge decks with some type of overlay. Published data for the chloride content of bridge decks with overlays are of little value in the determination of the

effective diffusion constant unless the samples were taken prior to the installation of the overlay.

In addition, the literature was searched for methods used to determine the effective chloride diffusion constant for concrete.

Beeby [20] reported that the corrosion process occurs in essentially two phases, one in which the passive layer is destroyed, the initiation phase, and the second which occurs from the time when corrosion begins until the concrete surface is disrupted. This occurrence was summarized by Bazant [21] as:

$$T_{cr} = T_p + T_{cor} \quad (1)$$

where  $T_{cr}$  = time to cracking of the concrete surface as a result of corrosion,

$T_p$  = the time of depassivation or initiation of corrosion,

and  $T_{cor}$  = the corrosion time, steady state.

The chloride ion which may be present in the water, aggregates, or admixtures is considered to be insignificant as compared to the chloride diffusing through the concrete [22].

Attempts have been made for determining the time to depassivation. The time to depassivation was reported as a range of chloride accumulation between 1.0 and 1.3 lb  $Cl^-$ /cu yd at the reinforcement steel level by the Federal Highway Administration [23]. Clear [24,25]

estimated the chloride depassivation concentration at 0.2% by weight of cement. An average value for Clear's [24,25] estimate was reported by Cady and Weyers [10] as 1.2 lb Cl<sup>-</sup>/cu yd for 6½ sacks of cement per cubic yard of concrete.

Others have proposed methods for determining the effective diffusion constant. Browne [26] suggested that the chloride penetration into concrete exposed to a marine environment was according to Fick's Law:

$$\partial C / \partial t = D_C (\partial^2 C / \partial x^2) \quad (2)$$

where C = concentration of the chloride ion,

x = distance of depth below the equilibrium point of the chloride ion concentration,

t = time over which the chloride diffusion has taken place,

and D<sub>C</sub> = diffusion constant for the concrete.

A standard solution to this partial differential equation is given as [26],

$$C(x,t) = C_0 - C_0 \operatorname{erf}(x/2\sqrt{D_C t}) \quad (3)$$

where C<sub>(x,t)</sub> = chloride concentration at depth x after time t,

C<sub>0</sub> = the equilibrium chloride concentration at the surface,

and erf = the error function.

Estimates of the effective diffusion coefficient were also determined using Fick's Law by West and Hime [27], Weyers and Cady [28], and Smith [29].

West and Hime [27] used measured chloride content data to determine the effective diffusion constant and compared the predicted chloride content values with the measured values. The results indicated a good relationship between the predicted values versus the measured values with respect to depth. Their results also indicated that chloride contents with respect to depth would increase with time.

West and Hime [27] also noted that using this method to predict the time of depassivation could be used assuming the chloride sources and the diffusion coefficient remained constant with time. In addition to estimating the effective diffusion constant, they offered a means to project future profiles of chloride content and for estimating the time for chloride to reach the steel reinforcement.

Weyers and Cady [28] estimated values for  $D_c$ ,  $C_o$ , and  $C(x,t)$  based on Fick's Law and proposed a theoretical relationship between the amount of cover over the reinforcement steel and the time to depassivation. They noted the relationship was appropriate only after the diffusion process reached a steady-state condition.

Smith [29] used Fick's Law to investigate the effects of temperature, W/C ratio, and different pozzolanic admixtures on the effective diffusion constant. A computer

program written in Fortran was developed to calculate the effective diffusion constant in terms of the error function.

Fisher et al [30], decided to treat the reaction as chloride absorption, following a linear absorption isotherm. This was to account for chemical reactions which may occur along the diffusion path in addition to the physical process. In an effort to account for these reactions, the amount of chloride absorbed was estimated by the following equation:

$$S_C = kC_C \quad (4)$$

where  $S_C$  = the amount of chloride absorbed,

$k$  = the slope of the absorption isotherm,

and  $C_C$  = concentration of chloride of the solution.

This modified Fick's Law to be as follows:

$$\frac{\partial C_C}{\partial t} = \frac{(D_C)}{(k+1)} \left( \frac{\partial^2 C_C}{\partial x^2} \right) \quad (5)$$

where  $D_C$  = the diffusion coefficient in an aqueous solution,

and  $x$  = the depth from the concrete surface.

The solution to this equation is given by,

$$C_S^{tot} = p C_S - S_C \quad (6)$$

$$= (p - k) C_C \quad (7)$$

where  $C_S^{tot}$  = the total concentration of chloride in concrete,

and  $p$  = the porosity.

Additional factors which may indirectly influence the chloride concentration and ultimately depassivation have also been considered. Four mass conservation equations to simulate the diffusion process of chlorides in concrete were presented by Bazant [31] as:

$$\frac{\delta C_w}{\delta t} + \frac{\delta J_w}{\delta x} + \dot{w}_h = 0 \quad (8)$$

$$\frac{\delta C_o}{\delta t} + \frac{\delta J_o}{\delta x} - J_w \frac{\delta C_o}{\delta x} + \dot{m}_o = 0 \quad (9)$$

$$\frac{\delta C_c}{\delta t} + \frac{\delta J_c}{\delta x} - J_w \frac{\delta C_c}{\delta x} = 0 \quad (10)$$

$$\frac{\delta C_f}{\delta t} + \frac{\delta J_f}{\delta x} - J_w \frac{\delta C_f}{\delta x} + \dot{m}_f = 0 \quad (11)$$

where  $x$  = the distance from the surface of the concrete to the level of the reinforcement steel,

$C_w$ ,  $C_o$ ,  $C_c$ , and  $C_f$  represent values for the concentrations of water, oxygen, chloride, and ferrous hydroxide respectively,

$J_w$ ,  $J_o$ ,  $J_c$ , and  $J_f$  represent diffusional fluxes for water, oxygen, chloride, and ferrous hydroxide respectively,

$\dot{w}_h$  = distributed sink of capillary water,

$\dot{m}_o$  and  $\dot{m}_f$  represents the distributed sinks of oxygen and ferrous hydroxide (resulting from the chemical reaction) respectively,

and  $t = \text{time.}$

Equations for the diffusional fluxes of water, oxygen, chloride and ferrous hydroxide were presented as [31]:

$$J_w = - D_w \frac{\delta C_w}{\delta t} \quad (12)$$

$$J_o = - D_o \frac{\delta C_o}{\delta t} \quad (13)$$

$$J_c = - D_c \frac{\delta C_c}{\delta t} + D_c \frac{\delta \phi}{\delta t} \quad (14)$$

$$J_f = - D_f \frac{\delta C_f}{\delta t} \quad (15)$$

where  $D_w$ ,  $D_o$ ,  $D_c$ , and  $D_f$  represent the diffusivity values for water, oxygen, chloride, and ferrous hydroxide respectively,

and  $\phi = \text{the electric potential.}$

The four mass equations are related to the diffusion of water, oxygen, chloride, and ferrous hydroxide all of which may have an influence on the time to depassivation [31].

Bazant [31] used the four mass conservation equations to project a chloride concentration profile reported by Gjorv [32], following exposure to sea water at 27 years. The profile for the projected chloride concentrations closely approximated the actual values presented.

Bazant [31] noted that concentration profiles versus many different times would be needed to verify the relationship.

An alternate approach for calculating  $t_p$ , was also derived by Bazant [33] in terms of the error function - "the

well known solution". This method was based on the assumptions that the diffusion was uncoupled and nonlinear.

In this approach, Bazant surmised the chloride concentration profile was approximately parabolic in shape up to the depth corresponding to the varying penetration depth,  $x = H(t)$  for which he derived the following equations:

$$C_C \approx C_C^S \left(1 - \frac{x}{H}\right)^2 \quad \text{for } x \leq H \quad (16)$$

where  $C_C^S$  = the chloride concentration at the surface,  
and  $C_C$  = the chloride concentration at a depth,  $x$ .  
At the concrete surface,

$$\frac{\partial C_C}{\partial x} = \frac{2C_C^S}{H} \quad (17)$$

Ultimately, Bazant [33] derived the following equations in which the time to depassivation could be estimated "roughly" by:

$$t = \frac{H^2}{12 D_C} \quad (18)$$

$$t_p = \frac{1}{12 D_C} \left[ L / (1 - \sqrt{C_{cr}/C_C^S}) \right]^2 \quad (19)$$

where  $D_C$  = the diffusivity of chloride,  
 $C_{cr}$  = the critical chloride concentration,  
 $C_C^S$  = the chloride concentration at the surface,  
 $L$  = the concrete cover,  
 $t$  = time in years,

and  $t_p$  = time to depassivation in years.

Bazant considered the results from numerical examples using this alternative approach appeared reasonable, but no test results for verification of the approach were available [33].

In conclusion, several approaches for determining the effective diffusion constant have been proposed. No research efforts were identified for efforts toward ascertaining the most appropriate approach for calculating the effective diffusion constant. Consequently, the approach based on Fick's Law was selected as the most feasible approach for the scope of this research considering the previous studies by Brown [26], West and Hime [27], Weyers and Cady [28], and Smith [29].

## EXPERIMENTAL PROCEDURE

Four methods were selected from a review of the literature for the laboratory investigation to determine the chloride content in concrete. The variability in the amount of salt detected in concrete by different methods may be affected by:

1. The amount of salt present in the concrete.
2. The cement content.
3. Variations in reaction temperature.
4. Method error.
5. Operator error.

The experimental procedure to investigate the selected methods was formulated in an effort to minimize the effects of these errors upon final development of the field procedure for chloride content determination.

The procedure for calculating values for the effective diffusion constant was also selected from a review of the literature. One procedure for determining the effective diffusion constant based on Fick's Law was suggested by Smith [29]. How Lum [34] used a modified version of a program written in Fortran by Smith [29] to calculate the effective diffusion constant for bridges throughout the United States. The values for the effective diffusion constants, calculated by How Lum [34], were used in the variability analysis.

## LABORATORY EVALUATION OF TEST METHODS

The test methods selected for the laboratory investigation for the chloride content in concrete included the:

- o Specific ion electrode,
- o Spectrophotometric,
- o Digital titrator, and
- o Quantab titrator strips.

The laboratory investigation of the four selected test methods consisted of three phases: accuracy of the test method as compared to the standard test method, the effects of cement content, and effects of changes in temperature on the performance of the selected test method. The accuracy of the test method was used as one of the criteria for the selection of the field test method. Prior to field validation, the effects of cement content and temperature on the selected test method were determined. For the accuracy, cement content, and temperature tests, one foot square, six inch thick concrete specimens were cast at 10 chloride contamination levels. Specimens were cast at 0.0, 0.2, 0.4, 0.8, 1.6, 3.2, 6.4, 8.6, 10.8 and 12.8 lbs  $\text{Cl}^-$ /cu.yd. of concrete using a sodium chloride solution as the additive. Concrete slabs were cast at a water to cement ratio of 0.40, 0.47, and 0.50 with cement contents of 658, 640, and 552 lbs/cu.yd. of concrete, respectively at each of 10 chloride contamination levels. Subsequent to obtaining powdered

samples from each slab, the specimens were moist cured for 46 days. A rotary hammer drill with a three-quarter inch carbide drill bit was used to sample each of the concrete slabs. The extracted powder from the multiple drill holes in a single contaminated slab were combined, dried, and sieved over a number 50 sieve, and the retained 50 sieve material was discarded.

#### Laboratory Testing Procedure

For the four selected methods, a general laboratory procedure was written which included the number of specimens, specimen size, cement content, chloride content, and the number of samples per specimen. In addition, an outline of the procedure for determining the chloride content in concrete in accordance with AASHTO T260-84 [35] was included for the standard potentiometric titration method. Procedures for determining the chloride content for each of the selected methods were also outlined prior to their use. Details of these procedures are presented in the appropriate sections that follow.

#### Concrete Materials Evaluation

The fine aggregate and coarse aggregate were tested in accordance with ASTM C33 [36] for particle size distribution, fineness modulus, absorption, specific gravity, and dry rodded unit weight. The results of the evaluation of the fine and coarse aggregate material is

presented Appendix A, Aggregate Properties and Concrete Mixtures. The fine aggregate met ASTM specifications for the properties tested. However, the coarse aggregate did not meet the ASTM [36] or Virginia Department of Transportation (VDOT) [37] gradation specifications (see Table A2 in Appendix A).

Therefore, sieve analyses were performed on samples of coarse aggregate material from various local sources in an attempt to identify a material which met gradation specifications. However, these materials also did not meet gradation specifications. Since the coarse aggregate gradation is not critical to this phase of the study as long as the gradation and mineralogy remain relatively constant, the material from a local source was used in the laboratory investigation phase.

#### Concrete Mixtures

The concrete mixtures used to cast the chloride test specimens were proportioned to meet 1973 Pennsylvania Department of Transportation specifications [38]. Trial batches of the designed concrete mixture were tested to determine the slump, air content, yield, and workability. The results from these tests were used in adjusting the mixture proportions of the trial batch until the specifications for slump, air content, and water/cement ratio were met.

The concrete mix designs used to cast the concrete specimens are presented in Appendix A, Tables A4, A5, and A6. The bridge deck class concrete specimens ( $W/C=0.47$ ) were used in the initial series of chloride content tests to determine the accuracy of the four methods. The substructure and superstructure mixtures ( $W/C=0.50$  and  $0.40$  respectively) were used to prepare specimens for the determination of the effects of cement content. A single chloride contamination level was used to determine the effects of reaction temperature. Physical characteristics of the plastic concrete were determined for each batch and are presented in Appendix A, Table A7. Once cast, each of the specimens were placed in a curing room along with eight 3" X 6" compressive strength test cylinders.

For each batch, two compressive strength cylinders were tested at 7 and 28 days. Results of the compressive strength tests are also presented in Appendix A, Table A7.

#### PROCEDURE FOR TESTING THE CHLORIDE CONTENT OF CONCRETE

The testing procedures used in determining the chloride content of the specimens are summarized below for the standard potentiometric titration method and for the four selected methods (specific ion electrode, spectrophotometric, digital titrator, and Quantab titrator strips).

1. Standard potentiometric titration method, AASHTO T260-84 [35]: The standard potentiometric titration

procedure was used as the basis for comparison of the chloride content of the test samples for each of the four select methods. In following this procedure, 3 grams of the powdered concrete sample are weighed to the nearest milligram. Ten milliliters of 90 to 100 °C distilled water are added and the sample is swirled. Once the sample is in suspension, the slurry is ground to remove any lumps. The slurry is then transferred into a beaker. Three milliliters of concentrated nitric acid are added to the remaining sample material and is transferred quantitatively into the beaker while stirring the slurry constantly. The solution is increased to approximately 50 ml by adding hot distilled water.

Five drops of methyl orange indicator are added to the slurry and the pH of the sample is adjusted if needed. The solution is then covered with a watch glass and the stirring rod is left in the beaker. Next, the solution is heated, allowed to boil for one minute, and then is removed. The sample is filtered into another beaker and the beaker, filter paper, funnel, and stirring rod are rinsed thoroughly to ensure complete transfer of the chloride. Finally, the sample is allowed to cool to room temperature.

Four milliliters of 0.01N NaCl solution are added to the sample prior to titrating with 0.01N AgNO<sub>3</sub> solution. A chloride specific electrode, Orion Model 94-17B, and its

reference electrode, and a millivoltmeter were used in the determination of the titration end-point.

2. James Instruments, Model CL 500: The testing procedure developed by James Instruments [39] involves calibrating the specific ion electrode using three known standards by plotting the electrometer readings (millivolts) versus percent chloride on semilog graph paper. The powdered material is placed in an ampoule and is compressed to a level corresponding to a red indicator line. The sample is then added to a premeasured amount of the 'chloride extraction liquid' and is shaken for 15 seconds. The electrode is submerged into the solution and the electrometer reading is plotted. The corresponding chloride content of the sample may then be read directly from the calibrated graph.

The James Instrument used during the investigation is illustrated in Figure 1.

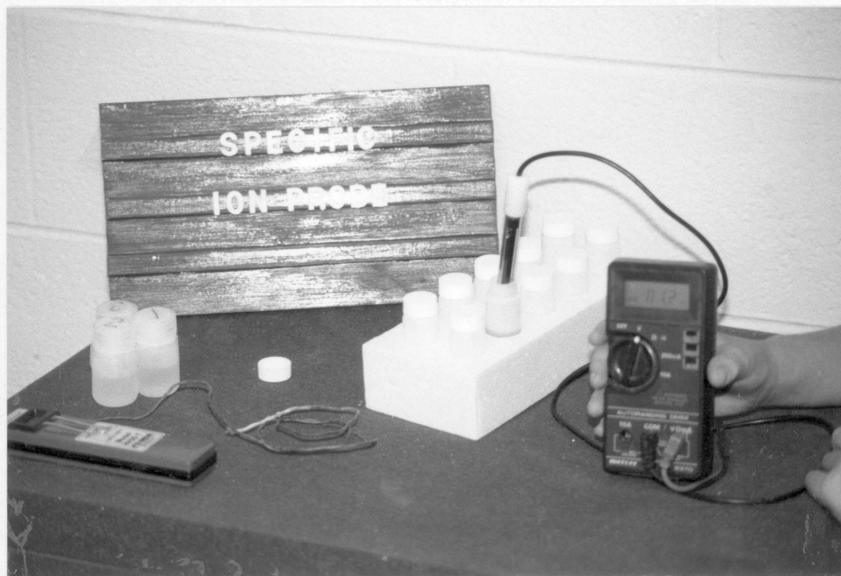


Figure 1. The Specific Ion Probe

3. DR/2000 spectrophotometer: The testing procedure for the DR/2000, developed by the Hach Company [40], includes specifying the chloride test procedure and entering the appropriate wavelength reading for the test into the instrument. Two sample cells are prepared, one with deionized water and the other with the filtered sample to be analyzed. Two milliliters of mercuric thiocyanate and 1.0 ml of ferric ion solution are added to each sample and swirled to mix. After mixing, the two samples are analyzed following a two minute waiting period. The blank cell is used to zero the instrument prior to analyzing the sample cell for chloride content.

The spectrophotometer used during the course of this investigation is presented in Figure 2.

CHIEFTAIN BOND

50% COTTON FIBER



Figure 2. The DR 2000 Spectrophotometer

4. CD-DT digital titrator: The analysis procedure for this method, as developed by the Hach Company [41], involves transferring the sample into an erlenmeyer flask and diluting if necessary with deionized water.

Diphenylcarbazone powder is added and swirled to mix. The sample is then titrated with mercuric nitrate to a light pink color. The number of digits required is used in calculating the chloride content.

Figure 3 presents the digital titrator used in this investigation.

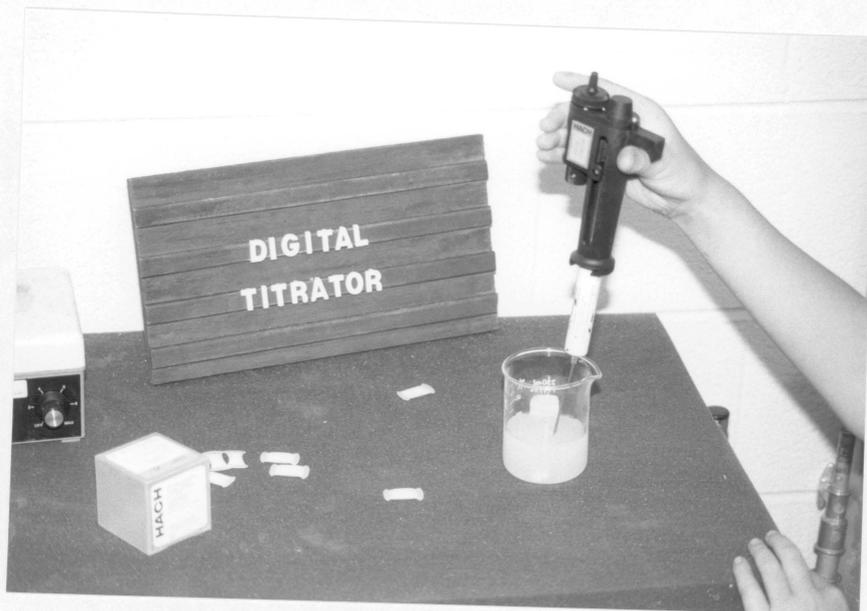


Figure 3. The Digital Titrator

5. Quantab titrator strips: The procedure in using the Quantab titrator strips, developed by Environmental Test Systems, Inc. [42], involves weighing 10 grams of the sample and adding 90 ml of boiling distilled water while stirring constantly. The sample is stirred for 30 seconds, and after one minute, is stirred again for another 30 second period. Filter paper is folded to a cone and is placed into the beaker to allow some of the sample to filter through. A Quantab titrator strip is placed in the filtered portion of the sample where it remains until a dark blue color forms at the top of the strip. The reading of the Quantab titrator strip is used in determining the chloride content from a calibrated chart.

Figure 4 presents the Quantab titrator strips used during the investigation.

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50% COTTON FIBER



Figure 4. The Quantab Titrator Strips

Details on the four selected chloride test methods are presented in Appendix B.

#### Chloride Content of Concrete Test Results

The standard potentiometric titration method was used to standardize the 0.01N silver nitrate and sodium chloride solutions used for the analysis of chloride in accordance with AASHTO T260-84 [35]. Tests for chloride content using the four select methods were performed on standardized solutions to verify their accuracy prior to testing the specimens. The test results for all four selected methods were within the range of accuracy specified by the manufacturers.

#### EFFECTS OF CEMENT CONTENT ON SELECTED TEST METHOD

The method selected for further investigation was tested to determine the effects differences in cement content representative of substructure and superstructure concrete have on measuring the chloride content. For this series of tests, the potentiometric titration procedure was used in accordance with AASHTO T260-84 [35] as the control method in determining the chloride content of the test specimens. However, the powdered concrete samples were screened and the material larger than 50 mesh was discarded prior to analyzing for chloride content.

The selected method was subjected to testing concrete having water to cement ratios of .40 and .50 at various

chloride contamination levels. Physical characteristics of plastic concrete for the specimens used in this series of tests is presented in Appendix B, Table A7.

Chloride contamination levels of 0.2, 0.8, 1.6, 3.2, 6.4, 8.6, 10.8, and 12.8 lb Cl<sup>-</sup>/cu yd of concrete were used for the specimens having a W/C = .40. Specimens having a W/C = .50 were tested at similar chloride contamination levels with one exception. One specimen containing no added chloride was tested in place of the 0.2 lb Cl<sup>-</sup>/cu yd. contamination level. The physical characteristics of plastic concrete for the concrete specimens are presented in Appendix B, Table A7.

#### EFFECTS OF TEMPERATURE ON SELECTED TEST METHOD

The selected method was also subjected to testing concrete at a single chloride contamination level to determine the effects of temperature in measuring the chloride content. Concrete representative of bridge decks (W/C = .47) was selected for this series of tests. The physical characteristics of plastic concrete for specimen C-4 containing 1.6 lb Cl<sup>-</sup>/cu yd is presented in Appendix B, Table A7.

Samples were tested at temperatures ranging from 34 to 130 °F using the procedure for the specific ion probe presented in Appendix B.

## EXPERIMENTAL PROCEDURE FOR FIELD VALIDATION

In accomplishing the task for field validation, three bridges were selected for testing from three different environmental exposure groups; Four bridges from a fourth environmental exposure group were selected.

The different environmental exposure groups were defined according to the soil freezing index. The soil freezing index combines elements of time and temperature into one component of measure in terms of degree days. One degree day is defined as one day having a mean air temperature corresponding to 1 °F below freezing or 31 °F [43].

The soil freezing index was considered appropriate for defining different environmental exposure groups as relative to the amount of salt being applied on the nation's bridges. The index combines the elements of time and temperature for a geographic area to identify the mean freezing index values across the United States [43].

Florida was selected to represent the coastal region with 0 freezing degree days per year. A non-coastal area of Virginia was selected having 0 freezing per days per year according to the index. The third environmental exposure group, Pennsylvania, has 0 to 500 freezing days per year, Wisconsin was designated to represent the fourth environmental exposure group having 1000+ freezing degree days per year [43].

Once the bridges were selected to test for chloride content for field validation of the selected method, a complete procedure was formulated to consist of:

- o Modifications of the procedure for the specific ion probe,
- o Development of the sampling procedure,
- o Determining the most viable method of weighing samples,
- o Determining whether samples needed to be crushed prior to analysis, and
- o Developing the final procedure for validating the accuracy of the results for the specific ion probe.

These objectives were accomplished by the procedures presented in the appropriate sections of this report.

#### Field Procedure for the Specific Ion Probe

During the laboratory investigation of the specific ion probe, certain trends were noticed in the readings obtained when measuring concrete specimens for chloride content. In addition, James Instruments, Inc. [44] was contacted regarding factors which may reduce the accuracy in the results obtained when using the specific ion probe. Subsequently, the procedure for measuring the chloride content was modified.

Modifications of the procedure for measuring the chloride content in concrete using the specific ion probe

involved observations of the trends in the readings on the electrometer by the operator. After waiting 3 minutes for the reading on the specific ion probe to stabilize, the following trends in the readings indicate the millivolt reading to be recorded:

Example 1	Example 2	Example 3
88.3	88.3	88.3
88.1	88.1	88.2
88.0	88.0	88.1
87.9	88.1	88.0
88.0	88.0	87.9
87.9	88.0	88.0
88.0	87.9	87.9

In the examples presented, the reading on the electrometer to record is 88.0 mV. However, any reading within 1 or 2 millivolts of this number will not significantly reduce the accuracy of the final results. The actual trends observed during use may not be exact, but should be similar.

Abnormal errors in the results using the specific ion probe may occur if certain precautions are not observed. The following precautions are recommended by James Instruments, Inc. to prevent the occurrence of this type of error [44]:

- o Turn the dial on the electrometer to read Volts; corresponding readings will result in millivolts.
- o Do not allow the electrometer battery to become weak. Irradic readings may result.

- o Refrain from storing the calibration liquids in direct sunlight or in very warm places.
- o Care should be taken to prevent contamination of the three calibration liquids. An extreme increase or decrease in the millivolt readings may indicate contamination of one or more of the liquids. Normally, calibration readings should be within  $\pm 5$  mV of previous readings and approximately 100 mV, 50 mV, and -5 mV for calibration liquids 1, 2, and 3 respectively.
- o Insure the distilled water does not become contaminated. Also, it is recommended that only salt-free distilled water is used.
- o Special care of the electrode is important. When not in use, the electrode should be submerged in distilled water to prevent clogging of the sensing device.
- o If the filling solution is allowed to remain in the electrode for extended periods of time, damage to the electrode may result. Clean and polish the electrode frequently to maintain consistent results.
- o Prior to using the electrode, observe the level of the filling solution. An insufficient amount of filling solution will result in readings that wander and reduce the repeatability of the

results. Sporadic readings have been noticed once the level fell below  $\frac{1}{4}$ " of the eyelet in the probe. For best results, it is recommended the probe be filled completely with filling solution prior to use.

- o The specific ion probe should be cleaned using distilled water only.
- o Never touch the tip of the probe.
- o Polish the tip of the probe using the polishing strip provided by the manufacturer at frequent intervals, depending on the amount of use.

The procedure for testing concrete specimens using the specific ion probe was modified prior to the field investigation in an attempt to improve the accuracy or repeatability of the measurements for chloride and to minimize errors in measuring the chloride associated with different operators. In addition, sampling and sample preparation procedures were developed for collecting the concrete specimens and preparation prior to testing.

#### Sampling Procedure

Obtaining a representative sample of the material to be analyzed is considered equally as important as the accuracy of the method used for analyzing concrete samples for chloride. The sampling procedure used for the first series of tests consisted of drilling  $\frac{3}{4}$ " holes and discarding the

plus 50 mesh material. This procedure was noted as a potential source of error in the comparative analysis between the amount of chloride measured using the standard potentiometric titration procedure and the amount of chloride actually added to the specimens.

The standard procedure for sampling in accordance with AASHTO T260-84 [35] provides that the drill bit utilized for the pulverizing method of sampling be of sufficient diameter to allow a representative sample. Utilizing a rotary hammer the hole is drilled to within  $\frac{1}{2}$ " of the desired drilling depth by setting the depth indicator on the drill. Next, the hole is cleaned thoroughly using a blow out bulb. The depth indicator is again reset for an additional  $\frac{1}{2}$ " drilling to collect the sample. For this portion of the drilling operation some users elect to use a bit which is  $\frac{1}{4}$ " less in diameter to prevent contamination from the sides of the drill hole. Ten grams of sample are collected from the powdered concrete remaining in the drill hole using a spoon and is placed in a sample container. Prior to testing, the sample is crushed until the entire sample passes through a No. 50 sieve.

In developing the sampling procedure for testing the thirteen bridges, the size distribution of the coarse aggregate was considered the primary factor influencing the test results for chloride. The sampling procedure selected for field use consisted of drilling holes using a rotary

impact drill and a  $1\frac{1}{8}$ " diameter Heller bit. This diameter was considered sufficiently large to negate or minimize influences the coarse aggregate ( $\frac{3}{4}$ " maximum size) may have on the test results. Centering the drill hole over a large aggregate particle was not considered likely. The Heller bit is designed to allow drill cuttings to be removed from the drilling surface by means of a vacuum system.

A vacuum system for collecting the concrete samples was selected to decrease the time required to collect samples when compared to the standard procedure provided in AASHTO T260-84 [35]. The sample collection system consists of a 2.25 hp Sears Craftsman wet and dry vacuum cleaner, a sample collection device, coffee filters, and plastic tubing connected to the Heller bit. A metal band with hooks was used to attach the vacuum cleaner to a crane for sampling the substructure components. The sample collection unit is illustrated in Figure 5. Refer to Appendix C for detailed drawings of the collection unit.

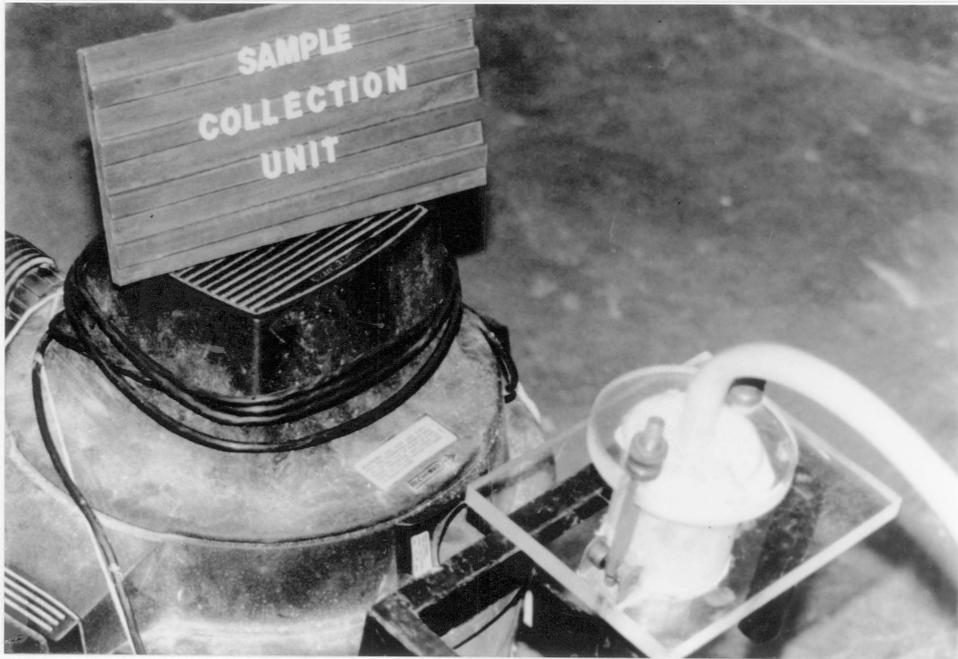


Figure 5. The Sample Collection Unit

Other equipment included a single phase generator and a pachmeter. The generator was used to supply electricity for the rotary impact drill and the vacuum system. A pachmeter is a device that operates on the principal of a magnetic field. It was used for determining the position of reinforcing steel in concrete surrounding the area around the holes to be drilled.

#### Weighing of Samples

Samples collected were weighed using the ampoules supplied by James Instruments, Inc [39]. The procedure for weighing involves placing the concrete powdered sample into an ampoule and compacting it completely to a red indicator line with the end of a pen. Powdered samples from two ampoules are used for the analysis of chloride. The accuracy in the procedure was determined to be  $3.04 \pm .05$  grams by How Lum [34]. Use of this procedure in the field was determined to be very time consuming. The time required to weigh the samples in the field ranged from 5 to 6 minutes.

The accuracy of two other methods for weighing specimens were also determined by How Lum [34]. A balance supplied by James Instruments, Inc. did not maintain sufficient accuracy for weighing the concrete specimens. However, the accuracy for a triple beam balance,  $3.04 \pm .05$  grams, was considered acceptable. Ultimately, the triple

beam balance was used to weigh the concrete specimens requiring 1½ to 2 minutes for each specimen.

#### Sample Preparation

The first twenty samples collected from a bridge in Pennsylvania was tested using the specific ion probe to compare the test results for chloride from samples that were crushed to pass a No. 50 sieve with samples that were not crushed prior to testing for chloride. The initial results indicated a good relationship between the test results. Therefore, the remaining specimens were not crushed in the field prior to testing for chloride.

Ultimately, twenty samples from one bridge in each of the four states were tested prior to crushing and after crushing to verify the relationship between crushing and not crushing the concrete powdered samples

#### Validation of the Specific Ion Probe

To validate the accuracy of the specific ion probe, twenty samples from one bridge in each of the four states ( a total of 80 samples) were tested for chloride content using the standard potentiometric titration procedure in accordance with AASHTO T260-84 [35]. The results of these tests, reported by Baden [45], were compared with the results obtained for the specific ion probe on samples that were not crushed.

The potentiometric titration results [45] were also compared with the test results of the specific ion probe for samples which were crushed to pass a No. 50 sieve prior to testing.

#### DIFFUSION COEFFICIENT DETERMINATION

Chloride ions may ingress into the concrete by means of capillary movement, cracks, or diffusion. Capillary systems which are generally considered to be discontinuous in cement pastes, are considered an insignificant means of chloride ingress even in arid environments [10]. Cracking of the concrete surface, however, provides a rapid means for chloride penetration.

If cracking is not present, the chloride ion permeates into the concrete by diffusion according to Fick's Law [26]

$$\partial C / \partial t = D_c (\partial^2 C / \partial x^2) \quad (20)$$

where  $C$  = concentration of the chloride ion,

$x$  = distance of depth below the equilibrium point of the chloride ion concentration,

$t$  = time over which the chloride diffusion has taken place, and

$D_c$  = diffusion constant for the concrete.

A standard solution to this partial differential equation is given as [26],

$$C(x,t) = C_0 - C_0 \operatorname{erf}(x/2\sqrt{D_c t}) \quad (21)$$

where  $C_{(x,t)}$  = chloride concentration at depth  $x$  after  
time  $t$

$C_0$  = the equilibrium chloride concentration  
at the surface

and  $\text{erf}$  = the error function

Determination of  $D_c$  and  $C_0$  for specific bridge components provides the means of calculating the time for the corrosion initiative of steel as reported by Cady and Weyers [10]. The diffusion constant,  $D_c$ , is a function of the water to cement ratio and temperature. Values of  $W/C = .45$  and an average ambient temperature of  $60^\circ\text{F}$  may be used for mean conditions [10]. An effective diffusion constant, which accounts for this variability, may be determined for a specific bridge deck from measured chloride concentrations as a function of depth [28].

Smith [29] developed a method for determining the effective diffusion constant based on Fick's Law. His procedure was adapted by How Lum [34] for use in determining the effective diffusion constant values for various bridges across the United States.

The variability of certain effective diffusion constants, calculated by How Lum [34], was examined for the bridges tested during the field validation of the specific ion probe. In addition, the effects of time on the diffusion constant was examined on four bridges in Pennsylvania.

## STATISTICAL ANALYSIS PROCEDURES

The statistical analysis procedures for evaluating the results obtained from the tests for chloride include the Wilcoxon Signed Rank, and regression analysis procedures. A summary of these procedures are included in the appropriate sections that follow.

### Analysis of Results for Chloride Content

Following completion of the laboratory testing on the four selected methods, appropriate statistical analysis procedures were used to analyze the results of the data. The statistical analysis of the test results from the laboratory procedure consisted of the Wilcoxon Signed Rank and regression analysis procedures.

#### Wilcoxon Signed Rank Procedure

The Wilcoxon Signed Rank procedure is basically a one-group test which does not require the data to be normally distributed. Instead, the assumptions are that the group of observations are independent and each observation comes from a symmetric distribution (observations are not required to come from the same distribution). This procedure is well known for hypothesis testing of means,  $\mu$ . In general, the testing procedure is as follows [46]:

Test the null hypothesis,

$$H_0: \mu = \mu_0$$

versus one of the following alternative hypotheses,

$$H_1: \mu > \mu_0$$

$$\mu < \mu_0$$

$$\text{or } \mu \neq \mu_0$$

where  $\mu$  = the true mean

$\mu_0$  = the estimated mean

Paired data may be treated by converting a two group problem to a one group problem by testing differences between the corresponding observations [46,47]. The following procedure may be used for converting two groups of data for analysis using the Wilcoxon Signed Rank procedure [46]:

Let  $D_i = X_{1i} - X_{2i}$  for each observation

where,  $X_{1i}$  and  $X_{2i}$  are results from corresponding observations from two groups of observations, groups 1 and 2.

Even without the normality assumption, the expected values for  $D_i$ ,  $X_{1i}$ , and  $X_{2i}$  are such that the following relationship may be written [46]:

$$\mu_D = \mu_1 - \mu_2$$

To test the null hypothesis,

$$H_0: \mu_1 - \mu_2 = \mu_{D0}$$

versus the alternative,

$$H_1: \mu_1 - \mu_2 \neq \mu_{D0}$$

Next, let  $Y_i = D_i$

Now the two groups of data have been converted to one group for testing as follows [46]:

$$\text{Let } W_i = |Y_i|$$

Rank order  $W_i$  from the smallest observation to the largest and let  $R(W_i)$  = the rank of  $W_i$

$$\text{Let } T^+ = \sum R(W_i) \text{ , for all } Y_i > 0$$

where  $T^+$  is the test statistic.

For testing the alternative hypothesis,  $H_1: \mu_D \neq \mu_{D0}$  where,  $\mu_D$  is the true mean for the difference between observations,

and  $\mu_{D0}$  is the estimated mean for the difference between the observations

reject  $H_0$ : if  $T^+ \geq n(n+1)/2 - A_{n,\alpha/2}$

or if  $T^+ \leq A_{n,\alpha/2}$

where  $A_{n,\alpha/2}$  = the table entry for

$n$  = the sample size,

$\alpha/2$  = column entry,

and  $\alpha$  = level of significance

Refer to the cited reference list for a more detailed discussion of the procedure [46,47].

### Regression Analysis

The regression analysis procedure incorporates the least squares method to determine the most appropriate relationship between two variables by fitting a straight line to the given data. The method of least squares may be

used to examine data and provides meaningful conclusions about any relationships which may exist [48].

Assuming a linear relationship between  $X_i$  the independent variable, and  $Y_i$  the dependent variable, the following statistical model may be written [46]:

$$Y_i = \beta_0 + \beta_1 X_i + e_i ; \text{ for } i = 1, 2, 3, \dots, n$$

where  $Y_i$ 's are random variables,

$X_i$ 's are known constants,

$\beta_0$  and  $\beta_1$  are unknown constants,

and  $e_i$ 's are independent random variables with an expected value of 0.

The errors of prediction,  $e_i$ , are assumed to independently and identically distributed belonging to a normal distribution with a mean,  $\mu = 0$  and a variance,  $\sigma^2 = 1$ . The errors,  $e_i$ , may include differences in the experimental units themselves, result from measurement errors, or indicate an inadequacy of the model (if the errors are not uniformly distributed) [46].

In linear regression, actual values for  $\beta_0$  and  $\beta_1$  are not known. Estimates for  $\beta_0$  and  $\beta_1$ ,  $b_0$  and  $b_1$  respectively, are chosen algebraically to minimize the errors of prediction,  $\sum e_i^2$ . This procedure is referred to as the least squares criterion [46].

Results from the regression analysis procedure provides estimates for  $b_0$  and  $b_1$ , and tests the following hypotheses

should be done to determine if [46]:

$H_0: b_0 = 0$  : null hypothesis  
versus  $H_1: b_0 \neq 0$  : alternative,  
and  $H_0: b_1 = 0$  : null hypothesis  
versus  $H_1: b_1 \neq 0$  : alternative.

Of particular importance is the test for  $b_1 = 0$ . If the null hypothesis is not rejected in this instance, then the relationship between the observation can not be explained by means of regression [46].

Other important relationships in a regression analysis are [46,48]:

R-sq or the coefficient of determination represents the proportion of the variation in the dependent variable that can be predicted from the independent variable, defined as [46]:

$$R^2 = SS_{reg} / SS_{tot}$$

R-sq(adj) or adjusted R-sq represents the proportion of the in the predicted variable which may be predicted from the independent variable in the population, defined as

$$R\text{-sq}(\text{adj}) = \frac{(df_{tot})R^2 - df_{reg}}{df_{res}}$$

For the analysis of variance, or ANOVA the following relationship exists [46]:

$$SS_{total} = SS_{reg} + SS_{res}$$

where  $SS_{total}$  represents the total variability among all observations.

$SS_{reg}$  represents the variability in Y that can be predicted by the values for X

$SS_{res}$  represents the variability of the observations which do not lie exactly on the regression line or line of best fit.

Other important parameters in the analysis of variance include [46]:

$$F_{obs} = MS_{reg} / MS_{res}$$

where  $MS_{reg} = SS_{reg} / df_{reg}$

$$MS_{res} = SS_{res} / df_{res}$$

$$df_{reg} = 1$$

$$df_{res} = n - 2$$

$$df_{tot} = n - 1$$

df refers to the degree of freedom

and n is the number of observations

The parameter  $F_{obs}$  may also be useful in determining if a relationship exists between values of Y and X which may be explained by means of a regression analysis (i.e. to determine if the estimated slope,  $b_1 \neq 0$ ) [46].

The microcomputer version of the Minitab has the capability of performing different statistical analysis procedures including the Wilcoxon Signed Rank procedure and regression analysis by least squares [49]. Consequently, Minitab was selected for analyzing the results obtained from the first series of laboratory tests.

## Analysis Procedure for the Effective Diffusion Constant

The effective diffusion constants determined by How Lum [34], were analyzed to determine the variability of the effective diffusion constant between bridges located in Pennsylvania, Wisconsin, Virginia, and Florida. The analysis also included the variability between different exposure groups within the study area. Finally an analysis to determine the variability of the effective diffusion constant with respect to time was performed.

The microcomputer version of Minitab [49] was used to perform regression analyze procedures and calculate means and standard deviations of the various data for the effective diffusion constant.

The t test procedure for paired data [46] was used to analyze the effective diffusion constants in determining the effects of time. Prior to using the t test procedure, the F test was incorporated into the analysis of variance to determine if the variances between each set of data were equal.

## EXPERIMENTAL RESULTS AND ANALYSIS

### RESULTS FOR THE CHLORIDE CONTENT OF CONCRETE

Four samples from each of the 10 chloride contamination levels were analyzed using the standard method and the four selected test methods. Presented are the results of the standard potentiometric titration procedure, specific ion probe, spectrophotometric, digital titrator, and the Quantab titrator strips.

#### Standard Potentiometric Titration Method

The results were compared to the actual chloride added to each of the chloride contaminated test specimens. The difference between the percent measured and the percent added is presented in Table 1.

Table 1. Comparison of the Potentiometric Titration Results  
and Amount of Chloride Added

<u>Specimen</u>	<u>Measured Chloride</u> <u>(% Cl<sup>-</sup>)</u>	<u>Added Chloride</u> <u>(% Cl<sup>-</sup>)</u>	<u>Measured Added</u> <u>Difference</u> <u>(% Cl<sup>-</sup>)</u>
C-0	.023	.000	.023
C-0	.018	.000	.018
C-0	.020	.000	.020
C-0	.020	.000	.020
C-1	.026	.005	.021
C-1	.027	.005	.022
C-1	.027	.005	.022
C-1	.021	.005	.016
C-2	.032	.010	.022
C-2	.028	.010	.018
C-2	.032	.010	.022
C-2	.033	.010	.023
C-3	.049	.020	.029
C-3	.045	.020	.025
C-3	.049	.020	.029
C-3	.049	.020	.029
C-4	.064	.040	.024
C-4	.063	.040	.023
C-4	.062	.040	.022
C-4	.060	.040	.020
C-5	.111	.080	.031
C-5	.115	.080	.035
C-5	.115	.080	.035
C-5	.112	.080	.032
C-6	.198	.161	.037
C-6	.191	.161	.030
C-6	.189	.161	.028
C-6	.191	.161	.030
C-7	.222	.214	.008
C-7	.222	.214	.008
C-7	.221	.214	.007
C-7	.220	.214	.006
C-8	.330	.270	.060
C-8	.330	.270	.060
C-8	.329	.270	.059
C-8	.328	.270	.058
C-9	.388	.324	.064
C-9	.396	.324	.072
C-9	.386	.324	.062
C-9	.386	.324	.062

### Specific Ion Probe

Table 2 presents the results of the specific ion probe test and the standard test method.

Table 2. Comparison of the Potentiometric Titration  
and the Specific Ion Probe Results at W/C = .47

<u>Specimen</u>	<u>Potentiometric Titration (% Chloride)</u>	<u>Specific Ion Probe Reading Millivolts</u>	<u>Calculated (% Chloride)</u>
C-0	.023	82.0	.012
C-0	.018	82.0	.012
C-0	.020	81.7	.012
C-0	.020	81.8	.012
C-1	.026	60.7	.029
C-1	.027	76.2	.015
C-1	.027	79.1	.013
C-1	.021	72.0	.018
C-2	.032	63.2	.026
C-2	.028	65.8	.023
C-2	.032	64.5	.024
C-2	.033	64.6	.024
C-3	.049	48.4	.048
C-3	.045	47.8	.050
C-3	.049	47.5	.050
C-3	.049	45.5	.055
C-4	.064	39.1	.072
C-4	.063	39.7	.070
C-4	.062	39.6	.071
C-4	.060	39.7	.070
C-5	.111	23.4	.142
C-5	.115	20.2	.163
C-5	.115	21.3	.155
C-5	.112	19.6	.167
C-6	.198	6.1	.299
C-6	.191	6.6	.293
C-6	.189	5.9	.302
C-6	.191	6.4	.295
C-7	.222	-0.2	.368
C-7	.222	-3.0	.413
C-7	.221	-1.6	.390
C-7	.220	-0.1	.366
C-8	.330	-8.1	.510
C-8	.330	-11.6	.590
C-8	.329	-9.2	.534
C-8	.328	-7.6	.500
C-9	.388	-15.2	.749
C-9	.396	-14.7	.733
C-9	.386	-15.5	.758
C-9	.386	-16.4	.788

### Spectrophotometric

Table 3 presents the test results of the ten concrete chloride ion concentration levels for the DR/2000 spectrophotometer and the standard test method.

Table 3. Comparison of the Potentiometric Titration and the Spectrophotometer Results at W/C = .47

<u>Specimen</u>	<u>Potentiometric Titration (% Chloride)</u>	<u>Spectrophotometer Chloride (Mg/L)</u>
C-0	.023	1.8
C-0	.018	2.7
C-0	.020	2.3
C-0	.020	2.1
C-1	.026	1.5
C-1	.027	2.3
C-1	.027	1.1
C-1	.021	2.0
C-2	.032	2.0
C-2	.028	2.1
C-2	.032	1.8
C-2	.033	1.8
C-3	.049	2.4
C-3	.045	2.9
C-3	.049	2.1
C-3	.049	2.6
C-4	.064	3.7
C-4	.063	2.8
C-4	.062	2.1
C-4	.060	3.4
C-5	.111	4.8
C-5	.115	6.2
C-5	.115	6.5
C-5	.112	7.9
C-6	.198	9.8
C-6	.191	9.9
C-6	.189	9.7
C-6	.191	9.5
C-7	.222	12.1
C-7	.222	11.7
C-7	.221	11.1
C-7	.220	8.0
C-8	.330	16.0
C-8	.330	16.2
C-8	.329	19.7
C-8	.328	17.7
C-9	.388	15.8
C-9	.396	17.5
C-9	.386	19.1
C-9	.386	18.1

### Digital Titrator

The results of the chloride test for the digital titrator and standard method are presented in Table 4.

Table 4. Comparison of the Potentiometric Titration  
and the Digital Titrator Results at W/C = .47

<u>Specimen</u>	<u>Potentiometric Titration (% Chloride)</u>	<u>Digital Titrator Chloride (Mg/L)</u>
C-0	.023	10.5
C-0	.018	5.0
C-0	.020	6.0
C-0	.020	7.0
C-1	.026	8.5
C-1	.027	11.0
C-1	.027	2.5
C-1	.021	7.0
C-2	.032	10.0
C-2	.028	5.0
C-2	.032	8.0
C-2	.033	8.0
C-3	.049	9.0
C-3	.045	7.5
C-3	.049	13.0
C-3	.049	10.0
C-4	.064	13.0
C-4	.063	14.0
C-4	.062	15.0
C-4	.060	12.0
C-5	.111	25.0
C-5	.115	16.0
C-5	.115	18.5
C-5	.112	22.0
C-6	.198	30.0
C-6	.191	30.0
C-6	.189	32.0
C-6	.191	32.5
C-7	.222	34.0
C-7	.222	31.0
C-7	.221	35.5
C-7	.220	33.0
C-8	.330	52.0
C-8	.330	55.0
C-8	.329	51.0
C-8	.328	50.0
C-9	.388	59.0
C-9	.396	63.0
C-9	.386	55.0
C-9	.386	56.0

### Quantab Titrator Strips

When using the procedure recommended by Enviromental Test Systems, Inc. [42], the titrator strips were somewhat difficult to read. The change in the color of the Quantab strip to white tended to indicate very low readings which were considered unrealistic for the amount of chloride present in the samples. As a result, the highest reading which indicated a change in color on the test strip was recorded for the result. The color change was not white, but a yellow to a yellowish brown.

Since these problems did occur, tests using the procedure for water soluble chlorides were not completed for all of the specimens. However, the results obtained from testing seven of the ten specimens are presented in Table 5.

Table 5. Comparison of the Potentiometric Titration and the  
 Quantab Titrator Strip Results for  
 Water Soluble Chloride at W/C = .47

<u>Specimen</u>	<u>Potentiometric Titration (% Chloride)</u>	<u>Quantab Titrator Strip (Water Soluble) Reading</u>
C-1	.026	2.80
C-1	.027	2.85
C-1	.027	2.85
C-1	.021	2.80
C-2	.032	2.65
C-2	.028	2.85
C-2	.032	2.85
C-2	.033	3.00
C-3	.049	3.00
C-3	.045	3.00
C-3	.049	3.00
C-3	.049	3.00
C-4	.064	3.10
C-4	.063	3.00
C-4	.062	3.20
C-4	.060	3.20
C-5	.111	3.60
C-5	.115	3.40
C-5	.115	3.40
C-5	.112	3.55
C-6	.198	3.95
C-6	.191	3.40
C-6	.189	3.80
C-6	.191	3.90
C-9	.388	5.35
C-9	.396	5.20
C-9	.386	5.65
C-9	.386	5.20

Four samples from each of ten chloride contaminated concrete specimens were tested for total chloride content (acid digestion procedure) using the Quantab titrator strips. The results obtained using this method are presented in Table 6.

During this series of tests, the Quantab readings were more distinct and easier to read. The white color on the strip represented higher readings from the previous procedure indicating that more chloride was being extracted from the sample. However, there were no secondary or partial color changes above the white colored area as observed using the manufacturer's procedure.

Table 6. Comparison of the Potentiometric Titration and the  
 Quantab Titrator Strip Results for  
 Acid Soluble Chlorides at W/C = .47

<u>Specimen</u>	<u>Potentiometric Titration (% Chloride)</u>	<u>Quantab Titrator Strip Reading</u>
C-0	.023	0.20
C-0	.018	0.80
C-0	.020	1.20
C-0	.020	0.60
C-1	.026	0.70
C-1	.027	0.60
C-1	.027	0.80
C-1	.021	0.60
C-2	.032	0.80
C-2	.028	0.80
C-2	.032	0.60
C-2	.033	0.60
C-3	.049	1.80
C-3	.045	0.80
C-3	.049	0.85
C-3	.049	1.00
C-4	.064	0.50
C-4	.063	1.25
C-4	.062	1.10
C-4	.060	1.40
C-5	.111	2.15
C-5	.115	2.00
C-5	.115	2.05
C-5	.112	1.95
C-6	.198	2.60
C-6	.191	2.80
C-6	.189	3.20
C-6	.191	3.00
C-7	.222	3.10
C-7	.222	3.40
C-7	.221	2.95
C-7	.220	2.85
C-8	.330	4.20
C-8	.330	3.40
C-8	.329	2.95
C-8	.328	2.85
C-9	.388	4.30
C-9	.396	3.80
C-9	.386	4.35
C-9	.386	4.55

## COMPARISON OF TEST METHODS FOR DETERMINING THE CHLORIDE CONTENT IN CONCRETE

Following completion of the first series of tests, the selected methods were compared to determine which method was more suitable for determining the chloride content of concrete specimens in the field. These comparisons were based on the accuracy in predicting results obtained using the standard potentiometric titration procedure, costs associated with the purchase of the equipment and chemicals necessary for analysis, the speed or time required to weigh, prepare, and analyze individual samples, the level of expertise required in using the procedure without sacrificing accuracy, and the limitations associated with using the procedure in the field.

## TEST RESULTS FOR THE EFFECTS OF CEMENT CONTENT

Two specimens were tested for chloride content at each chloride contamination level using the potentiometric titration procedure and the procedure for the specific ion probe presented in Appendix B. Table 7 presents the results of the specific ion probe test and the standard test method.

Table 7. Potentiometric Titration and  
Specific Ion Probe Results for  
Different Cement Contents

<u>Specimen</u>	<u>W/C</u>	<u>Potentiometric Titration (% Chloride)</u>	<u>Reading Millivolts</u>	<u>Specific Ion Probe Calculated (% Chloride)</u>
DC-1	.40	.021	76.8	.015
	.40	.020	76.6	.015
DC-3	.40	.037	53.7	.041
	.40	.039	52.5	.043
DC-4	.40	.064	35.6	.088
	.40	.064	37.2	.083
DC-5	.40	.083	27	.127
	.40	.091	25.6	.135
DC-6	.40	.158	9	.275
	.40	.154	6.1	.312
DC-7	.40	.184	1.9	.373
	.40	.188	.4	.398
DC-8	.40	.270	-3.2	.442
	.40	.264	-2.7	.433
DC-9	.40	.315	-9.2	.572
	.40	.315	-7.1	.522
EC-0	.50	.018	90	.008
	.50	.020	90.8	.008
EC-3	.50	.037	52.5	.039
	.50	.036	53.5	.038
EC-4	.50	.057	36.5	.078
	.50	.059	35.4	.081
EC-5	.50	.101	23.4	.135
	.50	.103	24	.132
EC-6	.50	.187	2.4	.329
	.50	.185	5.9	.284
EC-7	.50	.224	-2.9	.400
	.50	.230	-1.3	.374
EC-8	.50	.313	-7.5	.486
	.50	.312	-10.9	.562
EC-9	.50	.380	-13.4	.625
	.50	.384	-14.2	.646

TEST RESULTS FOR THE EFFECTS OF TEMPERATURE

Table 8 presents test results for determining the effects of reaction temperature in measuring chloride content with specific ion probe.

Table 8. Specific Ion Probe Test Results for  
the Effects of Reaction Temperature

<u>SPECIMEN</u>	<u>Test Temperature (deg F)</u>	<u>Specific Ion Probe Reading Millivolts</u>	<u>Ion Probe Calculated (% Chloride)</u>	<u>Reaction Temperature (deg F)</u>
C-4	34	30.7	.099	49
C-4	34	32.4	.092	37
C-4	34	34.6	.084	42
C-4	34	29.3	.105	39
C-4	40	32.5	.095	46
C-4	40	31.4	.100	44
C-4	50	29.0	.101	58
C-4	50	33.5	.083	58
C-4	50	32.8	.086	54
C-4	50	31.4	.100	57
C-4	50	35.6	.084	56
C-4	50	35.0	.086	59
C-4	50	38.5	.074	58
C-4	70	43.7	.059	77
C-4	70	42.2	.063	79
C-4	70	35.6	.084	79
C-4	70	43.5	.060	79
C-4	70	45.3	.055	83
C-4	70	44.9	.056	75
C-4	70	39.8	.069	80
C-4	70	40.9	.066	77
C-4	90	43.2	.060	92
C-4	90	41.1	.066	94
C-4	90	40.9	.066	94
C-4	90	42.7	.061	92
C-4	110	48.6	.047	113
C-4	100	48.8	.047	114
C-4	110	45.8	.054	114
C-4	110	47.3	.050	112
C-4	130	57.4	.033	123
C-4	130	53.8	.039	127
C-4	130	53.8	.039	127
C-4	130	51.5	.043	132

## RESULTS FROM FIELD VALIDATION FOR CHLORIDE

The following represents the results of the effort to accomplish the objective set forth for the field validation of the selected method. Presented in the appropriate sections that follow are the results from the field tests for field validation of the specific ion probe, and the appropriate statistical analysis procedures for these tests. Using the relationship for the specific ion probe determined from the field validation tests, chloride content values for the bridges tested were also determined.

### Test Results for the Specific Ion Probe

The following table presents the results of tests for chloride on eighty samples from bridges in Pennsylvania, Virginia, Florida, and Wisconsin. Table 9 presents the test results using the standard potentiometric titration procedure as reported by Baden [45], and the results for the specific ion probe on specimens which were not crushed and specimens crushed to pass a No. 50 sieve.

Table 9. Field Validation Test Results

<u>State</u>	<u>Bridge</u>	<u>Sample Number</u>	Potentiometric	Specific Ion Probe	
			Titration	Test Results	Test Results
			Results[45]	Crushed	NotCrushed
			(% Cl <sup>-</sup> )	(% Cl <sup>-</sup> )	(% Cl <sup>-</sup> )
PA	11-15/35 NB	B-1	.076	.088	.099
		2	.027	.038	.035
		3	.014	.023	.016
		4	.018	.021	.013
		G-1	.047	.055	.073
		2	.019	.025	.016
		3	.018	.025	.021
		H-3	.027	.037	.036
		I-1	.111	.164	.192
		2	.025	.043	.032
		P1-2	.280	.384	.505
		3	.081	.138	.125
		4	.019	.030	.016
		5	.014	.023	.014
		P2-2	.032	.047	.042
		3	.013	.025	.017
		4	.015	.024	.014
		C1-4	.015	.022	.016
		C2-3	.025	.034	.032
		4	.015	.025	.017
VA	I81 / 927 NB	A-1	.256	.314	.397
		2	.096	.149	.173
		3	.030	.050	.038
		4	.020	.023	.021
		B-1	.389	.460	.850
		2	.183	.419	.367
		3	.216	.322	.353
		D-1	.380	.641	.855
		3	.142	.288	.299
		4	.101	.181	.207
		5	.074	.123	.123

Table 9 Field Validation Test Results (Continued)

<u>State</u>	<u>Bridge</u>	Potentiometric Titration		Specific Ion Probe Test Results	
		<u>Sample Number</u>	<u>Results (% Cl<sup>-</sup>)</u>	<u>Crushed (% Cl<sup>-</sup>)</u>	<u>Not Crushed (% Cl<sup>-</sup>)</u>
		I-1	.220	.274	.378
		2	.124	.188	.226
		3	.051	.088	.081
		4	.021	.037	.033
		K-2	.107	.150	.152
		4	.025	.031	.025
		L-1	.258	.327	.525
		2	.142	.205	.263
		4	.039	.074	.056
FL	Rt 312	F1-6	.106	.144	.172
		F2-2	.503	.750	.849
		3	.333	.600	.502
		4	.206	.403	.486
		5	.231	.428	.399
		6	.144	.242	.212
		P1-1	.338	.363	.509
		2	.260	.406	.341
		3	.170	.250	.228
		4	.120	.174	.167
		5	.073	.101	.103
		6	.026	.038	.031
		P2-1	.146	.180	.245
		2	.120	.151	.164
		/3	.097	.129	.130
		4	.053	.074	.081
		5	.025	.042	.044
		6	.018	.026	.020
		P3-4	.013	.022	.015
		5	.011	.019	.013

Table 9 Field Validation Test Results (Continued)

<u>State</u>	<u>Bridge</u>	<u>Sample Number</u>	Potentiometric	Specific Ion Probe	
			<u>Titration Results</u> (% Cl <sup>-</sup> )	<u>Test Results</u> <u>Crushed</u> (% Cl <sup>-</sup> )	<u>Not Crushed</u> (% Cl <sup>-</sup> )
WI	Rt 252 WB	B-2	.453	.527	.717
		4	.220	.285	.294
		5	.132	.180	.167
		C-1	.319	.550	.591
		3	.265	.348	.341
		4	.148	.252	.264
		D-2	.344	.549	.757
		3	.365	.512	.605
		4	.220	.382	.381
		5	.138	.228	.223
		6	.054	.072	.067
		K-4	.144	.202	.184
		5	.101	.125	.117
		6	.040	.057	.045
		L-5	.155	.193	.219
		6	.077	.089	.086
		BW1-1	.605	.778	1.051
		3	.134	.208	.297
		4	.213	.282	.417
		5	.202	.275	.390

### Chloride Content of Various Bridges

The results from measuring the chloride content on thirteen bridges from Pennsylvania, Virginia, Florida, and Wisconsin using the specific ion probe are presented in Appendix D, Tables E1 to E13. Also presented are the predicted values for the chloride content for the potentiometric titration procedure.

### Effective Diffusion Constants of Various Bridges

The values of the effective diffusion constants for the thirteen bridges which were analyzed using the specific ion probe were calculated by How Lum [34] and are presented in Table 10. Also presented in Table 10 are values for the effective diffusion constants for bridges ES-2, ES-3, BS-6, and BS-7, also calculated by How Lum [34]. These bridges correspond to the bridges which were sampled in Pennsylvania approximately 4 years ago by Weyers and Cady [28].

Table 10. Effective Diffusion Constants for  
Various Bridge Components

<u>State</u>	<u>Bridge Location</u>	<u>Hole No.</u>	<u>Component</u>	$D_c$	$C_o$	$R^2$	$W/C$
FL	RTE 206	8	BEAM	.020	.550	99.80	.44
FL	RTE 206	9	BEAM	.015	1.600	99.80	.44
FL	RTE 206	10	DECK	.005	2.050	99.90	.44
FL	RTE 206	11	DECK	.015	0.650	100.00	.44
FL	RTE 206	12	DECK	.015	0.600	99.80	.44
FL	RTE 206	13	DECK	.035	0.700	97.00	.44
FL	RTE 206	1	COLUMN	.435	18.650	93.40	.44
FL	RTE 206	2	COLUMN	.045	11.500	98.60	.44
FL	RTE 206	3	COLUMN	.105	13.950	98.30	.44
FL	RTE 206	4	COLUMN	.020	2.350	99.50	.44
FL	RTE 206	5	COLUMN	.155	21.550	99.30	.44
FL	RTE 206	6	COLUMN	.095	12.250	91.70	.44
FL	RTE 206	7	CAP	.030	2.850	98.90	.44
FL	RTE 312	6	BEAM	.005	0.050	99.60	.44
FL	RTE 312	10	DECK	.025	0.850	94.40	.44
FL	RTE 312	11	DECK	.015	0.900	99.70	.44
FL	RTE 312	4	FOOTING	.300	20.150	94.40	.44
FL	RTE 312	5	FOOTING	.260	24.150	93.50	.44
FL	RTE 312	1	CAP	.130	13.700	99.30	.44
FL	RTE 312	2	CAP	.115	7.450	99.10	.44
FL	RTE 312	3	CAP	.035	5.850	99.10	.44
FL	RTE 312	8	CAP	.140	1.300	96.70	.44
FL	RTE 312	9	CAP	.110	0.500	87.20	.44
FL	RTE 295	5	BEAM	.020	0.300	96.10	.44
FL	RTE 295	6	BEAM	.005	1.050	99.30	.44
FL	RTE 295	1	COLUMN	.060	6.250	96.60	.44
FL	RTE 295	2	COLUMN	.030	0.900	99.70	.44
FL	RTE 295	3	COLUMN	.050	7.700	97.50	.44
FL	RTE 295	4	COLUMN	.030	1.000	98.30	.44
FL	RTE 295	7	CAP	.005	0.900	99.90	.44
FL	RTE 295	8	CAP	.015	0.350	95.70	.44
FL	RTE 295	9	CAP	.015	0.850	93.70	.44
FL	RTE 295	10	CAP	.015	0.700	98.70	.44
FL	RTE 295	12	CAP	.005	1.200	99.40	.44

Table 10 Effective Diffusion Constants for  
Various Bridge Components (Continued)

<u>State</u>	<u>Bridge Location</u>	<u>Hole No.</u>	<u>Component</u>	$D_c$	$C_o$	$R^2$	<u>W/C</u>
PA	11 & 15 NB	1	DECK	.080	3.950	75.60	.47
PA	11 & 15 NB	2	DECK	.035	15.700	99.90	.47
PA	11 & 15 NB	3	DECK	.015	12.700	99.70	.47
PA	11 & 15 NB	4	DECK	.015	16.950	99.80	.47
PA	11 & 15 NB	5	DECK	.010	13.050	99.80	.47
PA	11 & 15 NB	6	DECK	.020	3.350	97.50	.47
PA	11 & 15 NB	7	DECK	.030	7.150	98.80	.47
PA	11 & 15 NB	8	DECK	.020	9.900	97.80	.47
PA	11 & 15 NB	9	DECK	.020	4.650	97.10	.47
PA	11 & 15 NB	10	DECK	.020	9.450	98.50	.47
PA	11 & 15 NB	11	DECK	.015	5.300	86.10	.47
PA	11 & 15 NB	12	DECK	.010	16.700	97.40	.47
PA	11 & 15 NB	15	CAP	.160	0.700	89.40	.47
PA	11 & 15 NB	16	CAP	.035	2.750	93.20	.47
PA	11 & 15 NB	13	COLUMN	.045	10.500	99.60	.47
PA	11 & 15 NB	14	COLUMN	.035	18.350	99.80	.47
PA	11 & 15 SB	1	DECK	.015	5.950	85.00	.47
PA	11 & 15 SB	2	DECK	.025	5.200	98.50	.47
PA	11 & 15 SB	3	DECK	.020	4.850	98.50	.47
PA	11 & 15 SB	4	DECK	.020	4.250	80.40	.47
PA	11 & 15 SB	5	DECK	.040	9.250	98.80	.47
PA	11 & 15 SB	6	DECK	.015	11.750	97.20	.47
PA	11 & 15 SB	7	CAP	.040	31.250	98.60	.47
PA	11 & 15 SB	8	CAP	.010	24.550	99.50	.47
PA	11 & 15 SB	9	COLUMN	.155	1.250	93.90	.47
PA	11 & 15 SB	10	COLUMN	.140	1.900	94.30	.47
PA	I-180 WB	1	DECK	.015	6.100	99.60	.47
PA	I-180 WB	2	DECK	.035	19.450	98.90	.47
PA	I-180 WB	3	DECK	.025	28.400	99.80	.47
PA	I-180 WB	4	DECK	.015	22.400	99.10	.47
PA	I-180 WB	5	DECK	.010	7.850	97.80	.47
PA	I-180 WB	6	DECK	.015	9.950	98.70	.47
PA	I-180 WB	8	DECK	.015	20.150	99.90	.47
PA	I-180 WB	9	DECK	.010	13.350	98.40	.47
PA	I-180 WB	10	DECK	.125	0.850	90.10	.47
PA	I-180 WB	11	DECK	.010	9.200	96.70	.47
PA	I-180 EB	1	DECK	.040	6.200	88.60	.47
PA	I-180 EB	2	DECK	.030	12.200	99.40	.47
PA	I-180 EB	3	DECK	.015	3.350	95.70	.47
PA	I-180 EB	4	DECK	.010	19.700	98.80	.47
PA	I-180 EB	5	DECK	.015	15.200	99.40	.47
PA	I-180 EB	6	DECK	.015	14.100	99.70	.47

Table 10 Effective Diffusion Constants for  
Various Bridge Components (Continued)

<u>State</u>	<u>Bridge Location</u>	<u>Hole No.</u>	<u>Component</u>	$D_c$	$C_o$	$R^2$	<u>W/C</u>
VA	I81 NB 927	1	DECK	.015	15.600	98.80	.49
VA	I81 NB 927	2	DECK	.045	21.400	91.80	.49
VA	I81 NB 927	3	DECK	.075	9.750	93.90	.49
VA	I81 NB 927	4	DECK	.050	22.300	99.20	.49
VA	I81 NB 927	5	DECK	.020	18.200	98.40	.49
VA	I81 NB 927	6	DECK	.015	13.550	99.10	.49
VA	I81 NB 927	7	DECK	.010	31.800	99.40	.49
VA	I81 NB 927	8	DECK	.050	14.500	99.90	.49
VA	I81 NB 927	9	DECK	.025	13.000	98.10	.49
VA	I81 NB 927	10	DECK	.035	20.050	99.60	.49
VA	I81 NB 927	11	DECK	.010	22.250	100.00	.49
VA	I81 NB 927	12	DECK	.025	17.050	99.70	.49
VA	I81 NB 927	13	COLUMN	.065	5.000	97.80	.49
VA	I81 NB 927	12	COLUMN	.275	3.750	91.00	.49
VA	I81 NB 927	15	CAP	.160	5.850	97.80	.49
VA	I81 NB 927	16	CAP	.350	5.300	94.40	.49
VA	I81 NB 927	17	BEAM	.145	10.050	98.70	.49
VA	I81 NB 927	18	BEAM	.070	11.850	81.90	.49
VA	I81 NB 676	1	DECK	.035	22.350	98.20	.49
VA	I81 NB 676	2	DECK	.040	15.700	98.90	.49
VA	I81 NB 676	3	DECK	.025	9.700	92.80	.49
VA	I81 NB 676	4	DECK	.105	12.850	100.00	.49
VA	I81 NB 676	5	DECK	.045	11.650	97.40	.49
VA	I81 NB 676	6	DECK	.135	10.250	92.80	.49
VA	I81 NB 676	7	DECK	.005	8.350	99.70	.49
VA	I81 NB 676	8	DECK	.020	14.900	99.20	.49
VA	I81 NB 676	9	DECK	.010	9.500	99.70	.49
VA	I81 NB 676	10	DECK	.005	12.750	99.90	.49
VA	I81 NB 676	11	DECK	.010	11.200	99.40	.49
VA	I81 NB 676	12	DECK	.080	14.850	96.70	.49
VA	I81 NB 676	13	COLUMN	.030	5.050	99.60	.49
VA	I81 NB 676	14	COLUMN	.025	3.150	98.00	.49
VA	I81 NB 676	15	COLUMN	.230	8.950	84.20	.49
VA	I81 NB 676	16	CAP	.155	10.550	99.00	.49
VA	I81 NB 676	17	CAP	.525	9.650	99.60	.49
VA	I81 NB 676	19	BEAM	.005	5.850	97.90	.49
VA	I81 NB 676	20	BEAM	.005	1.750	98.50	.49
VA	I81 NB 670	1	DECK	.015	22.500	99.90	.49
VA	I81 NB 670	2	DECK	.035	10.550	99.60	.49
VA	I81 NB 670	3	DECK	.010	15.200	99.30	.49
VA	I81 NB 670	4	DECK	.010	15.250	97.70	.49
VA	I81 NB 670	5	DECK	.015	18.700	98.40	.49
VA	I81 NB 670	6	DECK	.011	15.250	97.60	.49
VA	I81 NB 670	7	DECK	.040	9.350	98.80	.49

Table 10 Effective Diffusion Constants for  
Various Bridge Components (Continued)

<u>State</u>	<u>Bridge Location</u>	<u>Hole No.</u>	<u>Component</u>	<u>D<sub>c</sub></u>	<u>C<sub>0</sub></u>	<u>R<sup>2</sup></u>	<u>W/C</u>
VA	I81 NB 670	8	DECK	.010	12.500	99.70	.49
VA	I81 NB 670	9	DECK	.005	22.500	97.40	.49
VA	I81 NB 670	10	DECK	.005	20.650	98.60	.49
VA	I81 NB 670	11	DECK	.005	17.300	98.80	.49
VA	I81 NB 670	12	DECK	.010	13.150	99.50	.49
VA	I81 NB 670	13	COLUMN	.170	8.500	94.90	.49
VA	I81 NB 670	14	COLUMN	.010	7.650	98.60	.49
VA	I81 NB 670	15	COLUMN	.070	9.050	98.90	.49
VA	I81 NB 670	16	BEAM	.005	16.050	97.10	.49
VA	I81 NB 670	17	BEAM	.005	8.250	98.40	.49
VA	I81 NB 670	18	BEAM	.010	16.100	99.20	.49
VA	I81 NB 670	19	CAP	.080	16.100	99.90	.49
WI	WI243	15	COLUMN	.030	2.450	64.40	.53
WI	WI243	16	COLUMN	.130	1.100	84.40	.53
WI	WI243	17	COLUMN	.045	1.950	90.80	.53
WI	WI243	1	DECK	.050	16.050	98.90	.53
WI	WI243	2	DECK	.070	17.500	95.80	.53
WI	WI243	3	DECK	.045	25.300	98.60	.53
WI	WI243	4	DECK	.045	22.050	97.00	.53
WI	WI243	5	DECK	.045	20.450	99.20	.53
WI	WI243	6	DECK	.080	13.550	98.40	.53
WI	WI243	7	DECK	.070	15.300	99.60	.53
WI	WI243	8	DECK	.065	16.850	99.80	.53
WI	WI243	9	DECK	.060	23.900	98.30	.53
WI	WI243	10	DECK	.070	19.050	98.30	.53
WI	WI243	11	DECK	.060	19.650	96.80	.53
WI	WI243	12	DECK	.105	15.800	98.10	.53

Table 10 Effective Diffusion Constants for  
Various Bridge Components (Continued)

<u>State</u>	<u>Bridge Location</u>	<u>Hole No.</u>	<u>Component</u>	<u>D<sub>c</sub></u>	<u>C<sub>o</sub></u>	<u>R<sup>2</sup></u>	<u>W/C</u>
WI	WI245	13	COLUMN	.115	11.400	92.50	.53
WI	WI245	14	COLUMN	.095	1.800	48.60	.53
WI	WI245	15	COLUMN	.105	17.650	98.70	.53
WI	WI245	16	COLUMN	.075	5.450	80.70	.53
WI	WI245	1	DECK	.145	16.300	94.60	.53
WI	WI245	2	DECK	.390	5.750	56.90	.53
WI	WI245	3	DECK	.100	14.650	98.20	.53
WI	WI245	4	DECK	.050	21.500	94.50	.53
WI	WI245	5	DECK	.060	17.500	98.60	.53
WI	WI245	6	DECK	.075	15.700	98.70	.53
WI	WI245	7	DECK	.085	20.350	99.00	.53
WI	WI245	8	DECK	.075	19.650	96.40	.53
WI	WI245	9	DECK	.110	13.550	94.90	.53
WI	WI245	10	DECK	.080	15.900	99.20	.53
WI	WI245	11	DECK	.100	18.500	99.90	.53
WI	WI245	12	DECK	.060	16.900	99.40	.53
WI	WI252	13	COLUMN	.060	12.400	96.10	.53
WI	WI252	14	COLUMN	.040	4.100	96.70	.53
WI	WI252	1	DECK	.080	19.100	97.00	.53
WI	WI252	2	DECK	.065	26.600	99.80	.53
WI	WI252	3	DECK	.090	16.300	95.30	.53
WI	WI252	4	DECK	.120	21.250	96.00	.53
WI	WI252	5	DECK	.075	21.000	98.30	.53
WI	WI252	6	DECK	.070	20.350	99.90	.53
WI	WI252	7	DECK	.090	26.350	96.00	.53
WI	WI252	8	DECK	.105	18.350	96.90	.53
WI	WI252	9	DECK	.075	21.300	97.40	.53
WI	WI252	10	DECK	.120	18.650	96.90	.53
WI	WI252	11	DECK	.070	19.350	98.80	.53
WI	WI252	12	DECK	.125	18.750	97.50	.53

Table 10 Effective Diffusion Constants for  
Various Bridge Components (Continued)

<u>State</u>	<u>Bridge Location</u>	<u>Hole No.</u>	<u>Component</u>	<u>D<sub>c</sub></u>	<u>C<sub>o</sub></u>	<u>R<sup>2</sup></u>	<u>W/C</u>
PA	ES-2	1	DECK	.095	11.3	98.66	.47
PA	ES-2	2	DECK	.035	6.9	99.95	.47
PA	ES-2	3	DECK	.065	7.8	99.78	.47
PA	ES-3	1	DECK	.0675	15.5	99.94	.47
PA	ES-3	2	DECK	.035	11.55	99.95	.47
PA	ES-3	3	DECK	.0475	9.35	98.31	.47
PA	BS-6	1	DECK	.035	10.6	99.22	.47
PA	BS-6	2	DECK	.025	6.4	99.87	.47
PA	BS-6	3	DECK	.035	8.4	99.96	.47
PA	BS-7	1	DECK	.0425	13.95	100.00	.47
PA	BS-7	2	DECK	.055	13.55	99.30	.47
PA	BS-7	3	DECK	.030	9.8	100.00	.47

Table 11 presents the values for the mean effective diffusion constant and standard deviation for the diffusion constants presented in Table 10. Effective diffusion constants having an  $R^2$  less than 80% were excluded from the data calculations. Also excluded from this table are the values presented for bridges ES-2, ES-3, BS-6, and BS-7 which analyzed during a different time period. The mean values for the temperatures reported were adapted from the appropriate weather reports [50,51].

Table 11. Mean Values for the Effective Diffusion Constants  
Presented for Various Bridges

<u>State</u>	<u>Bridge Component</u>	<u>Number of Values</u>	<u>Mean <math>D_c</math></u>	<u>Standard Deviation <math>D_c</math></u>	<u>Area Mean Temperature** (°F)</u>	<u>W/C</u>
FL	Subst	28	.0809	.1025	67.9	.44
PA	Deck	33	.02258	.02031	52.7	.47
VA	Deck	36	.02947	.02954	56.3	.49
WI	Deck	35	.07971	.02470	45.9	.53
FL	Deck	6	.01833	.01033	67.9	.44
PA	Subst	8	.0775	.0625	52.7	.50
VA	Subst	20	.1195	.1382	56.3	.49
WI	Subst	7	.08143	.03292	45.9	.53

\*\* Reference [50,51]

Table 12 presents the data for four bridge decks in Pennsylvania which current effective diffusion constants and effective diffusion constants on chloride data from the same bridge decks presented by Weyers and Cady [28] were calculated by How Lum [34]. The chloride data obtained from Weyers and Cady [28] were analyzed in April 1985, approximately 4 years previous to this analysis.

Table 12. Effective Diffusion Constant Values for  
Four Bridges in Pennsylvania  
at Different Time Periods

Bridge ES-2 PA180WB		Bridge ES-3 PA180EB		Bridge BS-6 11&15NB		Bridge BS-7 11&15SB	
.095	.015	.0675	.040	.035	.080	.0425	.015
.035	.035	.0350	.030	.025	.035	.0550	.025
.065	.025	.0475	.015	.035	.015	.0300	.020
	.015		.010		.015		.020
	.010		.015		.010		.040
	.015		.015		.020		.015
	.015				.030		
	.010				.020		
	.125				.020		
	.010				.020		
					.015		
					.010		

Note: ES-2 is the same bridge as PA180WB  
 ES-3 is the same bridge as PA180EB  
 BS-6 is the same bridge as 11&15NB  
 BS-7 is the same bridge as 11&15SB

Table 13 presents the mean values and standard deviations of the values for the effective diffusion constant presented in Table 12.

Table 13. Mean Effective Diffusion Constant Values for  
 Four Bridges in Pennsylvania  
 at Different Time Periods

<u>Bridge</u>	<u>Bridge Component</u>	<u>Number of Values</u>	<u>Mean <math>D_c</math></u>	<u>Standard Deviation <math>D_c</math></u>
ES-2	Deck	3	.0650	.0300
PA180WB	Deck	10	.0275	.0351
ES-3	Deck	3	.05000	.01639
PA180EB	Deck	6	.02083	.01158
BS-6	Deck	3	.03167	.00577
11&15NB	Deck	12	.02417	.01905
BS-7	Deck	3	.04250	.01250
11&15SB	Deck	6	.02250	.00935

Note: ES-2 is the same bridge as PA180WB  
 ES-3 is the same bridge as PA180EB  
 BS-6 is the same bridge as 11&15NB  
 BS-7 is the same bridge as 11&15SB

## ANALYSIS OF TEST RESULTS

### Standard Potentiometric Titration Method

The results obtained using the standard potentiometric titration method were compared with the known amounts of chloride added to each specimen. The amount of background chloride in each specimen was estimated to be the average of four tests on specimen C-0, as illustrated in Table 1. The Wilcoxon Signed Rank procedure for paired data [46] was used to determine if the amount of chloride measured using the potentiometric titration less the amount of chloride added was equivalent to the amount of background chloride. The test results presented in Table 1 were used for the comparison. Referring to Table 1, the following relationships are noted:

$$\begin{aligned} \text{Let the Difference} &= \text{Amount of chloride measured} - \\ &\quad \text{Added chloride} \\ \text{or Diff} &= \text{ACTUAL} - \text{ADDED} \end{aligned} \tag{22}$$

also, the average of four test results for background chloride (% Cl<sup>-</sup>) determined for specimen C-0) = .02025

The expression for the comparison of the data presented in Table 1 is given by the following hypothesis:

H<sub>0</sub>: Diff = .02025 : null hypothesis

H<sub>1</sub>: Diff > .02025 : alternative

The microcomputer version of Minitab was used to analyze the data with the Wilcoxon Signed Rank procedure. In the results that follow, 'median' may be substituted for the word 'mean' because of the symmetry assumptions associated with the test procedure [47,49]. Generally, the results from the analysis may be interpreted as follows:

The null and alternative hypotheses as restated by the Minitab statistical package is presented as:

TEST OF MEDIAN = 0.02025 VERSUS MEDIAN G.T. 0.02025

The results of the Wilcoxon Signed Rank procedure are presented in the following form:

	N	WILCOXON		ESTIMATED
	TESTS	STATISTIC	P-VALUE	MEDIAN_
ACTUAL-ADDED	40	667.0	0.000	0.027

where,

ACTUAL-ADDED represents the difference between the amount of chloride measured and the amount of chloride added.

N - refers to the number of observations in the group to be tested.

WILCOXIN STATISTIC - refers to the test statistic result calculated by Minitab equivalent to  $T^+$ , as defined previously.

P-VALUE - represents the amount of rejection region remaining above the Wilcoxon test statistic results (in this case 667).

and,

ESTIMATED MEDIAN - represents the estimated value for the true mean,  $\mu$ , for the samples tested.

The null hypothesis in a two sided test is rejected if the P-VALUE  $< \alpha$ . In this test,  $\alpha = .05$ .

The following results were obtained using Minitab for a 95% Confidence Interval of the estimated true mean value:

	ESTIMATED	ACHIEVED	
N	MEDIAN	CONFIDENCE	CONFIDENCE INTERVAL
ACTUAL-ADDED 40	0.0272	95.0	(0.0230, 0.0371)

where,

ACHIEVED CONFIDENCE - represents the level of significance for the confidence interval presented for the estimated median or true mean. In this case, a 95% confidence interval has been calculated for the estimated true mean.

CONFIDENCE INTERVAL establishes the lower and upper bound values for the estimated median or true mean.

As illustrated, results of the test procedure indicate the null hypothesis is rejected at a 5% level of significance as represented by the P-value ( $<.05$ ). The actual or true mean was estimated to be .0272.

The achieved confidence or 95% confidence interval for the estimated true mean,  $\mu_D$ , is  $(.0230 < \mu_D < .0371)$  as illustrated in the results from the analysis using Minitab.

The results of the Wilcoxon Signed Rank procedure suggests there is sufficient evidence to indicate that the difference in the amount of chloride added and the amount of chloride measured in the specimens is more than the amount of background chloride as determined by testing four samples from specimen C-0 for chloride. In essence, the results of this analysis indicate there were errors associated in adding chloride to the ten specimens, variations in the amount of background chloride were present in the aggregate materials or mixing water in each specimen, or different amounts of aggregates were present in each sample (resulting in different values for the amount of chloride present) as a result of the sampling procedure.

Since the results of the Wilcoxon Signed Rank analysis procedure were somewhat inconclusive, a regression analysis procedure was performed using Minitab to determine if a relationship exists between the difference in the amount of chloride added and the amount of chloride measured in the specimens, versus the amount of chloride added to the concrete. The following represents the results obtained from a regression analysis using Minitab.

The regression equation is given by:

$$\text{ACT-ADD} = 0.0160 + 0.103 \text{ ACTCL} \quad (23)$$

where ACT-ADD = Diff (as previously defined)

and ACTCL = the amount of chloride measured

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives  
were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p_
Constant	0.01604	0.002811	5.69	0.000
ACTCL	0.103	0.01468	7.02	0.000

s = 0.01180                  R-sq = 56.4%                  R-sq(adj) = 55.3%

where,

Coef represents the values for the intercept  
(Constant) and slope (ACTCL)

Stdev represents values for the standard deviation  
of the estimated values used in determining the 95%  
confidence interval for the slope and intercept values

t-ratio represents values for the t test of the  
null hypothesis and alternative stated previously

p\_ or P VALUE represents twice the amount of  
rejection region remaining above the value for the  
t test statistic results in a two sided test (in this  
case t = 5.69 for the constant or intercept and 7.02  
for the slope).

s represents the standard deviation of the MS or  
Mean Square Error presented in the Analysis of Variance  
Table below, commonly referred to as the standard

deviation of the predicted value about the regression [46,49] line

R-sq or the coefficient of determination represents the proportion of the variation in the predicted variable which may be explained from the independent variable. R-sq is generally considered a good measure of the strength of the relationship [46].

R-sq(adj) or adjusted R-sq represents the proportion of the in the predicted variable which may be predicted from the independent variable in the population [46]

The analysis of variance for the previous regression analysis as presented by Minitab is as follows:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	0.0068612	0.0068612	49.24	0.000
Error	38	0.0052950	0.0001393		
Total	39	0.0121562			

where,

DF represents the degrees of freedom due to Regression, Errors, and the Total degrees of freedom for the regression analysis procedure

SS represents the Sum of Squares error due to Regression, the Sum of Squares error due to the error of prediction, and the Total Sum of Square Errors.

MS represents the Mean Squared error due to Regression, and the Mean Square error due to the error of prediction

F value represents the value for a test statistic previously defined as [46]:  $F_{obs} = \frac{MS_{reg}}{MS_{res}}$ , useful in determining if a relationship exists between the values for the independent or X variables and the dependent or Y variables

p or P VALUE represents the amount of of rejection region above the value for the F test statistic results (in this case 49.24). For the F test, a p value  $< .05$  would reject the null hypothesis at a level of significance,  $\alpha = .05$ .

Results of the analysis indicate there is a relationship between the independent and the predicted values and that values for both the slope and the intercept in the regression equation are significantly different from 0 as indicated by the p value. As evidenced by the value of the coefficient of determination,  $R-sq = 56.4 \%$ , a little over half of the variation in the predicted variable may be explained by the independent variables.

The results of the analysis of variance indicate the error sum of squares for the predicted values are nearly as great as the error sum of squares for the regression. In summary, even though the F test indicates there is a relationship, the relationship is considered to be weak.

The following is a list of the unusual observations having the most influence in the regression analysis.

Unusual Observations

Obs.	ACTCL	ACT-ADD	Fit	Stdev.Fit	Residual	St.Resid
29	0.222	0.00790	0.03886	0.00220	-0.03096	-2.67R
30	0.222	0.00790	0.03886	0.00220	-0.03096	-2.67R
31	0.221	0.00690	0.03876	0.00219	-0.03186	-2.75R
32	0.220	0.00590	0.03866	0.00218	-0.03276	-2.82R

R denotes an obs. with a large standard residual.

Obs denotes the observation number in the analysis

ACTCL denotes the observations for the independent variable in the regression analysis

ACT-ADD denotes the dependent variable

Fit represents the predicted value for the dependent variable

Stdev. Fit or Standard deviation of Fit corresponds to the anticipated deviation in values estimated for the Fit or predicted values

Residual represents the error of the predicted value in relation to the actual value of the dependent variable for an individual observation related by:

$$\text{Residual} = \text{ACT-ADD} - \text{Fit}$$

St. Resid or Standard Residual refers to the number of standard residuals the observation lies away from the regression line

The results of the previous analyses indicate that the total variation between the difference or background

chloride and the actual amount of chloride added is not entirely do to errors associated with measuring or adding chloride to the specimens. When incorporating the results of the Wilcoxon Signed Rank procedure with the regression analysis, one may conclude that the total variation in the results observed is not entirely associated with operator and instrument errors in adding chloride to the ten specimens. In other words, the amount of background chloride present in the aggregate materials or mixing water varies in each specimen or from batch to batch when mixing concrete, or different amounts of aggregates were present in each sample as a result of the sampling procedure used.

In conclusion, when analyzing concrete samples, the effects of the plus 50 mesh material should be incorporated into the analysis procedure to provide more precise estimates for the actual amount of chloride present in the sample regardless of what testing procedure is used.

#### Specific Ion Probe

The samples analyzed using the specific ion probe, illustrated in Table 2, were tested at two different time intervals. Therefore it was necessary to calibrate the chloride specific electrode for each series of tests in accordance with the procedure recommended by James Instruments, Inc. [39]. A deviation from the recommended calibration procedure involved a regression analysis of the

results to determine the relationship between the chloride contents of the calibration liquids provided by James Instruments, Inc. and the readings obtained on the electrometer instead of determining the relationship graphically. This deviation was utilized to allow more precision in the results of the analysis. The result of the calibration tests are presented by the following regression analyses:

For the first series of tests the regression equation is:

$$\text{LOG}(\text{CL}\%) = - 0.410 - 0.0187 \text{ J1CAL} \quad (24)$$

where LOG(CL%) represents the  $\text{Log}_{10}$  of the percentage of chloride present in each of the three calibration liquids

and J1CAL represents the millivolt readings corresponding to each of the three calibration liquids

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
 versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives

were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-0.410	0.04902	-8.37	0.076
J1CAL	-0.0187	0.0007594	-24.64	0.026
s = 0.05734		R-sq = 99.8%		R-sq(adj) = 99.7%

Results of the regression analysis indicate the estimated value for the slope,  $-0.0187$  is significantly different from 0 at 5% level of significance. The value for the intercept,  $-0.410$ , though not statistically significant at the 5% level is still considered significant and of practical importance in the analysis procedure [46]. Accepting the null hypothesis at this level of significance could also result in a bias of the estimates since the true value for the intercept,  $\beta_0$ , is not known [48].

The coefficient of determination, R-sq, indicates 99.8 % of the variation in LOG%CL can be predicted from the independent variable, J1CAL (millivolt readings)

The following are the results of the analysis of variance for the regression analysis:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	1.9967	1.9967	607.20	0.026
Error	1	0.0033	0.0033		
Total	2	2.0000			

The results of the analysis of variance indicate the error sum of squares for the predict values are very small, much less than the error sum of squares for the regression. In summary, even the F test indicates there is a significant relationship, as indicated by the p value of .026.

For the second series of tests, the regression equation is given by:

$$\text{LOG}(\text{CL}\%) = - 0.438 - 0.0180 \text{ J2CAL} \quad (25)$$

where LOG(CL%) represents the  $\text{Log}_{10}$  of the percentage of chloride present in each of the three calibration liquids

and J2CAL represents the millivolt readings corresponding to each of the three calibration liquids

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives  
were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-0.43751	0.02848	-15.36	0.041
J2CAL	-0.0180402	0.0004324	-41.72	0.015

s = 0.03389      R-sq = 99.9%      R-sq(adj) = 99.9%

Results of the regression analysis indicate the estimated value for the slope, -.018 is significantly different from 0 at 5% level of significance as indicated by the p value, .015. The value for the intercept, -.438, is also statistically significant at the 5% level ( $p = .041$ ). Therefore, there is sufficient evidence to reject the null hypothesis for the slope and the intercept.

The coefficient of determination, R-sq, indicates 99.9 % of the variation in LOG%CL can be predicted from the independent variable, J2CAL ( millivolt readings)

The following are the results of the analysis of

variance for the regression analysis:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	1.9989	1.9989	1740.55	0.015
Error	1	0.0011	0.0011		
Total	2	2.0000			

The results of the analysis of variance indicate the error sum of squares for the predict values are very small, much less than the error sum of squares for the regression. In summary, the F test indicates there is a significant relationship, as indicated by the p value of .015.

The readings obtained from the James Instrument's specific ion probe presented in Table 2 were converted to percent of chloride using these equations. Figure 6 presents the results in Table 2 for the potentiometric titration procedure and the specific ion probe relative to a line of equivalence.

A regression analysis to determine the relationship between the results from the standard potentiometric titration procedure and the specific ion probe was performed using Minitab.

The regression equation using the specific ion probe for the data presented in Table 2 is given as:

$$\text{TITR}\% = 0.00459 + 0.493 \text{ \%JAM}^{\cdot 8} \quad (26)$$

where,

TITR% represents the values for % CL<sup>-</sup> obtained from the potentiometric titration procedure

%JAM represents the readings obtained using the specific ion probe which have been transformed using equations 3 and 4 presented earlier

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
 versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives  
 were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	0.004590	0.002966	1.55	0.130
%JAM <sup>0.8</sup>	0.493283	0.007795	63.28	0.000

s = 0.01265

R-sq = 99.1%

R-sq(adj) = 99.0%

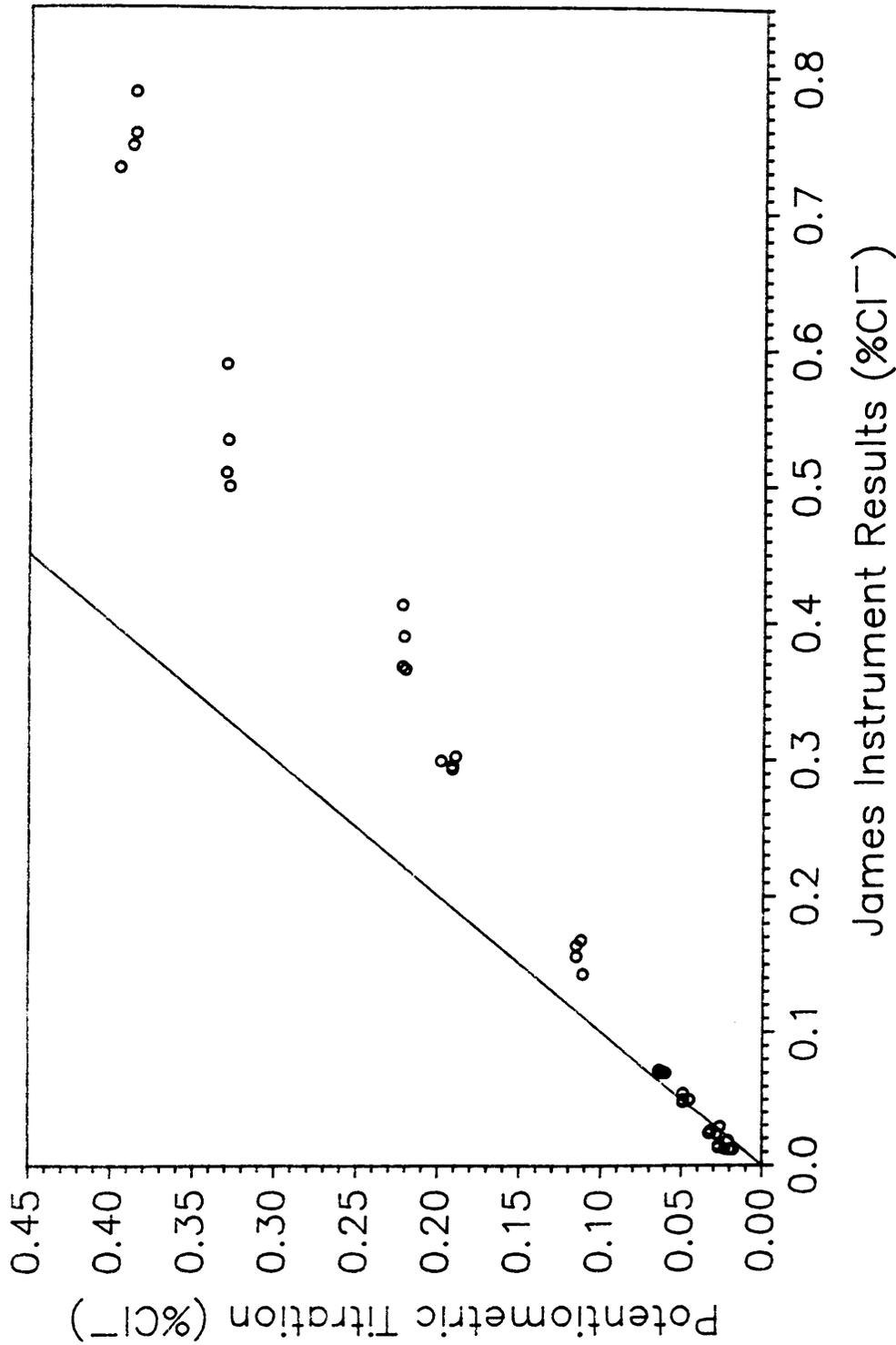


Figure 6. Equivalency Between the Potentiometric Titration and Specific Ion Probe Test Results

Results of the regression analysis indicate the estimated value for the slope, .493 is significantly different from 0 at 5% level of significance ( $p = 0$ ). The value for the intercept, .00459, though not statistically significant at the 5% level ( $p = .13$ ) is still considered significant and of practical importance in the analysis procedure [46]. Accepting the null hypothesis at this level of significance could result in a bias of the estimates since the true value for the intercept,  $\beta_0$ , is not known [48].

The coefficient of determination, R-sq, indicates 99.1 % of the variation in TITR% can be predicted from the independent variable, %JAM<sup>.8</sup> ( millivolt readings converted to percent chloride, %JAM)

The following are results of the analysis of variance for the regression analysis:

#### Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	0.64055	0.64055	4004.64	0.000
Error	38	0.00608	0.00016		
Total	39	0.64662			

The results of the analysis of variance indicate the error sum of squares for the predicted values are very small in relation to the error sum of squares for the regression. In summary, the F test indicates there is a significant relationship, as indicated by the p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

Unusual Observations

Obs.	%JAM <sup>.8</sup>	TITR%	Fit	Stdev.Fit	Residual	St.Resid
30	.493	.22200	.24775	.00259	-.02575	-2.08R
33	.584	.33000	.29255	.00309	.03745	3.05R
35	.605	.32900	.30325	.00322	.02575	2.11R
36	.574	.32800	.28782	.00304	.04018	3.27R
40	.827	.38600	.41238	.00470	-.02638	-2.25R

R denotes an observation with a large standard residual.

The regression analysis indicates a strong relationship exists between the results from the standard potentiometric titration procedure and the results obtained from tests conducted with the specific ion probe. Though the results of the analysis indicates the value for the intercept is not significantly different from 0 at the 5% level of significance, the intercept value obtained from the method of least squares is maintained to reduce the element of bias in the estimates [48] for the predicted value or the predicted results from the standard potentiometric titration procedure.

A plot of the results obtained using the specific ion probe as a function of the potentiometric titration results and the 95 percent confidence limits for the prediction interval is presented in Figure 7.

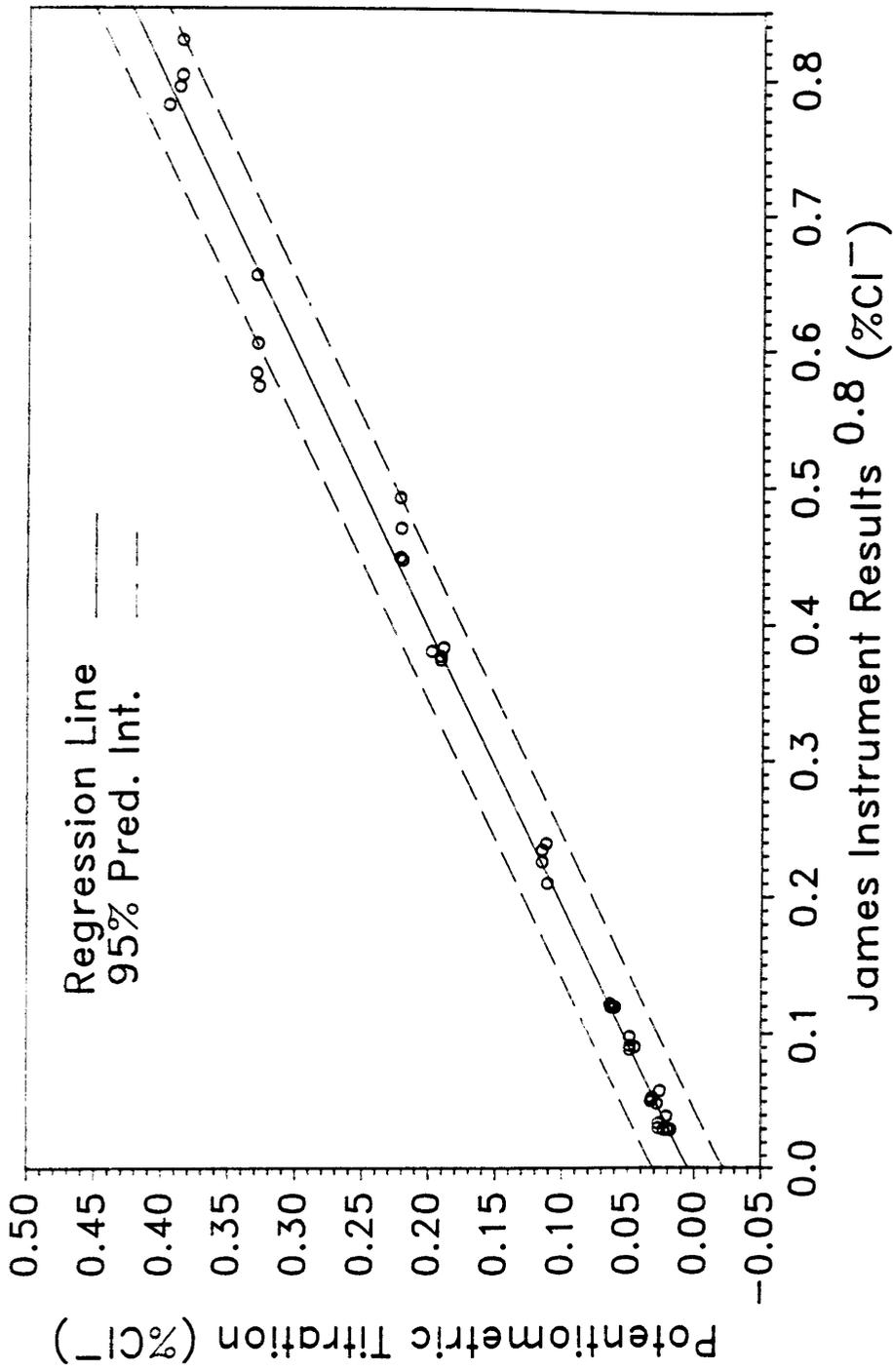


Figure 7. Potentiometric Titration Versus Specific Ion Probe

## Spectrophotometric

Samples analyzed using the spectrophotometer were also compared to the results obtained from the potentiometric titration procedure. Figure 8 presents the results in Table 3 for the potentiometric titration procedure and the spectrophotometer relative to a line of equivalence.

A regression analysis was performed on the data from Table 3 using the microcomputer version of Minitab. The results of the regression analysis procedure are presented below.

The regression equation for the spectrophotometer is:

$$\text{TITR\%} = - 0.00856 + 0.0207 \text{ SPECTRO} \quad (27)$$

where TITR% = results from tests using the standard

potentiometric titration procedure presented in Table 3

SPECTRO = results obtained from the spectrophotometer also presented in Table 3

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses

versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives

were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-0.008560	0.006140	-1.39	0.171
SPECTRO	0.0207322	0.0006477	32.01	0.000

s = 0.02467

R-sq = 96.4%

R-sq(adj) = 96.3%

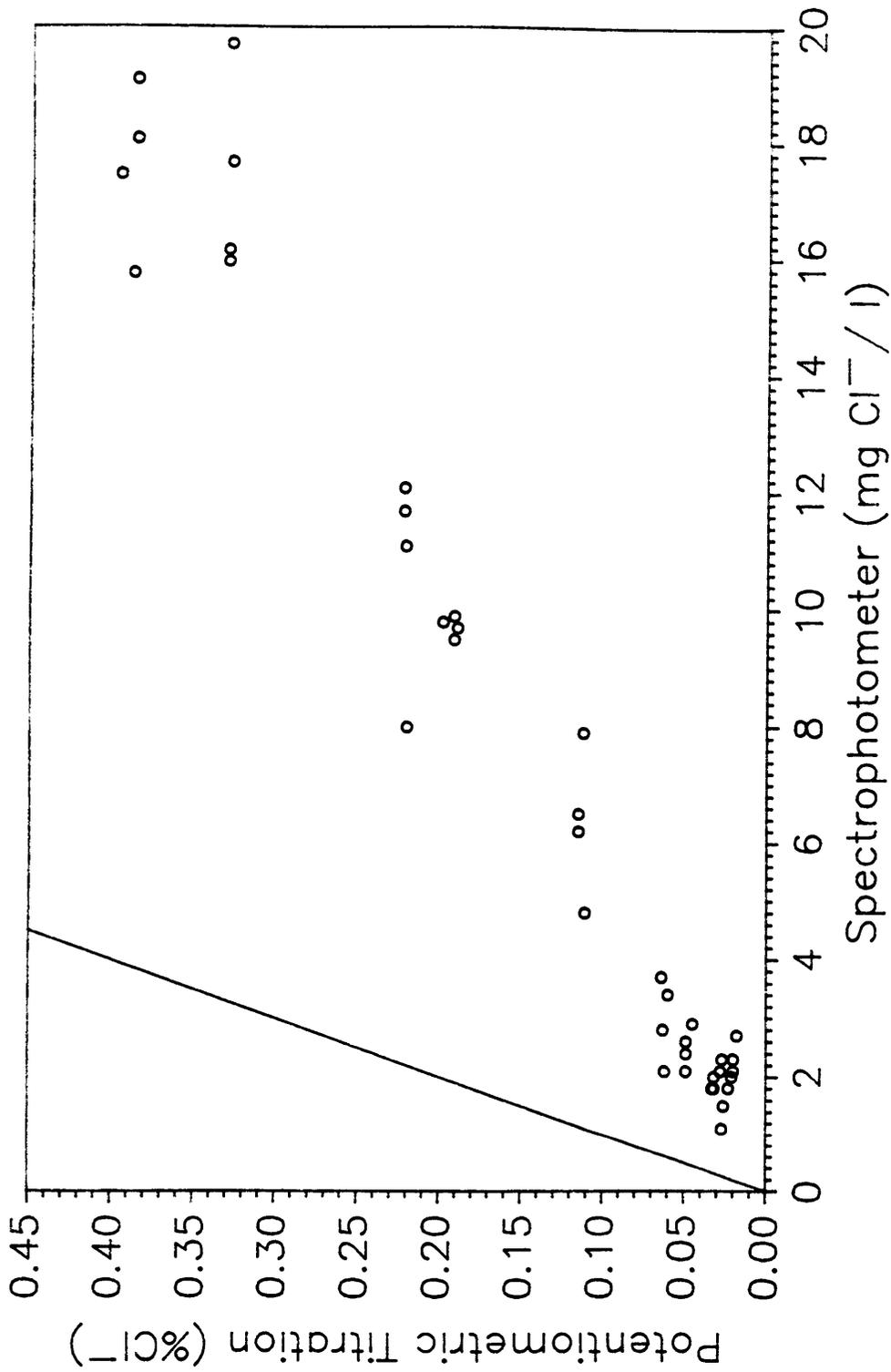


Figure 8. Equivalency Between the Potentiometric Titration and Spectrophotometer Test Results

Results of the regression analysis indicate the estimated value for the slope, .0207 is significantly different from 0 at 5% level of significance ( $p = .000$ ). The value for the intercept,  $-.00856$ , though not statistically significant at the 5% level is still considered significant and of practical importance in the analysis procedure [46]. Accepting the null hypothesis at this level of significance could also result in a bias of the estimates since the true value for the intercept,  $\beta_0$ , is not known [48].

The coefficient of determination, R-sq, indicates 96.4 % of the variation in TITR% can be predicted from the independent variable, SPECTRO (results from the spectrophotometer)

The following are the results of the analysis of variance for the regression analysis:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	0.62350	0.62350	1024.48	0.000
Error	38	0.02313	0.00061		
Total	39	0.64662			

The results of the analysis of variance indicate the error sum of squares for the predicted values (Error) are relatively small compared to the error sum of squares for the regression. In summary, the F test indicates there is a significant relationship having a p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

Unusual Observations

Obs.	SPECTRO	TITR%	Fit	Stdev.Fit	Residual	St.Resid
32	8.0	0.22000	0.15730	0.00393	0.06270	2.57R
35	19.7	0.32900	0.39987	0.00892	-0.07087	-3.08R
37	15.8	0.38800	0.31901	0.00674	0.06899	2.91R

R denotes an obs. with a large standard residual.

The regression analysis indicates a strong relationship exists between the results from the standard potentiometric titration procedure and the results obtained from tests conducted with the spectrophotometer. Though the results of this analysis indicates the value for the intercept is not significantly different from 0 at the 5% level of significance, the intercept value obtained from the method of least squares is considered significant enough to be maintained in order to reduce the element of bias in the estimates since the true value for the intercept,  $\beta_0$ , is not known [48] for the predicted value.

A plot of the spectrophotometer and the potentiometric titration results and the 95 percent confidence limits for the prediction interval is presented in Figure 9.

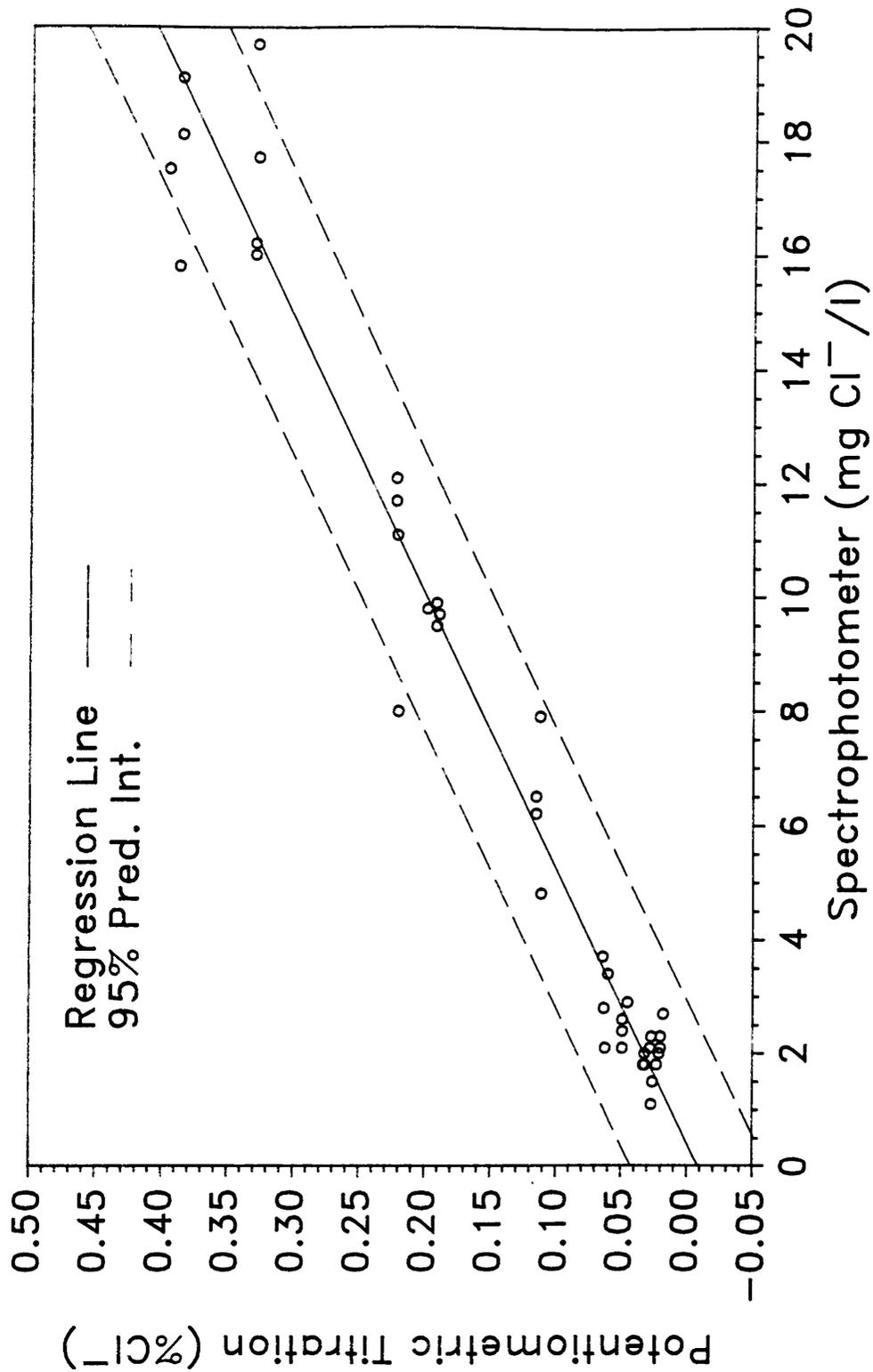


Figure 9. Potentiometric Titration Versus Spectrophotometer

## Digital Titrator

Samples analyzed using the digital titrator as presented in Table 4 were compared to the results obtained from the potentiometric titration procedure. The readings obtained during this series of tests were compared to corresponding potentiometric titration results. Figure 10 presents the results in Table 4 for the potentiometric titration procedure and the digital titrator relative to a line of equivalence.

A regression analysis was performed on the test results presented in Table 4 using Minitab. The results of the analysis procedures are presented below.

The regression equation for the digital titrator is given by:

$$\text{TITR}\% = - 0.0238 + 0.00694 \text{ DIGITAL} \quad (28)$$

where TITR% represents the results from tests using the standard potentiometric titration procedure presented in Table 4

DIGITAL represents the results obtained from the digital titrator also presented in Table 4

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives

were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-0.023797	0.004466	-5.33	0.000
DIGITAL	0.0069401	0.0001481	46.85	0.000

s = 0.01702

R-sq = 98.3%

R-sq(adj) = 98.3%

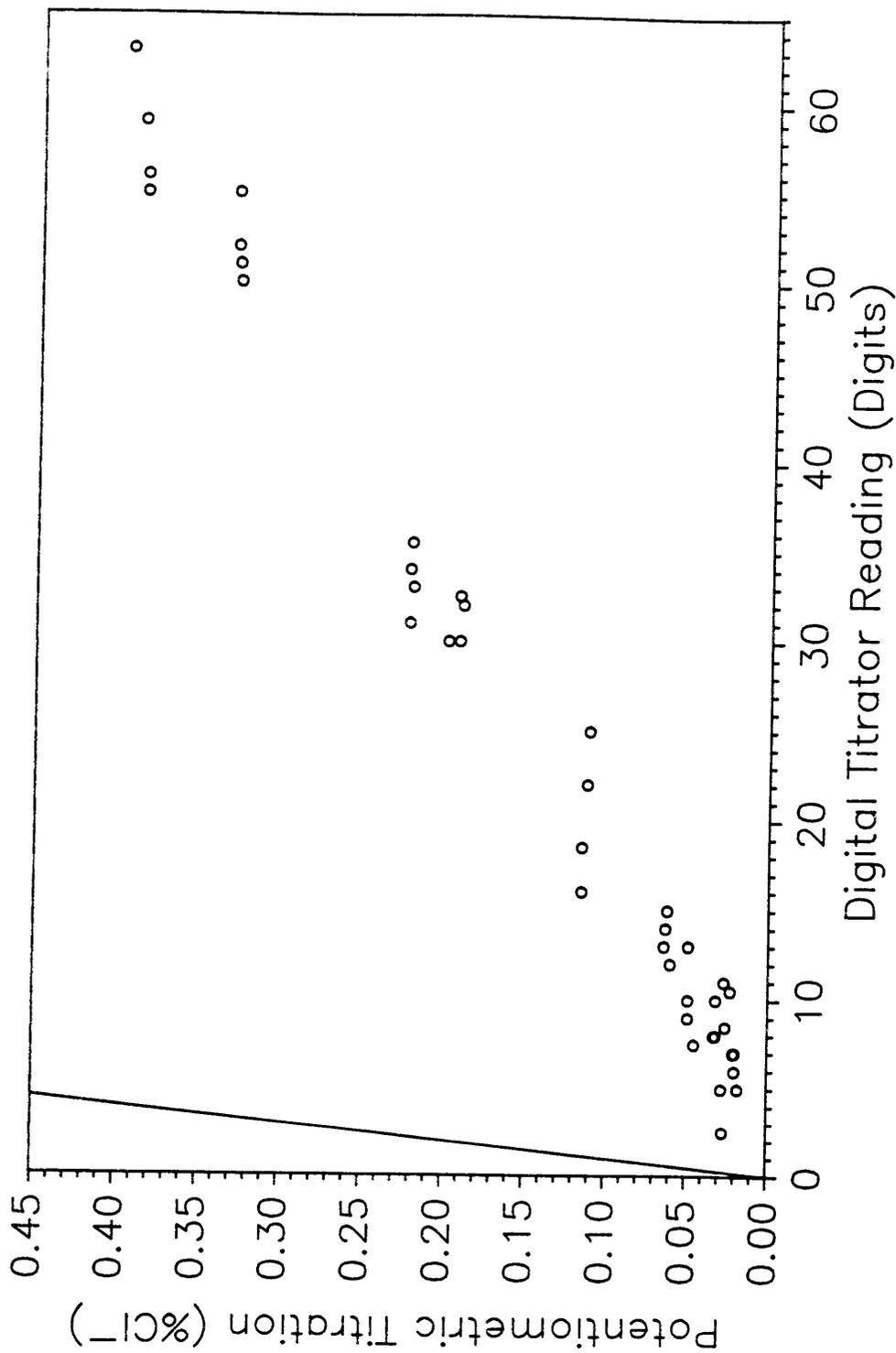


Figure 10. Equivalency Between the Potentiometric Titration and Digital Titrator Test Results

Results of the regression analysis indicate the estimated value for the slope, .00694 is significantly different from 0 at 5% level of significance as indicated by the p value, .000. The value for the intercept, -.0238, is also statistically significant at the 5% level ( $p = .000$ ). Therefore, there is sufficient evidence to reject the null hypothesis for the slope,  $b_1$ , and the intercept,  $b_0$  and conclude there is sufficient evidence to indicate that both values are different from 0.

The coefficient of determination, R-sq, indicates 98.3 % of the variation in TITR% can be predicted from the independent variable, DIGITAL (readings from the digital titrator).

The following are the results of the analysis of variance for the regression analysis:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	0.63562	0.63562	2194.54	0.000
Error	38	0.01101	0.00029		
Total	39	0.64662			

The results of the analysis of variance indicate the error sum of squares for the predict values are relatively small compared to the error sum of squares for the regression. In summary, the F test indicates there is a significant relationship, as indicated by the p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

Unusual Observations

Obs.	DIGITAL	TITR%	Fit	Stdev.Fit	Residual	St.Resid
7	2.5	.0270	-.00645	.00418	.03345	2.03R
21	25.0	.1110	.14971	.00269	-.03871	-2.30R

R denotes an obs. with a large st. resid.

The regression analysis indicates a strong relationship exists between the results from the standard potentiometric titration procedure and the results obtained from tests conducted with the digital titrator. Results of the analysis indicates values for both the slope and intercept are significantly different from 0 at the 5% level of significance.

A plot of the digital titrator as a function of the potentiometric titration results and the associated 95% confidence limits is presented in Figure 11.

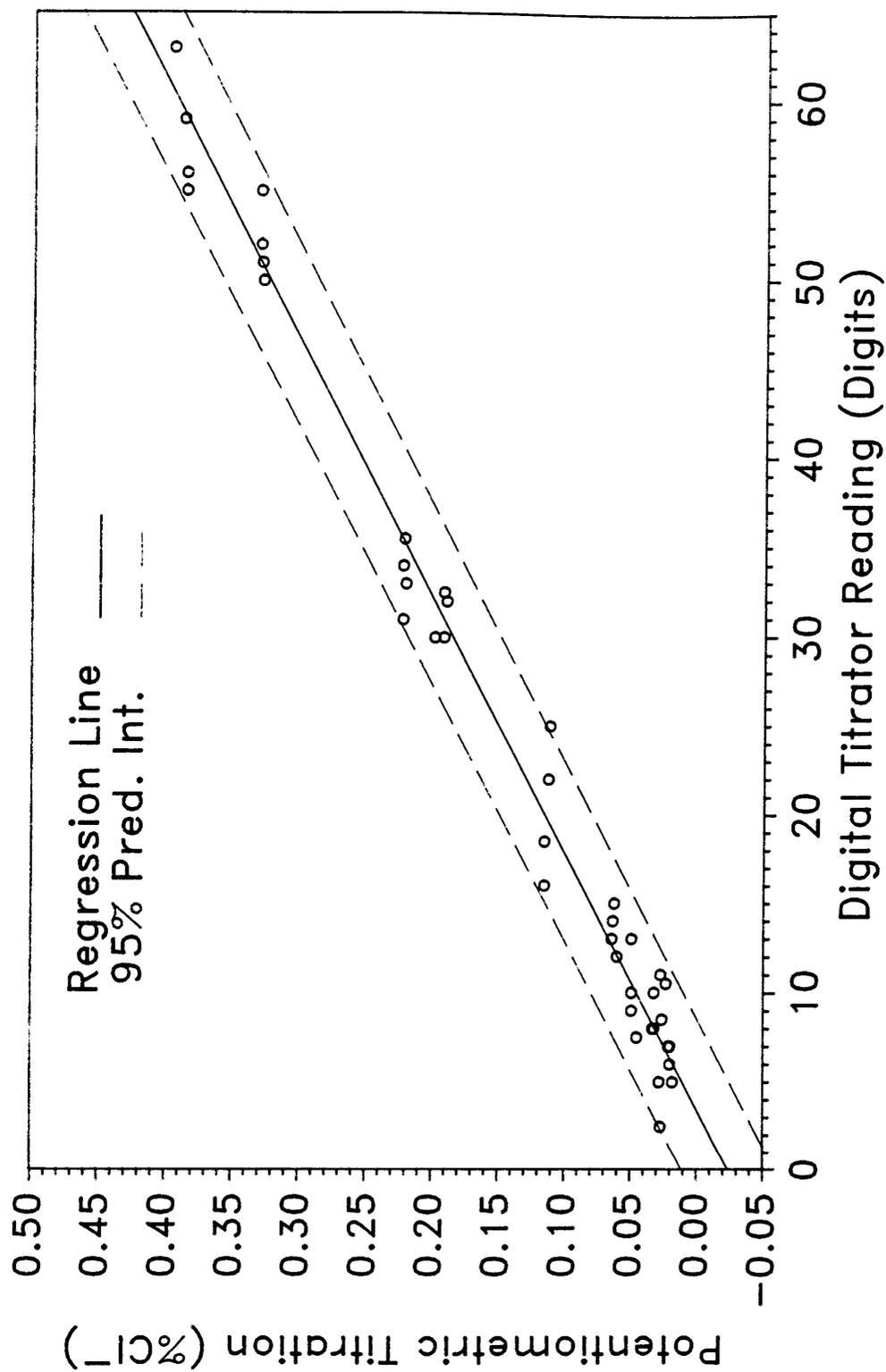


Figure 11. Potentiometric Titration Versus Digital Titrator

## Quantab Titrator Strips

Acid digested samples analyzed using the Quantab titrator strips were compared to the results obtained from the potentiometric titration procedure. In comparing the results, a regression analysis was performed using Minitab. The results of the analysis procedure is presented below.

The regression equation for the Quantab titrator strips is:

$$\text{TITR}\% = 0.00483 + 0.0427 \text{ QTAB}^{1.5} \quad (29)$$

where TITR% represents the results from tests using the standard potentiometric titration procedure presented in Table 6

QTAB represents the corresponding readings obtained for the Quantab titrator strips also presented in Table 6

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives  
were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	0.004828	0.009130	0.53	0.600
QTAB <sup>1.5</sup>	0.042723	0.002117	20.18	0.000

s = 0.03812      R-sq = 91.5%      R-sq(adj) = 91.2%

Results of the regression analysis indicate the estimated value for the slope, .0428 is significantly different from 0 at 5% level of significance ( $p = .000$ ). The value for the intercept, .00483, is not statistically significant at the 5% level ( $p = .600$ ). At this level of significance there is insufficient evidence to reject the null hypothesis that  $b_0 = 0$ .

The coefficient of determination, R-sq, indicates 91.5 % of the variation in TITR% can be predicted from the independent variable, QTAB (results from the Quantab titrator strips)

The following are the results of the analysis of variance for the regression analysis:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	0.59142	0.59142	407.10	0.000
Error	38	0.05521	0.00145		
Total	39	0.64662			

The results of the analysis of variance indicate the error sum of squares for the predicted values (Error) are small compared to the error sum of squares for the regression. In summary, the F test indicates there is a significant relationship having a p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

### Unusual Observations

Obs.	QTAB <sup>1.5</sup>	TITR%	Fit	Stdev.Fit	Residual	St.Resid
35	5.07	.3290	.22130	.00716	.10770	2.88R
36	4.81	.3280	.21039	.00689	.11761	3.14R
38	7.41	.3960	.32130	.01069	.07470	2.04R
40	9.71	.3860	.41948	.01496	-.03348	-.96X

R denotes an observation with a large standard residual.

X denotes an observation whose X value gives it large influence, but not having a large standard residual.

The regression analysis indicates a good relationship exists between the results from the standard potentiometric titration procedure and the results obtained from tests conducted with the Quantab titrator strips. Results of the analysis also indicates the value obtained for the intercept is not significantly different from 0 at the 5% level of significance.

A more appropriate relationship using the least squares method of regression is given by the following equation obtained using the microcomputer version of Minitab. The regression equation is given as:

$$\text{TITR}\% = 0.0436 \text{ QTAB}^{1.5} \quad (30)$$

where TITR% represents the results from tests using the standard potentiometric titration procedure presented in Table 6

QTAB represents the corresponding readings obtained for the Quantab titrator strips also presented in Table 6

Estimates for values of the and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_1 = 0$  : null hypotheses

versus  $H_1: b_1 \neq 0$  : alternative

were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
No Constant				
QTAB^1.5	0.043565	0.001385	31.46	0.000

s = 0.03776

Results of the regression analysis indicate the estimated value for the slope, .0436 is significantly different from 0 at 5% level of significance ( $p = .000$ ).

The following are the results of the analysis of variance for the regression analysis:

#### Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	1.4113	1.4113	989.71	0.000
Error	39	0.0556	0.0014		
Total	40	1.4669			

The results of the analysis of variance indicate the error sum of squares for the predicted values (Error) are small compared to the error sum of squares for the regression. In summary, the F test value (989.71) indicates there is a significant relationship having a p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

### Unusual Observations

Obs.	QTAB <sup>1.5</sup>	TITR%	Fit	Stdev.Fit	Residual	St.Resid
33	8.61	.330	.37498	.01192	-.04498	-1.26 X
35	5.07	.329	.22073	.00702	.10827	2.92R
36	4.81	.328	.20960	.00666	.11840	3.19R
37	8.92	.388	.38845	.01235	-.00045	-0.01 X
38	7.41	.396	.32271	.01026	.07329	2.02R
39	9.07	.386	.39525	.01256	-.00925	-0.26 X
40	9.71	.386	.42282	.01344	-.03682	-1.04 X

R denotes an obs. with a large standard residual.

X denotes an obs. whose X value gives it large influence.

The regression analysis indicates a good relationship exists between the results from the standard potentiometric titration procedure and the results obtained from tests conducted with the Quantab titrator strips.

A plot of the Quantab titrator strips number as a function to the potentiometric titration results and the 95 percent confidence limits for the predicted value is presented in Figure 13.

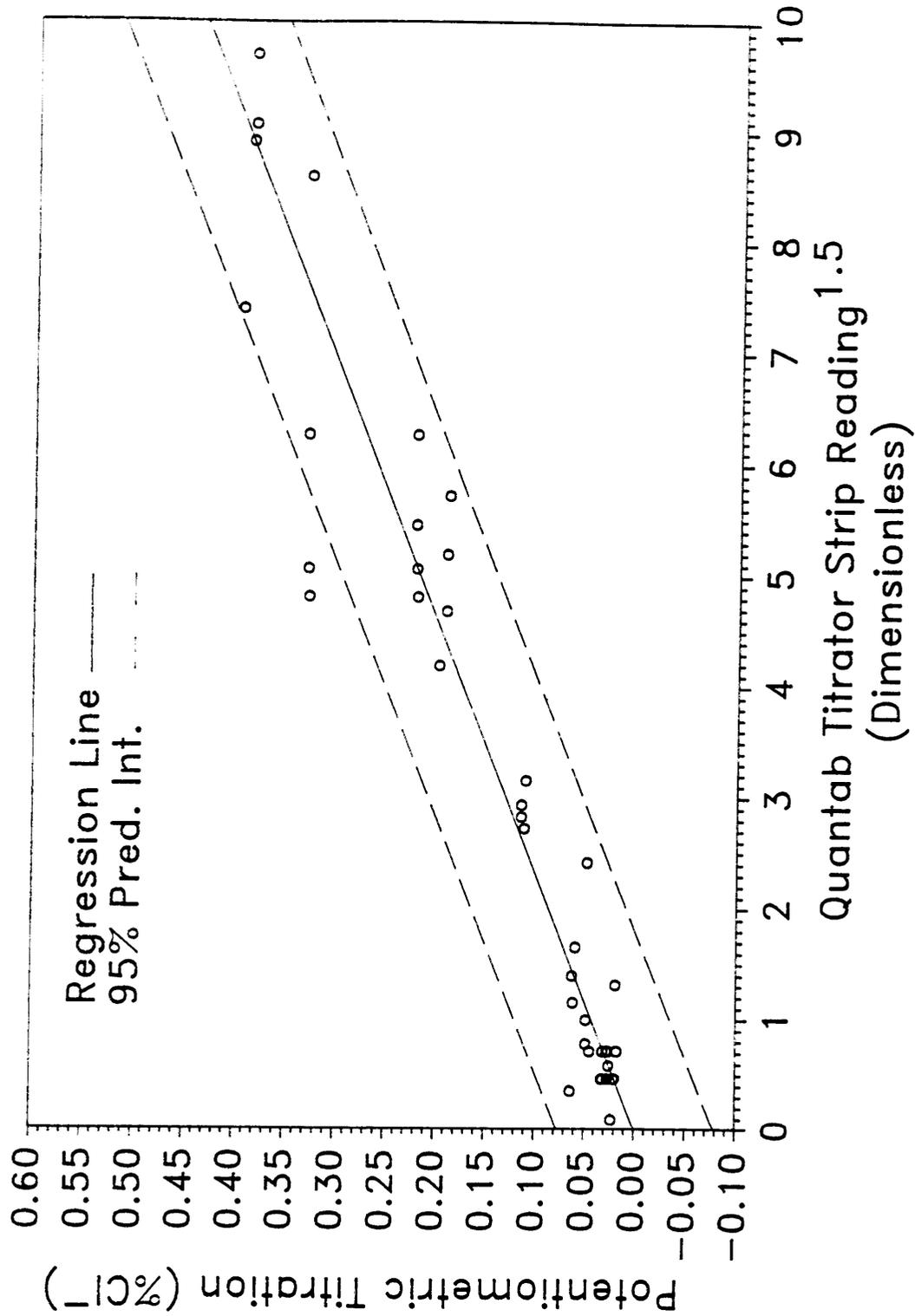


Figure 13. Potentiometric Titration Versus Quantab Titrator Strips

## COMPARISON OF THE SELECTED TEST METHODS

As indicated from an analysis of the results obtained from the first series of tests for chloride, the highest accuracy is achieved using the specific ion probe followed by the digital titrator, spectrophotometer, and the Quantab titrator strips, see Table 14. This is considered the most important criteria for selecting the appropriate method since errors in measuring the chloride content of bridges may influence the results obtained in estimating the extent of chloride contamination and in predicting the time to depassivation.

The initial cost of the specific ion probe was greater than the other selected methods. It should be noted that, hidden costs are associated with the spectrophotometer, digital titrator, and the Quantab titrator strips. Items such as filter paper, beakers, stirring rods, nitric acid, and deionized water are required for all three methods. Additional items such as funnels, a funneling apparatus, and pipets are required for the spectrophotometer and the digital titrator. Finally, a magnetic stirrer and magnetic stirring rods are desirable when using the digital titrator.

No special sample preparation is required for the specific ion probe. The probe can be calibrated in less than 10 minutes, following which samples can be weighed and analyzed in less than 10 minutes. This allows the operator seven minutes to weigh the sample prior to testing.

Table 14. Comparison of Test Methods

<u>Method</u>	Accuracy <u>R-sq (Adj)</u>	<u>*Cost</u>	<u>**Speed</u>	<u>***Level of Expertise</u>
Specific Ion Probe	99.0%	\$1860.90	1	Simple
Spectrophotometer	96.3%	\$1363.57	2	Medium
Digital Titrator	98.3%	\$ 156.74	3	Medium
Quantab Titrator Strips	91.2%	\$ 49.30	4	Simple

\*Cost represents the initial cost of the equipment and the chemicals necessary for analyzing 40 specimens.

\*\*Speed represents the order in which samples may be weighed, prepared, and tested using each method.

\*\*\*Level of expertise gives an indication of how complicated each procedure is to use.

The procedure for weighing and preparing the sample for the spectrophotometer and the digital titrator requires the same amount of time. Once the samples are prepared the analysis procedure using the spectrophotometer requires a little less time than the digital titrator. The operator must pay particular attention during the titrating procedure when using the digital titrator so the midpoint of the titration will be recognized.

The procedure for weighing and preparing the sample to be analyzed using the Quantab titrator strips does not require as much time as for the spectrophotometer and the digital titrator. However, sometimes the operator may be required to wait as much as 20 minutes before the results may be read using the Quantab titrator strips.

The analysis procedure for the specific ion probe was simplified for the operator when compared to the original procedure developed by James Instruments [39]. Essentially the operator will record readings displayed by the electrometer while following a step by step procedure. Two precautions must be followed in using this procedure to obtain accurate results. Otherwise, the procedure is simple, quick, and straightforward. Procedures for the spectrophotometer and the digital titrator are a little more complicated than for the specific ion probe. The operator not only needs to realize the importance of following the procedure completely, but also must be able to detect

interferences which may occur and compensate for them. The analysis procedure for using the Quantab titrator strip is a little more complicated than the procedure for the specific ion probe, but requires little technical expertise.

Considering each of the criteria, summarized in Table 14, for selecting the method for determining the chloride content of concrete, the specific ion probe was determined to be more suitable for the purpose intended than any of the remaining methods. First of all, the apparatus required for the spectrophotometer and the digital titrator is more suited for use in the laboratory than in the field. Secondly, both of these procedures require a certain level of expertise to achieve meaningful results. Finally, the Quantab titrator strips do not provide the accuracy that is desirable for determining the time to depassivation and, at times, may require an excessive amount of time for analysis.

#### ANALYSIS OF RESULTS FOR THE EFFECTS OF CEMENT CONTENT

The millivolt readings for the specific ion probe, presented in Table 7, were converted to percent of chloride using the procedure presented in the first series of tests for chloride content determination. Once the values for %  $\text{Cl}^-$  as measured with the specific ion probe were determined, the results presented in Table 7 were compared with the results from the first series of tests (Table 2).

An initial comparison of the results encompassed using the regression equation for the specific ion probe from the

first series of tests to convert the data in Table 7 to predicted values for %  $\text{Cl}^-$  obtained with the standard potentiometric titration procedure. Figure 14 presents the graphical results of this comparison. From Figure 14, it appears there is no significant effect of cement content on the performance of the specific ion probe. As illustrated in Figure 14, specific trends in the results obtained for specimens having water to cement ratios of .40 and .50, when compared to the results from the first series of tests, do not exist. In addition, the results presented in Table 2 and Table 7 indicated there was no correlation between the results obtained for chloride content using the specific ion probe and cement content.

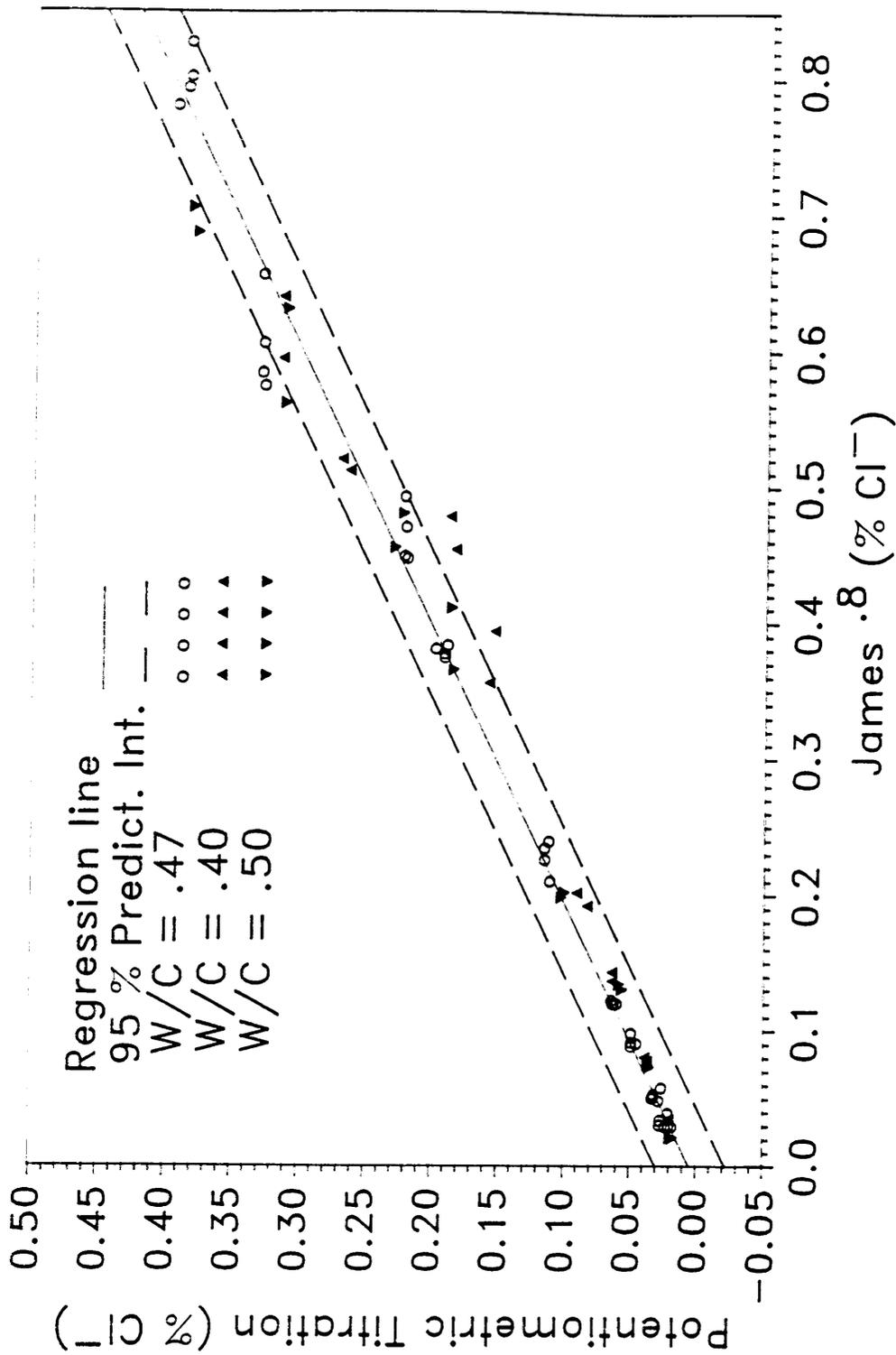


Figure 14. Potentiometric Titration Versus the Specific Ion Probe for Different Cement Contents

A regression analysis using Minitab was performed to determine the relationship between the results from the standard potentiometric titration procedure and the specific ion probe presented in Tables 2 and 7. Weak correlations between cement content and potentiometric titration results were noted during the analysis confirming earlier suspicions that the results from the specific ion probe were not significantly affected by differences in cement content.

The regression equation using the specific ion probe for the data presented in Tables 2 and 7 is given as:

$$\%TITRAT = 0.00047 + 0.499 \%JAMES^{.8} \quad (31)$$

where TITRAT% represents the results from tests using the standard potentiometric titration procedure presented in Tables 2 and 7

%JAMES represents the corresponding results for % Cl<sup>-</sup> obtained using the specific ion probe presented in Tables 2 and 7

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
 versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives  
 were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	0.000466	0.003010	0.15	0.878
%JAMES <sup>.8</sup>	0.499449	0.007888	63.32	0.000

s = 0.01620      R-sq = 98.3%      R-sq(adj) = 98.3%

Results of the regression analysis indicate the estimated value for the slope, .499 is significantly different from 0 at 5% level of significance ( $p = .000$ ). The value for the intercept, .000466, is not statistically significant at the 5% level ( $p = .878$ ). At this level of significance there is insufficient evidence to reject the null hypothesis that  $b_0 = 0$ .

The coefficient of determination, R-sq, indicates 98.3 % of the variation in TITRAT% can be predicted from the independent variable, %JAMES (results from the specific ion probe)

The following are the results of the analysis of variance for the regression analysis:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	1.0522	1.0522	4008.79	0.000
Error	70	0.0184	0.0003		
Total	71	1.0706			

The results of the analysis of variance indicate the error sum of squares for the predicted values (Error) are relatively small compared to the error sum of squares for the regression. In summary, the F test indicates there is a significant relationship having a p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

### Unusual Observations

Obs	%.JAME <sup>.8</sup>	%TITRAT	Fit	Stdev.Fit	Residual	St.Resid
33	0.584	0.33000	0.29203	0.00297	0.03797	2.38R
36	0.574	0.32800	0.28723	0.00291	0.04077	2.56R
50	0.394	0.15400	0.19717	0.00206	-0.04317	-2.69R
51	0.454	0.18400	0.22738	0.00229	-0.04338	-2.70R
52	0.479	0.18800	0.23947	0.00240	-0.05147	-3.21R
69	0.561	0.31300	0.28088	0.00284	0.03212	2.01R
71	0.687	0.38000	0.34339	0.00363	0.03661	2.32R

R denotes an obs. with a large st. resid.

The regression analysis indicates a good relationship exists between the results from the standard potentiometric titration procedure and the results obtained from tests to determine the effects of cement content using the specific ion probe. Results of the analysis also indicates the value obtained for the intercept is not significantly different from 0 at the 5% level of significance ( $p = .878$ ).

A more appropriate relationship using the least squares method of regression is given by the following equation obtained using the microcomputer version of Minitab. The regression equation is given as:

The regression equation is

$$\%TITRAT = 0.500 \%JAMES^{.8} \quad (32)$$

where TITRAT% represents the results from tests using the standard potentiometric titration procedure presented in Tables 2 and 7

%JAMES represents the corresponding results for % Cl<sup>-</sup> obtained using the specific ion probe presented in Tables 2 and 7

An estimate for value of the slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_1 = 0$  : null hypotheses

versus  $H_1: b_1 \neq 0$  : alternatives

was presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
No Constant				
%JAMES <sup>^8</sup>	0.500392	0.004969	100.70	0.000

s = 0.01609

Results of the regression analysis indicate the estimated value for the slope, .500 is significantly different from 0 at 5% level of significance ( $p = .000$ ). The value for the intercept was previously determined not statistically significant at the 5% level.

The coefficient of determination, R-sq, is irrelevant in the regression analysis since the value for the intercept was previously determined to be equal to zero.

The following are the results of the analysis of variance for the regression analysis:

#### Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	2.6249	2.6249	10139.63	0.000
Error	71	0.0184	0.0003		
Total	72	2.6433			

The results of the analysis of variance indicate the error sum of squares for the predicted values (Error) are very small compared to the error sum of squares for the

regression. In summary, the F test indicates there is a significant relationship having a p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

#### Unusual Observations

Obs	%.JAME <sup>.8</sup>	%TITRAT	Fit	Stdev.Fit	Residual	St.Resid
33	.584	.33000	.29211	.00290	.03789	2.39R
36	.574	.32800	.28731	.00285	.04069	2.57R
37	.793	.38800	.39692	.00394	-.00892	-0.57 X
38	.780	.39600	.39014	.00387	.00586	0.38 X
39	.801	.38600	.40104	.00398	-.01504	-0.96 X
40	.827	.38600	.41367	.00411	-.02767	-1.78 X
50	.394	.15400	.19708	.00196	-.04308	-2.70R
51	.454	.18400	.22734	.00226	-.04334	-2.72R
52	.479	.18800	.23945	.00238	-.05145	-3.23R
69	.561	.31300	.28094	.00279	.03206	2.02R
71	.687	.38000	.34357	.00341	.03643	2.32RX
72	.705	.38400	.35277	.00350	.03123	1.99 X

R denotes an obs. with a large st. resid.

X denotes an obs. whose X value gives it large influence.

The results indicate a strong relationship between the results obtained using the specific probe and the standard potentiometric titration procedure as evidenced by the F value of 10139.63.

Figure 15 presents a graphical presentation of the results from this analysis indicating a good relationship in predicting the results obtained with the potentiometric titration procedure from the results of the tests conducted using the specific ion probe.

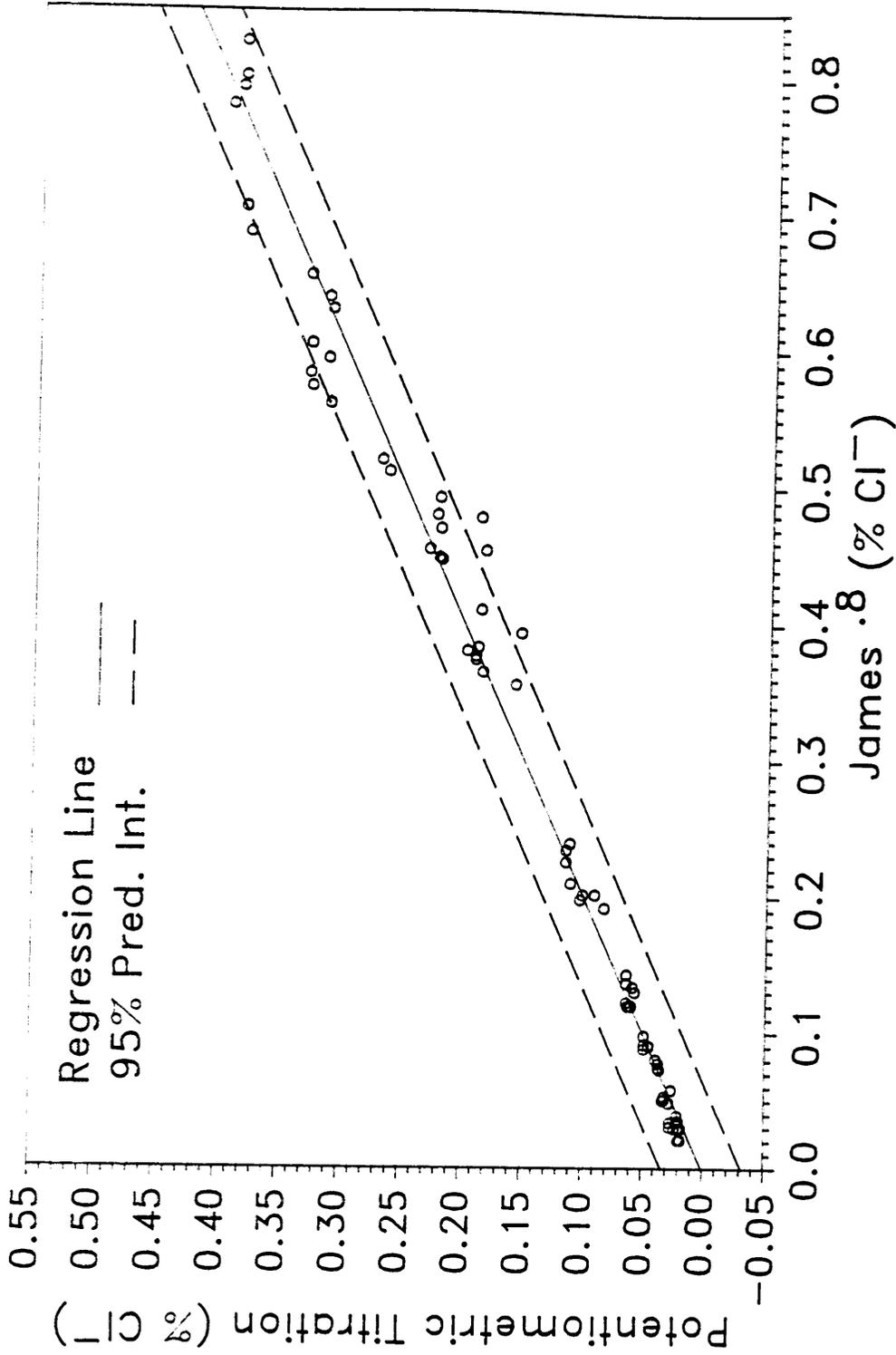


Figure 15. Potentiometric Titration Versus Specific Ion Probe for Effects of Cement Content

ANALYSIS OF RESULTS FOR THE EFFECTS OF  
REACTION TEMPERATURE

The millivolt readings for the specific ion probe, presented in Table 8, were converted to percent of chloride using the procedure presented in the first series of tests for chloride content determination. Once the values for % Cl<sup>-</sup> as measured with the specific ion probe were determined, the results presented in Table 8 were compared with the results for specimen C-4 from the first series of tests (Table 2) to determine the effects of temperature.

A regression analysis using Minitab was performed to determine the relationship between changes in the reaction temperature and the results from the specific ion probe.

The regression equation for the effects of temperature using the specific ion probe is given as:

$$\text{ACTUAL-\%JAMES} = - 0.103 + 0.0119 \text{ REACTTEMP}^{.5} \quad (33)$$

where ACTUAL-%JAMES represents the difference in the results from tests using the specific ion probe during the first series of tests as presented in Table 2 and the corresponding results presented in Table 8

REACTTEMP represents the various temperatures of the reaction resulting from the test procedure as presented in Table 8

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$

and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives  
were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-0.102990	0.008087	-12.74	0.000
REACTTEMP <sup>.5</sup>	0.0118683	0.0009076	13.08	0.000

s = 0.008288      R-sq = 84.7%      R-sq(adj) = 84.2%

Results of the regression analysis indicate the estimated value for the slope, .0119 is significantly different from 0 at 5% level of significance ( $p = .000$ ). The value for the intercept, -.103, is also statistically significant at the 5% level ( $p = .000$ ). At the 5% level of significance there is sufficient evidence to reject the null hypothesis that  $b_0 = 0$  and  $b_1 = 0$ .

The coefficient of determination, R-sq, indicates 84.7 % of the variation in the difference in the results from the first series of tests and the results presented in Table 8 can be predicted from the independent variable, REACTTEMP (the reaction temperature for the specific ion probe)

The following are the results of the analysis of variance for the regression analysis:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	0.011746	0.011746	171.01	0.000
Error	31	0.002129	0.000069		
Total	32	0.013876			

The results of the analysis of variance indicate the error sum of squares for the predicted values (Error) are small compared to the error sum of squares for the regression. In summary, the F test indicates there is a significant relationship having a p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

Unusual Observations

Obs.	RCTTMP <sup>.5</sup>	ACTL-%JAMS	Fit	Stdev.Fit	Resid	St.Resid
7	7.6	-.03006	-.01260	.00178	-.01745	-2.16R

R denotes an obs. with a large st. resid.

The results of the regression analysis indicates the relationship determined for the effects of temperature for the specific ion probe. Though not strong when compared to results from previous analyses, the relationship does exist and among other parameters, is evidenced by the F value of 171.01 (p = .000).

Figure 16 illustrates the relationship between the correction factor for the specific ion probe suggested by the results of the regression analysis and the reaction temperature.

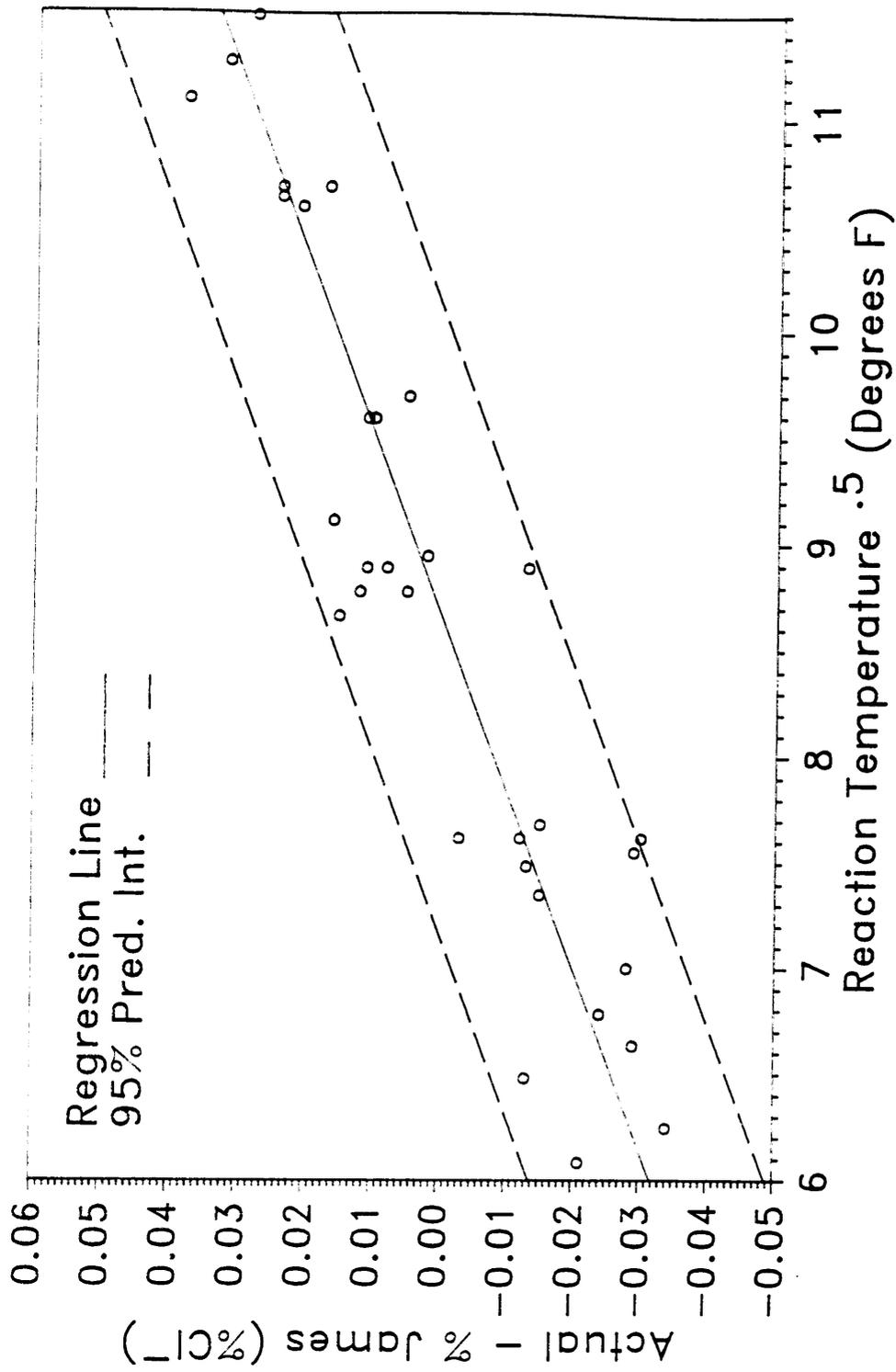


Figure 16. Temperature Correction for the Specific Ion Probe

## ANALYSIS OF FIELD VALIDATION TEST RESULTS

The results presented in Table 9 were analyzed to determine the relationships between the results for samples that were crushed versus the results obtained prior to crushing the specimens. Also, relationships between the results from the potentiometric titration procedure versus crushed sample results and results from samples that were not crushed were investigated using the regression analysis procedure. The results of the analysis procedure are presented in the appropriate sections that follow.

### Crushed and Non-Crushed Samples

The regression analysis equation for the relationship between samples which were crushed and not crushed is presented as:

$$1/\text{CRUSH} = - 0.723 + 1.96 \ 1/\text{NOTCR}^{.75} \quad (34)$$

where CRUSH represents the results of the analysis for %  $\text{Cl}^-$  using the specific ion probe on samples presented in Table 9 which have been crushed to pass a No. 50 sieve

and NOTCR represents the results of the analysis for %  $\text{Cl}^-$  using the specific ion probe on samples presented in Table 9 which have not been crushed

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives

were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-0.7228	.3704	-1.95	0.055
1/NOTCR <sup>.75</sup>	1.95730	.03407	57.45	0.000

s = 2.307      R-sq = 97.7%      R-sq(adj) = 97.7%

Results of the regression analysis indicate the estimated value for the slope, 1.957 is significantly different from 0 at 5% level of significance as indicated by the p value, .000. Therefore, there is sufficient evidence to reject the null hypothesis for the slope at the 5% level of significance.

The value for the intercept, -.723, is though not statistically significant at the 5% level ( $p = .055$ ) is still considered significant and of practical importance in the analysis procedure [46]. Accepting the null hypothesis at this level of significance could also result in a bias of the estimates since the true value for the intercept,  $\beta_0$ , is not known [48].

The coefficient of determination, R-sq, indicates 97.7 % of the variation in dependent variable, the inverse of the results for the samples that were crushed, can be predicted from the independent variable, representing the results obtained from samples prior to crushing.

The following are the results of the analysis of variance for the regression analysis:

### Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	17572	17572	3300.32	0.000
Error	78	415	5		
Total	79	17987			

The results of the analysis of variance indicate the error sum of squares for the predict values are very small, in relation to the error sum of squares for the regression. In summary, the F test result, 3300.32 indicates there is a significant relationship, as indicated by the p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

#### Unusual Observations

Obs.	$1/\text{NOTCR}^{.75}$	$1/\text{CRUSH}$	Fit	Stdev.Fit	Resid.	St.Resid
20	26.0	52.63	50.12	.671	2.515	1.14 X
44	26.0	47.62	50.12	.671	-2.497	-1.13 X
45	7.1	18.18	13.21	.259	4.968	2.17R
47	18.1	40.00	34.76	.436	5.242	2.31R
53	22.2	33.33	42.79	.555	-9.452	-4.22R
57	24.6	41.67	47.37	.627	-5.701	-2.57R
64	18.1	43.48	34.76	.436	8.720	3.85R

R denotes an obs. with a large st. resid.

X denotes an obs. whose X value gives it large influence.

The regression analysis indicates a strong relationship exists between the results for the samples that were tested with the specific ion probe prior to crushing and the results obtained after crushing the samples to pass a No. 50 sieve. Though the results of the analysis indicates the value for the intercept is not significantly different from

0 at the 5% level of significance, the intercept value obtained from the method of least squares is maintained to reduce the element of bias in the estimates [48] for the predicted value or the predicted results.

A plot of the results obtained using the specific ion probe illustrating the relationship between crushed and uncrushed samples and the 95 percent confidence limits for the prediction interval is presented in Figure 17.

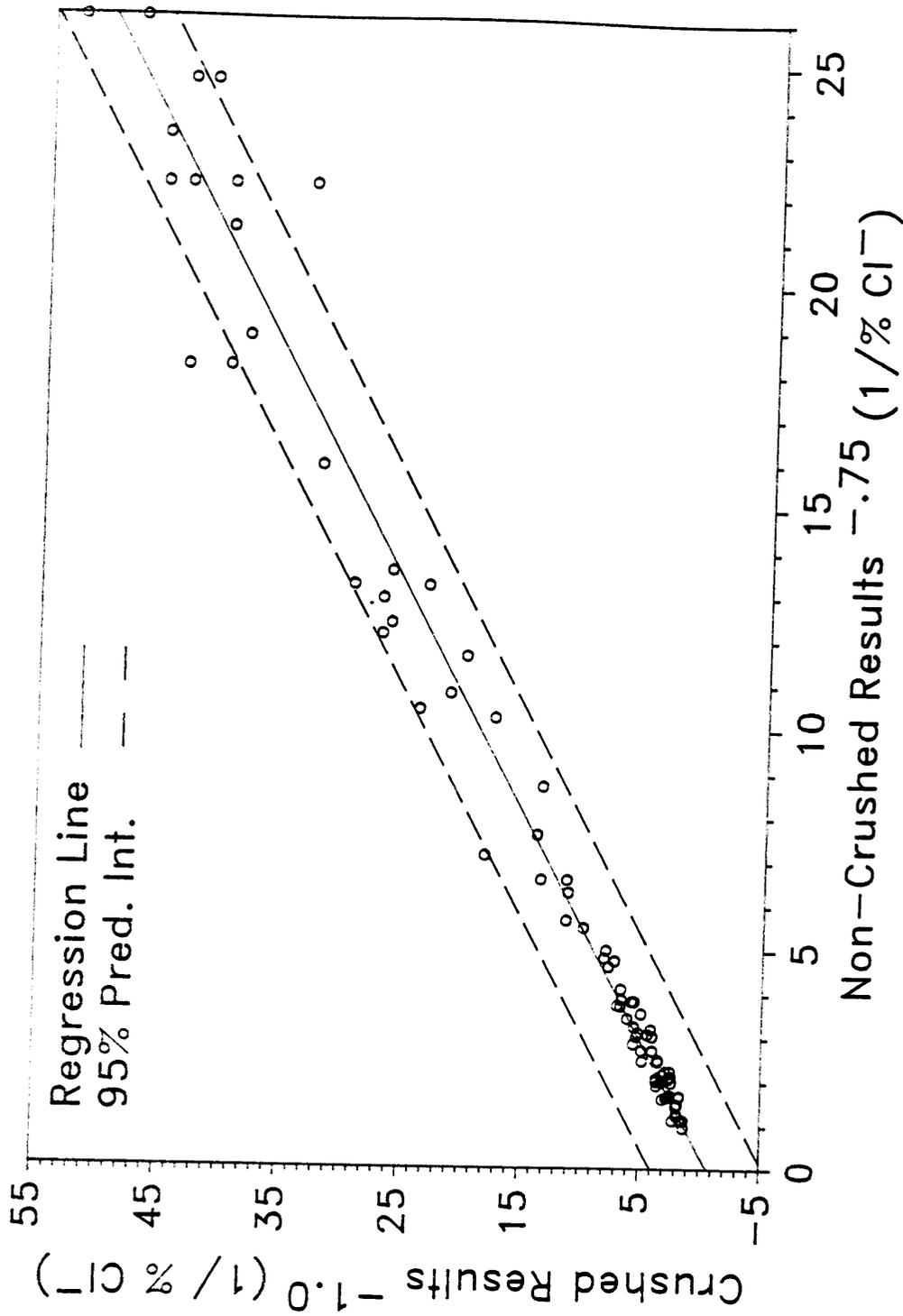


Figure 17. Specific Ion Probe: Crushed Versus Non-crushed Sample Results

## Potentiometric Titration and Crushed Samples

The relationship between the results of the standard potentiometric titration procedure and the specific ion probe for the crushed samples is given in the regression equation as

$$1/\%TITR = - 4.71 + 3.38 1/CRUSH^{.8} \quad (35)$$

%TITR represents the values for % CL<sup>-</sup> obtained from the potentiometric titration procedure

CRUSH represents the readings obtained using the specific ion probe which have been transformed using the procedure presented during the first series of tests

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses

versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives

were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-4.7091	0.8307	-5.67	0.000
1/CRUSH <sup>.8</sup>	3.37824	0.08029	42.08	0.000

s = 4.838

R-sq = 95.8%

R-sq(adj) = 95.7%

Results of the regression analysis indicate the estimated value for the slope, 3.378 is significantly different from 0 at 5% level of significance ( $p = 000$ ). The value for the intercept, -4.709, is also statistically

significant at the 5% level ( $p = .000$ ). Therefore, there is sufficient evidence to reject the null hypotheses for  $b_0$  and  $b_1$  at the 5% level of significance and conclude the values for the slope and intercept are different from 0.

The coefficient of determination, R-sq, indicates 95.8 % of the variation in the inverse of %TITR, the dependent variable can be predicted from the independent variable,  $1/CRUSH^{.8}$ .

The following are results of the analysis of variance for the regression analysis:

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	41433	41433	1770.38	0.000
Error	78	1825	23		
Total	79	43259			

The results of the analysis of variance indicate the error sum of squares for the predicted values are small in relation to the error sum of squares for the regression. In summary, the F test value, 1770.38, indicates there is a significant relationship in the regression analysis, as indicated by the p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

### Unusual Observations

Obs.1/cru <sup>.8</sup>	1/%TITR	Fit	Stdev.Fit	Residual	St.Resid	
19	21.2	76.923	66.864	1.199	10.059	2.15R
20	23.8	90.909	75.771	1.392	15.138	3.27RX
44	22.0	55.556	69.578	1.257	-14.023	-3.00R
56	19.1	76.923	59.907	1.055	17.017	3.60R
64	20.4	50.000	64.364	1.147	-14.364	-3.06R
77	16.1	40.000	49.691	0.855	-9.691	-2.04R

R denotes an obs. with a large st. resid.

X denotes an obs. whose X value gives it large influence.

The regression analysis indicates a good relationship exists between the inverse of results from the standard potentiometric titration procedure and the results obtained from tests conducted with the specific ion probe.

A plot of the results obtained using the specific ion probe as a function of the inverse of the potentiometric titration results and the 95 percent confidence limits for the prediction interval is presented in Figure 18.

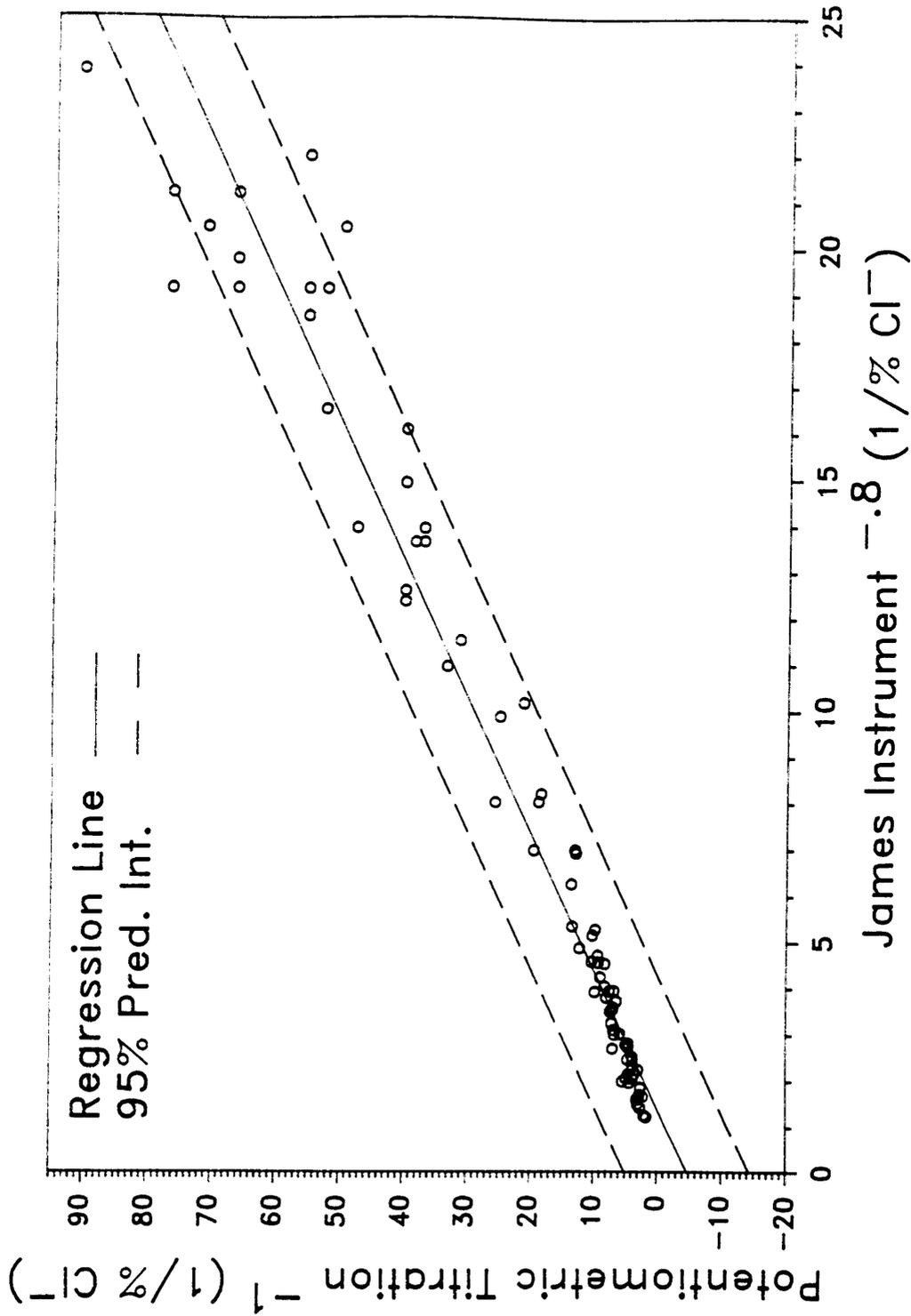


Figure 18. Potentiometric Titration Versus Specific Ion Probe for Crushed Samples

## Potentiometric Titration and Uncrushed Samples

The regression equation for the relationship between the results from the potentiometric titration procedure and the specific ion probe for samples that were not crushed is given as:

$$\%TITR = - 0.00895 + 0.513 \text{ NOTCR}^{.8} \quad (36)$$

where,

$\%TITR$  represents the values for  $\% \text{ CL}^-$  obtained from the potentiometric titration procedure

$\text{NOTCR}$  represents the results obtained using the specific ion probe for samples which have not been crushed

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives  
were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-0.008948	0.004806	-1.86	0.066
$\text{NOTCR}^{.8}$	0.51268	0.01272	40.31	0.000

$s = 0.02786$        $R\text{-sq} = 95.4\%$        $R\text{-sq(adj)} = 95.4\%$

Results of the regression analysis indicate the estimated value for the slope, .513 is significantly different from 0 at 5% level of significance ( $p = 000$ ). The value for the intercept, -.00895, though not statistically

significant at the 5% level ( $p = .066$ ) is still considered significant and of practical importance in the analysis procedure [46]. Accepting the null hypothesis at this level of significance could result in a bias of the estimates since the true value for the intercept,  $\beta_0$ , is not known [48].

The coefficient of determination, R-sq, indicates 95.4 % of the variation in %TITR can be predicted from the independent variable, NOTCR<sup>.8</sup> ( millivolt readings converted to percent chloride, NOTCR)

The following are results of the analysis of variance for the regression analysis:

#### Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	1.2609	1.2609	1624.93	0.000
Error	78	0.0605	0.0008		
Total	79	1.3215			

The results of the analysis of variance indicate the error sum of squares for the predicted values are small in relation to the error sum of squares for the regression. In summary, the F test value, 1624.93, indicates there is a significant relationship, as indicated by the p value of .000.

The following is a list of the unusual observations having the most influence in the regression analysis.

### Unusual Observations

Obs.	NOTCR <sup>.8</sup>	%TITRcl	Fit	Stdev.Fit	Residual	St.Resid
2	.88	.50300	.44080	.00812	.06220	2.33RX
4	.56	.20600	.27889	.00467	-.07289	-2.65R
21	.77	.45300	.38393	.00684	.06907	2.56R
25	.42	.26500	.20785	.00356	.05715	2.07R
27	.80	.34400	.40137	.00722	-.05737	-2.13R
37	1.04	.60500	.52454	.01007	.08046	3.10RX
65	.88	.38900	.44123	.00813	-.05223	-1.96 X
68	.88	.38000	.44334	.00818	-.06334	-2.38RX

R denotes an obs. with a large st. resid.

X denotes an obs. whose X value gives it large influence.

The regression analysis indicates a strong relationship exists between the results from the standard potentiometric titration procedure and the results obtained from tests conducted with the specific ion probe on samples that were not crushed. Though the results of the analysis indicates the value for the intercept is not significantly different from 0 at the 5% level of significance, the intercept value obtained from the method of least squares is maintained to reduce the element of bias in the estimates [48] for the predicted value or the predicted results from the standard potentiometric titration procedure.

A plot of the results obtained for the uncrushed samples using the specific ion probe as a function of the potentiometric titration results and the 95 percent confidence limits for the prediction interval is presented in Figure 19.

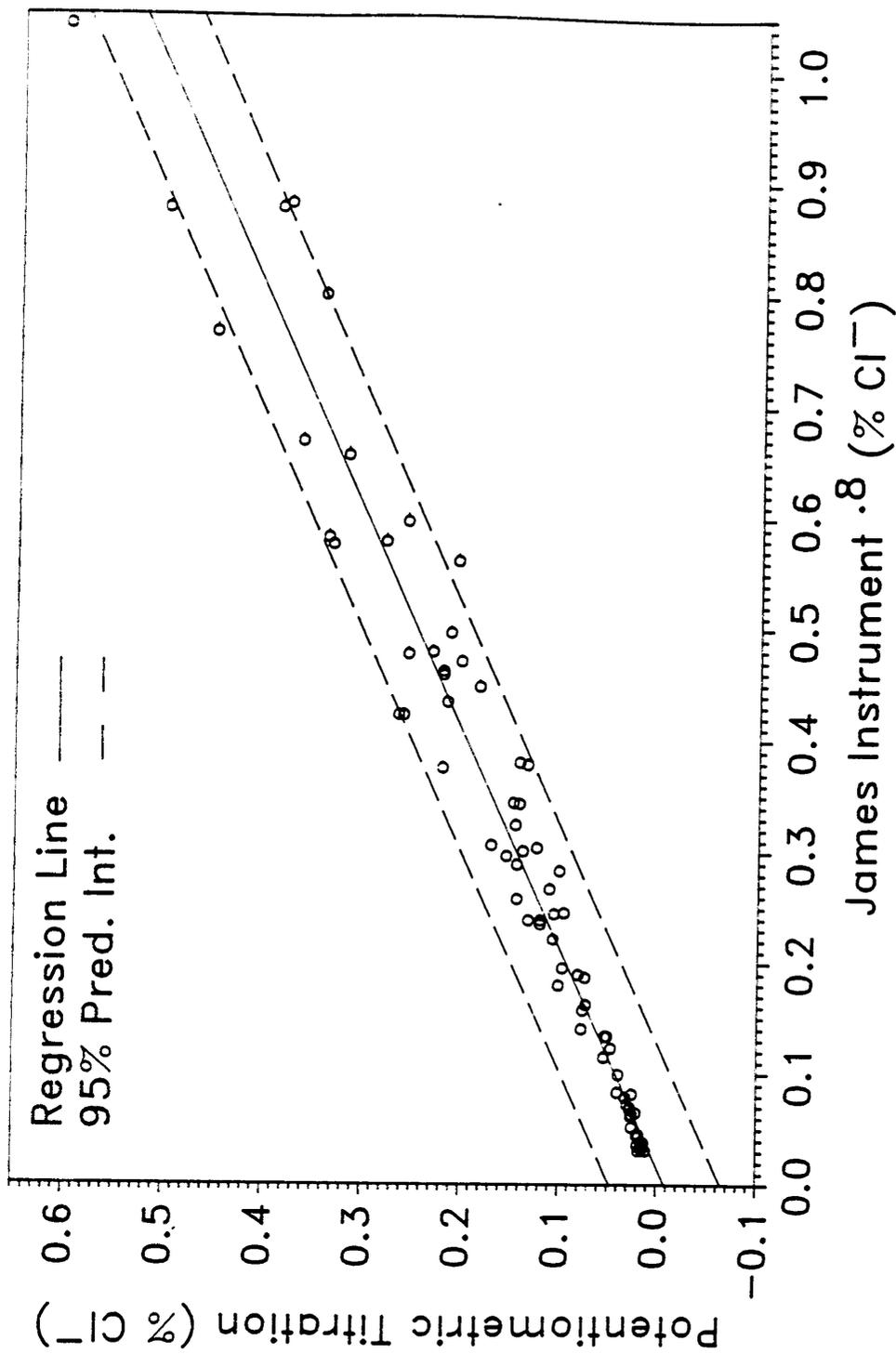


Figure 19. Potentiometric Titration Versus Specific Ion Probe for Uncrushed Samples

## ANALYSIS OF THE EFFECTIVE DIFFUSION CONSTANT

The analysis of the effective diffusion constants presented in Table 10 was essentially two fold. First, the results were analyzed to identify the effects of temperature and water to cement ratios on the diffusion constant. In theory, the effective diffusion constant is a function of temperature and water to cement ratio [10].

Secondly, an analysis was done to determine the effects of time on the values for the effective diffusion constant for a particular bridge component.

### Effects of Temperature

In examining the effects of temperature on the effective diffusion constant, the data presented in Table 11 for the substructure components in Florida and the deck components of Pennsylvania, Virginia, and Wisconsin were analyzed.

No relationship between temperature and the mean values for the effective diffusion constant were identified. The analysis indicated temperature and water to cement ratios for the different regions were highly correlated which prevented a multilinear regression analysis using both variables. For cement paste, Page et. al. [52] reported relationships between temperature and the effective diffusion constant existed for individual water to cement ratios.

### Effects of Water to Cement Ratios

Figure 20 illustrates the relationship between the mean values for the effective diffusion constant and the water to cement ratios of each bridge component for bridges from the four states.

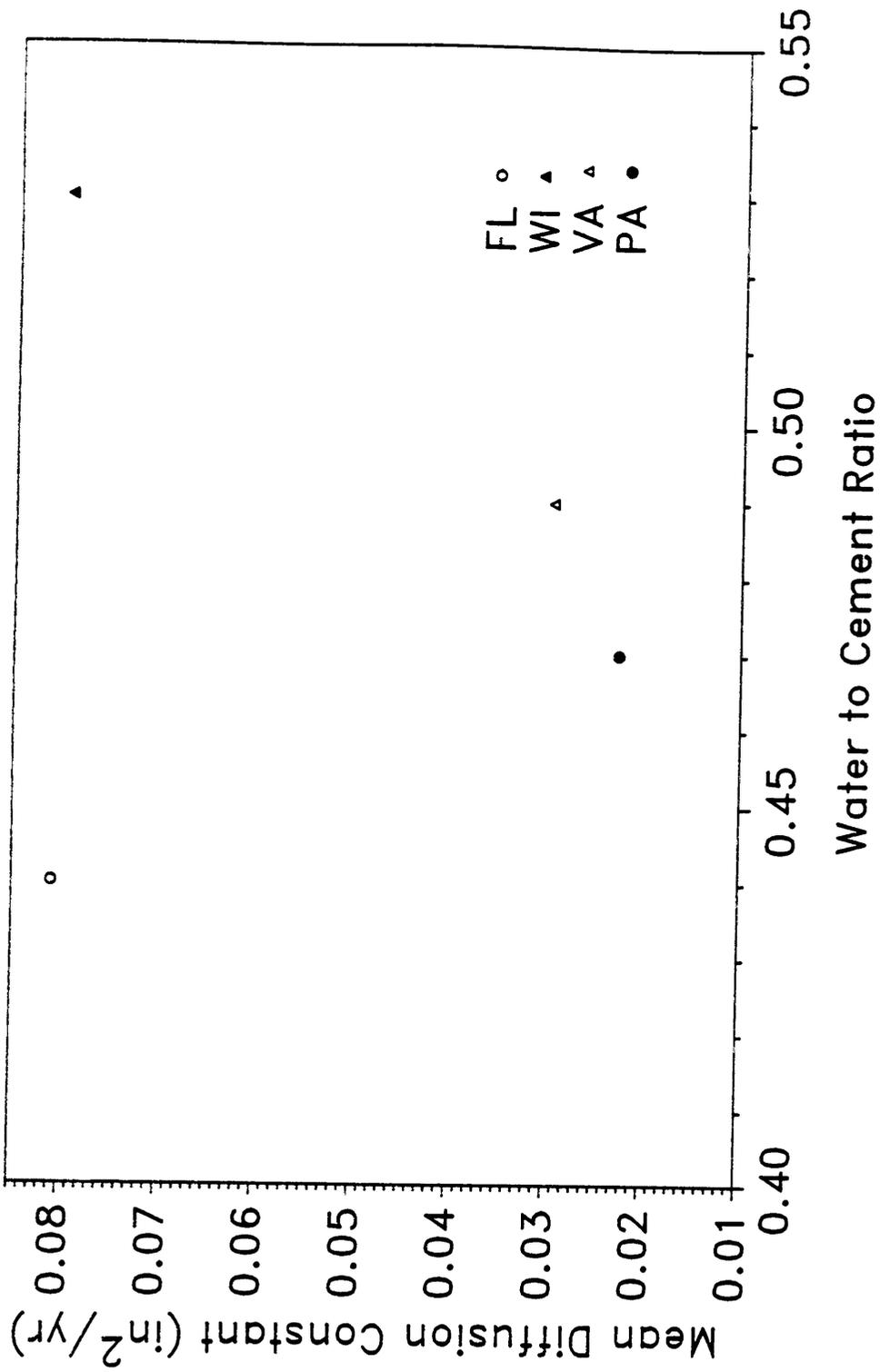


Figure 20. Mean Diffusion Constants Versus Water to Cement Ratios

As indicated from the data as presented in Figure 20, an irregularity in the results exists between the mean values for the effective diffusion constants and the corresponding water to cement ratios. A second plot excluded the data from the state of Florida. Figure 21 presents a plot of the mean value of the effective diffusion constant for the components versus the water to cement ratio of the component excluding the data obtained from the state of Florida.

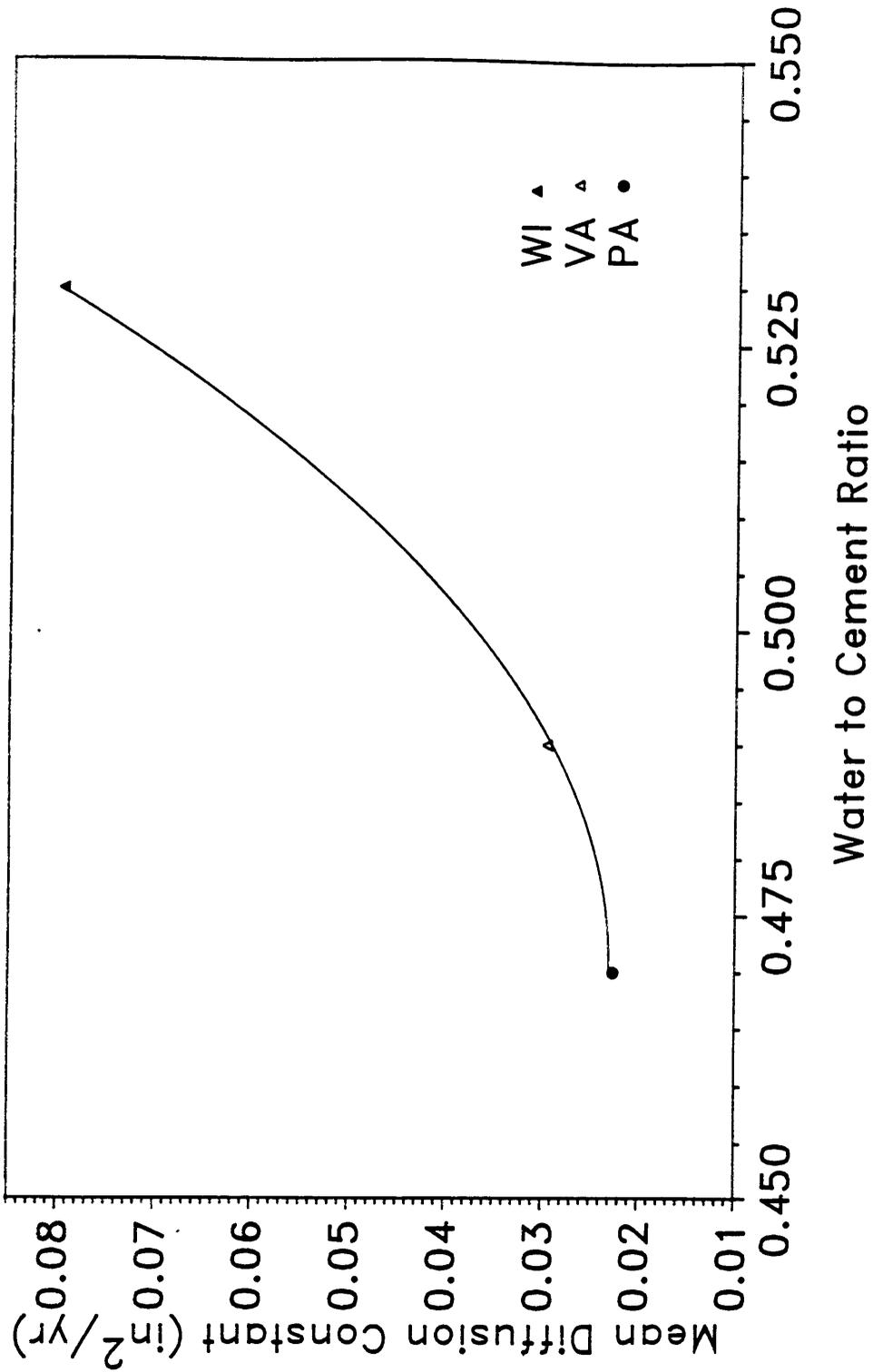


Figure 21. Mean Diffusion Constants Versus Water to Cement Ratios  
Excluding Florida

Figure 20 exhibited a discrepancy in the relationship in the data presented. The mean value for the effective diffusion constant for the substructures from Florida does not follow the general trend. One suspicion for the variance involves the softness of the aggregate which could be related to its porosity. When sampling the bridges located in Florida, the aggregate material was considered unusually soft. Therefore, the effective diffusion constant values which were calculated for the bridges in Florida may be discarded from the analysis.

The data presented in Figure 21 was analyzed to determine if relationships existed between the mean value for the effective diffusion constant and the water to cement ratio for each bridge. The following regression analysis indicates the relationship to be given as:

$$\text{Log}(D_c) = - 2.96 + 12.4 W/C^3 \quad (37)$$

where,

$\text{Log}(D_c)$  represents mean values for the effective diffusion constant for bridge decks in Virginia, Pennsylvania, and Wisconsin

$W/C$  represents the water to cement ratio of the bridges

Estimates for values of the intercept,  $b_0$  and slope,  $b_1$  and the appropriate test results for testing:

$H_0: b_0 = 0$ , and  $H_0: b_1 = 0$  : null hypotheses  
versus  $H_1: b_0 \neq 0$ , and  $H_1: b_1 \neq 0$  : alternatives

were presented by Minitab as follows:

Predictor	Coef	Stdev	t-ratio	p
Constant	-2.9613	0.1599	-18.52	0.034
W/C <sup>3</sup>	12.444	1.281	9.72	0.065

s = 0.04180      R-sq = 99.0%      R-sq(adj) = 97.9%

Results of the regression analysis indicate the estimated value for the slope, 12.444 is significantly different from 0 at a 10% level of significance (p = .065). The value for the intercept, -2.9613, is also statistically significant at a 10% level of significance (p = .034).

The coefficient of determination, R-sq, indicates 99.0% of the variation in Log(Dc) can be predicted from the independent variable, W/C<sup>3</sup> ( water to cement ratio).

The following are results of the analysis of variance for the regression analysis:

#### Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	0.16498	0.16498	94.44	0.065
Error	1	0.00175	0.00175		
Total	2	0.16673			

The results of the analysis of variance indicate the error sum of squares for the predicted values are relatively small in relation to the error sum of squares for the regression. Results from the F test also indicates there is a significant relationship at a 10% level of significance, as indicated by the p value of .065.

## Effects of Time

The first step in analyzing the effective diffusion values to determine the effects of time was to identify the most appropriate statistical procedure to be used. The t test and the Welch t test procedures were considered more suitable for the analysis depending on whether the variances for the samples of a particular bridge were equal or not. The F test procedure was used for determining the equality between variances [46].

The first step in the analysis procedure was to test for equal variances of the diffusion constant values for each bridge. The following are the results from the F test procedure [46].

ES-2	Deck	$n_1 = 3$	$\mu_1 = .0650$	$s_1 = .0300$
PA180WB	Deck	$n_2 = 10$	$\mu_2 = .0275$	$s_2 = .0351$

$H_0: \sigma_1^2 = \sigma_2^2$  : Null Hypothesis

$H_1: \sigma_1^2 \neq \sigma_2^2$  : Alternative Hypothesis

$$F_{obs} = s_1^2 / s_2^2 = .7305$$

$$F_{(2,9).975} = 5.71$$

$$F_{(2,9).025} = 1 / F_{(9,2).975} = 1 / 39.39 = .0254$$

Since  $.0254 < F_{obs} < 5.71$ ,  $H_0$  is accepted and conclude there is insufficient evidence to indicate the variance of the diffusion constants for this bridge are different. Therefore the t test will apply for comparing means.

For the t test procedure, the following hypothesis was tested to determine if the estimated mean values for the effective diffusion at each bridge had changed with time.

$H_0: \mu_1 - \mu_2 = 0$  : Null Hypothesis

$H_1: \mu_1 - \mu_2 \neq 0$  : Alternative Hypothesis

Test at  $\alpha = 5\%$  level of significance

$$t_{obs} = \frac{\bar{X}_1 - \bar{X}_2}{S_p \sqrt{1/n_1 + 1/n_2}}$$

where  $S_p = \sqrt{\frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2}}$

$S_p = .0342$  and  $t_{obs} = 1.664$

$t_{(11)} \alpha/2 = -2.201$  and  $2.201$

Since  $-2.201 < t_{obs} < 2.201$  the null hypothesis is accepted and conclude there is insufficient evidence to indicate the average values for the effective diffusion constants sampled at two different time periods on this bridge have changed.

ES-3	Deck	$n_1 = 3$	$\mu_1 = .05000$	$s_1 = .01639$
PA180EB	Deck	$n_2 = 6$	$\mu_2 = .02083$	$s_2 = .01158$

$H_0: \sigma_1^2 = \sigma_2^2$  : Null Hypothesis

$H_1: \sigma_1^2 \neq \sigma_2^2$  : Alternative Hypothesis

$F_{obs} = s_1^2 / s_2^2 = 2.0033$

$F(2, 5) .975 = 8.43$

$F(2, 5) .025 = 1 / F(5, 2) .975 = 1 / 39.3 = .02544$

Since  $.02544 < F_{obs} < 8.43$ ,  $H_0$  is accepted and conclude there is insufficient evidence to indicate the variance of the diffusion constants for this bridge are

different. Therefore the t test will apply for comparing means.

The following hypothesis was tested to determine if the estimated mean values for the effective diffusion at this bridge had changed with time.

$H_0: \mu_1 - \mu_2 = 0$  : Null Hypothesis

$H_1: \mu_1 - \mu_2 \neq 0$  : Alternative Hypothesis

Test at  $\alpha = 5\%$  level of significance

$$t_{obs} = \frac{\bar{X}_1 - \bar{X}_2}{S_p \sqrt{1/n_1 + 1/n_2}}$$

$$\text{where } S_p = \frac{\sqrt{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}}{n_1 + n_2 - 2}$$

$$S_p = .0131 \text{ and } t_{obs} = 3.149$$

$$t_{(7)} \alpha/2 = -2.365 \text{ and } 2.365$$

Since  $t_{obs} > 2.365$  the null hypothesis is rejected and concluding there is sufficient evidence to indicate the average values for the effective diffusion constants sampled at two different time periods on this bridge are different.

BS-6	Deck	$n_1 = 3$	$\mu_1 = .03167$	$s_1 = .00577$
11&15NB	Deck	$n_2 = 12$	$\mu_2 = .02417$	$s_2 = .01905$

$H_0: \sigma_1^2 = \sigma_2^2$  : Null Hypothesis

$H_1: \sigma_1^2 \neq \sigma_2^2$  : Alternative Hypothesis

$$F_{obs} = s_1^2 / s_2^2 = .0917$$

$$F_{(2,11).975} = 5.26$$

$$F_{(2,11).025} = 1/F_{(11,2).975} = 1/39.405 = .0254$$

Since  $.0254 < F_{obs} < 5.26$ ,  $H_0$  is accepted and conclude there is insufficient evidence to indicate the variance of

the diffusion constants for this bridge are different. Therefore the t test will apply for comparing means.

For the t test procedure, the following hypothesis was tested to determine if the estimated mean values for the effective diffusion at each bridge had changed with time.

$H_0: \mu_1 - \mu_2 = 0$  : Null Hypothesis

$H_1: \mu_1 - \mu_2 \neq 0$  : Alternative Hypothesis

Test at  $\alpha = 5\%$  level of significance

$$t_{\text{obs}} = \frac{\bar{X}_1 - \bar{X}_2}{S_p \sqrt{1/n_1 + 1/n_2}}$$

$$\text{where } S_p = \sqrt{\frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2}}$$

$$S_p = .0177 \text{ and } t_{\text{obs}} = .6576$$

$$t_{(13)} \alpha/2 = -2.160 \text{ and } 2.160$$

Since  $-2.160 < t_{\text{obs}} < 2.160$  the null hypothesis is accepted and conclude there is insufficient evidence to indicate the average values for the effective diffusion constants sampled at two different time periods on this bridge have changed.

BS-7	Deck	$n_1 = 3$	$\mu_1 = .04250$	$s_1 = .01250$
11&15SB	Deck	$n_2 = 6$	$\mu_2 = .02250$	$s_2 = .00935$

$H_0: \sigma_1^2 = \sigma_2^2$  : Null Hypothesis

$H_1: \sigma_1^2 \neq \sigma_2^2$  : Alternative Hypothesis

$$F_{\text{obs}} = s_1^2 / s_2^2 = 1.7873$$

$$F(2, 5) .975 = 8.43$$

$$F(2, 5) .025 = 1/F(5, 2) .975 = 1/39.3 = .02544$$

Since  $.02544 < F_{obs} < 8.43$  ,  $H_0$  is accepted and conclude there is insufficient evidence to indicate the variance of the diffusion constants for this bridge are different. Therefore the t test will apply for comparing means.

For the t test procedure, the following hypothesis was tested to determining if the estimated mean values for the effective diffusion at each bridge had changed with time.

$H_0: \mu_1 - \mu_2 = 0$  : Null Hypothesis

$H_1: \mu_1 - \mu_2 \neq 0$  : Alternative Hypothesis

Test at  $\alpha = 5\%$  level of significance

$$t_{obs} = \frac{\bar{X}_1 - \bar{X}_2}{S_p \sqrt{1/n_1 + 1/n_2}}$$

$$\text{where } S_p = \sqrt{\frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2}}$$

$$S_p = .0103 \text{ and } t_{obs} = 2.746$$

$$t_{(7)} \alpha/2 = -2.365 \text{ and } 2.365$$

Since  $t_{obs} > 2.365$  the null hypothesis is rejected and conclude there is sufficient evidence to indicate the average values for the effective diffusion constants sampled at two different time periods on this bridge have changed.

As indicated in the results from all four bridges, the variances of the effective diffusion constants for the different time periods between sampling remained equal at the 5% level of significance.

For the final analysis of the data, the t test [46] was used to determine if the average values for the effective

diffusion constants changed with time. The final results of the analysis indicated the effective diffusion constants did change with time for two of the four bridges investigated.

## CONCLUSIONS

The primary objective of this project was to develop a field procedure for determining the chloride content in concrete. This objective was accomplished by a preliminary investigation of four select methods to determine their effectiveness in measuring the chloride content of concrete followed by a subsequent analysis of the select method to determine the effects of cement content and reaction temperature in measuring chloride content. Ultimately, a field procedure for determining the chloride content in concrete was developed and validated in the field.

Results from the initial phase of the investigation indicated the Quantab titrator strips would provide a rough estimate of the chloride content in a concrete specimen.

The results of the tests using the spectrophotometer indicated that reasonable estimates of chloride content could be obtained particularly for low levels of chloride.

The digital titrator may also be used to obtain good estimates of chloride content in concrete specimens. This procedure allows accuracy in measuring the chloride content over a broader range of contamination levels than with the spectrophotometer.

The specific ion probe provided more accuracy in measuring the chloride concrete in chloride than the previous methods tested. In addition, less than 5 minutes

per specimen were required for the analysis procedure during the laboratory investigation.

The second phase of the investigation indicated the specific ion probe test results were not affected by different cement contents but were affected by changes in temperature. Consequently, a correction factor for temperature was developed for use in future tests.

Field validation of the specific ion probe indicated a good relationship between the results obtained with those obtained using the standard potentiometric titration procedure.

Following initial field validation tests using the procedure developed for the specific ion probe, the time required for analyzing the concrete specimens in the field ranged between 5 to 6 minutes per specimen. Under field conditions, 40 to 50 specimens may be analyzed in the field during an 8 hour work shift. Using the same procedure, 70 to 80 specimens can be analyzed in the laboratory in an 8 hour shift.

The increase in the number of specimens which may be analyzed in the laboratory versus in the field is primarily due to delays associated with moving to different locations to facilitate the sampling operation during the field operations.

A second objective of the investigation was to examine the variability in the effective diffusion constant. This

was accomplished by investigating the effects of temperature and the effects of the water to cement ratio on the mean values for the effective diffusion constants calculated from the tests for chloride obtained during field validation of the specific ion probe. In addition, the effects of time on the effective diffusion constant was evaluated by examining four bridges in which chloride contents were analyzed at two different time periods.

A significant relationship between temperature and the mean values of the effective diffusion constant for the bridges tested was not evident. The results, however, are not conclusive since Page et. al. [52] suggested the relationship exists at a particular water to cement ratio.

The results of the analysis indicated a significant relationship exists between the mean values for the effective diffusion constant and the water to cement ratio for the different areas tested. In addition, the mean temperature and water to cement ratios exhibited a high correlation which prevented interaction of the two variables in predicting values for the effective diffusion constant.

For the effects of time, mean values of the effective diffusion constant calculated for four bridges in Pennsylvania were compared at two separate time periods approximately 4 years apart. The results indicated variances of the effective diffusion constants for the different time

periods between sampling remained equal at the 5% level of significance.

For the final analysis of the data, the t test [46] was used to determine if the average values for the effective diffusion constants changed with time. The final results of the analysis indicated the effective diffusion constants did change with time for two of the four bridges investigated. Though not statistically significant for two of the four bridges, the mean values for the effective diffusion constant decreased with time for all four bridges. This occurrence is probably attributed to a decrease in the amount of deicing salt being applied to the bridges. Since the data reported by Weyers and Cady was obtained, the Pennsylvania Department of Transportation altered their policy regarding the application of deicing salt on the bridges tested. Previously, deicing salts were applied over the entire length of the bridges. Anymore, no salt is applied directly to the bridges. Additional salt contamination is due to vehicles tracking the salt from the highway onto the bridges.

## RECOMMENDATIONS FOR FUTURE RESEARCH

The following are suggestions for future research which may improve or enhance the results from this investigation:

1. The spectrophotometer and digital titrator were found unsuitable for determining the chloride content of concrete in the field. However, both procedures may be validated for future use in a laboratory environment.
2. The specific ion probe exhibited more variability in the estimates for chloride above the level of contamination corresponding to the maximum chloride concentration of the calibration liquids. An investigation of the procedure using an additional contamination level in the calibration liquids may be warranted.
3. The vacuum collection unit used during the field validation phase of the investigation is considered a prototype and could be modified. A smaller vacuum system may also be incorporated into the design.
4. Several methods have been proposed for calculating the effective diffusion constant. However, none of the identified research has compared the different procedures to determine which one is more effective in determining diffusion constants and for projecting the time of depassivation.
5. Additional research may be warranted concerning different factors which may influence the values for the effective diffusion constant to determine which factors are

in fact influential and which factors may be regarded less relevant.

6. Additional research may be needed to verify suspicions that mean values for the effective diffusion constant within a bridge component does not change with time. Results presented in this investigation are considered preliminary. An investigation to determine the factors which may cause the change in some cases may be warranted.

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APPENDIX A Aggregate Properties and Concrete Mixtures

Table A1. Fine Aggregate Properties

<u>Sieve Size</u>	<u>Cumulative Percent Passing</u>	<u>ASTM C33 Specifications</u>
3/8"	100	100
#4	99.9	95 to 100
#8	95.3	80 to 100
#16	75.7	50 to 85
#30	43.1	25 to 60
#50	12.8	10 to 30
#100	2.4	2 to 10
#200	.72	

Fineness Modulus	= 2.71	ASTM C33
Absorption, %	= .83	2.3 to 3.1
Bulk Dry Specific Gravity	= 2.54	

Table A2. Coarse Aggregate Properties

Source: Transportation Materials Laboratory

<u>Sieve Size</u>	<u>Cumulative Percent Passing</u>	<u>Specifications</u>	
		<u>ASTM C33</u>	<u>VDOT [37]</u>
1 ½"	100	100	100
1"	99.5	95 to 100	90 to 100
¾"	15.9	25 to 60	26 to 60
#4	1.1	0 to 10	0 to 7
#8	1.0	0 to 5	0 to 3

Dry Rodded Unit Weight, lbs/cu ft. = 99.0  
Absorption, % = 0.51  
Bulk Dry Specific Gravity = 2.79

Table A3. Coarse Aggregate Properties

Source: Area Suppliers

Sieve Size	Cumulative Percent Passing			Specifications ASTM C33
	<u>Marshall</u>	<u>Marshall</u>	<u>Atlantic</u>	
1 1/2"	100	100	100	100
1"	99.6	97.6	99.5	95 to 100
3/4"	14.2	16.4	21.0	25 to 60
#4	1.1	.6	2.4	0 to 10
#8	1.0	.6	2.0	0 to 5

Table A4. Concrete Mixture Proportions for  
 Bridge Deck Concrete, W/C = .47

<u>Ingredient</u>	<u>Dry</u> <u>(lb/cu yd)</u>	<u>Moisture</u> <u>Adjusted</u> <u>(lb/cu yd)</u>	<u>SSD</u> <u>(lb/cu yd)</u>
Water	301	304	301
Cement	640	640	640
Coarse Agg.	1740	1743	1749
Fine Agg.	1159	1172	1169
Total	3840	3858	3858
Air Entrainment	9.6 oz./		

Moisture Content  
 Coarse Aggregate = .16%  
 Fine Aggregate = 1.12%

Table A5. Concrete Mixture Proportions for  
Superstructure Concrete, W/C = .40

<u>Ingredient</u>	<u>Dry</u> <u>(lb/cu yd)</u>	<u>Moisture</u> <u>Adjusted</u> <u>(lb/cu yd)</u>	<u>SSD</u> <u>(lb/cu yd)</u>
Water	263	273	263
Cement	658	658	658
Coarse Agg.	1741	1742	1750
Fine Agg.	1239	1247	1249
Total	3901	3920	3920

Air Entrainment 10.5 oz.  
Water Reducer 22.5 oz.

Moisture Content  
Coarse Aggregate = .06%  
Fine Aggregate = .63%

Table A6. Concrete Mixture Proportions for  
Substructure Concrete, W/C = .50

<u>Ingredient</u>	<u>Dry</u> <u>(lb/cu yd)</u>	<u>Moisture</u> <u>Adjusted</u> <u>(lb/cu yd)</u>	<u>SSD</u> <u>(lb/cu yd)</u>
Water	276	294	276
Cement	552	552	552
Coarse Agg.	1741	1741	1750
Fine Agg.	1292	1293	1303
Total	3861	3881	3881

Air Entrainment 8.8 oz.

Moisture Content  
Coarse Aggregate = .02%  
Fine Aggregate = .10%

Table A7. Concrete Batch Test Results

Batch No.	W/C	Cl Content (lb/cu yd)	Unit Wt. (lb/cu ft)	Air (%)	Slump (in)	*Compressive Strength(psi)	
						7 day	28 day
C-0	.47	0.0	146.2	5.1	6 1/2	3600	4810
C-1	.47	.2	146.7	5.4	3	4250**	5250
C-2	.47	.4	146.7	5.2	3	4340**	5050
C-3	.47	.8	145.4	5.6	4 3/8	4140**	4910
C-4	.47	1.6	147.0	5.0	4	4350**	4810
C-5	.47	3.2	147.6	5.0	3 5/8	3700**	5240
C-6	.47	6.4	147.3	5.7	3 3/4	3700**	4900
C-7	.47	8.6	148.8	4.5	7 1/4	3960	4600
C-8	.47	10.8	148.3	5.2	4	4380	5080
C-9	.47	12.8	146.5	5.4	4 3/4	3690**	4990
DC-0	.40	0.0	147.6	6.0	2 1/4	5140	6170
DC-1	.40	.2	144.5	6.6	5 1/2	4540	5890
DC-2	.40	.4	147.0	5.5	3 3/4	5090	6400
DC-3	.40	.8	147.8	5.1	5 1/2	4850	6610
DC-4	.40	1.6	148.8	5.6	3	5130	6170
DC-5	.40	3.2	146.4	6.0	7	4860	6140
DC-6	.40	6.4	144.1	7.1	8	4940	5780
DC-7	.40	8.6	143.1	7.1	8 3/4	4840	5700
DC-8	.40	10.8	142.7	8.0	8	4840	5550
DC-9	.40	12.8	147.4	6.0	6 1/2	4820	5740
EC-0	.50	0.0	145.5	5.6	3 1/2	3750	4850
EC-1	.50	.2	146.0	5.7	3 1/2	3790	4690
EC-2	.50	.4	146.1	6.0	3 3/4	3870	4830
EC-3	.50	.8	146.6	6.0	3 3/4	3710	4580
EC-4	.50	1.6	146.6	5.2	3 3/4	3620	4480
EC-5	.50	3.2	147.0	5.3	4 1/4	3610	4230
EC-6	.50	6.4	146.3	5.5	4	3510	4290
EC-7	.50	8.6	146.8	5.6	4	3720	4250
EC-8	.50	10.8	146.2	5.4	5	3540	4440
EC-9	.50	12.8	146.8	5.6	4	3520	4610

\* Average of two cylinder tests

\*\* Indicate 8 day strength results

APPENDIX B Chloride Analysis Procedure for Selected Methods

James Instruments, Model CL 500

The procedure used for testing the chloride content of concrete specimens is as follows:

1. Calibration of the electrometer: Turn the electrometer on and set the dial to read V (volts). Insert the specific ion electrode into one of the standard solutions supplied with the instrument. Allow the electrometer reading (millivolts) to stabilize by waiting approximately  $1\frac{1}{2}$  to 2 minutes. Record the reading and the number corresponding to the standard solution. Repeat for the two remaining standard solutions. Calibration of the electrometer should be done each time the instrument is turned on.
2. Analyzing powdered concrete samples: Prior to weighing, stir the powdered sample thoroughly to insure a uniform mixture of chloride contaminated concrete powder. Weigh three grams of the powdered sample to the nearest 0.1 milligrams using an analytical balance. Place the three gram sample into the supplied chloride extraction liquid and shake for 15 seconds. Insert the electrode into the digested sample for a period of three minutes and record the millivolt reading.
3. Modifications of the original procedure suggested by James Instruments [39] included weighing each specimen using a precision balance, and increasing the time before reading the results on the electrometer to a minimum of 3 minutes.

After the three minutes, if the change in the millivolt reading resulted before the reading was recorded the new reading was recorded.

Spectrophotometer; Hach DR/2000

1. Sample Preparation: Prior to weighing, stir the powdered sample thoroughly to insure a uniform mixture of chloride. Weigh one gram of the powdered sample to the nearest 0.1 of a milligram using an analytical balance. Place the sample in a 250 ml beaker. Add approximately 5 to 10 milliliters of deionized water. Next, add 5 milliliters of concentrated nitric acid and stir vigorously for 10 seconds. After fizzing settles, transfer the sample to a second 250 ml beaker using a filtering apparatus and Whatman 40 filter paper. When filtering, rinse the sample in the filter 4 to 5 times to ensure a complete transfer of the chloride. Rinse the outer lip of the beaker. After filtering, rinse the outside of the filter paper 3 to 4 times and rinse the funnel and funnel tip thoroughly, allowing all of the filtrate to be transferred into the sample beaker. If a precipitant exists refilter the sample using the above procedure. After filtering, increase the sample volume to 100 milliliters using a graduated cylinder. Transfer the sample in the graduated cylinder back into the same 250 ml beaker.

2. Analyzing the filtrate: Specify the chloride test procedure on the spectrophotometer by pressing 7 0 enter.

Enter the appropriate wavelength reading (455) for the chloride test. Two sample cells are prepared, one with deionized water, and the other with the filtered sample to be analyzed. Pipet 25 milliliters of the sample into one of the same cells. Next, pipet 25 milliliters of deionized water into the matching sample cell. Add 2 milliliters of mercuric thiocyanate to each sample cell. Next, add 1.0 ml of ferric ion solution to each sample and swirl to mix. Observe the samples to determine if any interferences which may reduce the accuracy are present. Press shift time on the instrument. A two minute time period will start. During this waiting period, wipe the outside of the sample cells to remove any dirt or spots. Following the end of the two minute period. Place the sample cell containing no chloride into the slot provided in the instrument with the numbers on the cell facing to the left. Press zero on the instrument. Once the instrument provides a reading of 0 mg/L, remove the blank cell and place the cell containing the chloride sample into the instrument in a similar manner. Press read, wait, and record the results.

3. Modifications of the original procedure suggested by the Hach Company [40] involved the preparation of the sample prior to testing. The procedure developed by the Hach Company was applicable to water samples. Therefore, a procedure for extracting the chloride from the powdered concrete specimens was developed prior to analyzing the

samples using the procedure recommended by the Hach Company for analyzing the chloride content of water.

The spectrophotometer was developed mainly for detecting low levels of chloride. In addition this procedure is very sensitive to interferences resulting from low pH or acidic samples. Therefore a 1 gram sample was weighed and acid digested using .5 milliliters of concentrated nitric acid. Following the filtering process, the prepared sample of 100 milliliters was consistently above a pH of 3. Twenty-five milliliters of this sample was analyzed for chloride content. As a result, no interferences were detected throughout the testing procedure.

CD-DT Digital Titrator (Hach Company)

1. Powdered concrete sample preparation: Prior to weighing, stir the powdered sample thoroughly to insure a uniform mixture of chloride. Weigh one grams of the powdered sample to the nearest 0.1 of a milligram using an analytical balance. Place the sample in a 250 ml beaker. Add approximately 5 to 10 milliliters of deionized water. Next, add 0.5 milliliters of concentrated nitric acid and stir vigorously for 10 seconds. After fizzing settles, transfer the sample to a second 250 ml beaker using a filtering apparatus and Whatman 40 filter paper. When filtering, rinse the sample in the filter 4 to 5 times to ensure a complete transfer of the chloride. Rinse the outer lip of the beaker. After filtering, rinse the outside of

the filter paper 3 to 4 times and rinse the funnel and funnel tip thoroughly, allowing all of the filtrate to be transferred into the sample beaker. If a precipitant exists refilter the sample using the above procedure. After filtering, increase the sample volume to 100 milliliters using a graduated cylinder. Transfer the sample in the graduated cylinder back into the same 250 ml beaker.

2. Analyzing the filtrate: Remove 25 ml of the sample in the 250 ml beaker using a pipet. The sample now contains 75 ml. Place the 75 ml sample on a magnetic stirrer and insert a magnetic stirring rod. Using a pH probe, adjust the pH of the sample using perchloric acid (1:5 dilution) to 2.5 plus or minus .1. If necessary, sodium hydroxide (5N) may be used to adjust the pH upward. Once the pH is properly adjusted, add the contents from one of the chloride<sup>2</sup> powder pillows while stirring. The sample will turn from a clear to a bright yellow color. Next, insert one of the 1.128 N silver chloride containers into the digital titrator. Insert one of the titrating tubes into the tip of the silver chloride. Turn the dial until the silver chloride drips from the end of the red tube. Using a chemical free tissue, wipe the end of the tip to remove any silver chloride from the outside. Zero the dial on the digital titrator and insert the red tip into the 75 ml sample. Turn the dial to titrate the sample. Observe the first consistent color change, reddish yellow or brownish yellow, which indicates the midpoint of the

titration. Read and record the number of digits required for titrating the sample.

3. Modifications of the original procedure suggested by the Hach Company [41] involved the preparation of the sample prior to testing. The procedure developed by the Hach Company was applicable to water samples. Consequently, a procedure for extracting the chloride from the powdered concrete specimens was developed prior to analyzing the samples using the procedure recommended by the Hach Company for analyzing the chloride content of water.

Unlike the spectrophotometer, the digital titrator was developed for detecting somewhat higher levels of chloride. This procedure is also sensitive to interferences resulting from pH. Therefore a 1 gram sample was weighed and acid digested using 0.5 milliliters of concentrated nitric acid. Following the filtering process, the prepared sample of 100 milliliters was consistently above a pH of 3. Seventy-five milliliters of this sample was analyzed for chloride content. Prior to the analysis, each sample was adjusted to a pH ranging from 2.4 to 2.6. As a result, no interferences were detected throughout the testing procedure.

#### Quantab Titrator Strips (Standard)

1. Powdered concrete sample preparation: Prior to weighing, stir the powdered sample thoroughly to insure a uniform mixture of chloride. Weigh ten grams of the powdered sample to the nearest 0.1 of a milligram using a

precision scale. Place the sample in a 250 ml beaker. Add 90 ml of boiling distilled water while stirring constantly. Stir the sample stirred for 30 seconds, and after one minute, stir the sample again for another 30 second period. Fold Whatman 40 filter paper twice, into a cone, and place it into the beaker. Allow some of the sample to filter through.

2. Chloride analysis procedure: Place a Quantab titrator strip in the filtered portion of the sample and allow it to remain until a dark blue color forms at the top of the strip. Allow 5 to 10 minutes for the color to change. Read the Quantab titrator strip to the nearest .05 mark at the upper most point of the color change. This color may be white or a yellowish orange. Record the reading.

The procedure developed by Environmental Test Systems, Inc. [42] was used initially for analyzing the chloride content of the test specimens. This procedure provided an analysis of the water soluble chloride present in the specimens.

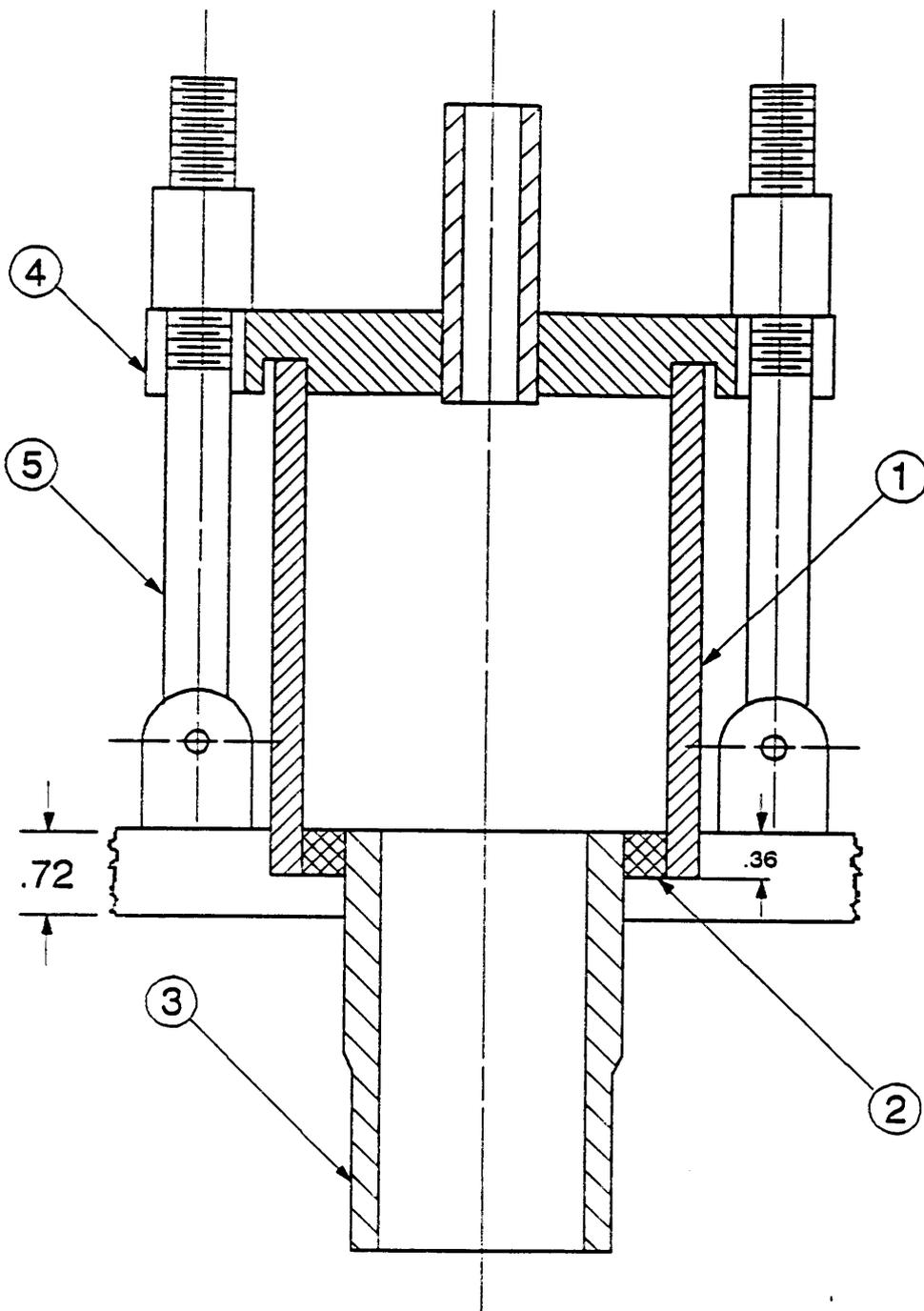
Since background chloride is also present in concrete bridge components, the procedure was modified to provide for an acid digestion of the sample prior to testing. Ten grams of the powdered samples were weighed, followed by the addition of 3 milliliters of nitric acid. After increasing the sample to 100 milliliters, the sample was tested for chloride content using the Quantab titrator strip. Prior to

testing each sample, a pin hole was inserted along the yellow line near the top of the strip as recommended by Environmental Test Systems, Inc. [42] to obtain more accurate results.

#### Quantab Titrator Strips (Modified)

1. Powdered concrete sample preparation: Prior to weighing, stir the powdered sample thoroughly to insure a uniform mixture of chloride. Weigh ten grams of the powdered sample to the nearest .1 of a milligram using an analytical balance. Place the sample in a 250 ml beaker. Add approximately 5 to 10 milliliters of distilled deionized water. Next, add 3 milliliters of concentrated nitric acid. Stir visciously for 15 seconds. Wait one minute and stir again for 15 seconds. After fizzing settles, fold the filter paper (Whatman 40) twice to form a cone. Place the filter paper into the beaker containing the sample. Allow some filtrate to filter into the cone. Using a stick pin, punch a whole in the top of the strip along the yellow line. Place the Quantab titrator strip in the filtered portion of the sample and allow it to remain until a dark blue color forms at the top of the strip. Allow 5 to 10 minutes for this to occur. Read the Quantab titrator strip to the nearest .05 mark at the uppermost portion of the white color change. Notice that this portion may not extend across the entire Quantab strip. Record the reading.

APPENDIX C Detailed Drawings of the Sample Collection Unit



All dimensions in inches

Figure C.1. Profile of the Sample Collection Unit

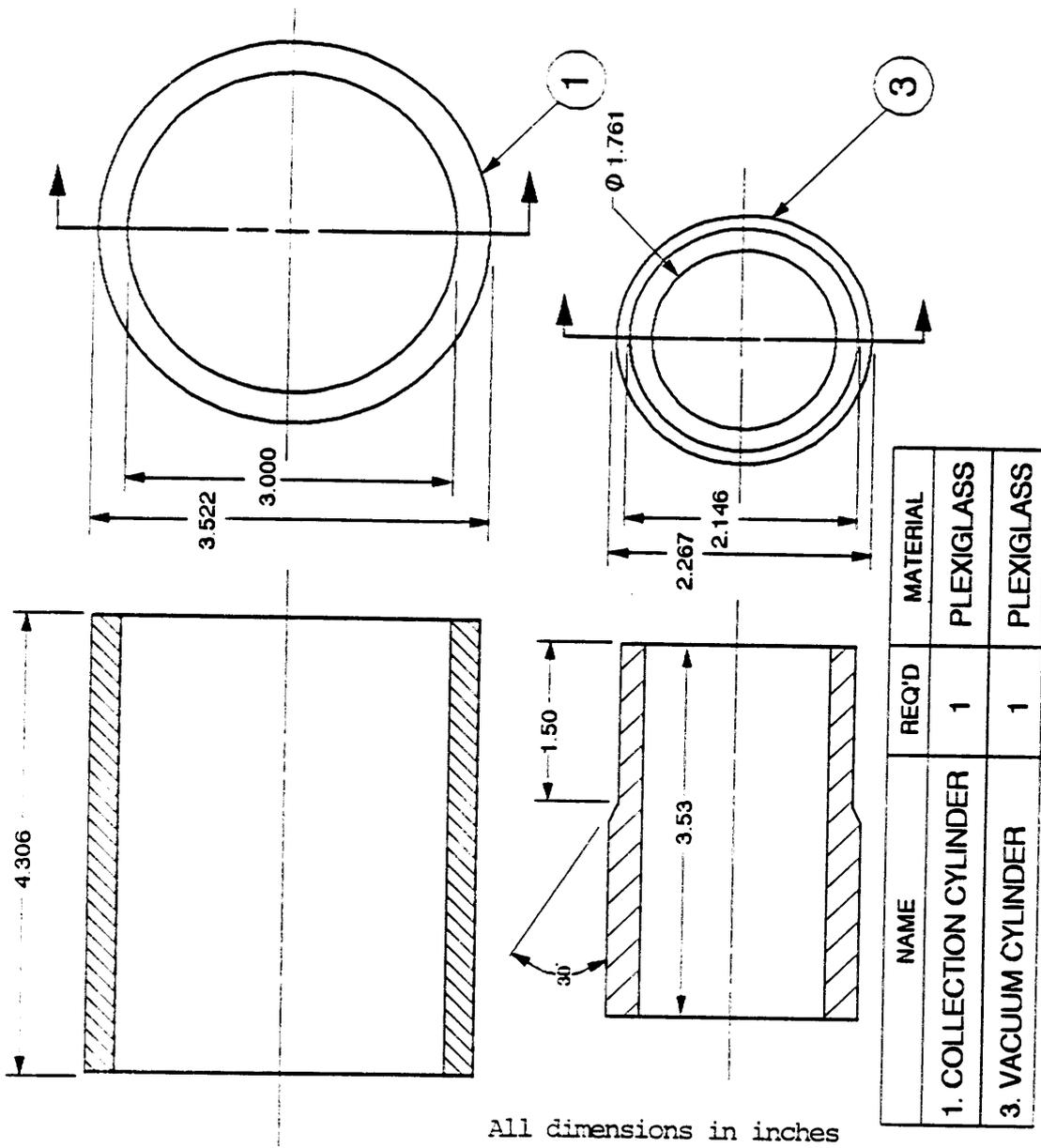
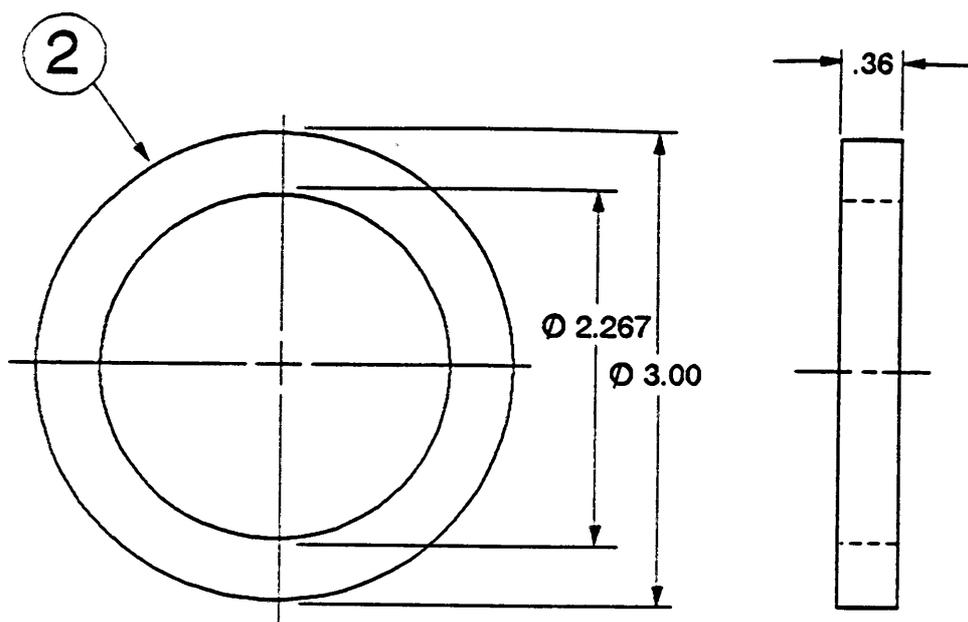


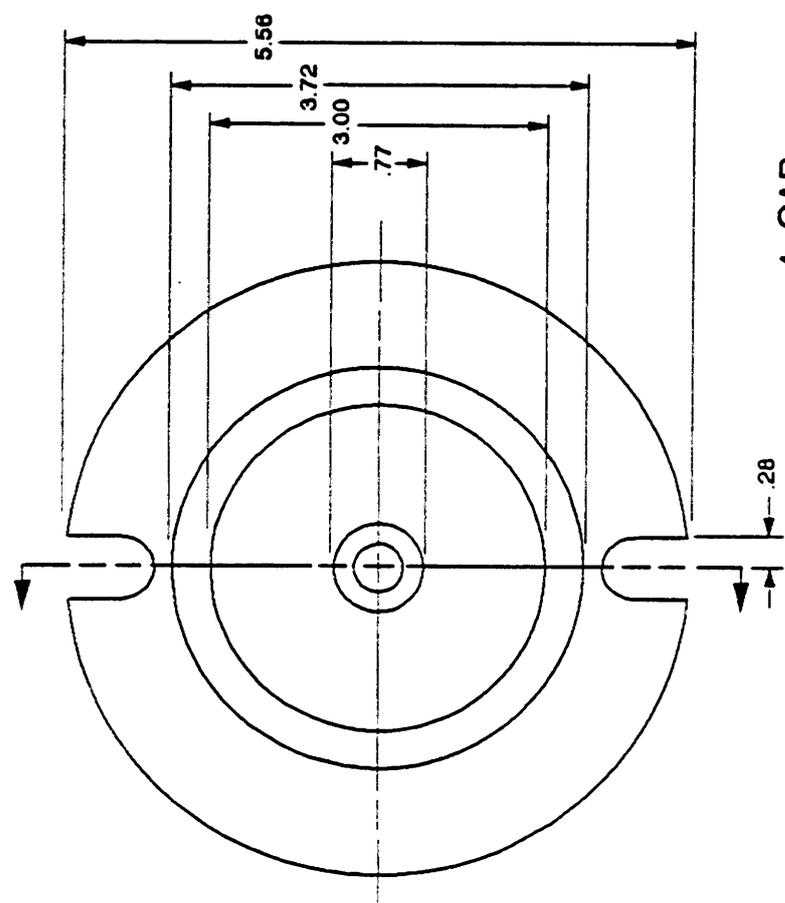
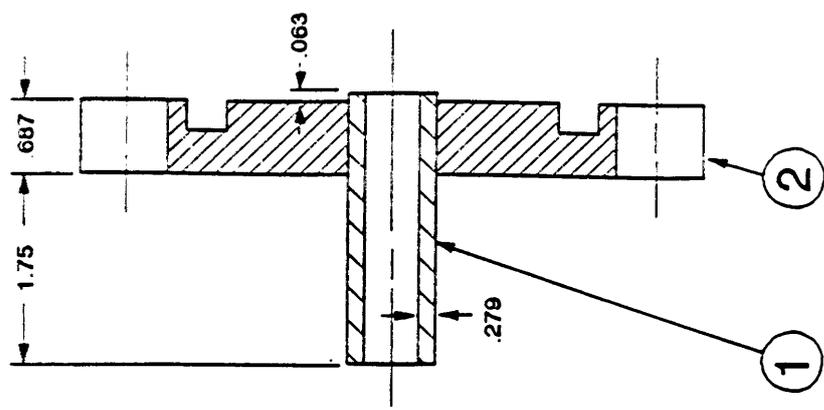
Figure C.2. Collection Cylinder and Vacuum Cylinder



NAME	REQ'D	MATERIAL
2. RING	1	PLEXIGLASS

All dimensions in inches

Figure C.3. Plexiglass Ring

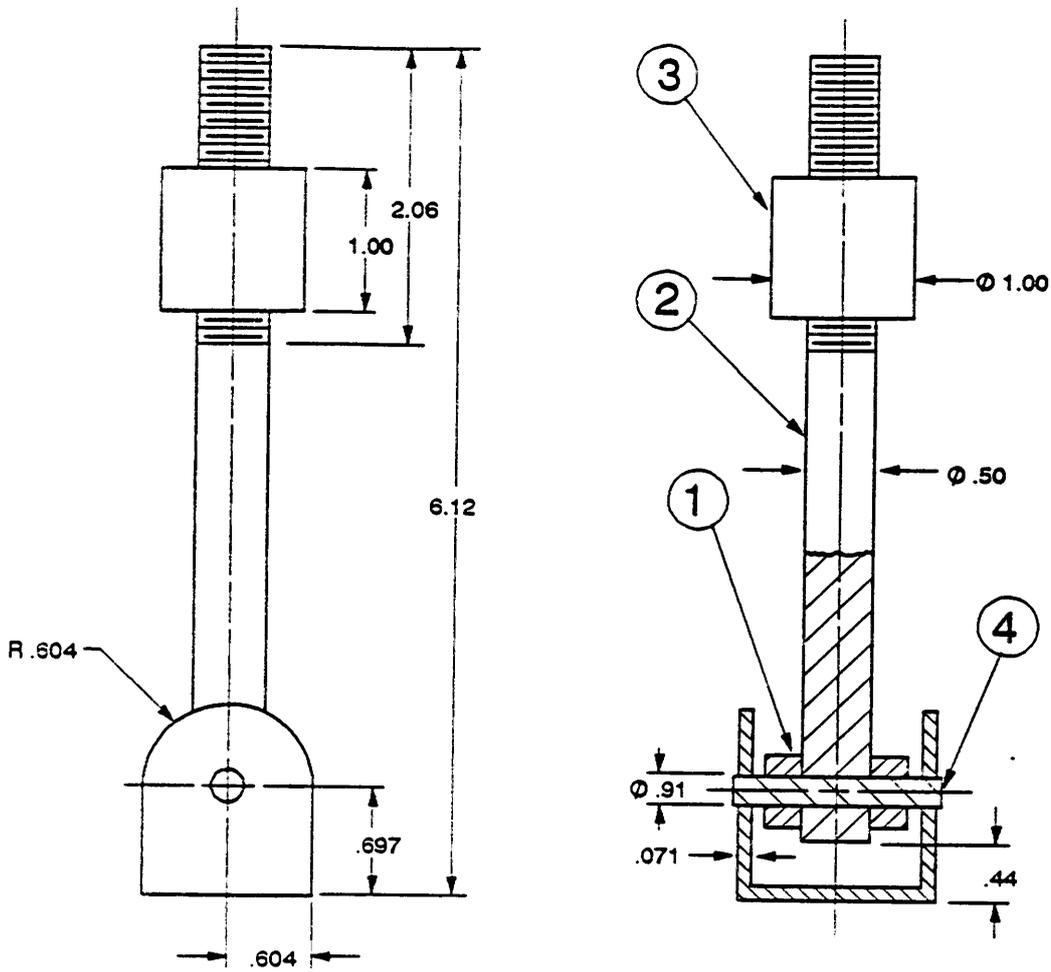


4. CAP

NAME	REQ'D	MATERIAL
1. ENTRY CYLINDER	1	PLEXIGLASS
2. CAP BASE	1	PLEXIGLASS

All dimensions in inches

Figure C.4. Entry Cylinder and Cap Base



### 5. CLAMP -- 2 REQ'D

NAME	REQ'D	MATERIAL
1. SPACER	2	RUBBER
2. SHAFT	1	STEEL
3. NUT	1	STEEL
4. SPRING PIN	1	STEEL

All dimensions in inches

Figure C.5. Clamping Mechanism

APPENDIX D. Chloride Content Results for Bridges in  
Pennsylvania, Virginia, Florida, and Wisconsin

Table D1. Predicted Chloride Content Values for  
 Pennsylvania Bridge PA 180 over 220 WB

Sample Number	Reading (M-Volts)	Specific Ion Probe*		Potentiometric Titration	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)*
A-1	27.8	66	.112	.07	2.68
2	59.3	71	.028	.02	.72
3	81.6	70	.008	.01	.26
4	108.5	69	0	0	.00
B-1**	3.5	72	.336	.30	11.66
2**	10.9	70	.242	.17	6.80
3**	34.2	70	.087	.05	2.05
4**	56.4	72	.033	.02	.83
C-1	-1.3	75	.388	.40	15.81
2	11.3	76	.228	.16	6.25
3	38.3	80	.076	.05	1.80
4	63.2	78	.027	.02	.71
D-1	1.5	65	.308	.25	9.93
2	26.6	64	.102	.06	2.42
3	44.6	65	.045	.03	1.10
4	73	61	.006	.00	.19
E-1	56.7	68	.026	.02	.75
2	99.7	64	0	.00	.00
3	104.9	64	0	.00	.00
4	LOST	LOST	LOST	LOST	LOST
F-1	25.1	68	.113	.07	2.70
2	62	65	.018	.01	.50
3	84.9	64	.012	.01	.35
4	107.5	68	0	.00	.00
G-1	19	79	.174	.11	4.40
2	52.1	80	.046	.03	1.12
3	80	84	.019	.01	.52
4	94.9	75	.007	.01	.22
H-1	5.7	76	.302	.24	9.63
2	37.7	70	.074	.04	1.76
3	60.6	74	.029	.02	.74
4	105.6	82	.009	.01	.28

Table D1. Predicted Chloride Content Values for  
 Pennsylvania Bridge PA 180 over 220 WB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe*		Potentiometric Titration	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)*
I-1	6.9	77	.288	.23	8.88
2	30.7	74	.104	.06	2.47
3	69.9	75	.020	.01	.54
4	93.3	76	.008	.01	.26
J-1	17.9	79	.180	.12	4.59
2	59.7	80	.034	.02	.87
3	88	80	.013	.01	.37
4	103.6	83	.010	.01	.31
K-1	LOST	LOST	LOST	LOST	LOST
2	87.1	75	.020	.01	.53
3	97.7	81	.010	.01	.31
4	111.9	83	.009	.01	.27
L-1	25.8	82	.132	.08	3.20
2	91.1	83	.014	.01	.39
3	116.6	89	.012	.01	.36
4	111.8	83	.009	.01	.28

\* Samples were crushed to minus 50 mesh

\*\* Actual sample numbers were:  
 B-1\*\* => B-4 depth 1<sup>3</sup>/<sub>4</sub>" to 2<sup>1</sup>/<sub>4</sub>"  
 B-2\*\* => B-1 depth 1<sup>1</sup>/<sub>4</sub>" to 3<sup>3</sup>/<sub>4</sub>"  
 B-3\*\* => B-2 depth 3<sup>3</sup>/<sub>4</sub>" to 1<sup>1</sup>/<sub>4</sub>"  
 B-4\*\* => B-3 depth 1<sup>1</sup>/<sub>4</sub>" to 1<sup>3</sup>/<sub>4</sub>"

Table D2. Predicted Chloride Content Values for  
 Pennsylvania Bridge PA 180 over 220 EB

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Potentiometric Titration	
		Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
A-1	25.2	83	.133	.093	3.67
2	31.5	81	.102	.074	2.91
3	85.8	83	.016	.009	.37
4	94.6	82	.012	.006	.23
B-1	6	81	.288	.180	7.11
2	26.1	83	.128	.090	3.55
3	62.9	81	.031	.023	.89
4	86.5	82	.015	.009	.34
C-1	11.9	81	.226	.147	5.80
2	LOST	LOST	LOST	LOST	LOST
3	LOST	LOST	LOST	LOST	LOST
4	LOST	LOST	LOST	LOST	LOST
D-1	50.2	82	.050	.038	1.48
2	89.6	84	.015	.009	.34
3	96.6	85	.013	.007	.28
4	110.3	81	.008	.002	.06
G-1	7.7	84	.270	.171	6.75
2	54.4	85	.044	.034	1.32
3	77.5	85	.021	.014	.57
4	94.4	83	.013	.006	.26
H-1	6.9	82	.272	.172	6.78
2	49.3	84	.051	.038	1.51
3	84.5	85	.017	.011	.42
4	100.9	84	.011	.005	.20
I-1	9.4	86	.248	.159	6.27
2	49	85	.052	.039	1.55
3	91.3	85	.014	.008	.32
4	108.5	86	.011	.005	.20

Table D3. Predicted Chloride Content Values for  
 Pennsylvania Bridge PA 11-15 over 35 NB

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Potentiometric Titration	
		Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
A-1	35.9	86	.088	.064	2.54
2	85.9	89	.019	.013	.50
3	85.3	86	.018	.011	.44
4	76.7	85	.021	.015	.58
B-1	33.3	88	.099	.071	2.82
2	62.3	87	.035	.026	1.02
3	94.5	88	.016	.009	.37
4	100.5	87	.013	.007	.29
C-1	37.6	85	.082	.060	2.38
2	70.8	86	.026	.019	.74
3	106.4	88	.013	.007	.27
4	95.6	88	.015	.009	.36
G-1	40.4	84	.073	.054	2.14
2	92.5	87	.016	.009	.37
3	81.2	89	.021	.015	.58
4	87.5	85	.016	.010	.39
H-1	12.7	83	.219	.143	5.64
2	27.6	85	.121	.086	3.38
3	60	85	.036	.027	1.07
4	78	84	.020	.013	.53
I-1	16	84	.192	.128	5.05
2	64.2	86	.032	.024	.94
3	77.7	85	.021	.014	.56
4	86.1	85	.017	.010	.41
P1-1	-23.1	81	.961	.488	19.23
2	-7.5	86	.505	.288	11.35
3	26.7	84	.125	.088	3.47
4	81.6	81	.016	.010	.39
5	90	83	.014	.008	.30

Table D3. Predicted Chloride Content Values for  
 Pennsylvania Bridge PA 11-15 over 35 WB (Continued)

<u>Sample Number</u>	<u>Reading (M-Volts)</u>	<u>Specific Ion Probe</u>		<u>Potentiometric Titration</u>	
		<u>Reaction Temperature (°F)</u>	<u>Calculated Chloride (%Cl<sup>-</sup>)</u>	<u>Predicted Values (%Cl<sup>-</sup>)</u>	<u>(lb/cu yd)</u>
P2-1	2.4	80	.332	.203	8.02
2	54.8	83	.042	.032	1.25
3	84.2	84	.017	.010	.41
4	88.7	82	.014	.008	.30
C1-1	60	83	.035	.026	1.02
2	70.6	84	.025	.018	.70
3	71	82	.023	.016	.65
4	90.4	86	.016	.009	.37
C2-1	51.2	85	.049	.037	1.46
2	57.2	85	.040	.030	1.18
3	62.5	83	.032	.024	.93
4	81.6	83	.017	.011	.43

Table D4. Predicted Chloride Content Values for  
 Pennsylvania Bridge PA 11-15 over 35 SB

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
A-1	30.8	71		.086	.063	2.50
2	29.6	80		.097	.070	2.78
3	57.9	79		.031	.023	.91
4	78.2	78		.014	.008	.32
B-1	-4.7	80		.401	.238	9.38
2	15.8	83		.173	.117	4.62
3	39.6	83		.067	.050	1.97
4	71.6	84		.022	.015	.60
C-1	11.3	83		.208	.137	5.40
2	49.4	80		.044	.033	1.31
3	84.1	82		.014	.008	.32
4	85.7	79		.012	.006	.22
D-1	3	81		.291	.182	7.19
2	41.3	79		.060	.045	1.78
3	80.8	81		.015	.009	.35
4	89.1	81		.012	.006	.22
E-1	17.7	80		.158	.108	4.27
2	69.1	82		.022	.016	.62
3	93.1	80		.010	.004	.15
4	99.7	81		.009	.003	.11
F-1	44.4	80		.055	.042	1.64
2	74	80		.018	.012	.48
3	88.3	80		.012	.006	.22
4	92.7	80		.010	.004	.17

Table D4. Predicted Chloride Content Values for  
 Pennsylvania Bridge PA 11-15 over 35 WB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Potentiometric Titration	
		Temperature (°F)	Calculated Chloride (%Cl-)	Predicted Values (%Cl-)	(lb/cu yd)
G-1	19.3	80	.152	.105	4.13
2	41.9	82	.062	.047	1.84
3	78.5	80	.016	.010	.38
4	81.8	82	.016	.009	.37
H-1	14.7	79	.183	.123	4.84
2	47.5	82	.050	.038	1.50
3	66.4	83	.026	.019	.74
4	82	83	.016	.010	.39
I-1	35.7	81	.079	.058	2.30
2	67.8	81	.023	.016	.64
3	86.1	81	.013	.007	.26
4	93.7	85	.013	.007	.27
J-1	17.1	86	.173	.117	4.63
2	50.7	85	.046	.035	1.37
3	69	83	.023	.016	.65
4	90.2	84	.013	.007	.29
K-1	37.5	88	.078	.058	2.27
2	83	87	.018	.012	.46
3	81	82	.016	.009	.37
4	75.1	83	.019	.013	.51
L-1	12.4	85	.210	.138	5.44
2	61.5	85	.032	.023	.92
3	80.9	86	.018	.012	.47
4	74.4	83	.020	.013	.52

Table D4. Predicted Chloride Content Values for  
 Pennsylvania Bridge PA 11-15 over 35 WB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Titration Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
P1-1	6.8	82	.263	.167	6.59
2	25.3	79	.120	.085	3.35
3	45	82	.055	.041	1.63
4	66.2	83	.026	.018	.73
P2-1	-8.1	83	.494	.283	11.15
2	13	80	.201	.133	5.26
3	41.6	80	.062	.046	1.82
4	63	83	.029	.021	.83
C1-1	70.5	82	.022	.015	.59
2	73.9	77	.016	.010	.39
3	79.6	78	.014	.007	.29
4	80.6	78	.013	.007	.28
C2-1	42.7	78	.058	.043	1.71
2	63.8	77	.024	.017	.67
3	76.6	77	.014	.008	.33
4	76.7	78	.015	.009	.35

Table D5. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 927 NB

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
A-1	-2.8	96		.397	.236	9.31
2	17.6	94		.173	.117	4.62
3	61.5	95		.038	.028	1.12
4	91.6	97		.021	.014	.57
B-1	-21.1	95		.850	.441	17.40
2	-1.1	91		.367	.221	8.73
3	.1	96		.353	.214	8.43
4	17.1	94		.177	.119	4.70
5	27.3	96		.120	.085	3.36
C-1	2.1	95		.324	.200	7.86
2	5	97		.289	.181	7.15
3	17.3	98		.178	.120	4.73
4	30.9	98		.106	.076	3.01
D-1	-21.2	98		.855	.444	17.49
2	LOST	LOST		LOST	LOST	LOST
3	4.2	97		.299	.186	7.35
4	13.4	98		.207	.137	5.39
5	26.7	96		.123	.087	3.42
E-1	-9.3	95		.519	.295	11.62
2	6.1	95		.276	.174	6.86
3	39.9	96		.076	.056	2.22
4	79.2	98		.026	.019	.75
F-1	.9	97		.342	.208	8.22
2	23.1	96		.141	.098	3.86
3	63.8	97		.037	.028	1.08
4	89.4	97		.022	.015	.59
G-1	-17.7	98		.739	.394	15.52
2	8.7	97		.249	.160	6.30
3	62.6	100		.040	.030	1.18
4	89.1	97		.022	.015	.60
H-1	-8.7	96		.507	.289	11.39
2	1	95		.339	.207	8.17
3	13.6	99		.206	.136	5.37
4	25.8	101		.130	.091	3.60

Table D5. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 927 NB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Titration Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
I-1	-1.5	99	.378	.227	8.94
2	11.2	98	.226	.147	5.80
3	38.4	97	.081	.059	2.34
4	68.3	97	.033	.024	.96
J-1	-15.5	100	.675	.366	14.42
2	-4.5	98	.427	.251	9.89
3	11.3	100	.226	.147	5.81
4	32.4	95	.099	.071	2.82
K-1	-7.9	100	.493	.282	11.13
2	21.3	97	.152	.105	4.12
3	57.6	99	.045	.034	1.33
4	82.5	98	.025	.018	.70
L-1	-9.5	98	.525	.298	11.73
2	7.5	100	.263	.168	6.60
3	23.6	100	.141	.098	3.86
4	50.6	101	.056	.042	1.66

Table D5. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 927 NB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
P1- 1	19.7	90		.167	.114	4.49
2	28.7	89		.114	.081	3.21
3	33.2	90		.095	.069	2.73
4	44.6	89		.060	.045	1.78
P2- 1	22.6	85		.145	.100	3.96
2	28.4	88		.115	.082	3.23
3	30	91		.109	.078	3.09
4	32.4	88		.097	.070	2.78
C1- 1	14	85		.211	.139	5.46
2	17.4	94		.187	.125	4.94
3	23.7	90		.141	.098	3.87
4	32.9	90		.119	.085	3.34
C2- 1	14.9	90		.206	.136	5.35
2	LOST	LOST		LOST	LOST	LOST
3	22.1	91		.152	.105	4.12
4	22.9	91		.147	.102	4.00
B1- 1	.7	88		.381	.228	9.00
2	5.1	90		.315	.195	7.68
3	9.4	90		.261	.166	6.55
4	17.3	87		.184	.123	4.86
B2- 1	-.8	93		.411	.243	9.57
2	2	91		.362	.219	8.62
3	23.2	91		.145	.100	3.96
4	18.8	91		.175	.118	4.65

Table D6. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 676 NB

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Titration Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
A-1	-12.8	93	.752	.399	15.74
2	-4.3	92	.518	.294	11.60
3	12.5	94	.253	.162	6.38
4	35.9	92	.096	.070	2.75
5	71.4	92	.029	.021	.83
B-1	-4.4	93	.521	.296	11.66
2	5.5	94	.341	.208	8.19
3	16	93	.218	.142	5.62
4	36.6	91	.093	.068	2.67
5	60.5	94	.041	.031	1.21
C-1	11.7	92	.260	.166	6.54
2	19	92	.191	.128	5.03
3	53.4	93	.051	.038	1.51
4	69	93	.031	.023	.91
D-1	-3.2	92	.494	.283	11.15
2	2.9	95	.381	.228	9.00
3	9.7	93	.284	.178	7.04
4	17.3	93	.206	.136	5.36
5	24.6	93	.152	.105	4.13
E-1	3.3	93	.374	.224	8.85
2	9.9	93	.282	.177	6.99
3	24.4	95	.155	.106	4.19
4	44.2	96	.072	.054	2.12
F-1	3.6	94	.369	.222	8.76
2	6.9	93	.320	.197	7.78
3	9	94	.293	.183	7.23
4	20	94	.185	.124	4.88
5	30.2	95	.122	.087	3.42
G-1	32.4	95	.112	.080	3.16
2	74.7	93	.027	.020	.77
3	87.4	94	.021	.014	.57
4	93.9	94	.019	.012	.49

Table D6. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 676 NB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Potentiometric Titration	
		Temperature (°F)	Calculated Chloride (%Cl-)	Predicted Values (%Cl-)	(lb/cu yd)
H-1	1.1	94	.411	.243	9.58
2	17.3	94	.207	.136	5.37
3	35	93	.100	.072	2.86
4	81.6	94	.024	.017	.65
5	82.9	93	.022	.016	.61
I-1	18.8	92	.193	.129	5.07
2	45.3	94	.068	.051	2.01
3	79.2	92	.024	.017	.66
4	80.2	92	.023	.016	.64
J-1	21.9	92	.169	.115	4.54
2	63.4	95	.038	.029	1.13
3	93.3	97	.021	.014	.56
4	93.1	97	.021	.014	.56
K-1	14.3	94	.234	.152	5.98
2	44.9	92	.068	.051	2.00
3	77.9	92	.024	.017	.68
4	83.8	92	.021	.015	.58
L-1	-4.7	96	.530	.300	11.82
2	-2.1	96	.474	.273	10.77
3	9.8	94	.233	.178	7.02
4	18.5	94	.197	.131	5.15
5	30.7	94	.119	.085	3.34
P1A-1	23.7	94	.144	.100	3.93
2	35.2	92	.089	.065	2.57
3	46.2	92	.058	.044	1.73
4	64.1	93	.033	.024	.96
P2-1	36	92	.086	.063	2.50
2	43.2	87	.062	.047	1.84
3	59.9	91	.036	.027	1.06
4	75.4	91	.023	.016	.64
P3-1	4.1	89	.329	.202	7.95
2	4.3	91	.327	.201	7.92
3	8.5	93	.273	.173	6.81
4	17.1	92	.188	.126	4.96

Table D6. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 676 NB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Reaction Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
C1-1	- .8	90		.409	.242	9.54
2	3.5	92		.339	.207	8.17
3	9.7	90		.258	.164	6.48
4	13.4	88		.218	.143	5.63
C2-1	LOST	LOST		LOST	LOST	LOST
2	2	92		.363	.219	8.63
3	4.1	91		.330	.202	7.98
4	7	92		.291	.182	7.19
B1-1	24.1	82		.134	.094	3.69
2	-2.4	89		.438	.256	10.10
3	29.6	89		.110	.079	3.10
4	-2.4	88		.438	.256	10.09
B2-1	37.7	92		.081	.060	2.35
2	69.1	94		.029	.021	.84
3	73.2	92		.025	.018	.71
4	86.4	94		.020	.014	.53
B3-1	59.4	90		.036	.027	1.06
2	82.5	91		.020	.013	.52
3	87.4	93		.019	.013	.50
4	87.2	92		.019	.012	.48

Table D7. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 670 NB

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
A-1	-9.7	93		.615	.339	13.35
2	12.3	88		.232	.150	5.93
3	42.2	94		.070	.052	2.07
4	68.8	95		.030	.022	.88
5	97.5	96		.018	.012	.47
B-1	4.9	96		.326	.200	7.89
2	16.2	95		.200	.133	5.24
3	27.9	93		.122	.087	3.41
4	44.9	94		.064	.048	1.88
5	71.7	95		.028	.021	.81
C-1	5.6	93		.314	.194	7.65
2	28.6	93		.119	.084	3.33
3	62.2	94		.036	.027	1.06
4	100.5	97		.018	.012	.47
D-1	5.7	89		.310	.192	7.58
2	26	94		.133	.093	3.67
3	77.9	95		.025	.017	.69
4	98.5	95		.018	.011	.44
E-1	-4.7	90		.491	.282	11.10
2	15.5	94		.206	.136	5.36
3	63.6	96		.036	.027	1.05
4	83.5	94		.021	.015	.58
F-1	.5	93		.392	.234	9.21
2	20.9	96		.165	.113	4.44
3	76.6	96		.026	.019	.73
4	100.6	93		.016	.010	.38

Table D7. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 670 NB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
G-1	17	96		.194	.129	5.10
2	49.7	98		.056	.042	1.67
3	86.3	96		.022	.015	.58
4	88.8	94		.019	.013	.51
H-1	10.7	91		.255	.163	6.42
2	34.8	92		.095	.069	2.72
3	68.7	93		.030	.022	.88
4	101.8	92		.015	.009	.36
I-1	5.8	95		.317	.195	7.71
2	43.8	93		.068	.051	2.00
3	91.2	93		.019	.012	.48
4	96.2	94		.018	.012	.45
J-1	8.6	94		.280	.177	6.96
2	43.1	93		.070	.052	2.05
3	88.8	94		.020	.013	.53
4	89.9	94		.020	.013	.52
K-1	12.6	96		.238	.154	6.06
2	75.6	96		.027	.020	.78
3	91.6	96		.020	.014	.54
4	86.4	92		.020	.013	.52
L-1	8.8	93		.277	.175	6.90
2	39.7	91		.078	.058	2.27
3	60.6	90		.036	.027	1.08
4	85	94		.021	.015	.58
P1-1	3.3	84		.335	.205	8.08
2	10.6	88		.247	.158	6.25
3	13.2	91		.223	.145	5.73
4	18.1	90		.180	.121	4.79
P2-1	20.9	87		.159	.109	4.28
2	53	90		.046	.035	1.37
3	72.9	92		.026	.019	.74
4	81.8	89		.019	.013	.51

Table D7. Predicted Chloride Content Values for  
Virginia Bridge VA I81 over 670 NB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
P3-1	5.5	90		.308	.191	7.54
2	12.1	91		.233	.151	5.96
3	19.5	92		.171	.116	4.58
4	32.3	90		.101	.073	2.87
B1-1	13.2	88		.221	.144	5.69
2	87.9	92		.019	.012	.49
3	72	90		.025	.018	.72
4	80.3	90		.021	.014	.55
B2-1	31.4	89		.112	.080	3.16
2	82.7	90		.022	.015	.60
3	91.8	94		.021	.014	.55
4	96.3	96		.020	.014	.54
B3-1	3.5	88		.339	.207	8.17
2	31.8	86		.108	.078	3.07
3	55.3	90		.048	.036	1.42
4	69.2	91		.032	.023	.92
C1-1	-10.9	94		.617	.340	13.39
2	-4	86		.460	.267	10.52
3	5.3	90		.317	.195	7.71
4	14.4	90		.219	.143	5.66
C2-1	28.4	86		.124	.088	3.45
2	16.5	86		.199	.132	5.21
3	25.6	89		.140	.098	3.85
4	26	90		.139	.097	3.81

Table D8. Predicted Chloride Content Values for  
Florida Bridge FL 206

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Potentiometric Titration	
		Temperature (°F)	Calculated Chloride (%Cl-)	Predicted Values (%Cl-)	(lb/cu yd)
PC-1	-21.1	83	.828	.432	17.04
2	-19.1	85	.763	.404	15.94
3	-9.1	81	.502	.286	11.29
4	-4.4	81	.413	.244	9.61
5	-3.6	83	.401	.238	9.37
6	7	82	.258	.165	6.49
PC2-1	2	82	.317	.196	7.72
2	14.6	81	.188	.126	4.97
3	42.4	82	.062	.047	1.84
4	48.5	80	.048	.036	1.43
5	62.5	83	.030	.022	.88
6	85.9	82	.014	.008	.31
PO1-1	-7.2	81	.464	.268	10.58
2	-1.1	87	.364	.220	8.65
3	9.3	86	.237	.153	6.05
4	26.1	83	.119	.085	3.34
5	47.5	82	.051	.039	1.52
6	77.7	84	.019	.013	.50
PO2-1	46.7	83	.053	.040	1.59
2	64.7	85	.029	.021	.85
3	101.2	85	.012	.006	.22
4	111.7	82	.008	.002	.07
5	104	81	.008	.002	.09
P5-1	-22.7	84	.883	.455	17.95
2	-13.7	84	.610	.336	13.26
3	-6.9	86	.463	.268	10.57
4	2.4	85	.316	.195	7.70
5	20.8	82	.149	.103	4.06
P6-1	-1.6	80	.369	.222	8.76
2	-.7	80	.356	.215	8.49
3	LOST	LOST	LOST	LOST	LOST
4	36	80	.080	.059	2.34
5	59.9	85	.035	.026	1.04

Table D8. Predicted Chloride Content Values for  
Florida Bridge FL 206 (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl-)	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl-)	(lb/cu yd)
C1-1	39.4	83		.072	.054	2.12
2	58.2	84		.037	.028	1.09
3	66.7	82		.026	.019	.75
4	75.1	85		.022	.015	.60
5	83.2	83		.016	.010	.40
B1-1	68.1	82		.025	.018	.71
2	75.8	83		.020	.014	.54
3	90.8	85		.015	.009	.34
4	108.1	83		.009	.003	.13
5	113.8	83		.008	.002	.09
B2-1	55.7	81		.038	.029	1.13
2	71	81		.022	.015	.61
3	85.1	83		.015	.009	.37
4	101.7	82		.010	.004	.15
DA-1	74	90		.028	.020	.79
2	91.4	87		.017	.010	.41
3	97.5	91		.017	.011	.43
4	98	89		.016	.010	.38
DB-1	77.3	90		.025	.018	.72
2	92.1	92		.020	.013	.52
3	96.7	92		.018	.012	.46
4	98.5	89		.016	.010	.38
DC-1	79.3	91		.025	.018	.70
2	LOST			LOST	LOST	LOST
3	94.9	91		.018	.012	.46
4	97.6	92		.018	.012	.46
5	98.4	92		.018	.011	.45
DD-1	75.5	95		.030	.022	.86
2	82.9	94		.025	.018	.69
3	92.8	91		.019	.012	.49
4	99.2	93		.010	.012	.46

Table D9. Predicted Chloride Content Values for  
Florida Bridge FL 295 EB

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Potentiometric Titration	
		Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
P1-1	15.3	81	.188	.126	4.95
2	28.6	78	.107	.077	3.02
3	41.2	80	.065	.048	1.91
4	53.8	82	.041	.031	1.21
5	56.6	81	.036	.027	1.06
P2-1	68.5	83	.025	.018	.69
2	81.3	83	.017	.010	.41
3	89.2	81	.012	.006	.24
4	95.7	80	.010	.003	.14
P3-1	11	83	.226	.147	5.79
2	25.2	81	.125	.088	3.47
3	39.8	84	.071	.053	2.09
4	53.8	81	.040	.030	1.19
5	57.5	82	.035	.027	1.05
P4-1	66.6	82	.026	.018	.73
2	79.7	85	.019	.012	.49
3	96.6	84	.012	.006	.23
4	101.7	82	.009	.003	.13
B1-1	91.6	83	.013	.007	.26
2	104	84	.010	.004	.17
3	113.9	84	.009	.003	.11
4	118.5	85	.009	.003	.12
B2-1	81.3	81	.015	.009	.36
2	100.7	82	.010	.004	.14
3	116.1	83	.008	.002	.07
4	120.3	80	.006	0	0

Table D9. Predicted Chloride Content Values for  
Florida Bridge FL 295 EB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric Titration	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
C1-1	85	82	.014	.008	.32
2	106.8	83	.009	.003	.12
3	109.7	83	.009	.003	.10
4	114	83	.008	.002	.08
C2-1	93.9	85	.013	.007	.28
2	103.4	83	.010	.004	.15
3	108.4	84	.010	.004	.14
4	114.9	84	.009	.003	.10
C3-1	76.5	84	.020	.013	.52
2	90.6	82	.012	.006	.25
3	104.9	83	.010	.003	.14
4	106.6	85	.011	.004	.18
C4-1	81	85	.018	.012	.46
2	102.9	86	.012	.006	.23
3	114.5	82	.008	.001	.05
4	114.4	80	.006	0	0
C5-1	78.6	86	.020	.013	.53
2	101.8	81	.009	.003	.11
3	117.5	78	.005	0	0
4	121.5	78	.004	0	0
C6-1	83.5	84	.016	.010	.40
2	104.4	83	.010	.004	.14
3	113.4	84	.009	.003	.11
4	121.2	78	.004	0	0

Table D10. Predicted Chloride Content Values for  
Florida Bridge FL 312

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
P1-1	-10.1	85		.509	.290	11.43
2	-.3	83		.341	.208	8.20
3	9.7	84		.228	.148	5.85
4	17.5	84		.167	.114	4.48
5	29.7	83		.103	.074	2.92
6	62.8	84		.031	.023	.90
P2-1	7.9	84		.245	.158	6.22
2	18	85		.164	.112	4.42
3	24.1	86		.130	.091	3.60
4	35.9	83		.081	.060	2.35
5	52.3	84		.044	.034	1.32
6	76.8	84		.020	.014	.53
P3-1	21.9	85		.141	.098	3.87
2	36	85		.082	.060	2.38
3	57.2	83		.037	.028	1.09
4	84.1	82		.015	.009	.35
5	95.3	84		.013	.007	.26
6	96.5	84		.012	.006	.25
F1-1	-22	83		.826	.431	17.00
2	-18.9	85		.729	.389	15.35
3	-16.7	85		.666	.362	14.26
4	-.7	86		.348	.212	8.35
5	2.4	87		.308	.191	7.53
6	16.9	86		.172	.117	4.60
F2-1	-29.9	86		1.144	.562	22.16
2	-22.6	87		.849	.441	17.39
3	-9.8	83		.502	.286	11.29
4	-9	83		.486	.279	10.99
5	-4	87		.399	.237	9.34
6	11.7	86		.212	.139	5.49
B1-1	99.1	88		.015	.009	.35
2	115.3	90		.013	.007	.28
3	118.6	86		.010	.004	.16
4	110.7	88		.013	.007	.26

Table D10. Predicted Chloride Content Values for  
Florida Bridge FL 312 (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
B1A-1	95.5	91		.018	.012	.46
2	108.6	91		.015	.009	.34
3	119.4	93		.015	.008	.33
4	121.6	92		.014	.008	.30
C1-1	54.5	87		.048	.036	1.43
2	60	91		.042	.032	1.26
3	62.8	90		.038	.029	1.13
4	74.1	90		.028	.020	.80
5	86.9	93		.022	.016	.61
6	96.9	94		.019	.013	.51
C2-1	74.4	91		.028	.021	.81
2	77.2	93		.027	.020	.79
3	84.2	93		.024	.017	.65
4	90	91		.020	.013	.52
DA-1	72.6	93		.031	.023	.89
2	80.9	91		.024	.017	.67
3	95.5	90		.017	.011	.43
4	94.8	93		.019	.013	.51
DB-1	73.5	91		.029	.021	.83
2	86	89		.020	.014	.54
3	93.5	89		.017	.011	.43
4	94.1	89		.017	.011	.43

Table D11. Predicted Chloride Content Values for  
Wisconsin Bridge WI 243 WB

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Titration Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
A-1	-6.3	87	.521	.296	11.65
2	11.8	87	.244	.157	6.19
3	23	88	.155	.106	4.19
4	42.5	88	.072	.053	2.11
5	72.4	89	.027	.019	.76
B-1	-8.8	90	.581	.323	12.74
2	-1.8	91	.434	.254	10.01
3	18	92	.192	.128	5.05
4	46.6	90	.063	.047	1.86
5	38.9	88	.082	.061	2.39
C-1	-16.1	92	.792	.417	16.43
2	-3.9	89	.472	.273	10.74
3	22.9	91	.157	.108	4.25
4	43.9	94	.072	.053	2.11
5	70.3	95	.032	.024	.94
D-1	-12.9	89	.690	.372	14.68
2	1.1	92	.385	.230	9.07
3	26	91	.139	.097	3.82
4	51.1	90	.054	.040	1.59
5	32.4	83	.103	.074	2.93
E-1	-11	91	.638	.349	13.76
2	5	91	.327	.201	7.91
3	19.9	89	.176	.119	4.69
4	54.1	89	.048	.036	1.42
5	55.7	90	.046	.034	1.36
F-1	-3.1	92	.458	.266	10.48
2	5.1	92	.326	.200	7.89
3	18.9	90	.184	.124	4.87
4	40.9	94	.080	.059	2.33
5	42.3	85	.070	.053	2.07
G-1	-6.1	90	.519	.294	11.61
2	5.6	94	.320	.197	7.78
3	17.7	94	.196	.130	5.13
4	38.2	94	.088	.065	2.55
5	48.8	93	.060	.045	1.78

Table D11. Predicted Chloride Content Values for  
Wisconsin Bridge WI 243 WB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric Titration	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (lb/cu yd)
H-1	-8.3	93	.571	.319	12.56
2	4.1	92	.340	.207	8.17
3	18.2	95	.192	.128	5.06
4	37.8	90	.087	.064	2.52
5	45.6	87	.063	.047	1.87
I-1	-16.3	94	.800	.420	16.56
2	-6.5	94	.530	.300	11.81
3	11.4	90	.250	.160	6.32
4	37.4	91	.089	.065	2.57
5	61.6	92	.039	.029	1.15
J-1	-11.3	93	.647	.353	13.93
2	-2	90	.437	.255	10.07
3	11.8	92	.242	.156	6.15
4	40.9	95	.082	.061	2.39
5	50.3	91	.058	.043	1.71
K-1	-14.5	91	.694	.374	14.74
2	5.1	87	.312	.193	7.62
3	11.3	92	.247	.159	6.25
4	32.7	93	.109	.078	3.09
5	58.9	90	.043	.032	1.27
L-1	-9.9	90	.575	.321	12.64
2	-1.8	90	.415	.245	9.65
3	9.4	90	.265	.168	6.63
4	20.4	93	.174	.117	4.63
5	47.8	88	.061	.046	1.80

Table D11. Predicted Chloride Content Values for  
Wisconsin Bridge WI 243 WB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl-)	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl-)	(lb/cu yd)
A1-1	52.7	99		.058	.043	1.71
2	63	99		.043	.032	1.26
3	72.3	104		.037	.027	1.08
4	68.7	102		.038	.029	1.14
A2-1	39.1	102		.092	.067	2.65
2	56.2	104		.055	.041	1.62
3	73.2	105		.036	.027	1.08
4	75.7	102		.033	.024	.96
PC1-1	41.2	97		.083	.061	2.41
2	58.1	103		.051	.039	1.52
3	75.5	104		.034	.026	1.01
4	55.7	102		.054	.041	1.61
PC2-1	47.6	96		.066	.049	1.94
2	57.2	100		.051	.038	1.51
3	59	98		.047	.035	1.40
4	60.9	101		.046	.035	1.38
PO1-1	40.3	93		.079	.059	2.31
2	56.8	101		.049	.037	1.46
3	59.2	102		.046	.035	1.38
4	64.3	103		.041	.031	1.22
PO2-1	51.4	97		.056	.042	1.67
2	76.3	97		.028	.021	.81
3	70.9	96		.031	.023	.92
4	39.6	91		.081	.060	2.35

Table D12. Predicted Chloride Content Values for  
Wisconsin Bridge WI 245 WB

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric Titration	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
A-1	-14.4	91	.673	.365	14.37
2	.2	87	.371	.223	8.79
3	1.4	90	.355	.215	8.48
4	15	90	.207	.137	5.38
5	31.5	92	.111	.079	3.13
B-1	14.4	83	.207	.137	5.39
2	16.8	88	.192	.128	5.04
3	12.9	92	.226	.147	5.80
4	20	90	.170	.116	4.55
5	49.3	92	.059	.044	1.75
C-1	-7.2	95	.505	.288	11.36
2	.3	97	.376	.225	8.88
3	12.7	94	.229	.149	5.87
4	27.8	95	.129	.091	3.58
5	58.9	95	.045	.034	1.35
D-1	-13.6	96	.718	.385	15.16
2	10.3	88	.259	.165	6.51
3	13.2	94	.233	.151	5.96
4	37	95	.092	.067	2.64
5	65.8	96	.036	.027	1.07
E-1	-7.3	100	.552	.310	12.23
2	3.1	94	.354	.215	8.46
3	21.5	97	.168	.114	4.51
4	49	96	.060	.045	1.79
5	67.9	95	.034	.025	.99
F-1	-7.3	98	.551	.310	12.20
2	7.1	90	.297	.186	7.31
3	18.9	96	.186	.125	4.92
4	28.4	88	.123	.087	3.43
5	61.6	94	.040	.030	1.17
G-1	-14.9	91	.756	.401	15.81
2	-1.1	91	.421	.248	9.76
3	6.6	96	.292	.183	7.21
4	26.2	94	.138	.096	3.80
5	49.7	95	.058	.044	1.73

Table D12. Predicted Chloride Content Values for  
Wisconsin Bridge WI 245 WB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
H-1	-14	90		.661	.360	14.17
2	-4.9	92		.459	.266	10.49
3	9.2	89		.260	.165	6.52
4	47.2	90		.062	.047	1.84
5	66.8	93		.035	.026	1.03
I-1	-4.6	89		.451	.263	10.35
2	-1	93		.393	.234	9.23
3	12.3	93		.232	.151	5.93
4	31.5	88		.108	.078	3.07
5	51	91		.055	.042	1.64
J-1	-9	90		.540	.304	12.00
2	.9	92		.364	.219	8.65
3	16.4	96		.200	.132	5.22
4	36.6	97		.095	.069	2.73
5	57.8	94		.046	.035	1.37
K-1	-14.7	95		.683	.369	14.56
2	-4.5	95		.453	.263	10.39
3	8.8	94		.267	.169	6.68
4	23.5	94		.151	.104	4.11
5	36	86		.090	.066	2.60
L-1	-9.5	94		.553	.311	12.25
2	5.7	93		.301	.187	7.39
3	19.7	91		.173	.117	4.61
4	45.8	91		.066	.049	1.94
5	72.4	93		.030	.022	.88

Table D12. Predicted Chloride Content Values for  
Wisconsin Bridge WI 245 WB (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe Reaction		Calculated Chloride (%Cl <sup>-</sup> )	Potentiometric Titration Predicted Values	
		Temperature (°F)			(%Cl <sup>-</sup> )	(lb/cu yd)
PO-1	3.5	87		.369	.222	8.76
2	5.7	91		.341	.208	8.19
3	18.4	89		.205	.136	5.35
4	34.6	88		.110	.079	3.10
5	62.6	86		.039	.030	1.17
PO2-1	39.9	88		.090	.066	2.59
2	58.3	88		.047	.035	1.40
3	60.5	88		.044	.033	1.30
4	47.6	87		.067	.050	1.98
5	68.7	87		.033	.025	.97
PC1-1	-11.2	89		.669	.363	14.31
2	1.5	87		.400	.238	9.37
3	8.3	88		.305	.190	7.48
4	28.4	84		.136	.095	3.75
5	36.9	91		.102	.074	2.91
6	65.7	92		.039	.030	1.17
PC2-1	25.4	95		.160	.110	4.32
2	35.8	88		.171	.116	4.57
3	53.7	93		.058	.044	1.72
4	60.9	88		.043	.032	1.28
5	62.7	91		.042	.032	1.26
A1-1	49.4	97		.069	.052	2.04
2	64.3	100		.046	.035	1.37
3	61.9	98		.048	.036	1.42
4	47.8	91		.069	.052	2.04
5	67.5	102		.040	.030	1.19
A2-1	23.1	95		.162	.111	4.36
2	34.4	102		.110	.079	3.10
3	59.2	103		.050	.038	1.50
4	64.2	101		.043	.032	1.28
5	78	97		.029	.021	.84

Table D13. Predicted Chloride Content Values for  
Wisconsin Bridge WI 252

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric Titration	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
A-1	-18.8	88	.785	.414	16.32
2	-7	84	.479	.276	10.86
3	-4.9	91	.443	.258	10.19
4	7.5	86	.264	.168	6.61
5	28.1	80	.111	.079	3.13
6	42.1	84	.066	.049	1.94
B-1	-26.9	89	1.102	.546	21.50
2	-16.6	89	.717	.384	15.15
3	-5.6	90	.455	.264	10.42
4	5	88	.294	.183	7.23
5	19	88	.167	.113	4.47
6	46.4	87	.058	.043	1.71
C-1	-12	86	.591	.328	12.92
2	-10.4	92	.556	.312	12.30
3	1.5	92	.341	.208	8.21
4	7.5	86	.264	.168	6.61
5	32.2	84	.097	.070	2.77
6	49.2	86	.052	.039	1.54
D-1	-20.4	89	.840	.437	17.24
2	-17.9	89	.757	.402	15.83
3	-12.4	93	.605	.334	13.17
4	-1.2	91	.381	.228	8.98
5	11.7	88	.223	.146	5.75
6	43.5	92	.067	.050	1.98
E-1	-21.2	89	.869	.449	17.72
2	-9.5	91	.535	.302	11.92
3	-4.6	92	.438	.256	10.10
4	6.2	92	.282	.177	6.99
5	27.4	91	.121	.086	3.39
6	57.6	90	.041	.031	1.22
F-1	-19.3	94	.806	.423	16.66
2	-11	90	.569	.318	12.53
3	-1.1	91	.379	.227	8.95
4	13.5	99	.215	.141	5.55
5	26.8	91	.124	.088	3.46
6	44.7	87	.061	.046	1.82

Table D13. Predicted Chloride Content Values for  
Wisconsin Bridge WI 252 (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Titration Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
G-1	-25.6	93	1.046	.523	20.61
2	-22.3	92	.911	.467	18.42
3	-15.5	87	.684	.370	14.57
4	4.1	91	.306	.190	7.50
5	14.4	91	.202	.134	5.28
6	44.7	86	.061	.046	1.80
H-1	-18.1	87	.762	.404	15.92
2	-9.7	87	.537	.303	11.95
3	-3.8	92	.424	.249	9.83
4	-.3	90	.366	.221	8.70
5	18	90	.175	.118	4.65
6	50.9	92	.052	.039	1.56
I-1	-19.7	91	.817	.428	16.86
2	-15.6	92	.690	.372	14.67
3	.6	90	.353	.214	8.44
4	5.2	93	.294	.184	7.25
5	24.7	93	.136	.095	3.75
6	55.1	92	.046	.034	1.36
J-1	-16.9	87	.725	.388	15.28
2	-15.2	88	.676	.366	14.43
3	-7	88	.481	.277	10.91
4	2.2	88	.329	.202	7.96
5	13.6	88	.207	.137	5.38
6	40.8	89	.072	.054	2.12
K-1	-17.3	91	.740	.394	15.54
2	-10.6	88	.558	.313	12.34
3	-1.5	90	.385	.230	9.06
4	16.6	89	.184	.123	4.87
5	28.7	94	.117	.083	3.29
6	53.8	88	.045	.034	1.34
L-1	-18.6	88	.779	.411	16.21
2	-11.7	89	.585	.325	12.82
3	-9.8	88	.540	.305	12.00
4	1.1	89	.345	.210	8.28
5	12.2	88	.219	.143	5.65
6	36.6	91	.086	.063	2.48

Table D13. Predicted Chloride Content Values for  
Wisconsin Bridge WI 252 (Continued)

Sample Number	Reading (M-Volts)	Specific Ion Probe		Potentiometric Titration	
		Reaction Temperature (°F)	Calculated Chloride (%Cl <sup>-</sup> )	Predicted Values (%Cl <sup>-</sup> )	(lb/cu yd)
P1-1	- .6	98	.420	.247	9.74
2	3.3	103	.361	.218	8.60
3	18	99	.200	.133	5.23
4	34.6	101	.108	.078	3.07
5	44.5	104	.079	.058	2.30
P2-1	26.8	104	.146	.101	3.98
2	39.4	102	.092	.067	2.65
3	49.4	104	.068	.050	1.99
4	58.1	99	.050	.037	1.47
5	59.1	103	.050	.038	1.50
BW1-1	-23.8	93	1.051	.525	20.69
2	-7.2	91	.533	.301	11.87
3	7.1	88	.297	.185	7.31
4	-1.1	92	.417	.246	9.69
5	.6	93	.390	.232	9.16
BW2-1	-21.4	94	.953	.485	19.11
2	-14	93	.704	.378	14.92
3	-5.3	93	.494	.283	11.16
4	-1.5	90	.422	.248	9.79
5	2.2	93	.365	.220	8.69
A1B-1	-7.8	95	.548	.308	12.15
2	12.6	97	.244	.157	6.19
3	26.1	100	.147	.102	4.02
4	38.4	95	.092	.067	2.64
5	55.4	94	.051	.039	1.53
A1Cl-1	-19.9	93	.896	.461	18.16
2	-10.7	95	.617	.340	13.38
3	-2.4	94	.440	.257	10.14
4	11.9	94	.249	.160	6.30
5	30	94	.124	.088	3.46

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