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Process improvement to the anodize line through design of experiments

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Process Improvement to the Anodize Line through Design of Experiments

by

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ABSTRACT

The goal of this project is to analyze the anodize line and make process improvements which directly affect the product, the results being reduced defects, lower variability in the process and product, faster cycle time, reduced costs, and higher profits. Possible defect conditions were identified, tracked, and analyzed in order to determine the greatest problem. After recognizing a prime improvement opportunity, a design of experiments was conducted with the purpose of showing relationships between key process parameters and product characteristics. Finally, recommendations were made to raise the quality level of the anodize line, with the information that was gained throughout the entire project and design of experiments.

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INTRODUCTION

The introduction section is to introduce the reader to the anodizing process and Design of Experiments.

Anodizing

Definition

Anodizing or anodic oxidation is an electrolytic process for oxidizing aluminum to produce an improved surface quality. "Aluminum without some surface treatment is like good wood without varnish. The wood is strong and may make a good structural member, but it does not look as good as it could, and it is susceptible to wear and weather." [1] Anodizing is like varnishing in the example above. It adds to the quality of the aluminum by making it more resistant to the environment.

Exposing aluminum to air produces a thin oxide film, that is 0.1-0.4 x 10^{-6} inches (0.25-1.0 x 10^{-2} μm) thick. Anodizing will produce a thicker oxide coating than the film formed naturally in air. With a thicker coating, aluminum has improved physical and chemical properties which allows for expanded applications. Some of the improved properties include excellent resistance to marine and atmospheric corrosion, abrasion resistance, electrical insulation, and the ability to be colored. [2]

Anodizing occurs when an electrochemical conversion occurs from metallic aluminum to aluminum oxide, Al_2O_3. This conversion requires a source of direct current passing through a suitable acid electrolyte which will produce oxygen ions. The most commonly used electrolyte is a dilute sulfuric acid solution, but chromic acid, oxalic acid, phosphoric acid plus additives, and other specialized electrolytes with limited applications are also possibilities.

Applications[3]

Anodic coatings are widely applied to aluminum because of its unique response to anodizing. There are many advantages gained from anodizing aluminum. The following is a list of principal functions for anodizing.

- Undercoat for organic coatings, electroplated metallic coatings, and solid lubricants
- Corrosion resistant coating
- Coloring (a wide range of colors, for example, black, bronze, purple, orange)
- Antimark applications
- Heat reflection and radiant heat absorption
- Wear resistance and lubrication
- Electrical resistance
- Abrasion resistance
- Thermal resistance
- Marine resistance

To get an even better idea of the applications for anodized aluminum, Table 1 below lists some of the more important applications in present-day industrial practice.

Table 1. Industrial Applications of Anodized Aluminum[4]

Industry	Application
Building	Decoration, protection of exterior building components, structural members, storefronts, entrance ways, window frames, ceiling panels, handrails, hardware, telephone booths.
Transportation	Auto: Headlight bezels, grills, window frames, garish moldings, brake pistons. Air: Aircraft instrument panels, landing gear, propellers, fuel pumps, wing skins, structural components, rivets, instruction plates, trim.
Consumer Durable Goods	Refrigerator: trim, shelves, evaporators, appliance trim, cooking utensil covers, baking pans, name plates, furniture, giftware, costume jewelry, firearm/military components.
Lighting	Reflectors for highway and stadium lights, indoor lighting fixtures.
Electrical	Capacitors, insulated wire and strip conductors.
Other	Machine Components

The Process

Understanding the anodizing process is simplified by looking at the flow of the process. A flow chart is shown below in figure 1.

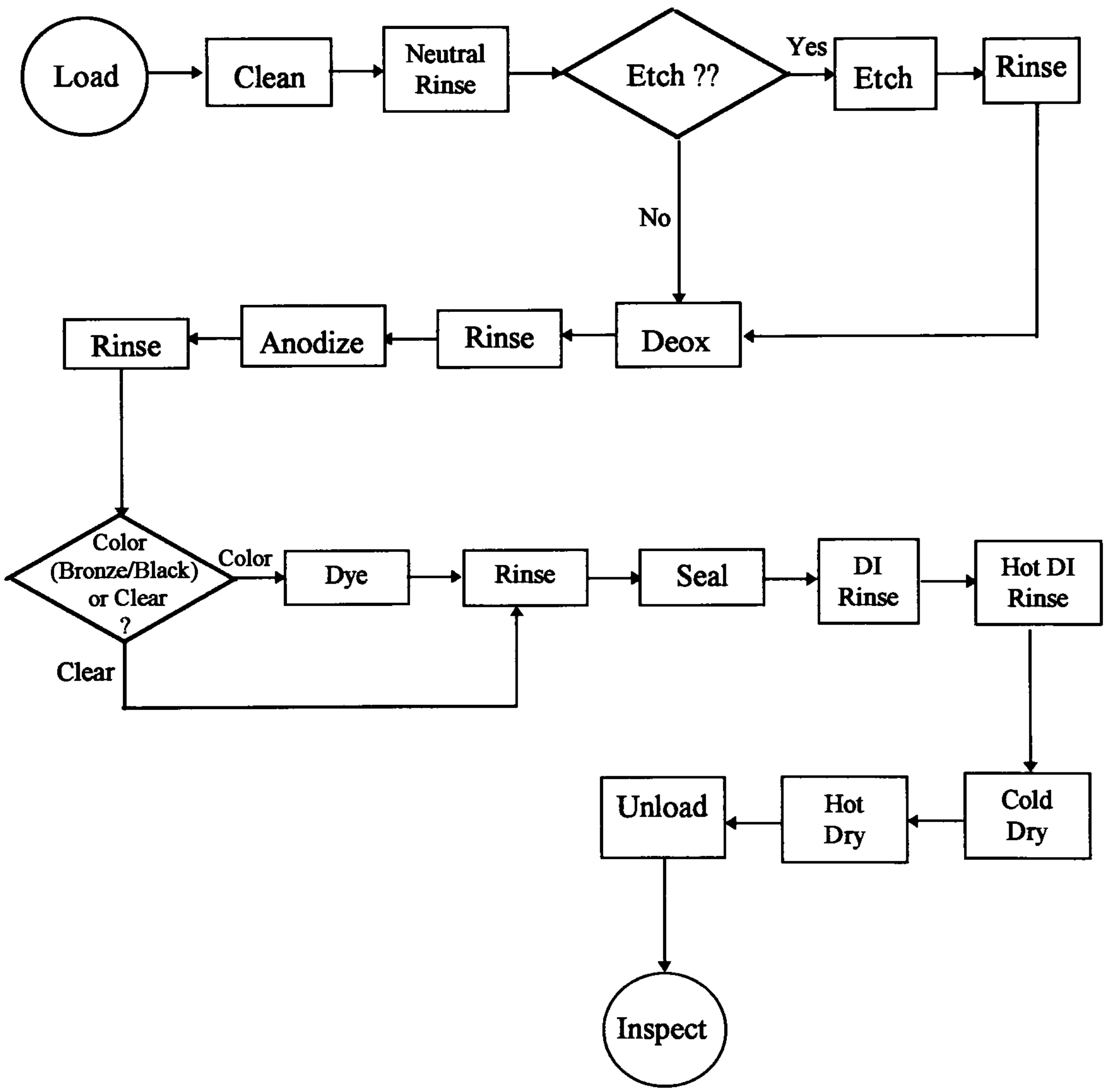


Figure 1. Anodize Process Flow

Types of Anodic Coatings

Anodic coatings are classified as barrier or porous depending on the solvent action of the electrolyte on the naturally occurring oxide layer. Deciding on the type of coating is based on the application of the part being anodized.

Barrier-type

Electrolytes with little or no capacity to dissolve the oxide form barrier-type coatings. These type of coatings are thin (less than one ten thousandth of an inch), compact, nonporous, and electrically resistant. In addition, with suitable etching conditions, high capacitance is obtainable. Sodium borate/boric acid electrolytes are examples of this type of film producer. Electrical capacitors have barrier-type layer.

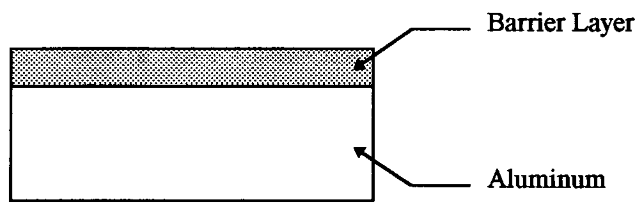


Figure 2. A cross-section of a barrier-type coating

Porous-type

Porous type of coatings are formed in an electrolyte with high solvent action on the natural oxide. The formed film consists of a porous outer portion and a thin barrier portion adjacent to the metal. Porous-type coatings have wide ranges of applications ranging from decorative purposes to protective, wear resistant purposes. [5]

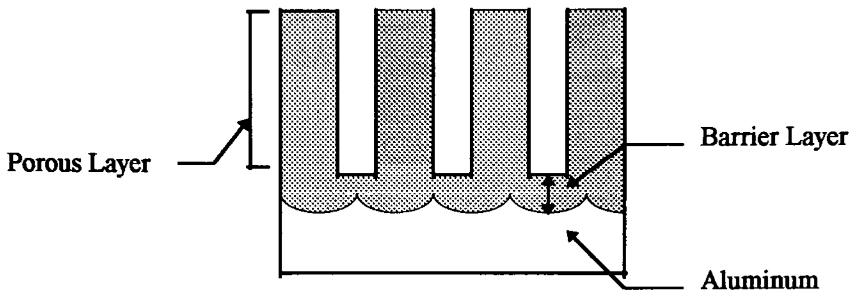


Figure 3. A cross-section of a porous type coating

Coating Structure of the Porous-type

The structure of the anodic coating is a group of hexagonal-shaped oxide cells each having a central pore that extends to a thin compact barrier layer of oxide. The barrier layer is continuously transformed into the porous form during the anodizing process. The cell size equals twice the cell wall thickness plus the central pore diameter. There are approximately a million cells per square inch.[6] The cell structure of the oxide layer formed from sulfuric, oxalic, chromic, and phosphoric acids are similar, but vary in dimensions (see Table 2).

Table 2. Pore Diameter and Cell Wall Thickness of Several Oxide Coatings[7]

Electrolyte	Pore Diameter (Angstrom)	Wall Thickness (Angstrom/volt)
15% sulfuric acid, 50 °F	120	8.0
2% oxalic acid, 75 °F	170	9.7
3% chromic acid, 100 °F	240	10.9
4% phosphoric acid, 75 °F	330	10.0

The pore diameter is completely dependent on the type of electrolyte whereas the wall thickness is highly dependent on the applied voltage and slightly dependent on the electrolyte. Additionally, the coating thickness depends on the same two main factors: applied voltage and the electrolyte. For example sodium borate/boric acid electrolytes and 300-500 volts are conditions that produce a thin film with a thickness that is less than 0.0001 inch. A sulfuric acid solution and 12-24 volts are conditions that produce thicker films of up to 0.001 inch. Other factors affecting the thickness are the current density and time in the anodizing tank. [8]

Mechanism

The anodizing process is dissimilar to electroplating in the way that the coating forms. Porous-type anodic films start on the outside surface of the metal anode and progress inward, so that the last-formed coating is near the metal-coating interface and the first-formed layer is on the surface. By contrast, the metal being plated in the electroplating process is a conducting substrate acting as the cathode in the electrolytic cell. A metallic coating is deposited on the surface of the substrate and grows outwards. Furthermore, when anodizing, additional metallic materials are not being added to the aluminum, instead a conversion of the surface is occurring.

The mechanism of forming the barrier coatings is ionic. Aluminum ions combine with the oxygen ions of the electrolyte. The barrier thickness represents the distance though which the ions can penetrate the layer of oxide under the influence of the applied potential. Therefore the voltage is the driving force behind the ions, and determines the thickness of the barrier layer. For barrier-type coatings, a limiting thickness is reached and current flow ceases. Porous-type coatings do not reach a limiting thickness due to the solvent action of the electrolyte. However, a barrier layer with a thickness that is equal to fourteen times the applied voltage times a factor less than unity determined by the electrolyte will still exist between the metal and base of the pores for a porous film. [9]

Barrier Layer Thickness = (14)(Voltage)(Electrolyte Factor)

Theory of Design of Experiments

Definition

Design of experiments (DOE) is a systematic approach to experimentation that allows an efficient and effective effort towards improving the quality and productivity of a process. The goal of DOE is to understand the relationship between process parameters and product characteristics, save experimentation time, decrease scrap rates, decrease production times, decrease inventory, and save costs associated with each of these. [10]

Qualifications

DOE is a useful problem solving process in many different situations, for example: 1) there exists a part with high nonconformity or many defects, 2) there exists a process with high nonconformity, 3) a new machine, process or part is being implemented, or 4) a new machine is being purchased.[11]

A nonconformity is a departure from specification requirements. A defect is any variation of a required characteristic of the product or its parts, which is far enough removed from its target value to prevent the product from fulfilling the physical and functional requirements of the customer. [12] The quality of a product or process increases as the number of defects decrease. A measure of the quality of the process or product is a defect per unit (DPU).[13]

$$\text{DPU} = \frac{\text{\# of Defects found at Any Acceptance Point}}{\text{\# of Units (parts) processed through that Acceptance Point}}$$

The most common application of a DOE in the manufacturing area is that which deals with a part being produced at a high DPU (case 1). Other times a process is producing too many defects, regardless of the part being produced (case 2). In this situation a representative part of the process is chosen for the experiment, and the results of the experiment are related to all parts produced by the process. Case three addresses problems before they happen. By way of DOE, insight into a new process can be gained. Experimentation will teach how the variables of the process will affect the critical parameters of the parts being produced. Lastly, before purchasing a new machine a DOE is a good idea in order to test to see if the machine does what it is desired. By running a DOE, the machine can be tested for output, variability, ease of use, set-up time, and overall machine performance.

Basic General Procedure[14]

There are ten steps in a designed experiment. They are as follows:

1. Brainstorm
2. Design the experiment
3. Obtain materials and clean machine
4. Conduct experiment/collect data
5. Clean the data
6. Analyze the data
7. Interpret the results
8. Confirmation run
9. Write report
10. Present results

The first step of any design of experiment project is brainstorming. A team of experts should be gathered for a brainstorming session in order to discuss the problems associated with the process at hand. The team should consist of different skill levels including operators, maintenance, engineers, managers, and other experts. Several key questions need to be answered during the brainstorming step. These include:

- What is the project goal?
- What is the project objective?
- What are the outputs/responses of the process?
- What are the inputs/factors of the process?
- What are the levels of the inputs?
- Which inputs are inter-related to each other?
- What parts/material are going to be used for the experiment?
- How many parts can be produced during the experiment? [15]

The brainstorming has resulted in a list of responses (outputs), factors (inputs), and levels for the factors of the process. Having completed this first step, the list of factors and responses is used to design an efficient experiment, and create a Design of Experiment sheet, step 2.

There are several different experiments that may be chosen, but the goal is to select the most economical design that will render the most information about the process. See Appendix B for types of designs.

The third step is to obtain materials and clean the machine, which is pretty self-explanatory. Obtaining materials simply means ordering and receiving the desired number of parts for the experiment. Cleaning the machine means doing any necessary maintenance or adjustments to the machine before experimentation.

After brainstorming, designing the experiment, obtaining the parts, and preparing the machine, the experiment is run at the levels indicated on the Design of Experiments sheet (step 4). Parts need to be tagged, recorded, and measured for the response. If the response is quantitative data, then this is an easy task. On the contrary, if the response is attribute or qualitative data and requires judgment, then measuring the response is difficult and not a recommended practice. For example, it is simple to obtain a thickness value for an anodic coating using a permascope. If there were no measuring tools, assigning a thickness to each test sample by visual means would be impossible. To summarize, attribute data is not recommended for analysis, and should be replaced by quantitative data if possible.

Bad parts will be made during the DOE. The idea of a designed experiment is to change process parameters in order to induce changes in the final part. Both good and bad parts are expected to be made allowing one to see where the optimal settings are located.

Step five of the whole design of experiments process is cleaning the data. Checking the accuracy of the data is important to ensure that mistakes did not occur in the transmission of the data. The result of this step is a list of the factor settings of each experimental run and the resulting responses.

Now the data are ready to be analyzed and interpreted (steps 6 and 7) for two items: 1) relationships between factors and responses and 2) significant verses insignificant factors. An empirical equation, describing the relationship between the factors and the responses, is also obtained from the data analysis. This equation will be used to predict what the process will produce at various factor levels. The “true” functional relationship between the response and the factors, the mechanistic model, is often too complicated to allow parameter estimation, but it can be approximated by an empirical (polynomial) model.

Step eight is to conduct a confirmation run. By doing this, the results, theories, and suggested optimal settings attained from the design of experiment and are verified. Finally, the purpose of the two remaining steps, nine and ten, is to inform the team of the results so that the results in a written report and an oral presentation. A plan to keep the process under statistical control can be established at this point. [16]

JMP Analysis

JMP (Statistical Software for the Macintosh from SAS Institute Inc.) is a software package capable of performing the DOE analysis. The results to look at from the JMP output are the Summary of Fit, the Analysis of Variance (ANOVA), the Parameter Estimates, the Effect Test, the Lack of Fit, and the Leverage Plots.

Summary of Fit [17]

RSquare
RSquare Adj
Root Mean Square Error
Mean of Response
Observations (or Sum of Weights)

RSquare (R^2) is the coefficient of determination and measures the percent of the corrected total sum of the squares that is explained by all of the terms in the model (except for the intercept term). The equation that calculates R^2 is given by:

$$R^2 = \text{Model sum of squares/Corrected total sum of squares}$$

$$R^2 = \text{SS Model/SS C Total}$$

The R^2 value is constrained between 0 and 1. Multiplying R^2 by 100 yields the DOE Equation Prediction Rating. An RSquare value of 0.95-1.0 is desired. The higher the RSquare value the more "adequate" the model is.

RSquare Adj (R^2 Adjusted) adjusts RSquare to make it more comparable over models with different numbers of parameters by using degree of freedom in its computation. It is a calculation of mean squares instead of sums of squares and is calculated by

$$R^2\ Adj = 1 - \frac{Error\ Mean\ Square}{C\ Total\ Mean\ Square}$$

where, Error mean square is found in the ANOVA table found on page 17-19.

C Total mean square = C Total SS/C Total DF

(C Total SS and C Total DF found in the ANOVA table)

Root Mean Square Error (Root MSE) is an estimate of the standard deviation of errors about the fitted regression model (random error). The calculation for this value is:

$$Root\ Mean\ Square\ Error = \sqrt{ErrorMeanSquare}$$

A prediction with the least amount of variability is desired. Therefore a small Root MSE value is desirable.

The Mean of Response is simply the mean of the responses, calculated by:

$$Mean\ of\ Response = \frac{\sum_{i=1}^{n} y\mathrm{i}}{n}$$

where,

n = The number of experimental runs

y_i = The i^{th} response value

The number of experimental runs, n, equals the number of Observations (or sum of weights) used in the fit.

Analysis of Variance (ANOVA) [18]

Degrees of Freedom (Model, Error, Total)
Sum of Squares (Model, Error, Total)
Model Mean Square
Error Mean Square
F Ratio
Prob>F

"The analysis of variance is a means for partitioning the total variability of the observed response variables into various components which can be attributed to known sources." [101] The total variability is broken down into the experimental error variability and the model variability. The experimental error variability represents the variability within the groups of response values. The model variability is the variability due to changing the factor levels and it represents the variability across the three factor levels (high, medium, and low).

Notation for the ANOVA table:

n = The number of experimental runs

p = The number of model parameters

y_i = The i^{th} response value

$\bar{y}$ = The average of the n response values

$\hat{y}_i$ = The i^{th} response predicted from the model

Σ = The summation from i=1 to n (over all the responses)

Table 3. The ANOVA Table

Source of Variability	Degrees of Freedom	Sum of Squares	Mean Square
Model	p-1	$\Sigma(\hat{y}_i - \bar{y})^2$	$\Sigma(\hat{y}_i - \bar{y})^2/(p-1)$
Error	n-p	$\Sigma(y_i - \hat{y})^2$	$\Sigma(y_i - \hat{y})^2/(n-p)$
Total	(p-1)+(n-p) = n-1	$\Sigma(y_i - \bar{y})^2$ OR $\Sigma(\hat{y}_i - \bar{y})^2 + \Sigma(y_i - \hat{y})^2$	

- Source of Variability- indicates the specific component of variability.
- Degrees of Freedom- represents the number of independent pieces of information used to estimate the particular component of variability.
- Sum of Squares (SS)- is the numerical estimate of the component of variability (unadjusted for the degrees of freedom). It is the sum of squares of the difference between the fitted response and the actual response.
- Mean Square- is an estimate of the variability contribution from the corresponding source of variability after adjusting for the degrees of freedom.
- In addition the ANOVA table has the F Ratio and the Prob>F.

F Ratio- is the Model mean square divided by the Error mean Square.

$$F\ Ratio = \frac{Model\ Mean\ Square}{Error\ Mean\ Square}$$

It estimates the following quantity:

$$\frac{\text{Experimental variability + Factor variability}}{\text{Experimental variability}}$$

- The larger the F Ratio (the further it deviates from one in the positive direction), the more evidence there is of significant factor effects.
- The F Ratio is the "F Value" for the test statistic and the Prob>F value (the p-value) is the significance level which are used to test the following hypotheses:

H_o: No factors have an effect on the response or

$$\beta_1 = \beta_2 = \beta_3 = \beta_i = 0$$

H_a: At least one factor has an effect on the response or

$$\beta_1 \text{ or } \beta_2 \text{ or } \beta_3 \text{ or } \ldots\ \beta_i \neq 0$$

where the βs are the coefficients of the main effects in the equation that will result from the DOE analysis. See Appendix B for more information.

A "Prob>F" value < 0.05 indicates sufficient experimental evidence to reject the null hypothesis (H_o).

Parameter Estimates[19]

Term
Estimate
Std Error
t Ratio
Prob>|t|

- Term is the parameter in the model being estimated.
- The Estimate values are estimations of the coefficients of the model found by least squares. For example,

  $$Smut = \beta_0\ (\text{intercept}) + \beta_1\ (\text{free sulfuric}) + \beta_2\ (\text{pH}) + \beta_3\ (\text{t seal}) + \beta_4\ (\text{t DI}) + \varepsilon$$

  where, β_0, β_1, β_2, β_3, and β_4 are the parameter estimates, where β_1 estimates the free sulfuric effect, β_2 estimates the pH effect, etc.
- The Std Error (the standard error of the estimate) is the square root of the estimated variance of the parameter estimate and is used to quantify the uncertainty or variability in the parameter estimates. In other words, it is an estimate of the standard deviation of the distribution of the parameter estimate.

- The t Ratio (t value) is computed as follows:

$$t = Estimate/Std\ Error$$

The hypothesis that is being tested by the test statistic, t, is:

Ho: The parameter = 0 (model term insignificant)

Ha: The parameter $\neq$ 0 (model term is significant)

- If "Prob>|t|" $\leq$ 0.05, then reject the null hypothesis and assume the model term is significant.

Effect Test [20]

Source
Sum of Squares
F Ratio
Prob>F

The "Effect Test" provides the same information as the "Parameter Estimates". It is a type III statistic meaning it presents a partial partitioning of the model sum of squares. The individual sums of squares are said to be partial in that each sum of squares represents the amount of variability the corresponding model terms would explain if it was the last term entered into the model. [21]

- The F Ratio, "F Value", test statistic is:

$$F = \frac{\textit{Sum of Squares (type III)/DF}}{\textit{Error Mean Square}}$$

The following hypothesis test is constructed to determine model term significance.

Ho: The variability explained by the model term is insignificant

Ha: The variability explained by the model term is significant.

- If "Prob> F" ≤ 0.05, then reject the null hypothesis and assume the model term is significant.

Lack of Fit [22]

Source
Sums of Squares (Lack of Fit, Pure Error, Total)
Lack of Fit Mean Square
Pure Error Mean Square
F Ratio
Prob>F
Max RSq

The lack of fit analysis provides a breakdown of the error sum of squares. The error sums or squares is made up of two components of variability, lack of fit error and pure error. To separate the total sum of squares into the lack of fit and pure error components there are four steps.

1. For each distinct factor combination which is replicated, compute a standard deviation, s, or variance, s^2, from the response values. If there are k distinct factor settings with replication, then there will be k variances computed. These k variances represent k estimates of the experimental variability or pure error.
2. A "total" pure error sums of squares is computed as:

   Pure Error Sum of Squares $= (df_1)(s_1^2) + (df_2)(s_2^2) + (df_3)(s_3^2) + \ldots + (df_k)(s_k^2)$

   where, df_k = degree of freedom for k^{th} factor setting

   s_k^2 = estimated variance for k^{th} factor setting

3. The lack of fit sum of squares is obtained by subtracting the pure error sum of squares from the total error sum of squares.

   lack of fit sum of squares = (total error sum of squares) - (pure error sum of squares)

4. The degree of freedom associated with pure error and lack of fit are obtained from the degrees of freedom chart. (The pure error degrees of freedom can also be computed by summing the degrees of freedom for each individual variance estimate).

The pure error and lack of fit sums of squares are divided by their respective degrees of freedom to obtain the pure error mean square and a lack of fit mean square. The pure error mean square is an estimate of the pure error variance. However, the lack of fit mean square is an estimate of the sum of the "pure error" variance and a "bias" component. The bias component represents the bias or error associated with using an inappropriate model to describe the true relationship.

Testing the bias significance, which reflects the lack of fit, is done with the following hypothesis test. The F ratio tests that the lack of fit error is zero, and is calculated by:

F Ratio = (Lack of fit mean square)/(Pure error mean square)

The hypotheses for the test are as follows:

H_o: The model is adequate, no lack of fit

H_a: The model is inadequate, lack of fit

A “Prob>F” value ≤ 0.05 implies rejection of the null hypothesis or lack of fit.

A lack of fit indicates that additional parameters should be added to the model. The F ratio estimates

$$\frac{\text{Experimental variability + Model bias}}{\text{Experimental variability}}$$

The larger the F statistic, the more evidence there is of a bias due to an under-specified model.

Max RSq is the maximum R^2 that can be achieved by a model using only the variables in the model. It’s calculation is:

$$Max\ R^2 = 1 - \frac{SS\ (Pure\ Error)}{SS\ (Total\ for\ whole\ model)}.$$

Leverage Plots [23]

Leverage plots graphically illustrate the significant parameters and at which level will produce the most favorable response. Essentially the leverage plot is a graphical display of the Effect Test.

Dotted confidence curves on the plots indicate whether the test is significant at the 5% level by showing a confidence region for the line of fit. If the confidence region between the curves contains the horizontal line then the effect is not significant. If the curves cross the line, the effect is significant.

KEY PROCESS PARAMETERS

The anodizing process has many parameters that are important to the production of good parts. One of the hardest anodizing parameters to control is the aluminum quality from suppliers. Because anodic oxidation involves a conversion of the aluminum surface into an oxide coating, the alloy and its metallurgical structure have important effects on the characteristics of the finished surface. Differences in coatings arise with the purity of the aluminum, the type and quantity of alloying elements, type of mill product, different production lots, interchanged manufacturer lots, type of fabrication, or different temper/aging treatments. All of these factors have significant effects on the appearance and functional properties of the finished parts. [24]

Properties of anodic coatings that are affected by alloy composition include appearance (color, reflectance, and transparency), continuity (protectiveness), abrasion resistance, weight density, porosity, dielectric strength, and composition. As far as appearance, pure aluminum will produce the most transparent anodic coating of all its alloys. Clear anodized coatings (not dyed) could look opaque, gray, gold, tan, or brown depending on the major alloying element. [25]

The aluminum alloy system assigns a four-digit numerical designation to each grade. The numerical designations for the alloy and cast alloy and the suffix designations are in Appendix A.

Racking is another important factor in the production of good anodic coatings. [26] Sufficient electrical contact between the rack and the parts is necessary to ensure that current flows to the part during anodizing. Rack design and part placement on the rack are important. A good rack design will hold parts securely, conduct current adequately, and carry a full load without shielding. For the most part, racks are made of titanium but may also be made of aluminum. Aluminum racks require stripping after each use. Titanium racks last longer but are more expensive and require larger contact area because of their lower electrical conductivity. Part position must allow for good drainage and avoidance of air pockets.

Having racked the parts, processing commences and parts are moved from tank to tank. There are many tanks involved in the anodizing process (Figure 1). Furthermore, there are several factors of each tank like concentrations, times, temperatures, etc. that have considerable contributions to the final products.

Adequate cleaning is the first required tank process operation. Because many organic compounds will act to resist etching and anodizing steps, they need to be removed. Control of cleaner concentration, temperature, and oil accumulation are all necessary.

Following the cleaning is rinsing. Actually, thorough rinsing must follow each chemical step in the sequence of tanks. The requirements for the rinse tanks are clean, flowing water and an overflow lip. Rinsing may be single or multiple tank rinses and they may be spray or immersion tanks.[27]

Deoxidizing is the step to follow cleaning and rinsing. During this step an acid solution at an elevated temperature removes nonuniform oxide films and contaminants from the parts to be anodized, that could not be remove during the cleaning.

Etching is a step that may or may not be used. Its purpose is to remove the natural shine and provide a soft, matte, textured appearance. On average etching is a 3-5 minute process in a nominally five percent sodium hydroxide solution at 90-120 °F.

The anodizing tanks have many key process parameters, the first being the chemical concentration. In the anodizing tank, the sulfuric acid solution is controlled in industry at a nominal fifteen percent solution. It is important that the temperatures are held within a couple of degrees in order to produce consistent coating properties. Current flow is also recommended to be controlled in the range from 12-16 amperes/ft^2. However many plants operate at fixed voltages instead. Agitation is another factor essential to the process in order to provide a uniform solution temperature throughout the tank. Cathode location can have different effects on the thickness' of the oxide coatings. The closer the surfaces are to the cathode, the thicker the anodic coating will be.

When desirable, dying is carried out next. The most important parameters in the dye tank are the dye concentration, pH, and temperature. Agitation is needed to keep concentrations and temperatures uniform. Also, time is a mentionable variable. Longer immersion times will promote deeper dye penetration into the pores of the oxide coating. Contamination is one last parameter. Impurities such as aluminum, sulfates, and iron affect absorption characteristics and dye life.

The last important chemical tank is the seal tank. Without sealing parts, they become subject to lower corrosion resistance, staining, and bleeding. The important factors involved in the sealing process include time, pH, concentration of nickel and fluoride, temperature, agitation, and contaminants.

Table 4. Summarization of Key Process Parameters

Metal Quality
- Impurities
- Alloy Composition
- Processing
- Temper/aging treatment

Cleaning Tank
- Cleaner Concentration
- Temperature
- Oil Accumulation
- Time

Anodizing
- Sulfuric Acid Concentration
- Aluminum Concentration
- Temperature
- Current
- Agitation
- Cathode Location
- Time

Sealing
- pH
- Nickel Concentration
- Fluoride Concentration
- Temperature
- Agitation
- Contaminants
- Time

Racking
- Electrical Contact
- Rack Design
- Part Placement
- Rack Material

Rinse Tanks
- pH
- Temperature (for some tanks)
- Water Flow
- Impurities
- Time

Etching
- NaOH Concentration
- Temperature
- Time

Deoxidizing
- Acid Concentration
- Temperature
- Time

Dying
- Dye Concentration
- pH
- Temperature
- Agitation
- Contamination
- Time

DEFECTS

Because of the complexity of the anodizing process, there are many possibilities where defects can occur on the line. Different defects that occur include, lost contact, smut, overanodizing, white spots, bleed out, bent parts, crashed parts, burnt parts, and staining. Causes for these defects may be due to materials, manpower, methods, machines, or measurements.

The key process parameters of the anodizing process were discussed in the previous section. The possible defects that can occur when these parameters are not at their optimum are discussed next.

Poor racking or poor contact due to insufficient rack-contact area or loose contacts can cause iridescent appearance on clear parts, blue appearance on black dyed parts, powdery coatings, burning, and other problems. Operator's technique and proper rack design play an important role in producing good contact.

Cleaning was the next variable to the process that was considered. When the cleaner concentration is too low white spots or staining may result. A dried-on foam pattern may result if the temperature of the solution in the tank is too high. Overly vigorous agitation produces excessive foam which stays on the rack and parts.

In the deoxidizing tank, white spots, film, or "smut" result when all the contaminates are not removed from the surface of the parts. The same thing can happen if the etch does not remove all of the contaminating elements. Furthermore, if the etch solution temperature is too high, caustic burning results. Caustic burning is a non-uniform etch pattern that is a rejectable product condition.

Many factors are involved in the anodizing tank. Too high of a sulfuric acid concentration may cause smutty, overanodized parts or burnt parts, and too low of a sulfuric acid concentration may cause white spotted parts. The concentration of the aluminum in the tank is important for the conductivity that is necessary in the oxide film formation. Too high or too low a concentration can cause overanodizing or underanodizing respectfully. Extended time in the tank may result in smut on the parts. High anodizing temperatures will produce a softer coating, leading to dye bleeding. Low current could cause white spots or dye bleeding as well. Air agitation is necessary to prevent part burning.

Generally, the dye tank is a low maintenance tank, and therefore tank life can be many years. As contaminants increase over many years, the dye "spoils" and this will cause defective colored parts. Black parts, for example, would have a blue tint. Concentrations and pH play a role in getting the right color too.

Sealing the parts is a place in the process where numerous things can and do go wrong. Low nickel concentrations and low pH leads to dye bleeding. High pH, nickel precipitation, and too much time yield smutty product. On the contrary, low temperatures, pH, and sealing time produce an inadequate seal.

Figures 4-12 graphically illustrate possible anodizing defects and the explanation for these defects.

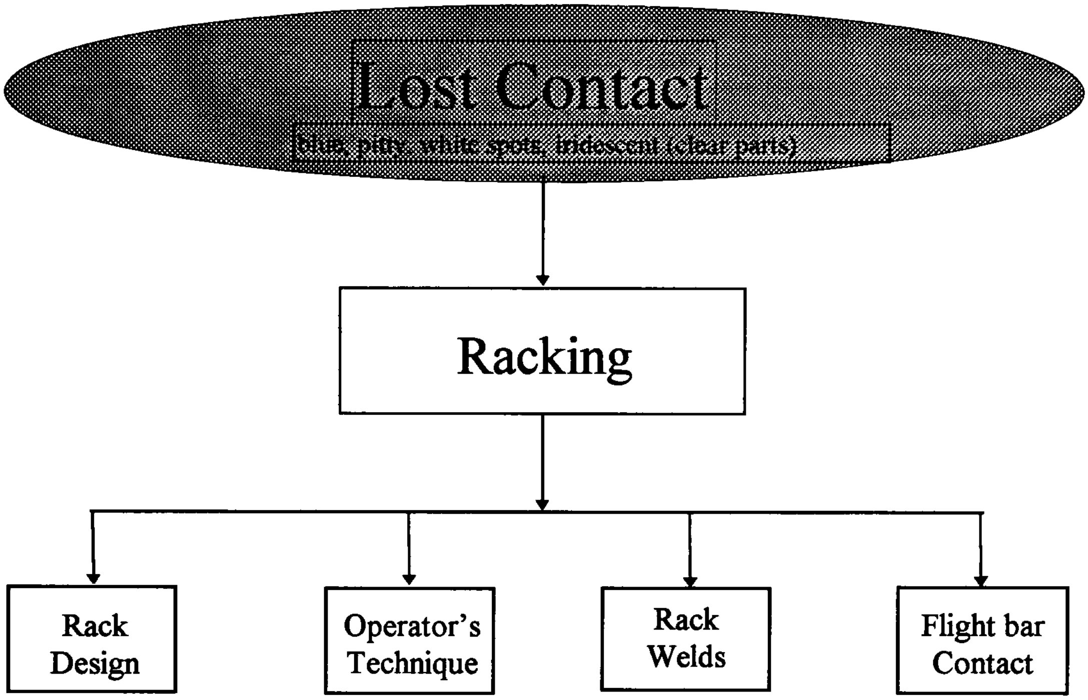


Figure 4. Causes for Lost Contact

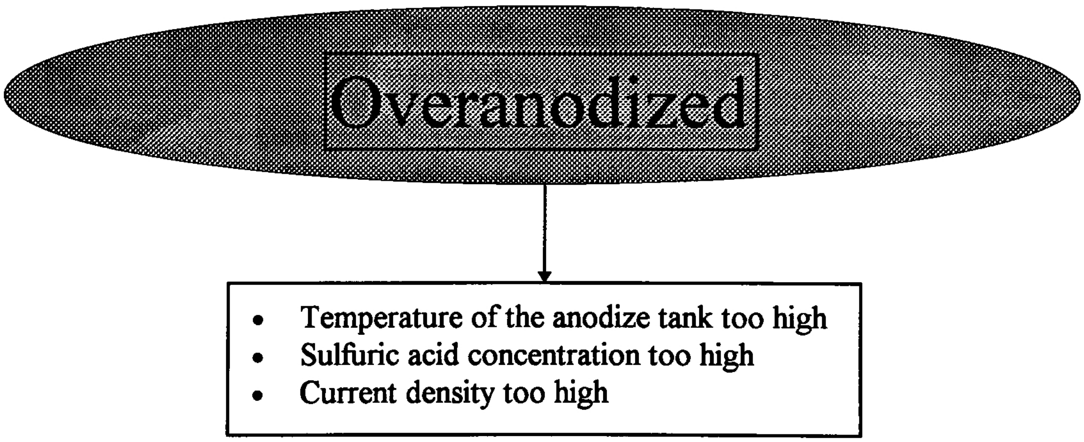


Figure 5. Causes for Overanodizing

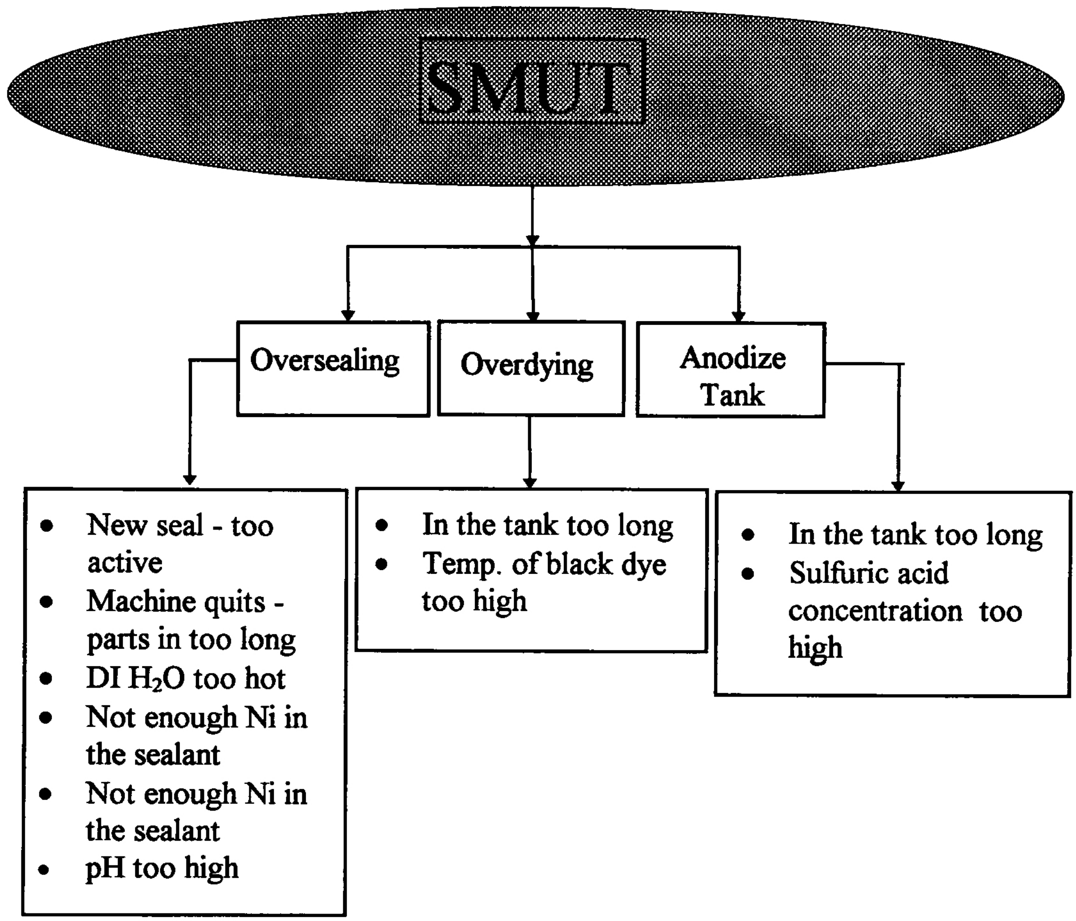


Figure 6. Causes for Smut

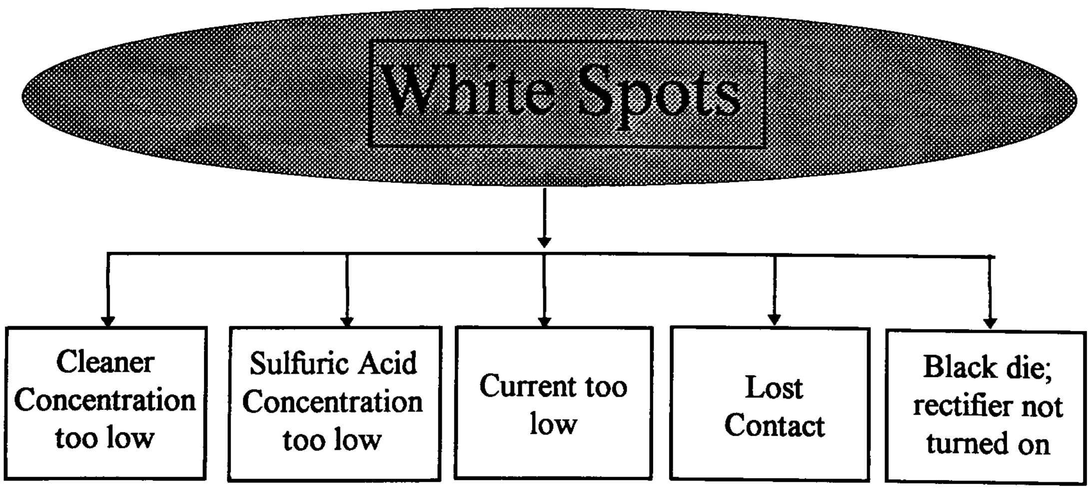


Figure 7. Causes for White Spots

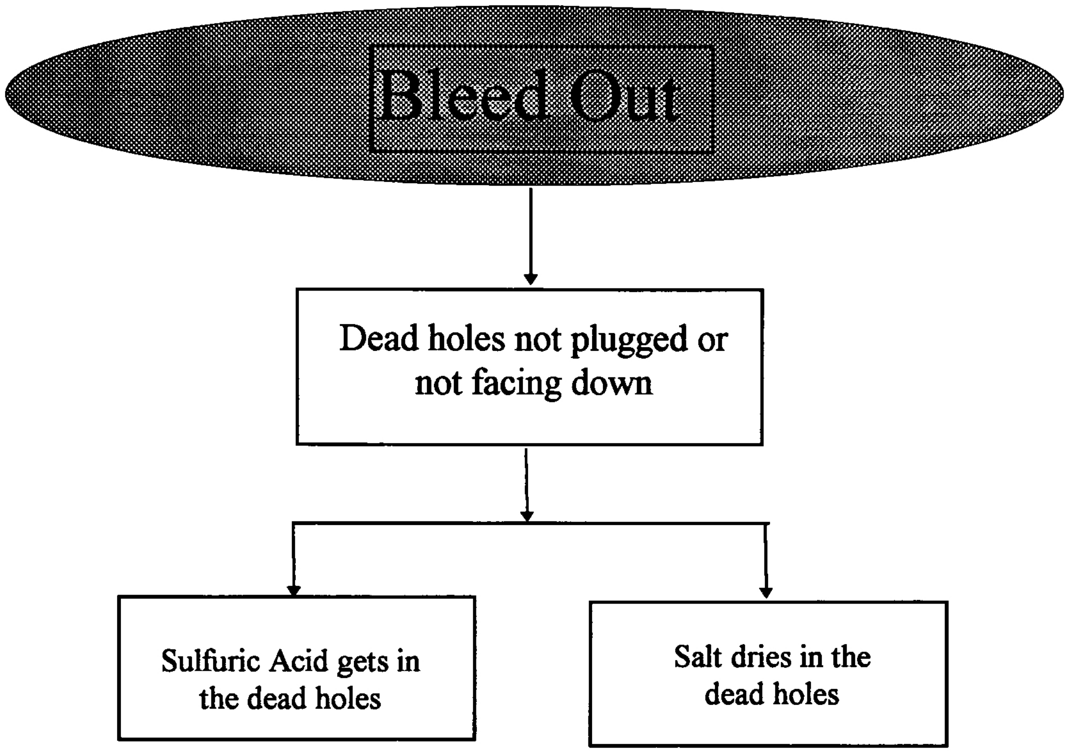


Figure 8. Causes for Bleed Out

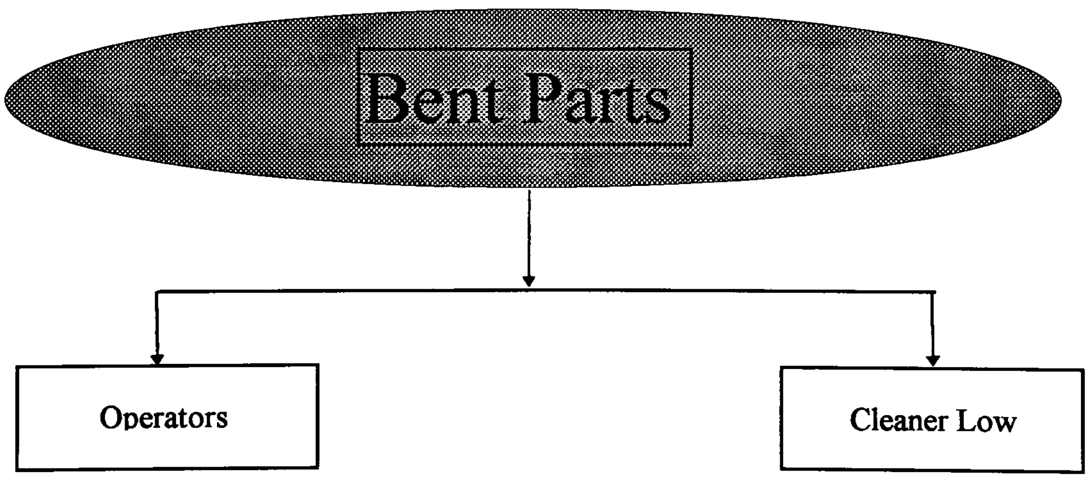


Figure 9. Causes for Bent Parts

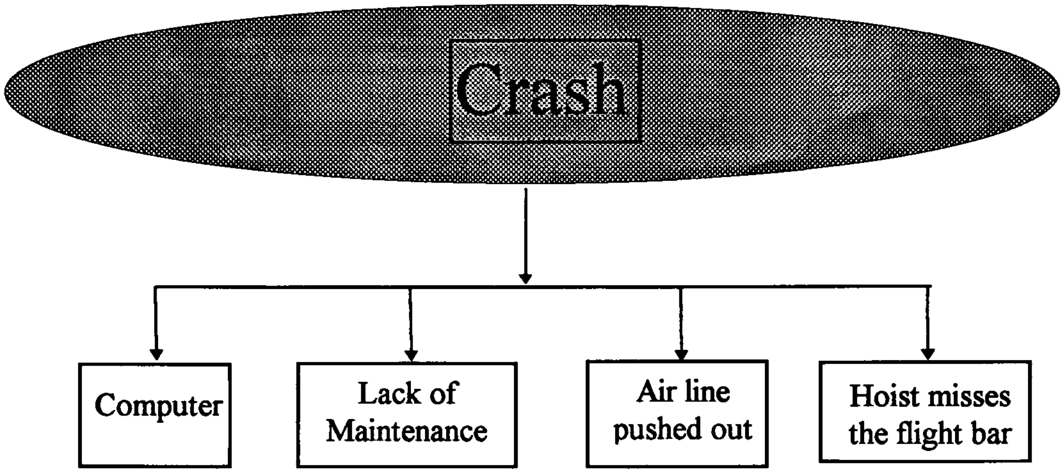


Figure 10. Causes for Crashes

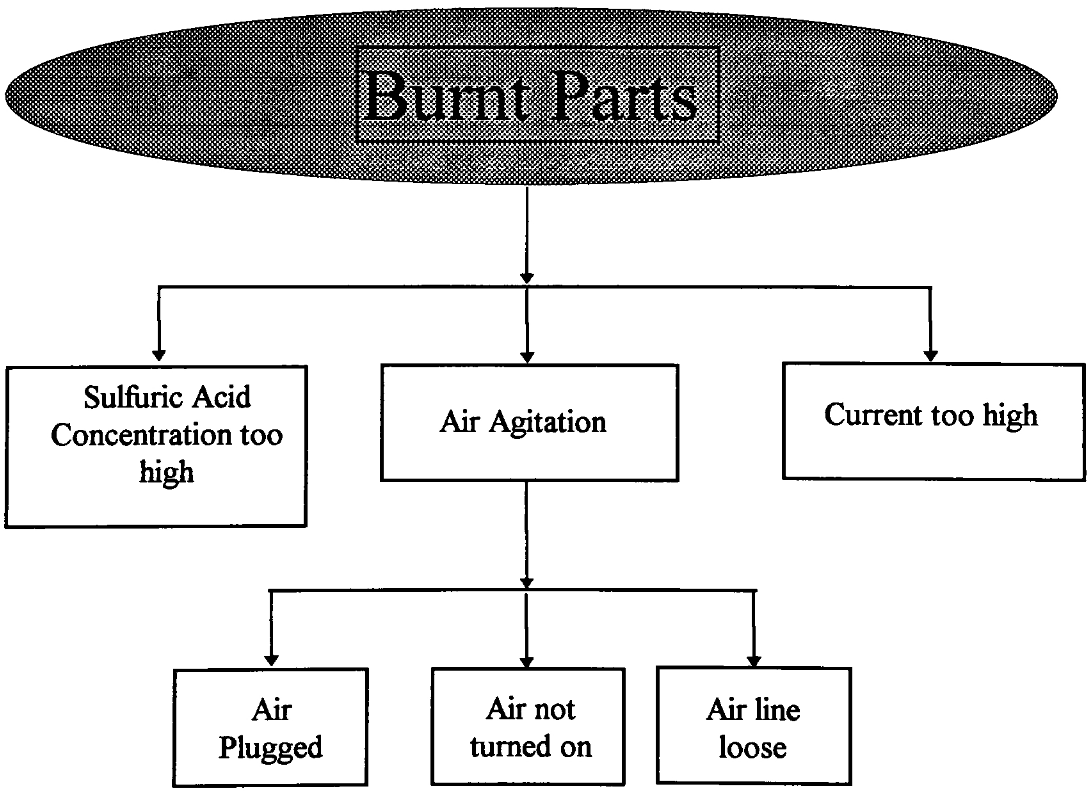


Figure 11. Causes for Burnt Parts

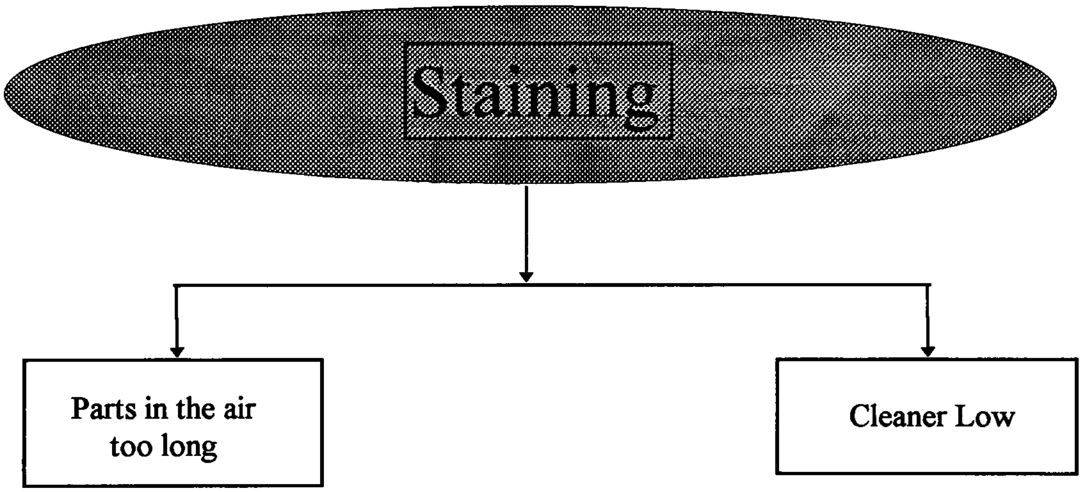


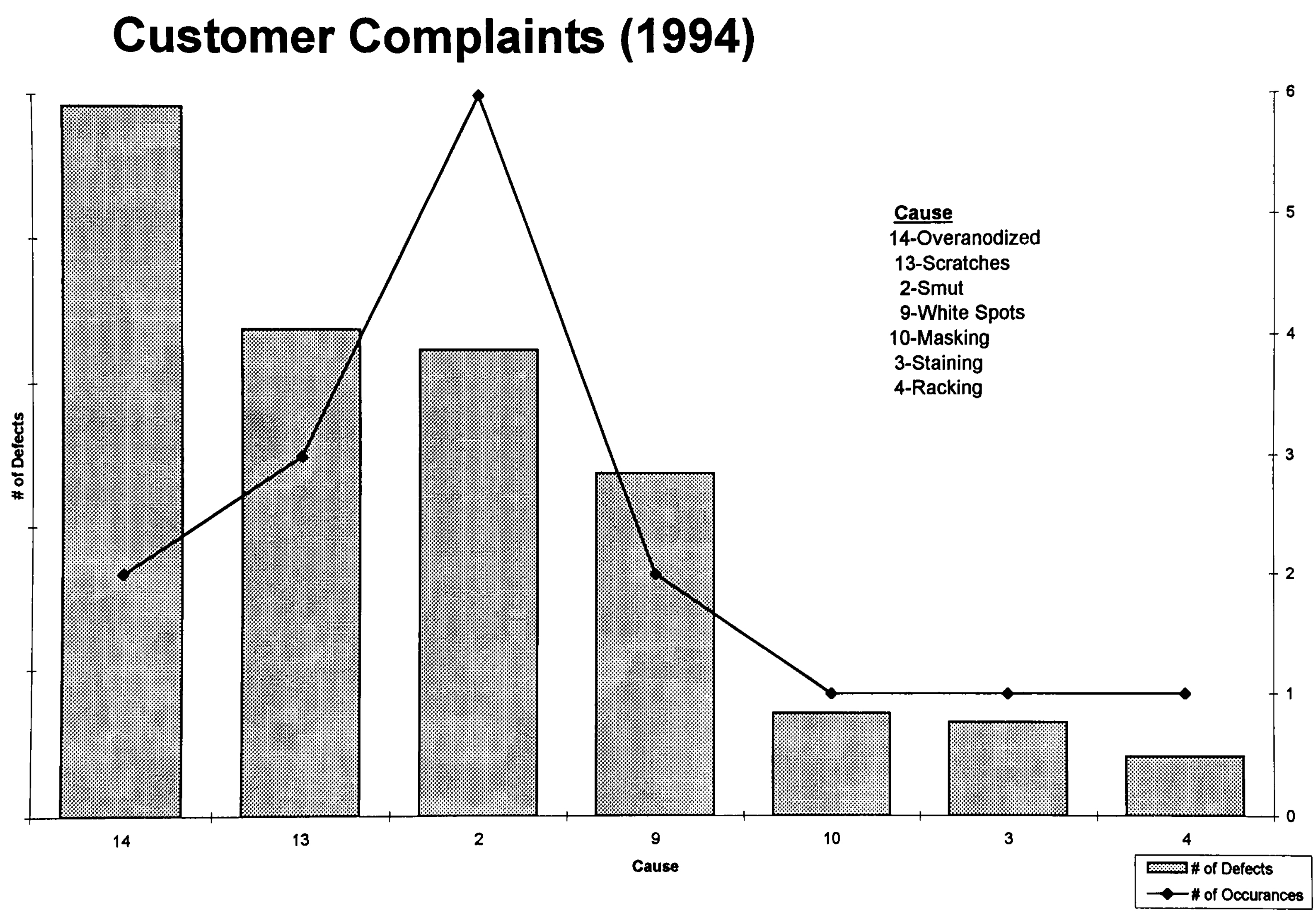
Figure 12. Causes for Staining

A pareto chart analysis was used to determine the top defects of the anodize process. A pareto analysis is a technique for prioritizing types or sources of problems. It "separates the 'vital few' from the 'trivial many' and provides help in selecting directions for improvement."[28] These data were collected over a time span of one year based on customer complaints.

Table 5. Customer Complaints

Defect	# of occurrences	Percentage of occurrences	Percentage of defective parts
Over anodized (film)	2	12.5 %	31.4 %
Scratches	3	18.8 %	21.6 %
Smut	6	37.5 %	20.7 %
White Spots	2	12.5 %	15.2 %
Masking	1	6.25 %	4.5 %
Bleed Out	1	6.25 %	4 %
Rack Marks	1	6.25 %	2.6 %

See figure 13 for the graph showing the pareto analysis. Based on the number of occurrences, smut is the biggest problem and accounted for six out of the sixteen reported defects that reached the customer. In addition it was in the top three for the number of defective parts.



Customer Complaints (1994)
# of Defects
Cause
14-Overanodized
13-Scratches
2-Smut
9-White Spots
10-Masking
3-Staining
4-Racking
0
1
2
3
4
5
6
14
13
2
9
10
3
4
Cause
# of Defects
# of Occurances

Figure 13

Another set of data was collected on the scrap/rework for ten weeks, and this data is represented below in table 6 and figure 14.

Table 6. Scrap/Rework

Defect	# of occurrences	Percentage of occurrences	Percentage of defective parts
Lost Contact	25	73.5 %	43 %
Color	1	2.9 %	20 %
Crash	1	2.9 %	17 %
White Spots	1	2.9 %	11 %
Bent	3	8.8 %	5 %
Dirty Parts	2	5.9 %	2 %
Racking	1	2.9 %	1 %

From this scrap/rework data for ten weeks it is apparent that lost contact parts are the number one problem with both the greatest number of defective parts and occurrences. This data was determined to be incomplete because everything was not being recorded, especially the reworked parts. For example there were large lots of smutty parts many of which were repaired and were not recorded, according to operators.

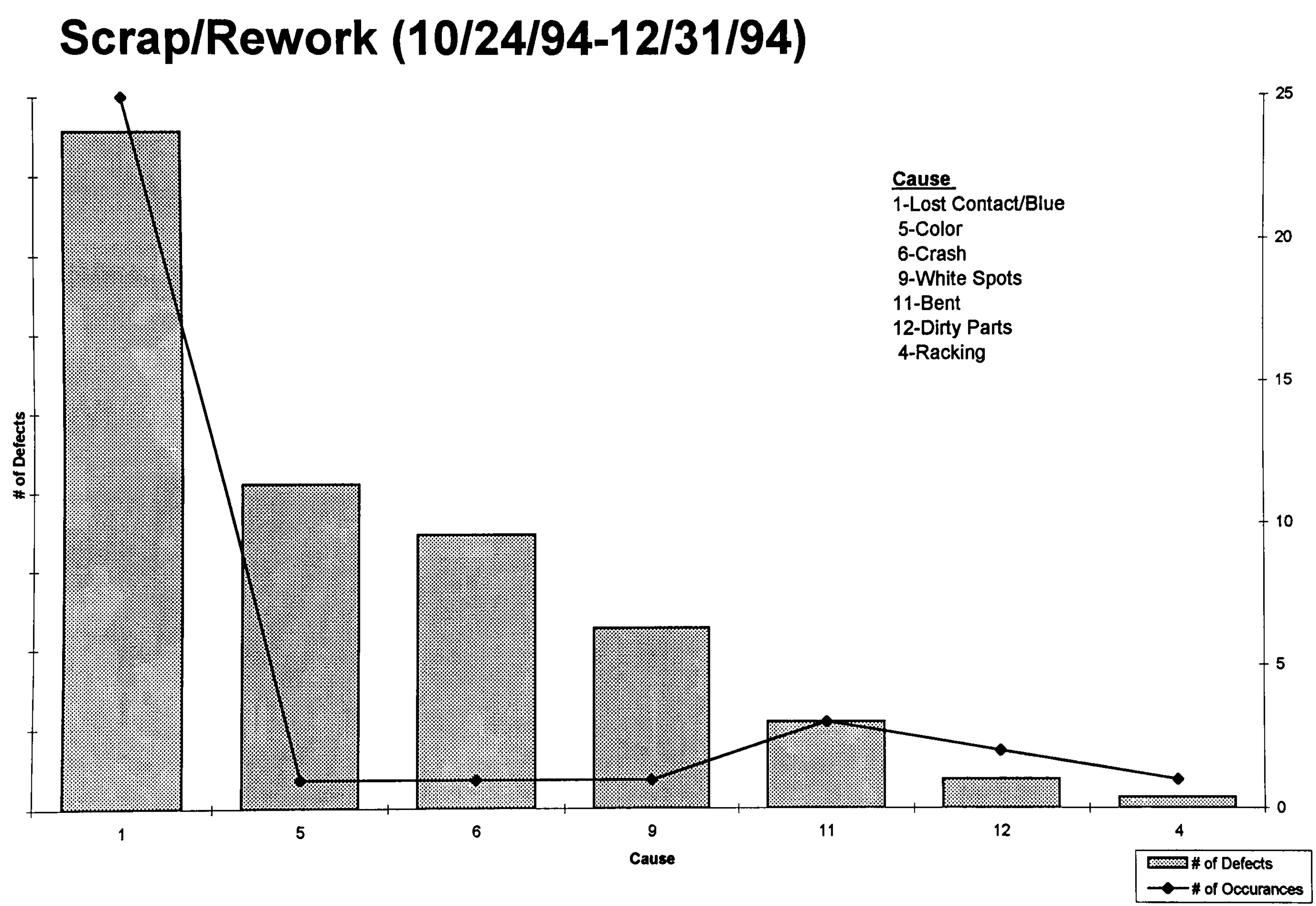

Scrap/Rework (10/24/94-12/31/94)
Cause
1-Lost Contact/Blue
5-Color
6-Crash
9-White Spots
11-Bent
12-Dirty Parts
4-Racking
# of Defects
25
20
15
10
5
0
1
5
6
9
11
12
4
Cause
# of Defects
# of Occurances

Figure 14

DESIGN OF EXPERIMENTS

Pre-experiment

Reduction of defective parts is the goal of the project. The question of which defect to tackle needed to be resolved by brainstorming.

Brainstorm

The first step of the DOE process is brainstorming. For the brainstorming session a group of experts consisting of two production supervisors, the most experienced line operator, maintenance, the department's quality coordinator, a chemical engineer, and two other anodizing experts attended.

Project Objective

Many objectives were to be accomplished during the team meeting. The primary goal of the meeting was to agree on a project objective, and this goal was met. The project objective is to test the hypothesis that smut is a function of six factors: anodize temperature, seal temperature, hot DI rinse temperature, free sulfuric acid concentration in the anodize tank, fluoride concentration in the seal tank, and the pH of the seal.

Response Variable (Output)

A combination of factors was involved in deciding on the project objective, the customer complaint data, scrap/rework data, operator interaction, and team members consensus. Before the team meeting, lost contact appeared to be the obvious choice to base the experiment on because of the scrap/rework data and pareto analysis that showed 25 occurrences during the ten week interval of data collection. However, there were several reasons why this was not selected by the team. First of all, lost contact is a problem that is well understood. Basically it is a racking problem due to the racks themselves, the operators, or the contacts as shown in previous section (Figure 4). Secondly, there were no lost contact parts that slipped through to the customer in 1994.

The problem selected was smut, the second largest problem. Because smut formation depends upon many factors such as, chemistries, times, and temperatures, the learning potential was immense. With the complexity of the smut problem comes many conflicting opinions, that need resolution. Furthermore, it was the problem with the most occurrences of dissatisfied customers. Six of sixteen (37.5%) complaints were received due to smut in 1994, meaning there were six occasions where customers received smutty parts. Also, operators admitted that they were having a large smutting problem, that was not recorded on the data log sheets. For these reasons, smut was chosen as the response variable (though others were added later).

Factors (inputs)

In the brainstorming session, the key parameters of the anodize process which may be involved in producing smut were determined as follows:

Table 7. Possible Variables that Cause Smut (Brainstorm)

Seal	• Time • Temperature • Concentration of fluoride • Concentration of nickel • Contamination • pH • Age/activity
Anodize	• Time • Temperature • Sulfuric acid concentration • Aluminum concentration • Current density
Black Dye	• Time • Temperature
Material	• Alloy Composition • Temper/Aging Treatments

Table 7 was then arranged into constants and variables for the experiment and is shown below in table 8.

Table 8. Experiment Variables and Constants (First Draft)

Variables	Constants
Seal temperature	Material
Anodize temperature	All times
Hot DI rinse temperature	Black dye tank conditions
Sulfuric acid concentration	Current density
Fluoride concentration	pH hot DI rinse
pH of the seal	Contamination
	Age/activity of the seal
	Aluminum concentration in the anodize tank

Concluding the results of the meeting, a full project objective was developed. The hypothesis to be tested is that smut is a function of six factors: anodize temperature, free sulfuric acid concentration in the anodize tank, seal temperature, seal pH, fluoride concentration in the seal tank, and the temperature in the distilled water rinse after the seal. The material, the number of parts, and the levels of input were going to be determined outside the meeting with a few people rather than the whole team.

Revelations Since the Brainstorming:

Material

Four questions pertaining to the material/parts selection needed to be addressed so that material could be ordered in time to run the experiment. They were: 1) which aluminum alloy, 2) what form: coupons or actual production parts, 3) how many pieces, and 4) how fast can the parts be obtained.

A 6000 series aluminum, 6061, was chosen as the material because it is the "purest" aluminum anodized at the company. An actual production part was chosen over coupons because of unknown tempers that are unrepresentative of production parts. A window part was selected because 1) it is made of 6061 aluminum, 2) smut problems are occurring with these parts, 3) high production lots are run of these parts, 4) there exists 1000 parts available free of charge. Furthermore, the theory was that the chosen parts were sensitive to the process indicating when the process was out of control. Therefore, if the smut problem could be solved for these parts, then it could be resolved for any other parts as well.

Number of Parts

Calculations showed that at least one rack of seventy two parts needed to be produced per run to be successful. The calculation was based on the minimum amperage and the total area of material to be anodized.

Experiment-First Design Proposal

The first experiment designed was a six factor 2^{6-2} fractional factorial design with two to four center points. This means that this experiment would have 18 to 20 runs. With seventy two parts per run, at least an additional 440 parts needed to be ordered.

The levels of the experimental factors were determined by talking to experts from the team meeting as well as outside experts. The first experiment is shown below with the high level and low level indicated by pluses and minuses respectively.

Table 9. First Design Proposal

RUN	Temp. Anodize	Sulfuric Conc.	pH Seal	Temp. Seal	Fluoride Conc.	Temp. DI
1	-	-	-	+	-	+
2	-	-	-	-	-	-
3	-	-	+	+	+	-
4	-	-	+	-	+	+
5	-	+	-	-	-	+
6	-	+	-	+	-	-
7	-	+	+	-	+	-
8	-	+	+	+	+	+
9	0	0	0	0	0	0
10	0	0	0	0	0	0
11	+	-	-	-	+	-
12	+	-	-	+	+	+
13	+	-	+	-	-	+
14	+	-	+	+	-	-
15	+	+	-	+	+	-
16	+	+	-	-	+	+
17	+	+	+	+	-	+
18	+	+	+	-	-	-

2^{6-2} design type summary:

- Number of factors = 6
- Number of runs = 16 (without centerpoints)
- Resolution = IV
- Fractionation = 1/4
- Degree of confounding = Moderate
- Main effects confounded with 3-factor interactions and 2-factor interactions confounded with other 2-factor interactions
- Good for estimating main effects
- Time to run experiment = approximately 40 hours
- # of parts possible (for 20 runs) = 1440

A couple of design constraints presented themselves. First, the time to run an experiment is approximately two hours, not including the time for stabilizing the chemical composition of the tanks when additions are made or the tank temperatures (especially the seal tank) are changed. Time is important, because the machine's primary purpose is for production, not experimentation. A second constraining factor is the number of parts needed for the experiment. Ordering more parts would be expensive and take too long.

In order to solve these constraint problems, several solutions were possible. One solution would be to use a 2^{6-3} design, thus decreasing the number of runs and parts in half. This higher degree of fractionation results in a severe degree of confounding because the main effects are confounded with 2-factor interactions. The 2^{6-3} design is a screening experiment.

Another possibility is to reduce the number of factors from six. If five factors are chosen, with a 2^{5-1} design there are the same number of runs as the 2^{6-2} design and with a 2^{5-2} design there exist the same degree of confounding. Basically, there are no advantages gained with a five factor design. The payback comes in reducing the experiment to a four factor design.

A full factorial design is not viable because of the number of runs involved, but a 2^{4-1} fractional factorial is a feasible solution. A summary of a 2^{4-1} design type is below.

- Number of factors = 4
- Number of runs = 8 (without centerpoints)
- Resolution = IV
- Fractionation = 1/2
- Degree of confounding = Moderate
- Main effects confounded with 3-factor interactions and 2-factor interactions confounded with other 2-factor interactions.
- Good for estimating main effects
- Time to run experiment = approximately 18 hours
- # of parts (for 9 runs) = 648

With this design the time and resource constraints are satisfied. Moreover, the leverage that the main effects have on the process will be concluded, which is the objective of the experiment.

Two variables needed to be changed to constants to create the 2^{4-1} design. Changing the anodizing temperature from a variable to a constant was the first modification. This change was made because the low level was at a temperature that was not obtainable. The tank does not have a chiller and therefore the lowest temperature that can be reached is room temperature. Also common industry practice does not cool this tank. Since the low level is below room temperature, it can not be reached. The fluoride concentration in the seal tank was removed from the variable list because testing fluoride has long lead times and it is expensive. Table 10 is a revision of Table 8 and depicts the variables and constants for the final experiment.

Table 10. Revised Table 8. Final Experiment Variables and Constants.

Variables	Constants
Seal temperature	Material
Hot DI rinse temperature	All times
Sulfuric acid concentration	Black dye tank conditions
pH of the seal	Current density
	pH hot DI rinse
	Contamination
	Age/activity of the seal
	Anodize temperature
	Fluoride concentration
	Aluminum concentration in the anodize tank

Experiment-Final Design

Several modifications were made to the original design of experiments. As a result the project objective changed. The new objective was to test the hypothesis that the response variable, smut, is a function of free sulfuric acid in the anodize tank, seal pH, seal temperature, and hot DI water rinse temperature after the seal. The projected time of the revised experiment was two nine hour shifts, which was acceptable, and 648 parts, leaving 352 extras available for confirmation experiments. The final design is shown below.

Table 11. Final Design

RUN	Free Sulfuric Acid	pH Seal	Temperature Seal	Temperature DI Rinse
1	-	+	-	+
2	-	+	+	-
3	-	-	+	+
4	-	-	-	-
5	+	-	+	-
6	+	+	+	+
7	+	+	-	-
8	+	-	-	+
9	0	0	0	0

As before the minuses represent the low levels, the pluses represent the high levels, and the zeros represent the center settings.

The Experiment

As predicted the experiment took two nine hour shifts. These shifts were run back-to-back on a C shift and the following A shift, a total of eighteen hours (from 11 PM till 5 PM) on March 16^{th} and 17^{th}. Two advantages were gained by running two back-to-back shifts. First, no production is run on the C shift so that only one day of production was lost. Second, all constants in the process could be kept under control, because production would not be run in between experiments.

Response Variables

Measuring the smut response was a visual test. The level of smut was measured on a scale from zero to three with the following values:

0 - No Smut

1 - Little Smut

2 - Moderate Smut

3 - High Smut

Because quantitative data for the response variable is desired, alternatives for measuring the smut were being researched.

More response variables were added after running the experiment. They included the degree of blue, degree of darkness, and degree of seal. The blue response was added because it is an undesirable defect that showed up in a couple of runs. Darkness was added to tell how black the part was. The darkness data was gathered at the same time as the blue data and, therefore, took no extra time. The degree of seal was added because there is an acceptable standard that parts must meet. If parts don't seal then they are more susceptible to wear and paint fading.

A spectrophotometer is an instrument used to measure color intensity. Two different scales, blue-green and light-dark, were of particular interest. Using this instrument, the blue and darkness responses were measured yielding a continuous quantitative comparison of each run. On the blue-green scale, the negative numbers mean green and the positive numbers mean blue. Only the blue was visible, never the green. As the number increased, the more severe the blue becomes. The human eye can see tints of blue around a value of one.

The level of darkness was determined in the same way as the level of blueness, the lower the value the higher the darkness. A part that had zero smut, that was not run during the experiment, was measured for darkness and was higher than any of the experimental samples (meaning it was lighter than any of the experimental samples). Also a black standard used for calibration has a high number on the darkness scale. Therefore it is more desirable to have a higher value on the darkness scale.

Determining the degree of sealing was done by using a simple seal test, that measures the amount of material that is removed after sitting in a chemical for a period of time. To do the test, parts are weighed, immersed in acid for fifteen minutes, weighed again, and the weight loss is calculated. If the weight loss is greater than three mg/in^2 then the part is rejected. Consequently the lower the weight loss during the seal test, the better the part is sealed.

Post-experiment

A confirmation experiment was run a couple of weeks later to make sure that the results of the experiment were repeatable. Run number one was the repeated run. The settings for the confirmation run were low for the sulfuric acid concentration, high for the seal pH, low for the seal temperature, and high for the hot DI rinse temperature. The results were favorable because they turned out the same as run one.

Cleaning the data and analyzing the data was done using JMP. These were two easy steps in comparison to the one to follow, interpreting the results. The results of the experiment, the analysis, and the interpretation of the results are in the analysis section.

ANALYSIS

Experimental Results

To begin the data that were analyzed should be shown before anything else is discussed.

Table 12. Experimental Results

RUN	Free Sulfuric Acid	pH Seal	Temp Seal	Temp DI Rinse	Smut (0-3)	Blue	Wt. Loss (mg/in^2)	Darkness
1	-1	+1	-1	+1	1	-0.86	14.92	22.94
2	-1	+1	+1	-1	3	0.18	0.84	20.05
3	-1	-1	+1	+1	3	5.86	0.48	19.09
4	-1	-1	-1	-1	1	-0.13	8.37	19.0
5	+1	-1	+1	-1	3	8.49	0.29	19.49
6	+1	+1	+1	+1	3	0.9	0.06	19.05
7	+1	+1	-1	-1	2	-1.85	2.67	22.40
8	+1	-1	-1	+1	3	0.46	10.48	18.29
9	0	0	0	0	3	2.18	0.9	18.83
10*	-1	+1	-1	+1	1	-1.81	12.9	23.47

* Confirmation Run

Scatter Plots

A scatter plot of the data is one method of determining which factors might be important and needed in the model. Specifically, scatter plots of the response values verses the corresponding factor levels are called main effect plots. Main effect plots for smut (figures 15-18), blue (figures 19-22), degree of seal or weight loss (figure 23-26), and darkness (figures 27-30) are shown below.

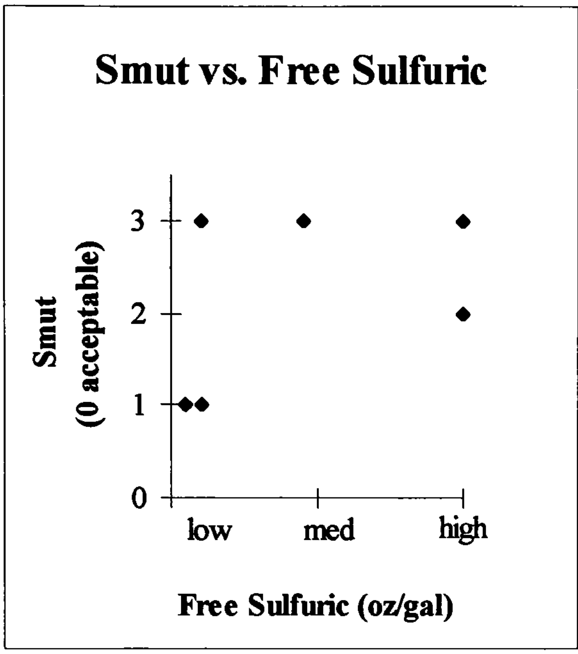


Figure 15

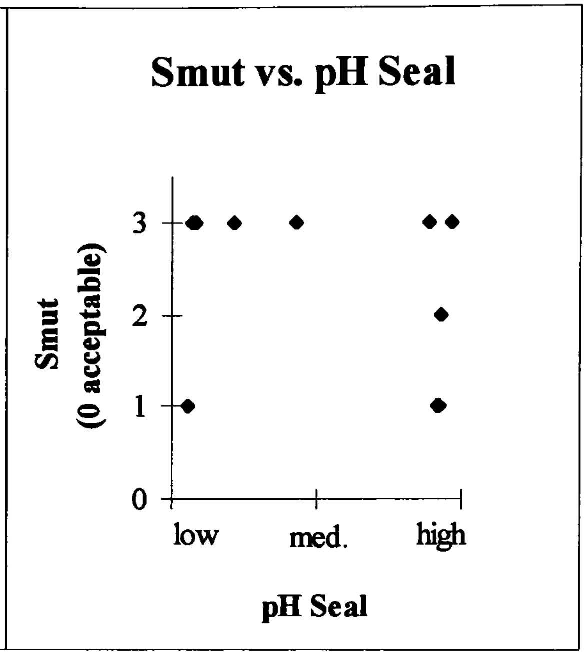


Figure 16

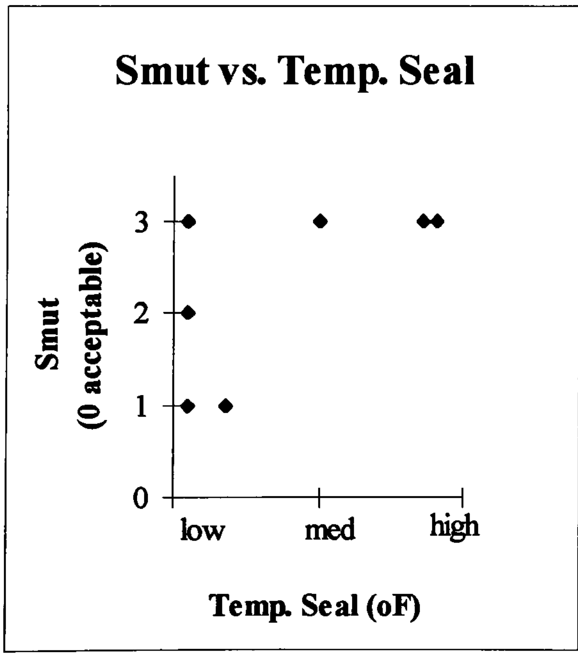


Figure 17

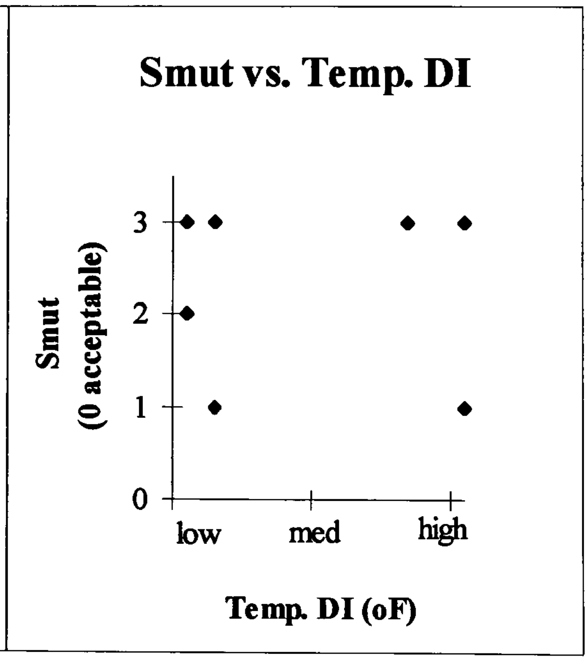


Figure 18

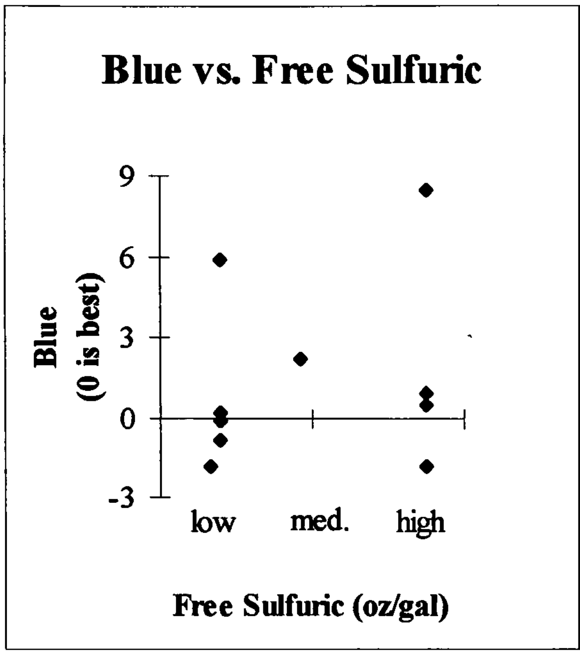


Figure 19

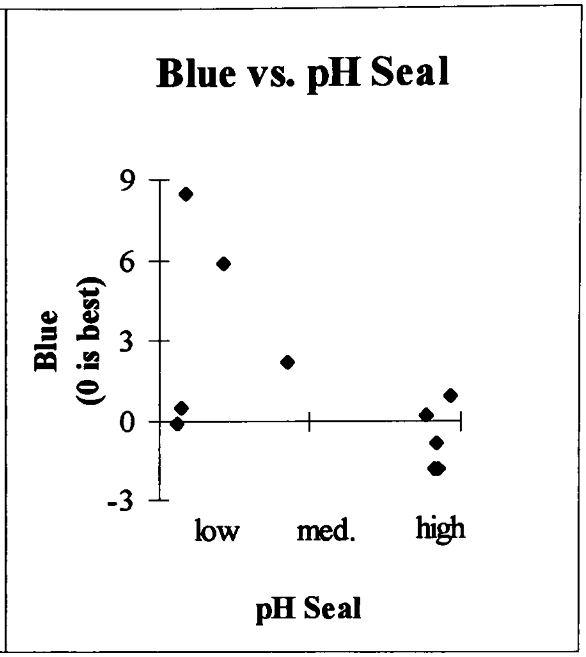


Figure 20

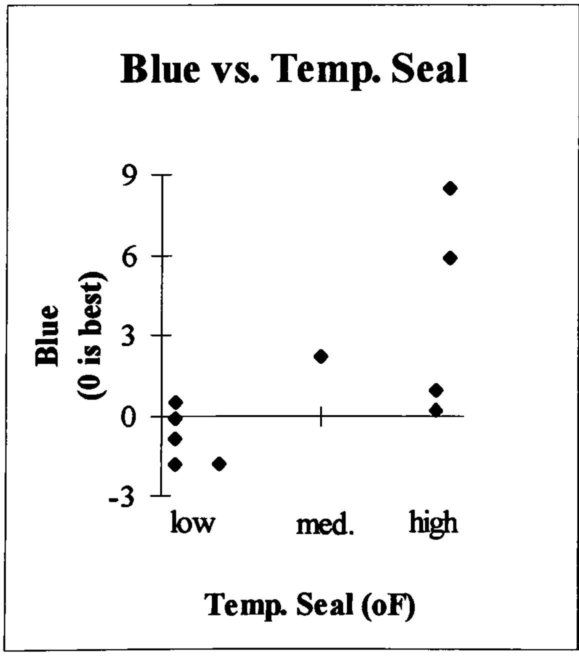


Figure 21

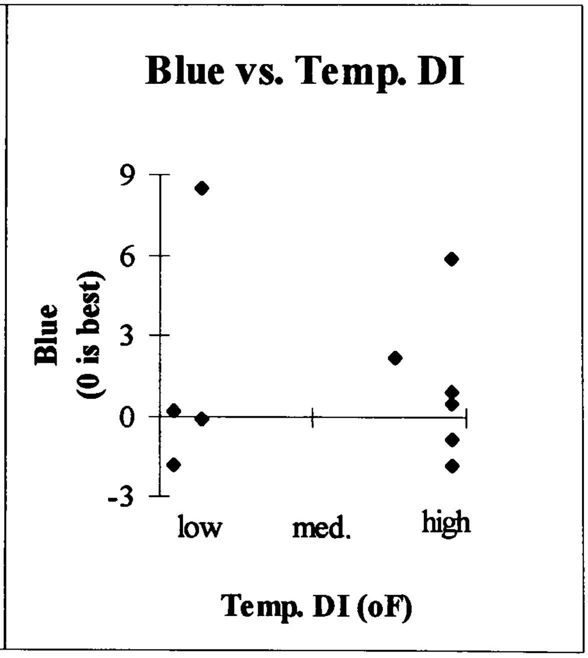


Figure 22

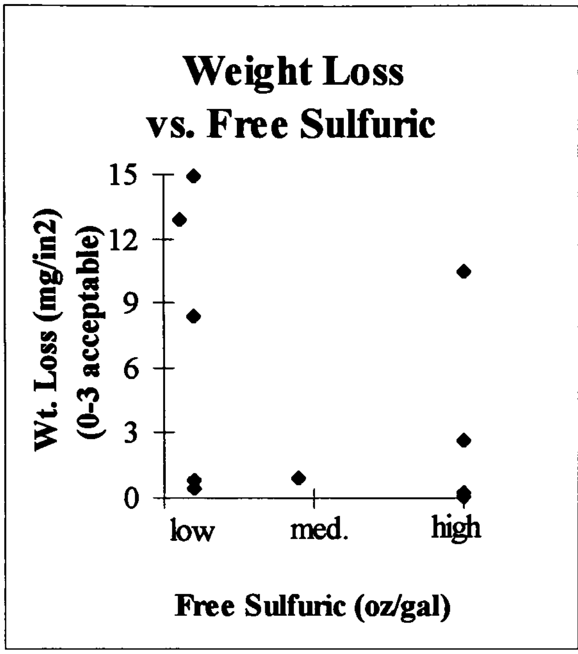


Figure 23

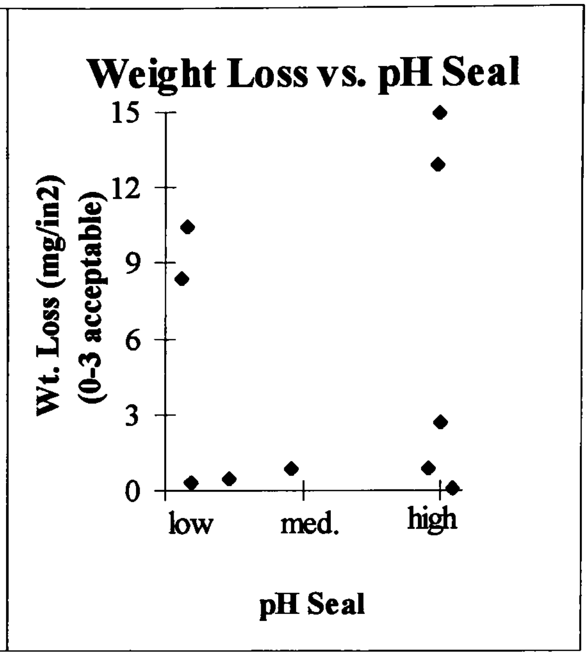


Figure 24

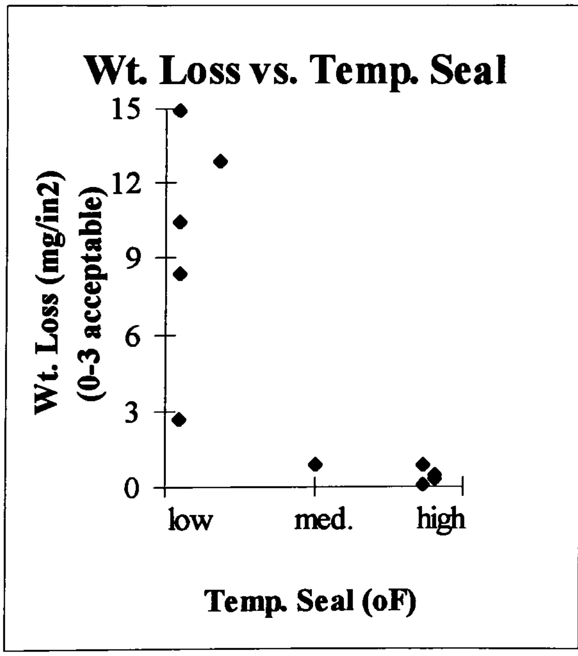


Figure 25

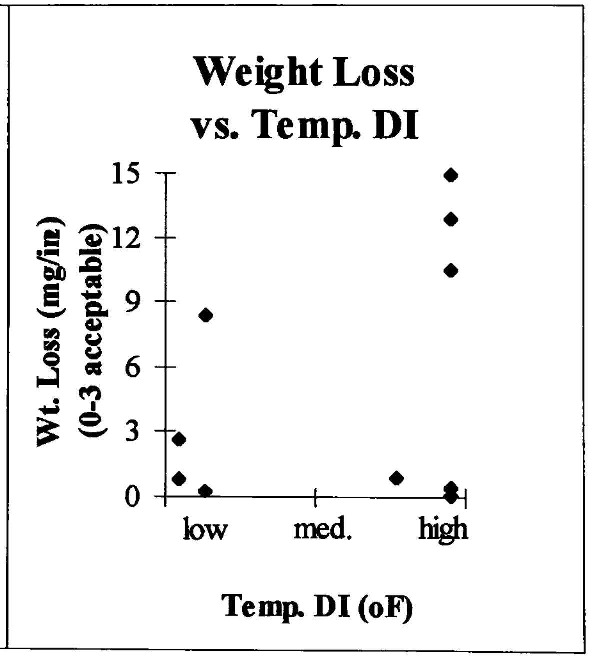


Figure 26

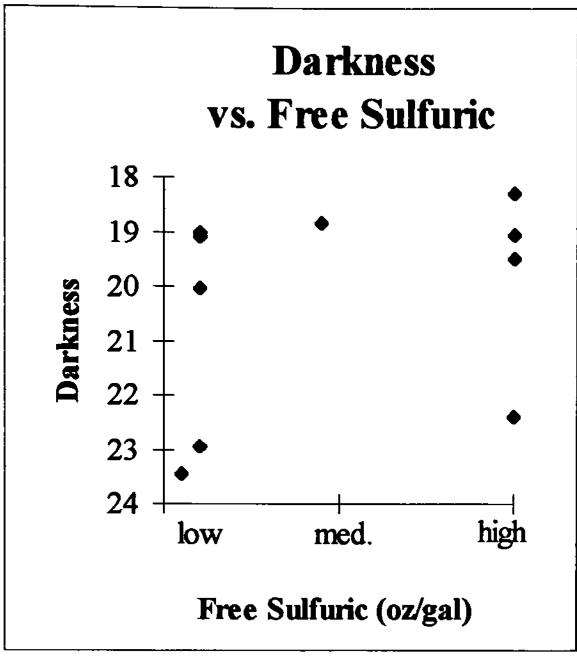


Figure 27

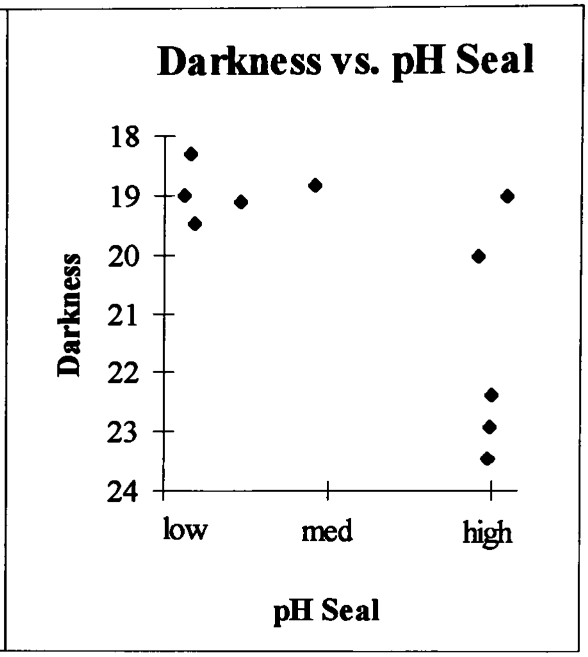


Figure 28

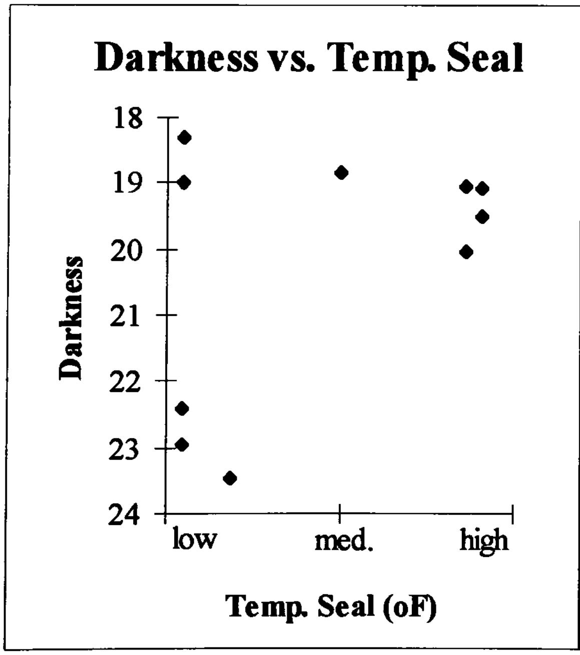


Figure 29

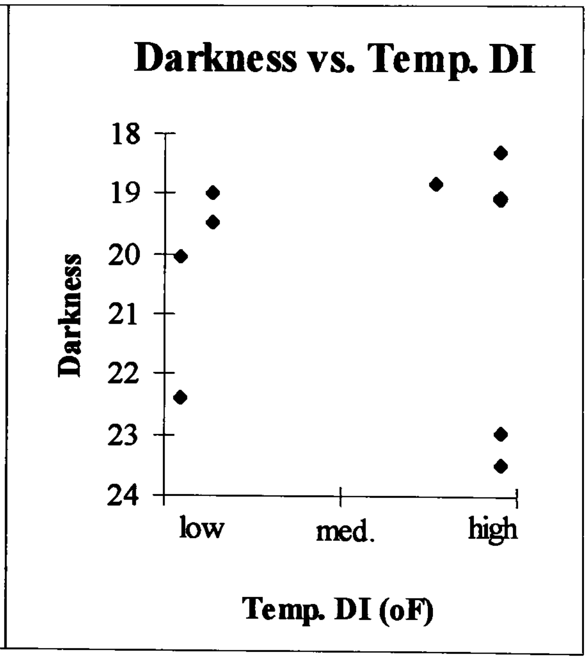


Figure 30

Looking at the smut data graphs, it appears that there exist a trend that higher sealing temperatures will produce more smut (Figure 17). The other variables indicate no solid trends in one way or the other, either because, 1) there are not enough data points, 2) there is no trend, 3) the effects of other factors on the response will produce what appears to be variability in the response when plotted against the one factor of interest, 4) interactions are present among the factor being plotted and the other factors and no relationship is showing up because of it, or 5) as predicted, the qualitative data is difficult to measure and assign a value to the samples. Quantitative data is needed.

The blue data shows a definite trend in the seal temperature again (Figure 21). It appears that as the temperature increases, the blue condition gets worse. The pH may have an effect in a way that higher pHs would appear to produce better parts that were not blue (Figure 20).

Figure 25, the weight loss (degree of seal) vs. temperature seal plot shows seal temperature significance once again. In this case, higher seal temperatures are producing better sealed parts. The other factors are not appearing to have large effects.

Finally, the darkness output shows that the pH of the seal and the temperature of the seal may be the most significant factors, where higher pHs and lower seal temperatures producing the better parts (Figures 28 and 29 respectively).

Since the temperature of the seal seems to play a role in all four response variables it was plotted against all the responses on one graph (Figure 31).

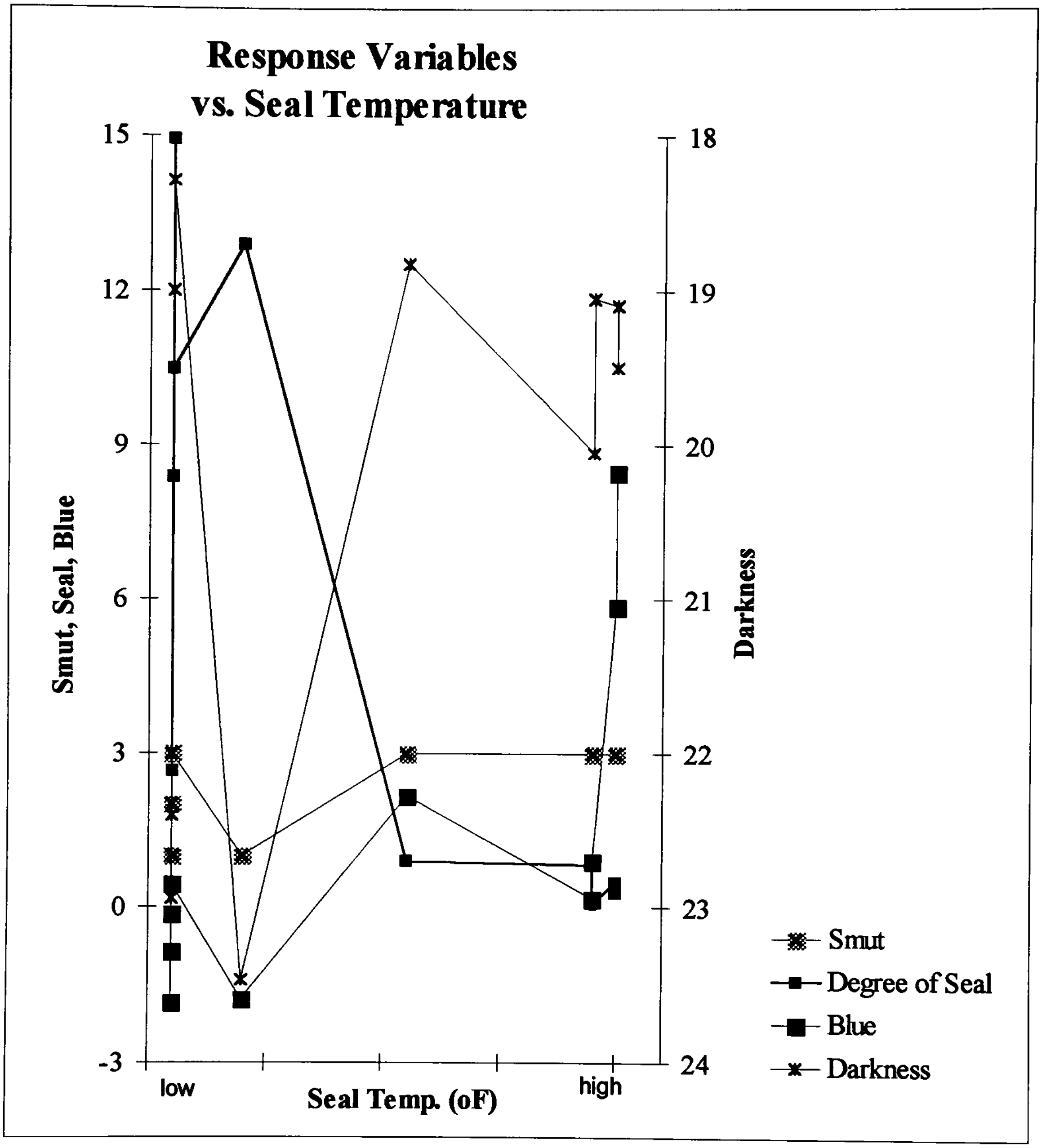


Figure 31. Response variables vs. Seal temperature

When plotting a graph of smut and degree of seal verses the temperature (Figure 32), it is seen that the two responses are producing good parts at different levels of the sealing temperature range. A compromise will have to be made to satisfy both the smut and sealing conditions.

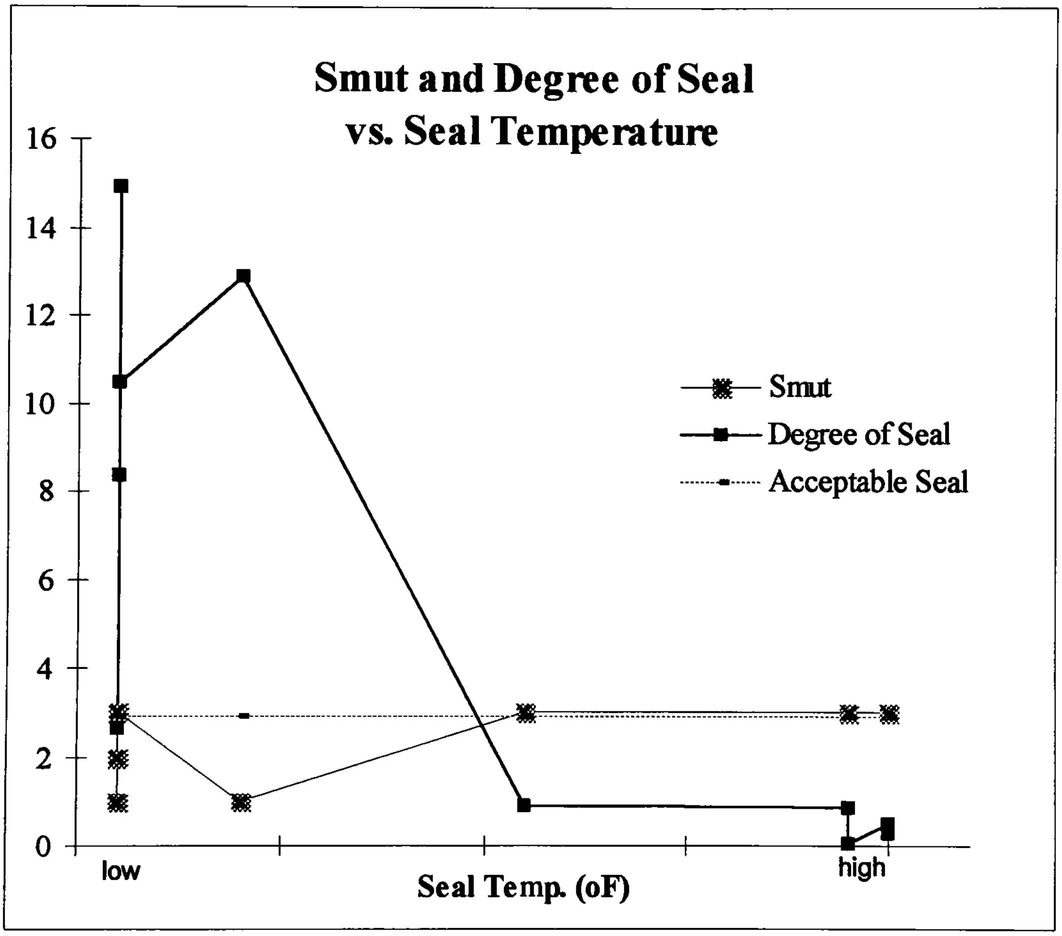


Figure 32. Smut and Degree of Seal vs. Seal Temperature

Residuals

With 3 or more factors scatter plots may be ineffective because of the effects of the other variables. Another type of plot, a residual plot, is generally more informative for evaluating a fitted model and is useful for any number of factors. A residual is the difference between an observed response value and the corresponding response value predicted from the polynomial model. Residual plots serve three purposes:

- to determine if the specified model is correct
- to determine if there are problems with the data
- to determine if the assumptions of regression are met.

If a trend exists in the residual plot, then an important parameter or factor effect has maybe been left out of the model. Problems with the data are indicated by outlier points, which are due to a recording error, an experimental error, or another reason. In general outliers should not be deleted from the data unless there is justification.

Experimental error (ε) is included in every model. The residual values are estimators of these true errors (ε) and can be used to determine if the assumptions of regression are met.

These assumptions are as follows:

1. If the model form is correct, then at any setting of the factors, the ε`s should be centered around zero [Mean of $\varepsilon = 0$]. The model should be able to predict equally well on average at any factor setting.
2. At any factor setting the variability of the ε`s should be equal [variance of ε = constant]. The experimental variability in the response must not change from factor setting to factor setting.
3. No cyclical trends when plotted against the experimental run order exist [ε`s independent of one another].
4. The ε`s must follow a normal distribution.

Table 13 shows the residual values obtained from JMP.

Table 13. Residual Values

Run	Residual Smut	Residual Blue	Residual Sealing	Residual Darkness
1	-0.04631	0.420789	1.383379	-0.16947
2	-0.03087	-0.03614	0.248919	0.063688
3	-0.09262	-0.10842	0.746758	0.191063
4	-0.03087	-0.03614	0.248919	0.063688
5	-0.03087	-0.03614	0.248919	0.063688
6	-0.09262	-0.10842	0.746758	0.191063
7	-0.03087	-0.03614	0.248919	0.063688
8	-0.09262	-0.10842	0.746758	0.191063
9	0.493997	0.57825	-3.98271	-1.01901
10	-0.04631	-0.52921	-0.63662	0.360532

Using the data in the table the assumptions for regression can be checked. The first set of graphs is the residuals of the responses verses the corresponding response (Figures 33-36), which is used to check for trends in the data, centering around zero, and the variance of the residuals. Next, the residual values were plotted verses the run number (Figure 37-40) to check that residuals are independent of each other. Normality plots of the residuals were completed to verify the fourth and last assumption.

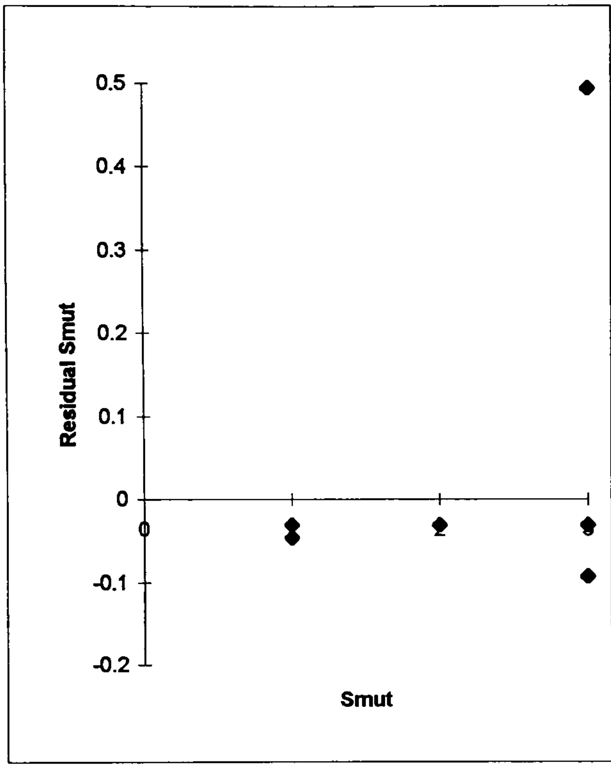


Figure 33. Residual Smut vs. Smut

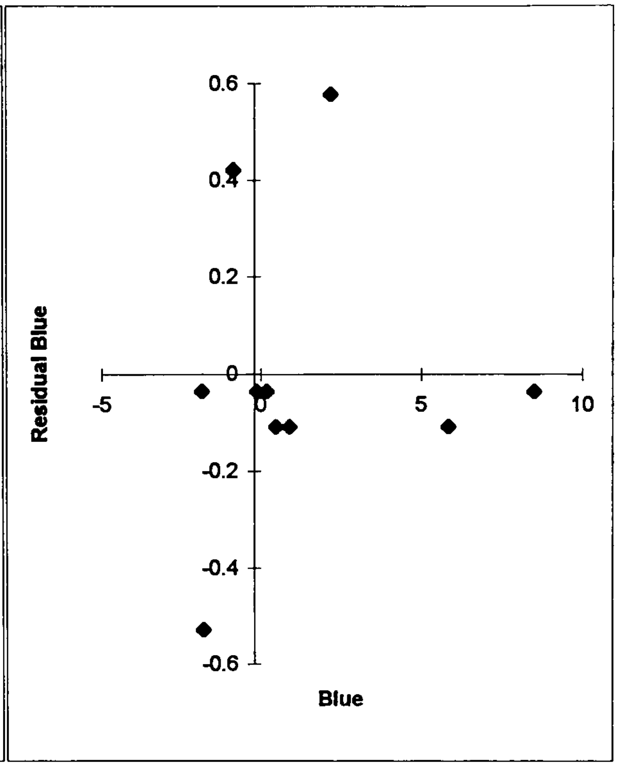


Figure 34. Residual Blue vs. Blue

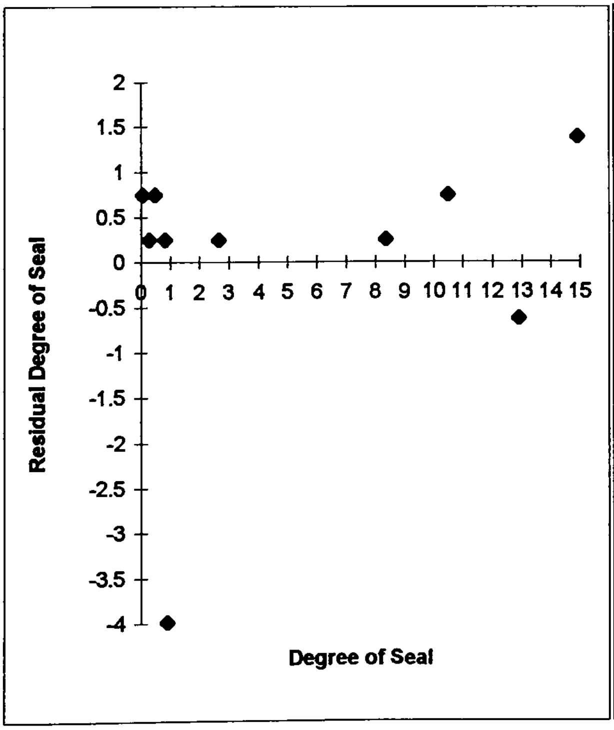


Figure 35. Residual Degree of Seal vs. Degree of Seal

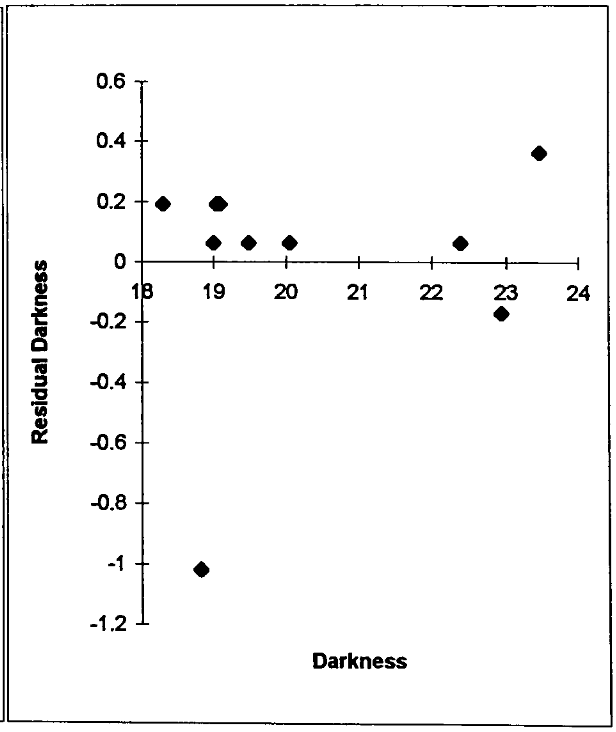


Figure 36. Residual Darkness vs. Darkness

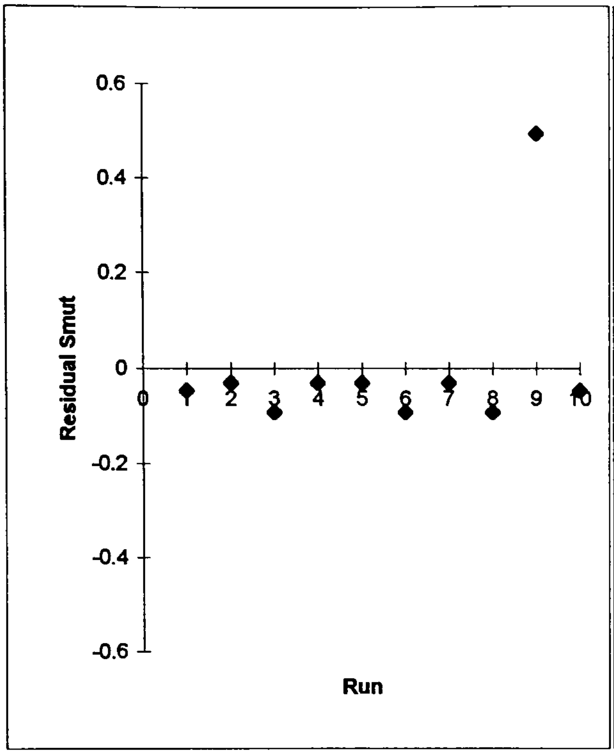


Figure 37. Residual Smut vs. Run

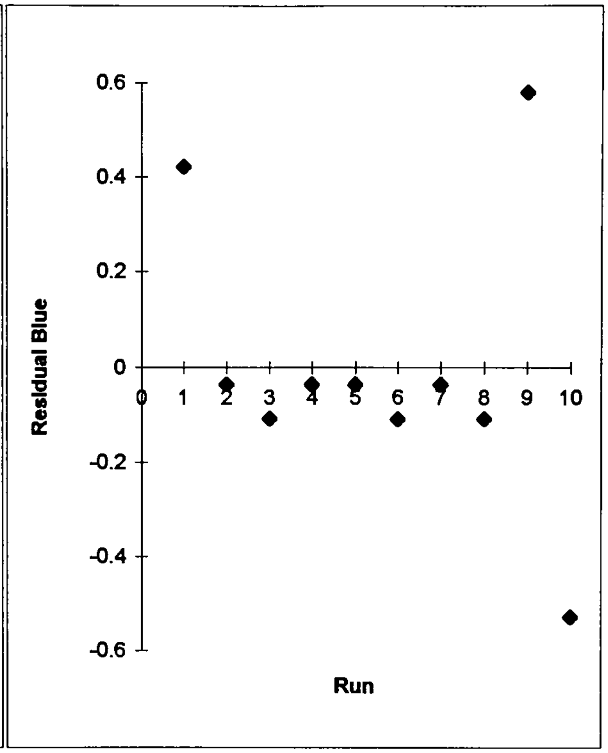


Figure 38. Residual Blue vs. Run

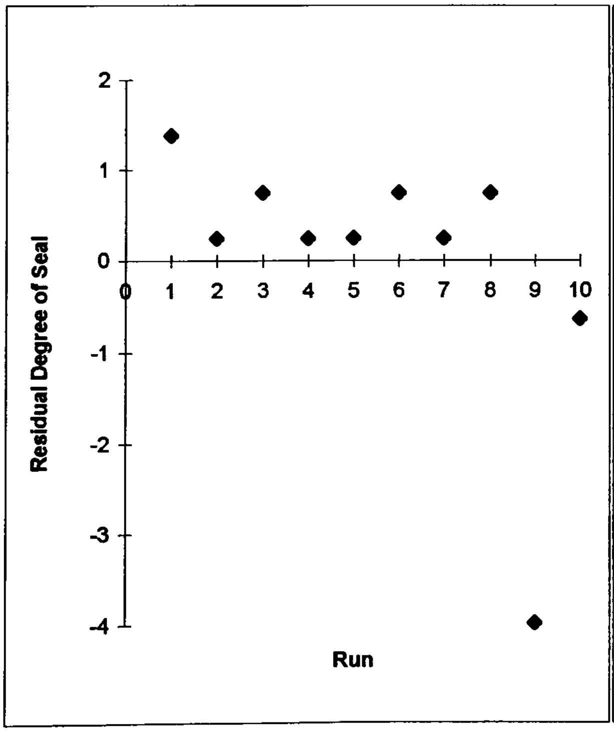


Figure 39. Residual Degree of Seal vs. Run

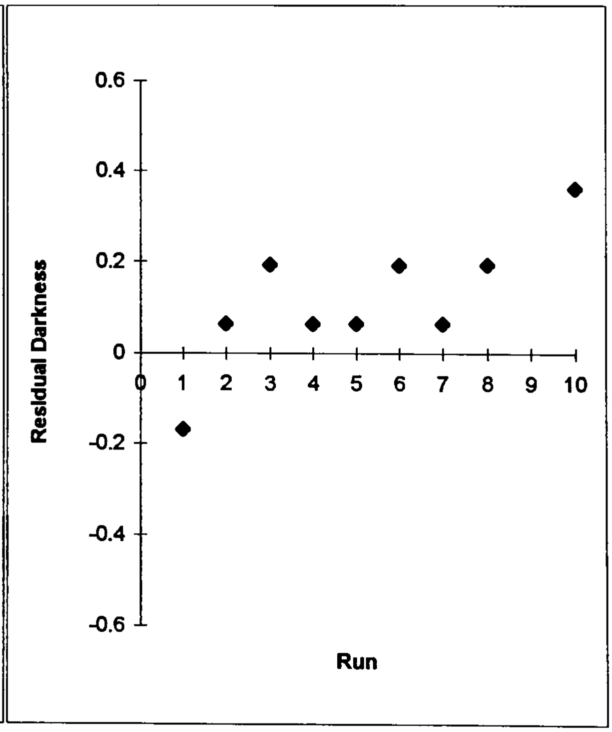
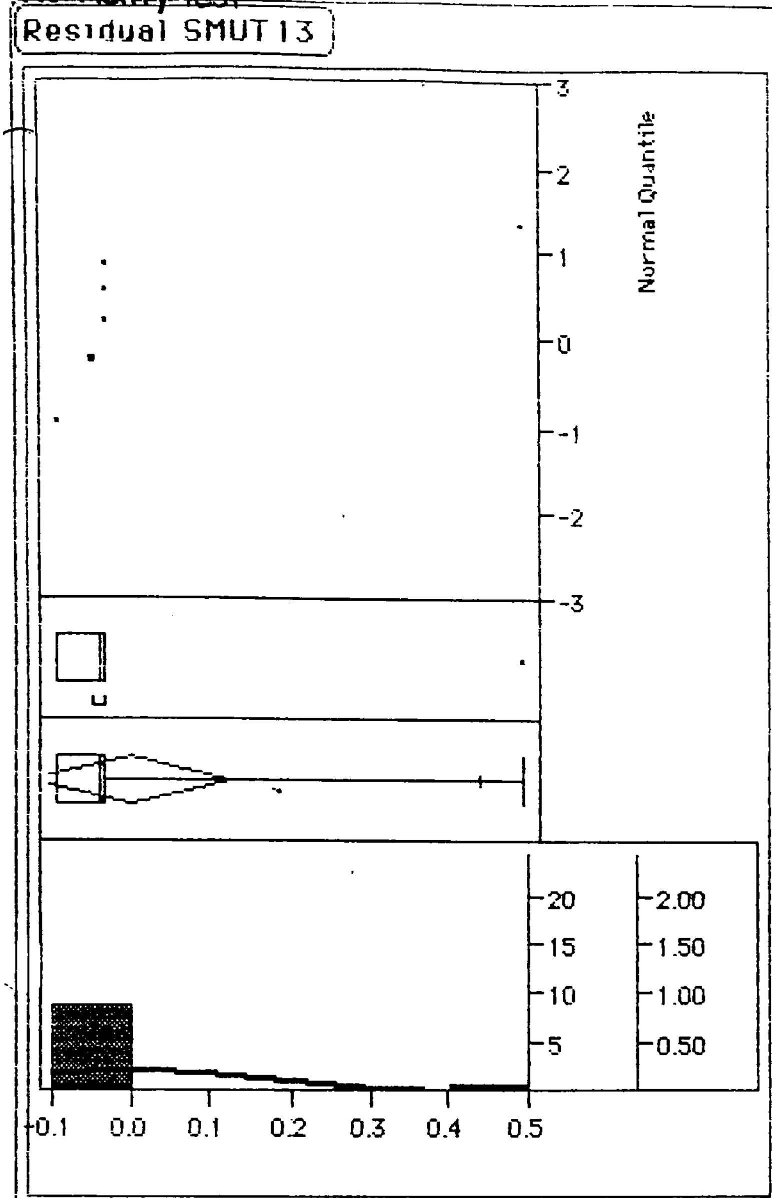


Figure 40. Residual Darkness vs. Run

Normality Test



Quantiles

maximum	100.0%	0.49400
	99.5%	0.49400
	97.5%	0.49400
	90.0%	0.44151
quartile	75.0%	-0.0309
median	50.0%	-0.0386
quartile	25.0%	-0.0926
	10.0%	-0.0926
	2.5%	-0.0926
	0.5%	-0.0926
minimum	0.0%	-0.0926

Moments

Mean	-0.00000
Std Dev	0.17571
Std Err Mean	0.05557
upper 95% Mean	0.12570
lower 95% Mean	-0.12570
N	10.00000
Sum Wgts	10.00000

Test for Normality

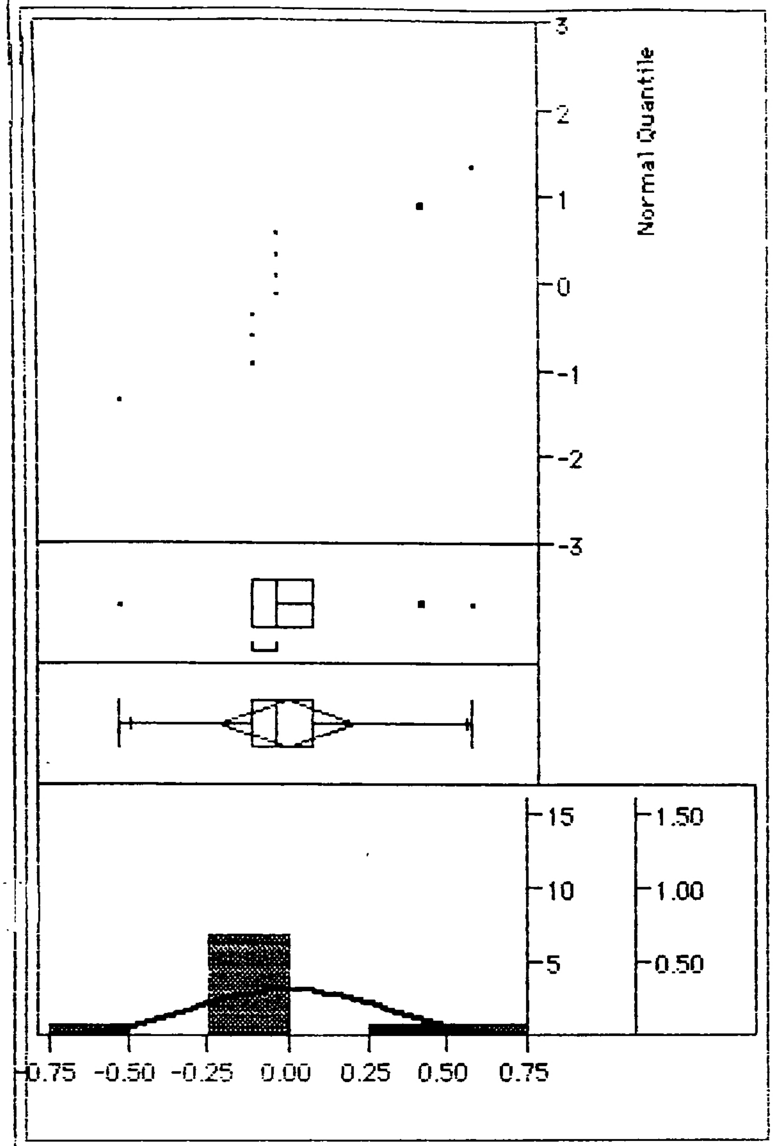
Shapiro-Wilk W Test

W	Prob<W
0.494320	0.0000

Figure 41.

Normality Test

Residual BLUE



Quantiles

maximum	100.0%	0.57825
	99.5%	0.57825
	97.5%	0.57825
	90.0%	0.56250
quartile	75.0%	0.07809
median	50.0%	-0.0361
quartile	25.0%	-0.1084
	10.0%	-0.4871
	2.5%	-0.5292
	0.5%	-0.5292
minimum	0.0%	-0.5292

Moments

Mean	-0.00000
Std Dev	0.30405
Std Err Mean	0.09615
upper 95% Mean	0.21750
lower 95% Mean	-0.21750
N	10.00000
Sum Wgts	10.00000

Test for Normality

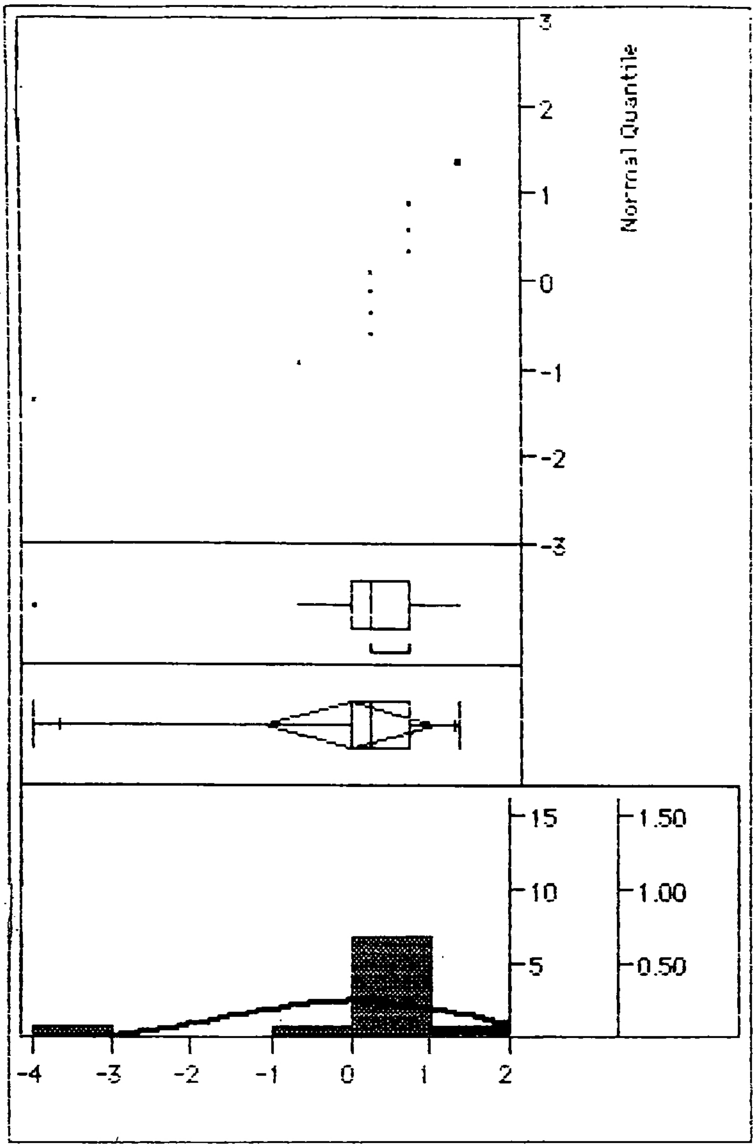
Shapiro-Wilk W Test

W	Prob<W
0.831267	0.0339

Figure 42.

Normality Test

Residual SEALING



Quantiles

maximum	100.0%	1.3834
	99.5%	1.3834
	97.5%	1.3834
	90.0%	1.3197
quartile	75.0%	0.7468
median	50.0%	0.2489
quartile	25.0%	0.0275
	10.0%	-3.6481
	2.5%	-3.9827
	0.5%	-3.9827
minimum	0.0%	-3.9827

Moments

Mean	0.00000
Std Dev	1.49450
Std Err Mean	0.47260
upper 95% Mean	1.06911
lower 95% Mean	-1.06911
N	10.00000
Sum Wgts	10.00000

Test for Normality

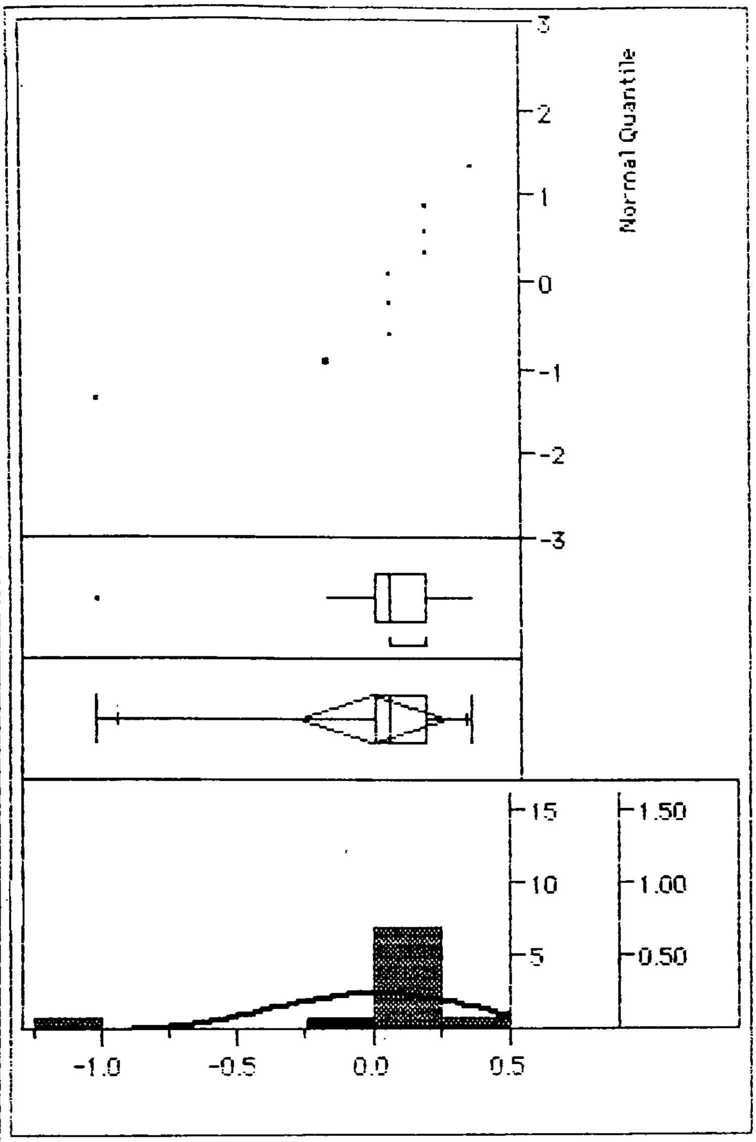
Shapiro-Wilk W Test

W	Prob<W
0.676249	0.0006

Figure 43

Normality Test

Residual DARKNESS



Quantiles

maximum	100.0%	0.3605
	99.5%	0.3605
	97.5%	0.3605
	90.0%	0.3436
quartile	75.0%	0.1911
median	50.0%	0.0637
quartile	25.0%	0.0054
	10.0%	-0.9341
	2.5%	-1.0190
	0.5%	-1.0190
minimum	0.0%	-1.0190

Moments

Mean	0.00000
Std Dev	0.38338
Std Err Mean	0.12124
upper 95% Mean	0.27426
lower 95% Mean	-0.27426
N	10.00000
Sum Wgts	10.00000

Test for Normality

Shapiro-Wilk W Test

W	Prob<W
0.681226	0.0007

Figure 44

Conclusions from the residual plots:

- No trends exist implying that terms have not been left out of the model.
- Run number nine appears to be an outlier. A probable cause for this outliers is that all four variables (and three different tanks) were changed between run 8 and run 9. Time constraints and large tank sizes prevented complete tank stabilization before run 9. When drastic temperature or chemical changes are made to the tanks on the anodize line, it takes a long time to bring the large tanks to equilibrium again. Because of this, deleting run nine from the data set is justifiable.
- The mean of the residuals is zero in all cases. Therefore the mean of $\varepsilon = 0$. However the points are not evenly distributed around the mean.
- The dispersion of the residuals about zero could be better. The variance of $\varepsilon \neq$ constant.
- In figure 40, there appears a trend when the residuals are plotted verses the run numbers. Therefore ε's are dependent of one another.
- The normal probability plots and the tests for normality show that all of the residuals do not follow a normal distribution. JMP uses the Shapiro-Wilk W test as the test for normality. For this test, the null hypothesis (H_o) is that the distribution is normal and the alternative hypothesis (H_a) is that the distribution is not normal. Assume that if the probability is less than or equal to .05 (prob $\leq$ .05) then reject the null hypothesis. H_o is rejected for all four response cases implying normality is not satisfied.

The residual data was re-evaluated removing run number nine since it was an outlier. The following is the results.

Table 14. Residual Values Excluding Run #9

Run	Residual Smut	Residual Blue	Residual Sealing	Residual Darkness
1	0.06667	0.92429	0.58	-0.09286
2	0.13333	-0.0325	0.32	0.0725
3	-0.1333	-0.8986	-0.32	-0.34429
4	-0.1333	0.0325	0.32	-0.0725
5	0.13333	0.89857	-0.32	0.34429
6	-0.1333	0.0325	0.32	-0.0725
7	-0.1333	-0.8986	-0.32	-0.34429
8	0.13333	-0.0325	0.32	0.0725
10	0.06667	-0.0257	-1.17	0.43714

Residual Data Excluding Run # 9

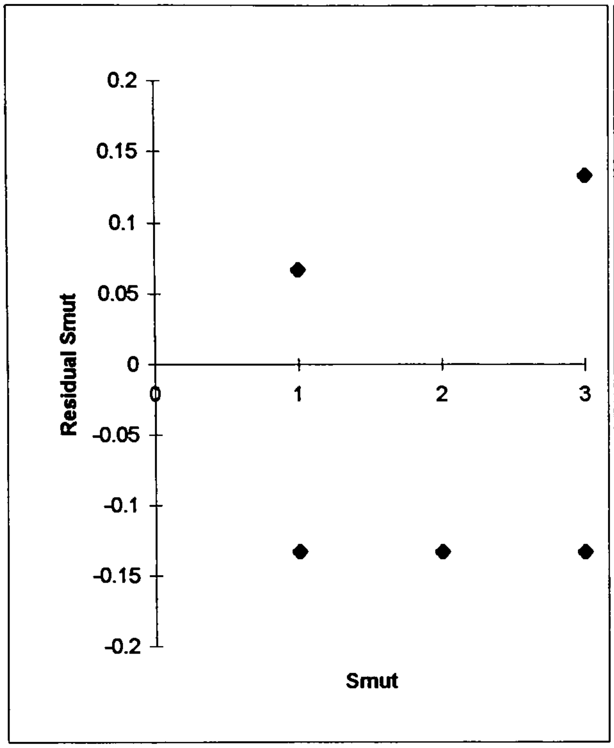


Figure 45. Residual Smut vs. Smut

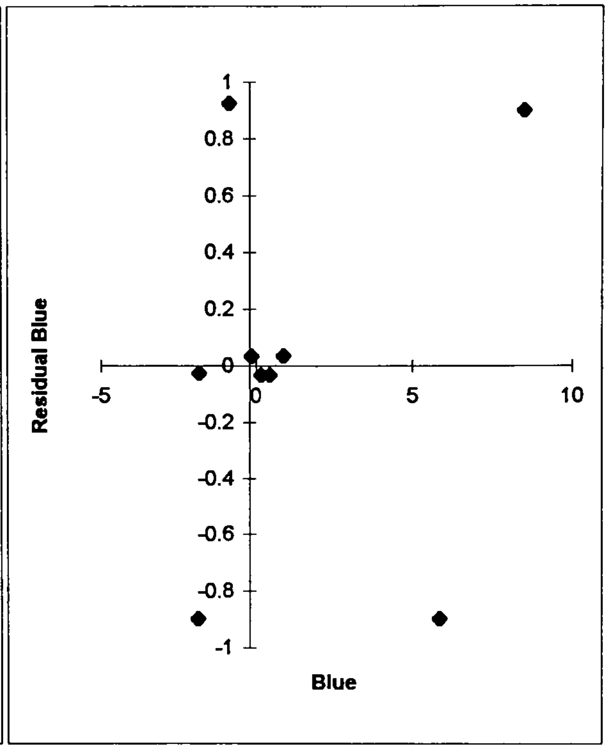


Figure 46. Residual Blue vs. Blue

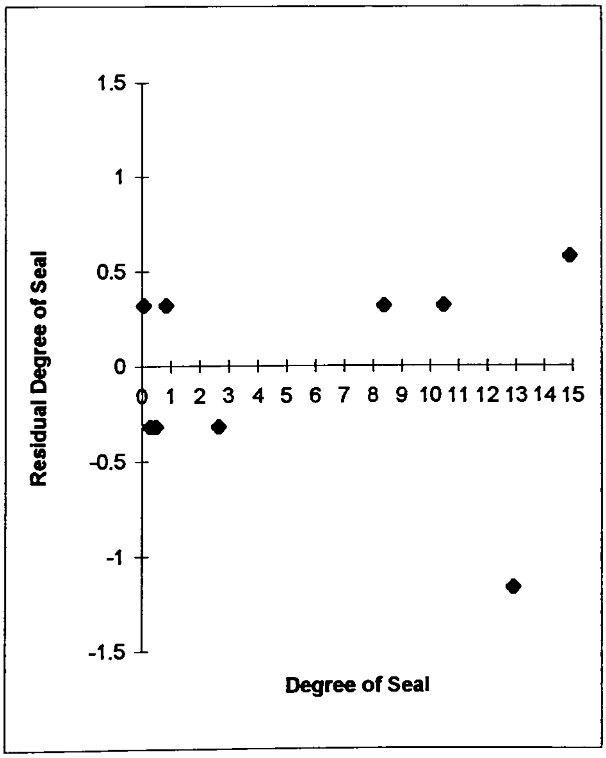


Figure 47. Residual Seal vs. Seal

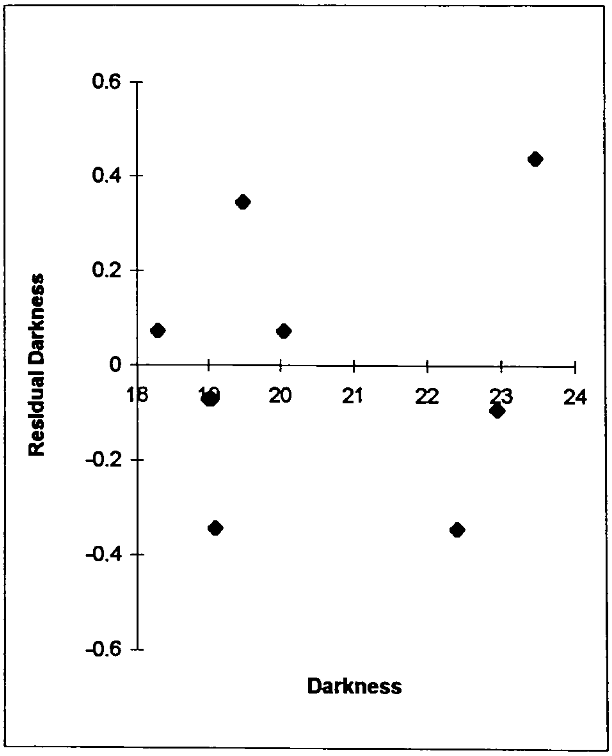


Figure 48. Residual Darkness vs. Darkness

Residual Data Excluding Run # 9

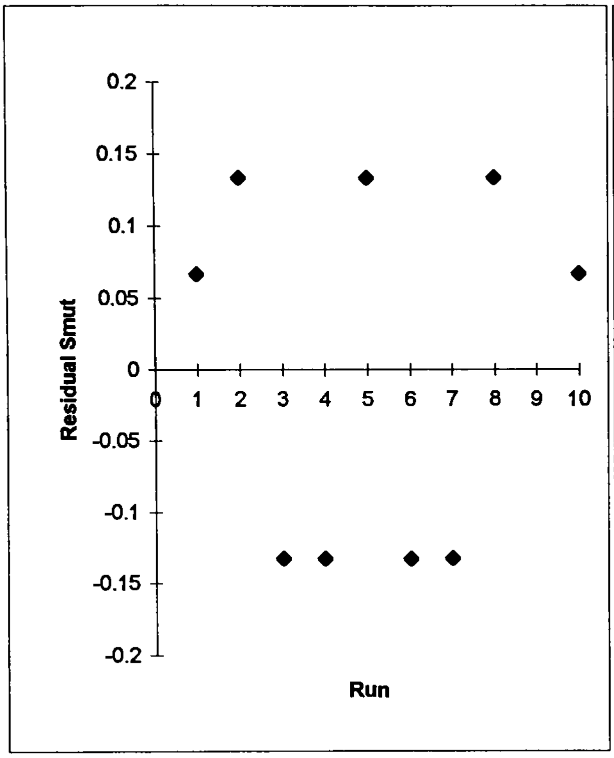


Figure 49. Residual Smut vs. Run

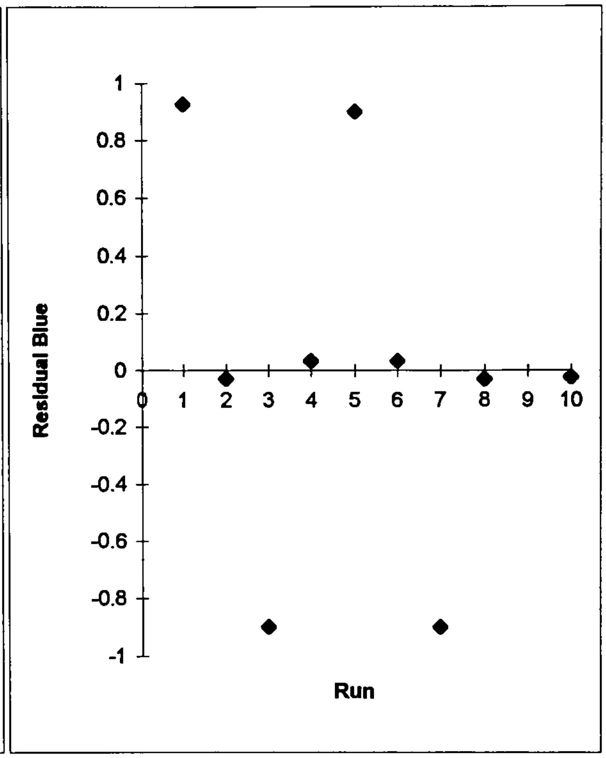


Figure 50. Residual Blue vs. Run

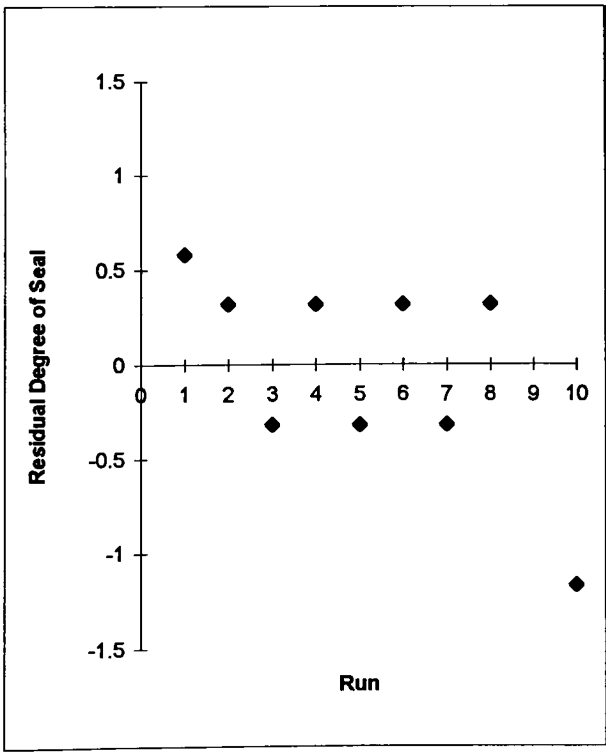


Figure 51. Residual Degree of Seal vs. Run

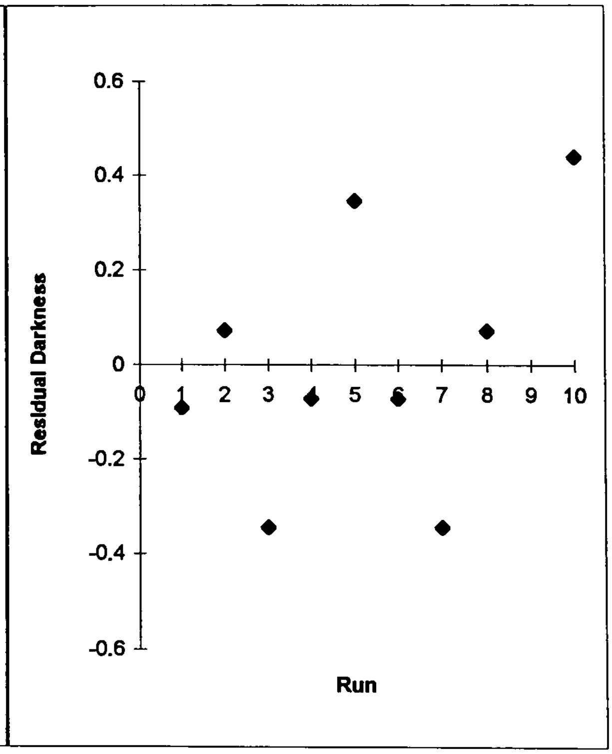
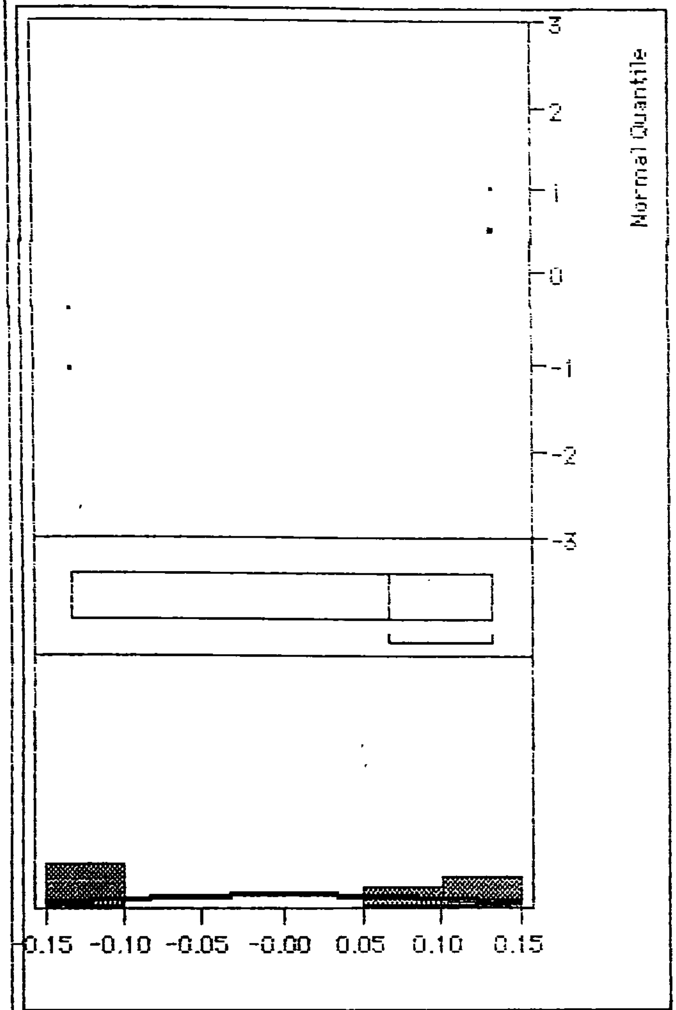


Figure 52. Residual Darkness vs. Run

Normality Test II

Residual SMUT133



Quantiles

Moments

Mean	0.000000
Std Dev	0.129099
Std Err Mean	0.043033
upper 95% Mean	0.099236
lower 95% Mean	-0.099236
N	9.000000
Sum Wgts	9.000000

Test for Normality

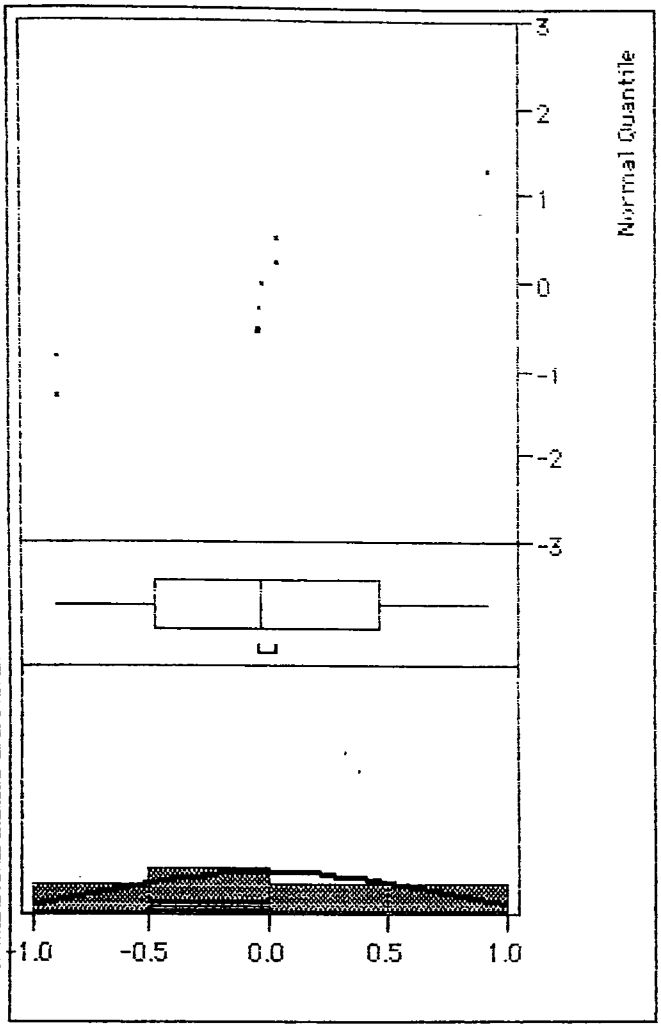
Shapiro-Wilk W Test

W	Prob<W
0.751277	0.0061

Figure 53

Normality Test II

Residual BLUE3



Quantiles

Moments

Mean	0.000000
Std Dev	0.640457
Std Err Mean	0.213486
upper 95% Mean	0.492304
lower 95% Mean	-0.492304
N	9.000000
Sum Wgts	9.000000

Test for Normality

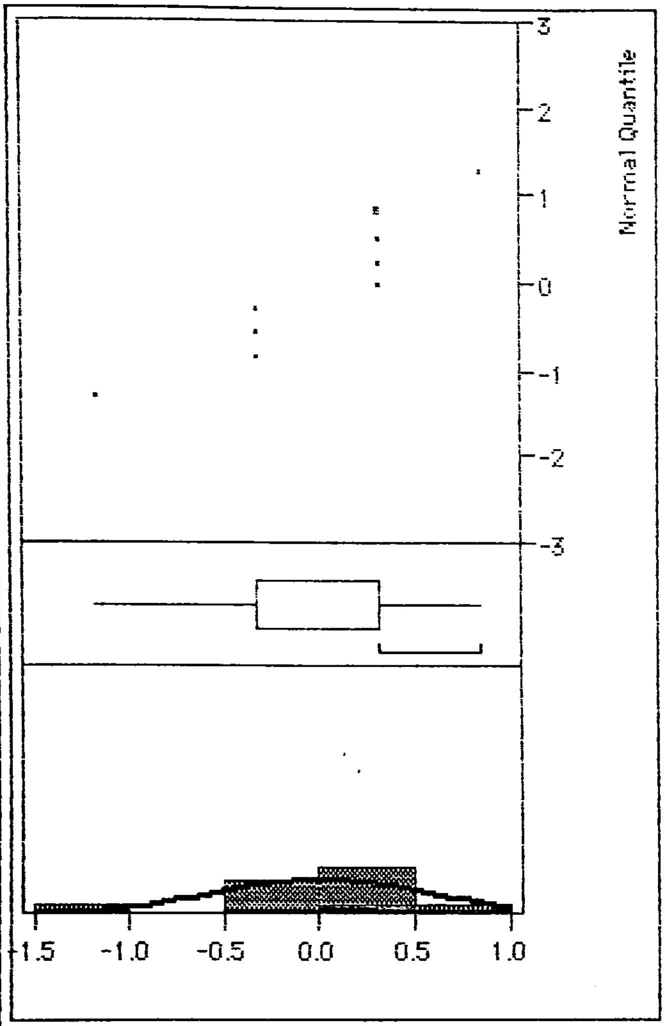
Shapiro-Wilk W Test

W	Prob<W
0.855732	0.0851

Figure 54

Normality Test II

Residual SEALING4



Quantiles

Moments

Mean	0.000000
Std Dev	0.592474
Std Err Mean	0.197491
upper 95% Mean	0.455420
lower 95% Mean	-0.455420
N	9.000000
Sum Wgts	9.000000

Test for Normality

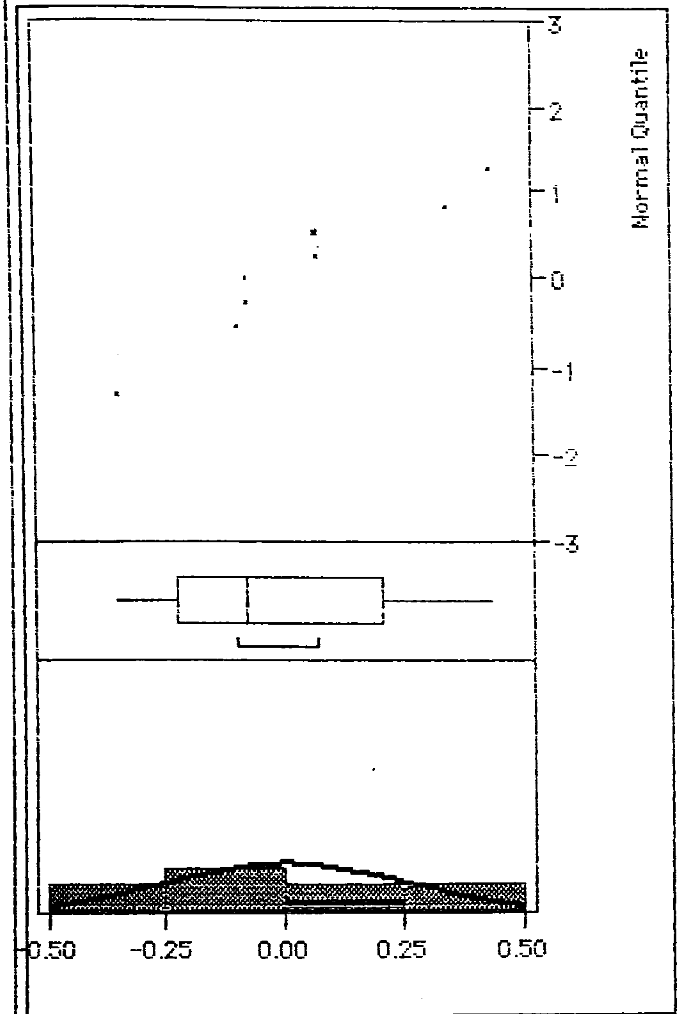
Shapiro-Wilk W Test

W	Prob<W
0.892237	0.2067

Figure 55

Normality Test II

Residual DARKNES3



Quantiles

Moments

Mean	0.000000
Std Dev	0.268407
Std Err Mean	0.089469
upper 95% Mean	0.206318
lower 95% Mean	-0.206318
N	9.000000
Sum Wgts	9.000000

Test for Normality

Shapiro-Wilk W Test

W	Prob<W
0.925874	0.4369

Figure 56

Conclusions from the residual plots excluding run #9:

- No trends exist implying that terms have not been left out of the model.
- No outliers.
- The mean of the residuals is zero in all cases (mean of $\varepsilon = 0$).
- The dispersion of the residuals about zero could be better. The variance of $\varepsilon \neq$ constant.
- No "strong" cyclical trends when the residuals are plotted verses the run numbers. Therefore ε`s are independent of one another.
- The normal probability plots and the tests for normality show that all of the residuals follow a normal distribution except smut (Figure 53).

t-Test[29]

In addition to checking the residuals excluding run number nine, run one and run ten, which were at the same factor settings, were checked to see that they were not statistically different. This analysis was done with a two sample t test. For this test the null hypothesis and alternative hypothesis were:

H_o: $\mu_1 - \mu 2 = \delta$ (Samples 1 and 2 are statistically the same)

H_a: $\mu_1 - \mu 2 \neq \delta$ (Samples 1 and 2 are statistically different)

$$t = \frac{\bar{x}_1 - \bar{x}_2 - \delta}{s_p\sqrt{(1/n_1)+(1/n_2)}}$$

reject the null hypothesis if $|t| \geq t_{\alpha/2,\nu}$

where, $\bar{x}_1$ = the mean of sample 1

$\bar{x}_2$ = the mean of sample 2

$\delta = 0$

n_1 = the sample size of sample 1

n_2 = the sample size of sample 2

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

s_1 = standard deviation of sample 1

s_2 = standard deviation of sample 2

$t_{\alpha/2,\nu}$ is looked up on a table containing values of $t_{\alpha,\nu}$ found in Appendix C

$\alpha = 0.05$ $\nu = n_1 + n_2 - 2$

The results of the t-test are shown in Table 15 and 16.

Table 15. t-Test Calculation Values

Response	n_1	n_2	x_1 mean	x_2 mean	s_1	s_2	s_1^2	s_2^2	s_p^2	s_p
Smut	50	50	1.2745	1.1961	0.45	0.4	0.2	0.16	0.18	0.43
Blue	5	5	-0.86	-1.81	0.56	0.86	0.31	0.74	0.53	0.72
Seal	2	2	13.392	13.175	2.16	0.39	4.68	0.15	2.42	1.55
Darkness	5	5	22.94	23.47	0.64	1.23	0.41	1.52	0.97	0.98

Table 16. t-Test Final Results

Response	t	Abs(t)	$t_{\alpha/2,v}$	Statistically the Same ?
Smut	0.92	0.92	1.96	Yes
Blue	2.072	2.072	2.306	Yes
Seal	0.14	0.14	4.303	Yes
Darkness	-0.851	0.851	2.306	Yes

The results show that run one and ten are the same inferring that nothing significant occurred in the process between the first run and the last confirmation run. As a result the experiment should be able to analyzed to get the main effects significant to smut formation, blue tinting, unacceptable seal, and dark parts.

JMP DOE Analysis

The data was run through the JMP design of experiments analysis. The results gained from each of the response variables will now be discussed. As a reminder, the objective for doing the design of experiments was to estimate the effects that free sulfuric acid concentration, seal pH, seal temperature, and DI water rinse temperature have on smut primarily and blue, seal, and darkness secondly. Design units (-1,0,1) were used to do the analysis because finding the exact equation (the parameter estimates) of the model was not the purpose of the experiment.

Each response was analyzed twice. Analysis I includes all the data. Analysis II did not include run number nine and some of the insignificant two-factor interactions (determined from Analysis I). The important results are summarized in tabular form and the theory behind the values are discussed in the introduction. The actual JMP output is in Appendix D - Appendix H.

Smut

Because the test for smut was a visual test it was the hardest response to evaluate. For this reason it was evaluated as both a continuous response and an ordinal response. Continuous values are treated as continuous measurement values, and ordinal values are treated as discrete categorical values that have an order. [30] The same results are concluded either way. The temperature of the seal is the parameter showing significance in forming smut. Lower seal temperatures mean better parts.

Ordinal

As an ordinal response the probability of getting a one at the lowest seal temperature is about 95 %. From the middle to the high seal temperatures there is a 100% probability that parts with a rating of three will be produced.

The other factors are not as significant as the seal temperature. In fact the temperature of the DI rinse is showing no change from the low to the high temperatures. Table 17 shows the predicted chance that a 1 or a three will be produced (in percents) at the low, medium, and high factor levels. See Appendix D for the JMP output.

Table 17. Smut Results (As an Ordinal Response Variable);

% Opportunity of Producing 1s and 3s for Each Factor at Three Levels

	Free Sulfuric		Seal pH		Seal Temp.		DI Temp.	
	1s	3s	1s	3s	1s	3s	1s	3s
Low	65%	20%	15%	78%	95%	1%	32%	57%
Med	25%	60%	27%	63%	0%	100%	32%	57%
High	10%	80%	50%	38%	0%	100%	32%	57%

Continuous

When considering smut as a continuous variable, the seal temperature is the significant factor, showing consistency with the ordinal results. Table 18 below shows the results for the continuous response analysis.

Table 18. Smut Analysis Results (As a Continuous Variable)

	Analysis I Includes All Runs & Interactions	Analysis II Excludes Run 9 & Insignificant Interactions
Summary of Fit	Very Good Predicting Model	Very Good Predicting Model
Rsquare	0.956833	0.982353
Root Mean Sq. Error	0.418121	0.258199
Analysis of Variance		
F Ratio	6.3331	18.5556
Prob>F	0.1431	0.0520
Significant Factors	Seal Temp.	Seal Temp.
Lack of Fit F Ratio Prob>F	------------	------------

Blue

Analysis I for the blue turned out more favorable than analysis II. Regardless, the results were the same for both.

Table 19. Blue Analysis Results

	Analysis I Includes All Runs & Interactions	Analysis II Excludes Run 9 & Insignificant Interactions
Summary of Fit Rsquare Root Mean Sq. Error	Very Good Predicting Model 0.991817 0.644977	Very Good Predicting Model 0.967475 1.045862
Analysis of Variance F Ratio Prob>F	Significant terms exist 34.6293 0.0283	Significant terms exist 17.8474 0.0193
Significant Factors	Seal Temp. Seal pH Seal Temp * Seal pH	Seal Temp. Seal pH Seal Temp * Seal pH
Lack of Fit F Ratio Prob>F	NO 0.8437 0.5270	NO 3.1360 0.3708

Degree of Seal

Like the scatter plots indicated the seal temperature is significant for yielding good sealed parts.

Table 20. Degree of Seal Analysis Results

	Analysis I Includes All Runs & Interactions	Analysis II Excludes Run 9 & Insignificant Interactions
Summary of Fit	Very Good Predicting Model	Very Good Predicting Model
Rsquare	0.952841	0.990247
Root Mean Sq. Error	2.696649	1.184947
Analysis of Variance		Significant terms exist
F Ratio	5.7728	33.8455
Prob>F	0.1556	0.0290
Significant Factors	Seal Temp.	Seal Temp.
Lack of Fit	NO	NO
F Ratio	6.1286	0.3764
Prob>F	0.2444	0.6497

Darkness

Analysis II gives better results than analysis I. It has a better prediction rating and indicates the presence of significant terms.

Table 21. Darkness Analysis Results

	Analysis I Includes All Runs & Interactions	Analysis II Excludes Run 9 & Insignificant Interactions
Summary of Fit Rsquare Root Mean Sq. Error	Very Good Predicting Model 0.959975 0.813269	Very Good Predicting Model 0.981272 0.438307
Analysis of Variance F Ratio Prob>F	No Significant terms 6.8526 0.1332	Significant terms exist 33.8455 0.0086
Significant Factors	------------	Seal pH Seal Temp. Seal pH * Seal Temp.
Lack of Fit F Ratio Prob>F	NO 8.4184 0.2113	NO 1.5518 0.4937

Correlations

Another analysis that can be done is a correlation between all the inputs and outputs. The JMP output for this analysis can be found in appendix H and the summary of the results can be found below.

Table 22. Significant Pairwise Correlations (Including run 9)

SMUT	SEAL TEMPERATURE
BLUE	SEAL TEMPERATURE
DARKNESS	SEAL PH
DARKNESS	SMUT
SEAL	SEAL TEMPERATURE
SEAL	SMUT

Summary of Results

Significant Factors

Significant main effects were the desired results of the DOE. These results are summarized in the table below.

Table 23. Significant Main Effects

	Smut	Blue	Seal	Darkness
Significant Factors	Temp. Seal	Temp. Seal pH Seal	Temp. Seal	Temp. Seal pH Seal
Best Level of the Significant Factors	Low	Low Medium/High	High	Low High

Prediction Profile

Prediction profiles were created using the significant factors, seal pH and seal temperature. Minimizing the smut problem occurs at high pHs and low temperatures of the seal. In addition, part darkness is minimized (remembering that the higher the darkness value, the better the part). This is not a viable solution though because the parts will not seal at these conditions (3 is the maximum number acceptable for sealing) , and parts with green tints may result (0 is desirable). Figure 61 illustrates the first prediction profile described above.

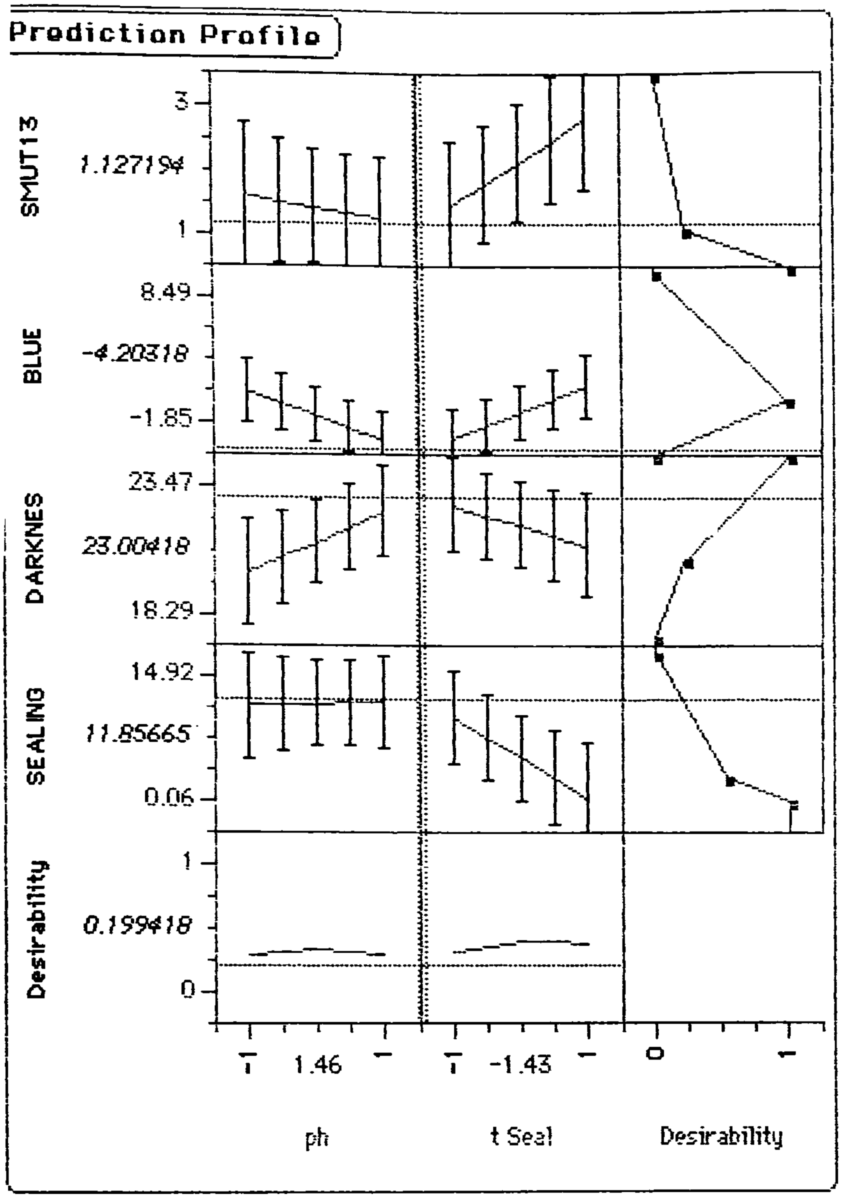


Figure 57. Prediction Profile
Minimized Smut

With the second prediction profile (figure 62) the desirability increases by 46%. The smut and darkness gets worse while the seal and blue get better, but overall predict more favorable results. The pH remains at the high level while the seal temperature should be set to the mid-high level.

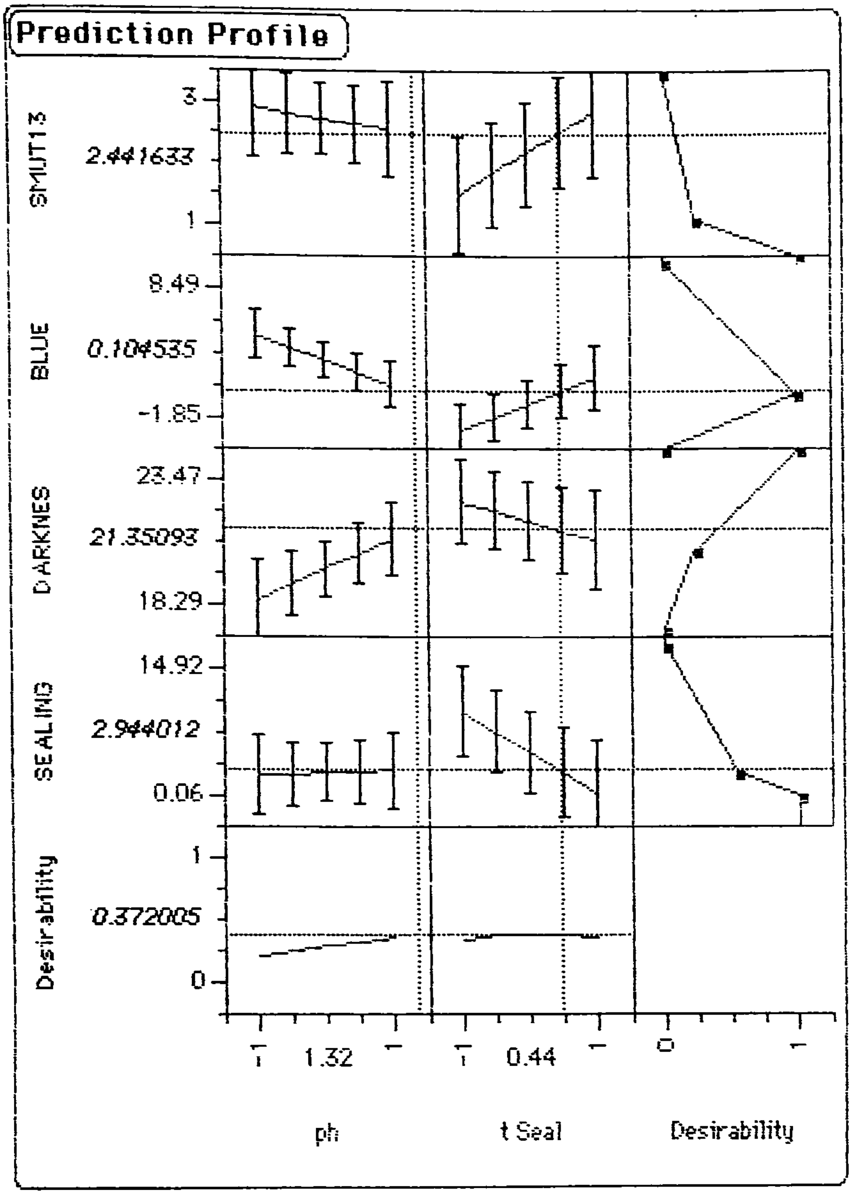


Figure 58. Prediction Profile
Most Desireable

RECOMMENDATIONS

There are many recommendations that can be made after having gone through the design of experiments on the anodizing line. They are listed below and then expanded upon.

1. Create a process window for the process when the same parts for the experiment are run.
2. Research and develop a method to measure the fluoride concentration in the seal tank.
3. Take a chemical analysis of the fluoride level on a regular basis.
4. Make control charts of the data that is collected from the fluoride analysis.
5. Purchase a rectifier for the lab.
6. Do another design of experiments to include the seal's fluoride level, the age/activity of the seal, the anodizing temperature and disregard the free sulfuric acid concentration and the DI rinse temperature after the seal.
7. Investigate the correlation between smut and darkness. Perhaps a test for smut has now been discovered.
8. Buy a spectrophotometer.

The first recommendation is to create a process window. A process window is similar to a two factor design of experiment. Everything in the process is held stable and under control. A process window depicts how two process parameters affect part characteristics.[31] The two recommended process parameters for the anodize line are seal temperature and seal pH, because they are the two factors that had significant effects on the process. For a process window, one variable is on the x-axis and the other variable is on the y-axis. Different levels and combinations of the two factors are run and the responses that are produced are recorded and plotted. As long as the process is under control the process window is a powerful tool for process development, improvement, and optimization. An example of a process window is illustrated below (Figure 63). Note that the data is not real. The white "window" is the location in the process where good parts will be produced. The optimum settings for the process would be in the middle of the "window".

Advantages to a process window are as follows:

- Data can be collected from production runs. Machine time does not have to be taken up by experimentation alone.
- Optimal settings can be determined for the two process parameters.
- It can be determined if the process is capable of producing "good" parts within the ranges of the process variables where the optimum is thought to be. The possibility exists that there is no open "window", and all combinations of the two variables cause defects.

The white area represents the area where good parts are produced.

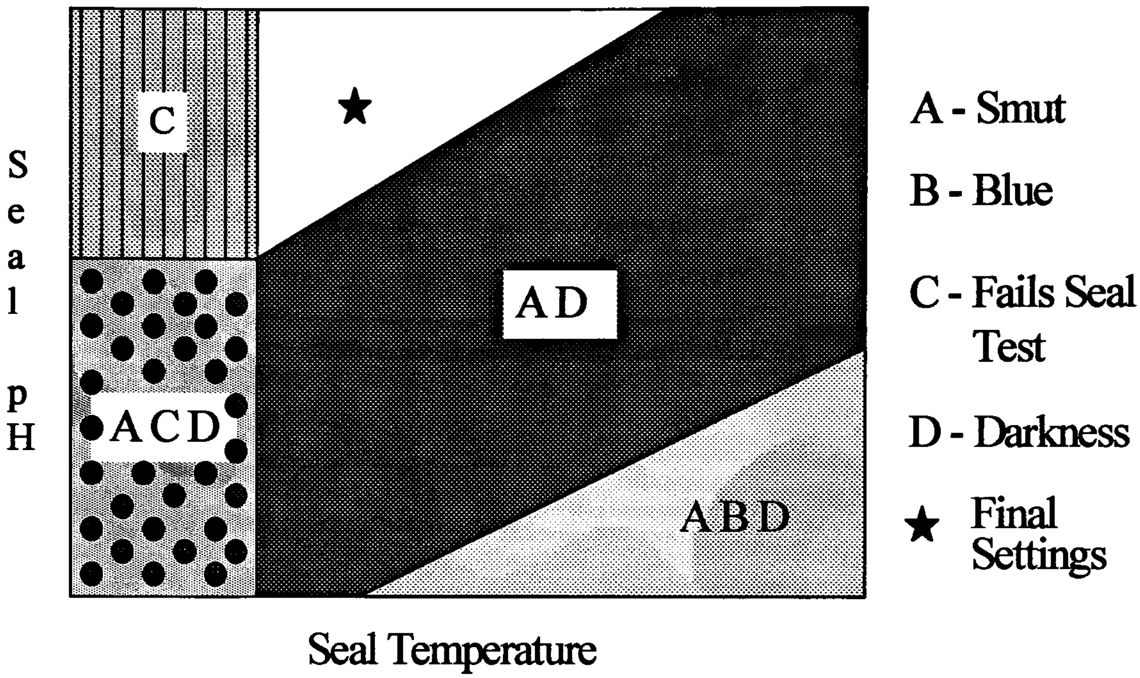


Figure 63. Process Window Example

The next three recommendations all deal with the fluoride concentration in the seal tank. Through research and talking to experts this is a key process parameter, and it is important to monitor it. Not enough research has been done in the field to determine all the effects of too high or too low a fluoride level. With a means to measure and monitor the levels of fluoride, more knowledge will be gained of the process.

The next recommendation is to purchase a rectifier for the lab. Without it, reliable experimentation is impossible thus forcing all experimentation to occur on the production line and because of this, too much production time is lost. A rectifier would provide a means to experiment more at lower cost.

Because the seal's fluoride level, the seal's age/activity, and the anodizing temperature were not included in the DOE, another experiment may be run to include these three factors. The time to do this experiment would be before dumping the seal tank. This strategy would allow obtaining old seal from the dumping and new seal from the making of a new tank. Of course this DOE would not be able to be run without accomplishing the first three recommendations first.

There appears to be a correlation between smut and darkness. If so, the test for darkness could be a test for smut, which would eliminated the visual test for smut. An acceptable smut standard between the customer and the supplier could be established, rather than relying on a visual measure. Graphs below support the hypothesis that smut and darkness are the same. Since only ten data points are available from the experiment, more testing should be done to prove the hypothesis.

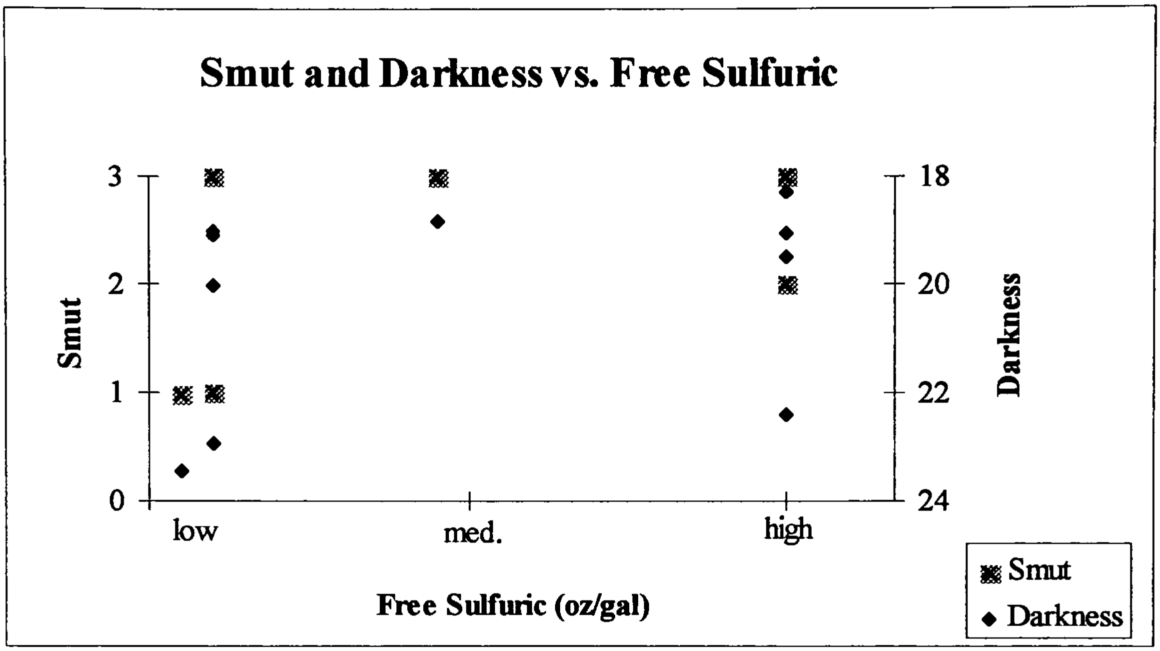


Figure 64

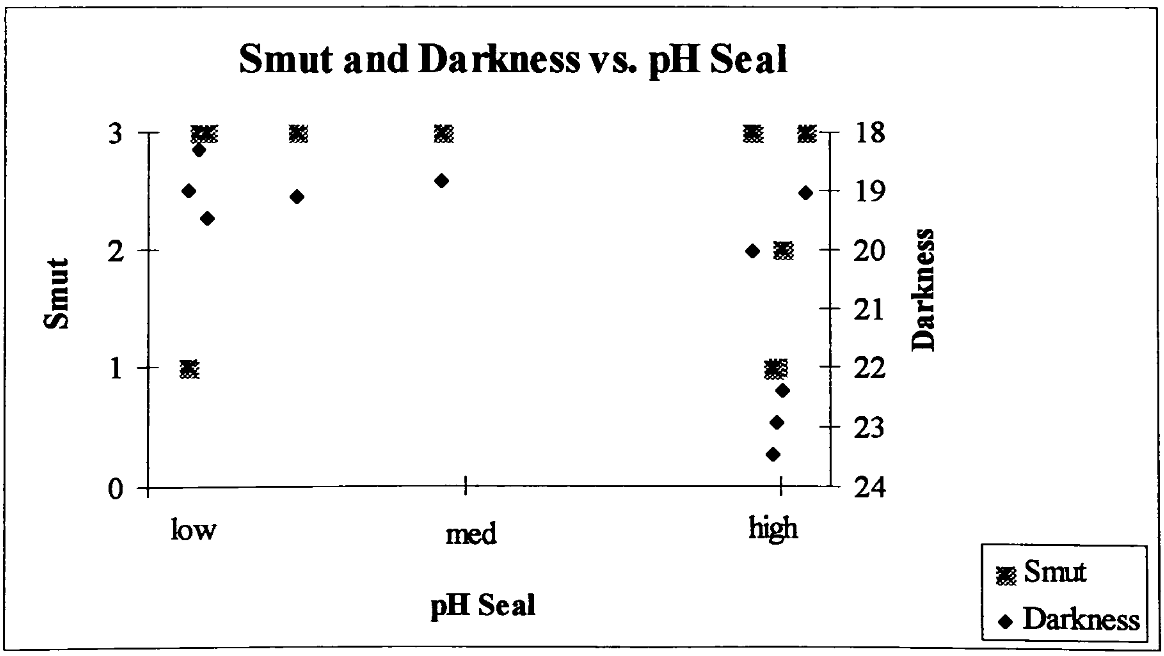


Figure 65

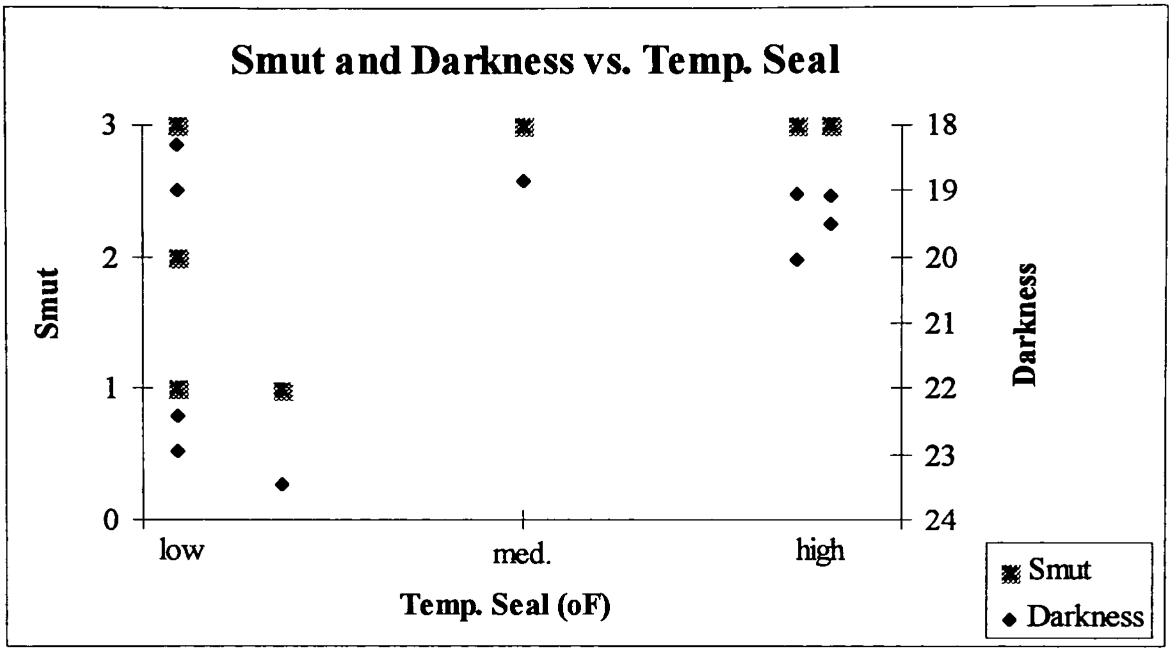


Figure 66

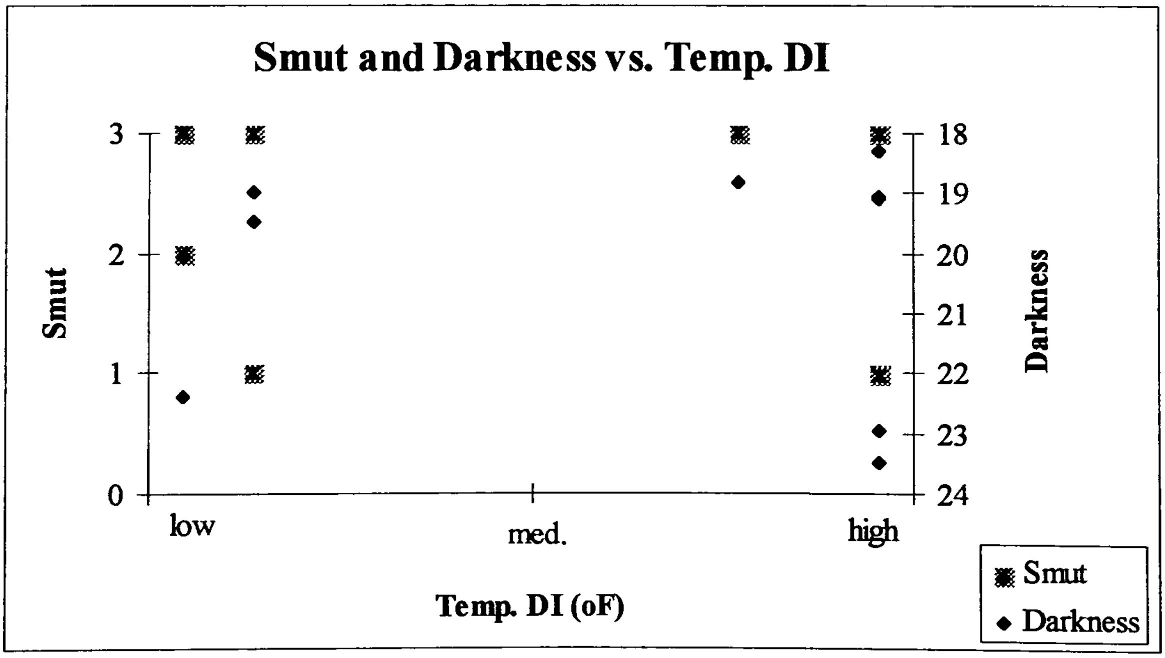


Figure 67

The seventh recommendation leads to the eighth recommendation to buy a spectrophotometer provided the hypothesis is true. Not only is darkness data measured using a spectrophotometer, but also blue data is measured. Once again an acceptable standard could be established with the customer for both blue and smut. In addition, product capability information could be supplied to the customer.

Appendices

Appendix A

Aluminum Alloy Compositions and Designations

Wrought Aluminum

The aluminum alloy system assigns a four-digit numerical designation to each grade. The first digit of the four-digit number indicates the major alloying element. Table below lists the alloy grougs. Whenever the aluminum is 99.0 % or greater then it falls into the 1000 series group. The alloy group in the 2000 series through the 7000 series is dependant on the alloying element with the highest mean precentage. If the greatest mean percentage is common to more than one element, then the group choice will be in order of group sequence Cu, Mn, Si, Mg, Mg_2Si, Zn, or others. The following table is the alloy designations of the Aluminum Association that is most commonly used in the United States.

Table A1. Wrought Al Alloy Groups First Digit Designation[32]

Major Alloying Element	Designation
99.0% or greater of pure Aluminum	1XXX
Copper	2XXX
Manganese	3XXX
Silicon	4XXX
Magnesium	5XXX
Magnesium and Silicon	6XXX
Zinc	7XXX
Other Element	8XXX
Unused Series	9XXX

The second digit of the four-digit designation indicates impurity limits or modifications to the original alloy. Finally, the last two digits identify the aluminum alloy or indicate the aluminum purity. The last two digits of the 1XXX series indicates the aluminum content above 99% in hundredths. For example, 1040 alloy contains 99.4% aluminum.

Cast Aluminum

The cast aluminum alloy designations are different than the wrought aluminum designations. They are designated by three digits a period and another digit. Sometimes a letter prefix is used to signify alloy or impurity limits. Like wrought alloys the first digit indicates the major alloying element. The second and third digits identify the alloy within a group. Finally the last digit after the decimal point indicates the final form, either 0 for a casting or 1 for an ingot. Designations are found in the table below.

Table A2. Cast Al Alloy Groups First Digit Designation[33]

Major Alloying Element	Designation
99.5% or greater of pure Aluminum	1XX.X
Copper	2XX.X
Silicon + Copper or Manganese	3XX.X
Silicon	4XX.X
Magnesium	5XX.X
Unused Series	6XX.X
Zinc	7XX.X
Tin	8XX.X
Other Element	9XX.X

Special Treatment

When additional treatments are done on the aluminum, they must be specified by a suffix.

Table A3. Alloy Suffix Designations[34]

XXXX	F	As fabricated, no special controls
	W	Solution heat treated (used only on alloys that naturally age harden)
	O	Annealed (wrought alloys only)
	H	Strain hardened (cold worked to increase strength), wrought alloys only
	T	Thermally treated to produce effects other than F, O, or H

The H letter is followed by one, two, or three digits indicating the degree of cold working.

XXXX-H1 Strain hardened only

XXXX-H2 Strain hardened and partially annealed

XXXX-H3 Strain hardened and stabilized by low-temperature thermal treatments

XXXX-H--2 Quarter-hard

XXXX-H--4 Half-hard

XXXX-H--6 Three quarters hard

XXXX-H--8 Full hard

The T is followed by one, two, or three digits to indicate various thermal treatments. [35]

XXXX- T1	Cooled from a hot working temperature and naturally aged
XXXX- T2	Annealed (cast products only)
XXXX- T3	Solution treated and cold worked
XXXX- T4	Solution treated and naturally aged
XXXX- T5	Cooled from a hot work temperature and furnace aged
XXXX- T6	Solution treated and furnace aged
XXXX- T7	Solution treated and stabilized
XXXX- T8	Solution treated, cold worked, and furnace aged
XXXX- T9	Solution treated, furnace aged, and cold worked
XXXX- T10	Cooled from an elevated temperature, furnace aged, and cold worked
XXXX-T42	Solution treated from O or F temper and naturally aged
XXXX-T51	Stress relieved by stretching
XXXX-T510	Stress relieved by stretching with no further processing
XXXX-T511	Stress relieved by stretching and minor straightening
XXXX-T52	Stress relieved by compression
XXXX-T54	Stress relieved by stretching and compression
XXXX-T62	Solution treated from O or F temper and furnace aged

Appendix B

Types of Designs

There are several different types of Designs of Experiments. Full factorial, fractional factorial, response surface, and screening are the most popular. Different experiments will lead to different results. For this reason it is important to clearly define and understand the objective of the experiment. It is desireable to get the maximum information with the minimum amount of experimental runs.

Full Factorial Design

A factorial design is an experimental plan consisting of all possible combinations of the factors and levels. For the most part two level factor designs are the most popular. The general form for these design types is 2^k, where k is the number of factors at two levels. Therefore, a three factor design would consist of $2x2x2 = 2^3 = 8$ experiments.

There are many advantages of a factorial design over other experiments. First, this design type requires relatively few runs per factor studied with the most efficient estimate of factor effects over the experimental region of interest. The factorial design can be run in an iterative and sequential manner. Because of this advantage, a fraction of the factorial design can be run to look at a large number of factors superficially. When more detailed information is needed additional experiments can be added to the existing fraction of the factorial. Another positive point is that the data collected from a factorial experiment is simple and easy to manipulate and interpret with calculations and graphical analysis. In addition the designs provide an efficient means for collecting the data that is to be analyzed. Lastly the factorial type designs can be easily blocked to eliminate the effects of bias error.

In order to generate a factorial design a systematic ordering plan is needed to list all of the possible factor setting combinations. However, when it comes time to run the experiments, random order is desired. The computer is a helpful tool for outputing a random experiment with all of the factor setting combinations once the variable names and levels are inputed. If a computer and/or the necessary program is unavailable, then the factorial type design must be done by hand. For the first factor, the low level and the high level alternate for the total number of experiments. For the second factor, the lows and highs are alternated in groups of two. For the third factor, the levels are alternated in groups of four. The alternating group size increases by a power of two with each addition of a factor, and the last factor will alternate its levels in group sizes equal to half the total number of experiments. Table B1 depicts the ordering patterns, where the plus is the high level and the minus is the low level.

Table B1. Full Factoral Design Ordering Patterns.

RUN	Factor A	Factor B	Factor C	Factor D
1	+	+	+	+
2	-	+	+	+
3	+	-	+	+
4	-	-	+	+
5	+	+	-	+
6	-	+	-	+
7	+	-	-	+
8	-	-	-	+
9	+	+	+	-
10	-	+	+	-
11	+	-	+	-
12	-	-	+	-
13	+	+	-	-
14	-	+	-	-
15	+	-	-	-
16	-	-	-	-

Groupings = 2^1 Groupings = 2^2 Groupings = 2^3 Groupings = 2^4

Notice factor D, the last factor has a group size of half of the total experiment size. This table would be randomized when running the experiment.

A polynomial model can be estimated from the experimental data. The model consists of an intercept (β_o), the main effects and their coefficients ($\Sigma x_n\beta_n$), and all possible interactions and their coefficients. For example for the 2-level, 4-factor design above the model would be $y = \beta_o + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_1 x_2 + \beta_6 x_1 x_3 + \beta_7 x_1 x_4 + \beta_8 x_2 x_3 + \beta_9 x_2 x_4 + \beta_{10} x_3 x_4 + \beta_{11} x_1 x_2 x_3 + \beta_{12} x_1 x_2 x_4 + \beta_{13} x_1 x_3 x_4 + \beta_{14} x_2 x_3 x_4 + \beta_{15} x_1 x_2 x_3 x_4$. The polynomial model is a Taylor series expansion which is a mathematical equation used to approximate a complex function within a specified region. The higher order terms (β_{15} being the highest) generally contribute less to the predictive ability of the model.

<u>Fractional Factorial Experiment</u>

Like the name implies, a fractional factorial design has a fraction of the runs of a full factorial design. Because there are less runs some information is lost like the effects of interactions between factors. Fractionation may be considered to be a structured losing of data, because it is determined ahead of time what information is being lost. A fractional factorial design of experiment is created by fractionating the full factorial design in a structured method. For a two level factorial design, it is fractionated by factors of $1/2^n$ (n=1,2,3,...). For example, a 2-level, 4-factor factorial design has 2^4 runs (16 runs). Running eight of the total sixteen runs would represent half of the full factorial [$(1/2)\ 2^4 = (1/2^1)(2^4) = (2^{-1})(2^4) = 2^{4-1} = 2^3 = 8$ runs. The general notation for fractional 2-level designs is 2^{k-p}, where two indicates that all the factors are at two levels, the k represents the total number of factors, and the p represents the degree of fractionation.

The greatest advantage of using a fractional factorial over a full factorial is the resource savings. When time and/or costs are tight, then fractional factorial designs are good choices provided that the information that is lost is not essential. Recall from above the polynomial model for the 2-level, 4-factor design was $y = \beta_o + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_1 x_2 + \beta_6 x1\ x3 + \beta_7 x_1 x_4 + \beta_8 x_2 x_3 + \beta_9 x_2 x_4 + \beta_{10} x_3 x_4 + \beta_{11} x_1 x_2 x_3 + \beta_{12} x_1 x_2 x_4 + \beta_{13} x_1 x_3 x_4 + \beta_{14} x_2 x_3 x_4 + \beta_{15} x_1 x_2 x_3 x_4$. With a 2^{4-1} fractional factorial design only eight of the parameters of the experiment will be determined. The higher order interactions are the ones that will not be predicted (($\beta_{11} x_1 x_2 x_3$, $\beta_{12} x_1 x_2 x_4$, $\beta_{13} x_1 x_3 x_4$, $\beta_{14} x_2 x_3 x_4$, $\beta_{15} x_1 x_2 x_3 x_4$, and three of the two way interactions). The problem may occur in trying to determine which three of the six two-way interactions are being predicted. because, they are confounded. With some knowledge of the process, this becomes an easily determined problem.

There are two steps involved in generating a fractional factorial design. The first is to determine the number of factors that would be involved in a full factorial design with the number of experiments for the fractional factorial design. For example for a 2^{4-1} fractional factorial design eight experiments are involved. A full factorial of the same size would consist of three factors (2^3). Next this full design with three of the four factors using plus ones and negative ones is generated. Taking the example from above and making it a fractional design would yield the following for the first step:

RUN	Factor A	Factor B	Factor C
1	+	+	+
2	-	+	+
3	+	-	+
4	-	-	+
5	+	+	-
6	-	+	-
7	+	-	-
8	-	-	-

The second step assigns appropriate plus and minus ones to the fourth factor (factor D). This is accomplished by using a generator, which will generate the needed information. Generator information is found in table B2. So for a 2^{4-1} fractional factorial design the generator is 4= $^{+}/_{-}$ 123, meaning that the three factors are multiplied together resulting in two halves of the full 2^4 factorial design as shown below.

Factor D = $^{+}$(Factor A)(Factor B)(Factor C)

RUN	Factor A	Factor B	Factor C	Factor D
1	+	+	+	+
2	-	+	+	-
3	+	-	+	-
4	-	-	+	+
5	+	+	-	-
6	-	+	-	+
7	+	-	-	+
8	-	-	-	-

Factor D = -(Factor A)(Factor B)(Factor C)

RUN	Factor A	Factor B	Factor C	Factor D
1	+	+	+	-
2	-	+	+	+
3	+	-	+	+
4	-	-	+	-
5	+	+	-	+
6	-	+	-	-
7	+	-	-	-
8	-	-	-	+

What is learned from one design above should be the same that is learned from the other. The problem with a fractional factorial design is that confounding factor effects will exist as mentioned earlier. When two or more factor effects are confused with one another, or linked together due to fractionation, then they are confounded. The number of runs an experiment has determines the number of factor effects that can be estimated. For example the 2^4 design estimates sixteen effects, whereas a 2^{4-1} design can only estimate eight of the sixteen effects. The table below is a good summary of factorial designs.

Table B2. Fractionation Table [58]

Number of Factors	Number of Runs	Resolution	Design Type	Fractionation	Fractional Design Generators
3	8	--	2^3	full	None
	4	III	2^{3-1}	1/2	3=12
4	16	--	2^4	full	None
	8	IV	2^{4-1}	1/2	4=123
	32	--	2^5	full	None
5	16	V	2^{5-1}	1/2	5=1234
	8	III	2^{5-2}	1/4	4=12,5=13
	64	--	2^6	full	None
6	32	VI	2^{6-1}	1/2	6=12345
	16	IV	2^{6-2}	1/4	5=123,6=234
	8	III	2^{6-3}	1/8	4=12,5=13,6=23
	64	VII	2^{7-1}	1/2	7=123456
7	32	IV	2^{7-2}	1/4	6=1234,7=1245
	16	IV	2^{7-3}	1/8	5=123,6=234,7=134
	8	III	2^{7-4}	1/16	4=12,5=13,6=23,7=123
	64	V	2^{8-2}	1/4	7=1234,8=1256
8	32	IV	2^{8-3}	1/8	6=123,7=124,8=2345
	16	IV	2^{8-4}	1/16	5=234,6=134,7=123,8=124
	8	III	2^{8-5}	1/32	4=23,5=24,6=13,7=12,8=1234

The third column of the fractionation table (Table B2) is the design resolution, which is a quick indicator of the worst degree of counfounding for the fractional factorial design. The meaning to each roman numeral is below.

Table B3. Design Resolution

Resolution	Degree of Confounding	Applications	Confounding
III	Severe	Screening	Main effects confounded with 2-factor interactions
IV	Moderate	Estimate effects	Main effects with 3-factor interactions, 2-factor with other 2-factor interactions
V	Good	Response surface	Main effects with 4-factor interactions, 2-factor with 3-factor interactions
V+	Excellent	Response surface	Main effects with 5-factor interactions or higher 2-factor with 4-factor interactions or higher

In general, the higher the degree of fractionation, the lower resolution number, and the higher the degree of confounding. Furthermore as the degree of confounding increases, the amount of bias in the estimates of selected factor effects also increases. The confounding variables are determined by the computer.

<u>Screening</u>

The screening is simply a highly fractionated, severely confounding fractional factorial design of resolution III. Screening experiments are useful for determining the most important factors that affect a particular response when there are many factors (6-

30), little knowledge is known about interactions, and resources are limited. With a screening experiment, obtaining a model is not the objective, whereas determining the main effects is. Knowing the significant main effects can lead to another experiment with only the important factors.

<u>Response Surface Design</u>

A response surface design or central composite design is a combination of two different designs: a factorial design and a one factor at a time design. See Figure B1.

The fac.orial portion provides information about the main effects and interactions while the axial portion provides information of the curvature effects and some on the main effects. When high quality prediction is required, a response surface design is useful because it will predict quadratic terms. For example, the polynomial model,

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_1 x_2$$

becomes

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_1 x_2 + \beta_4 x_1^2 + \beta_5 x_2^2$$

with a response resurface design.

Beside producing a better prediction model, another advantage to this design type is that the factorial portion can be completed and then the axial portion can be added later. All the advantages of a factorial design also apply to a response surface design.

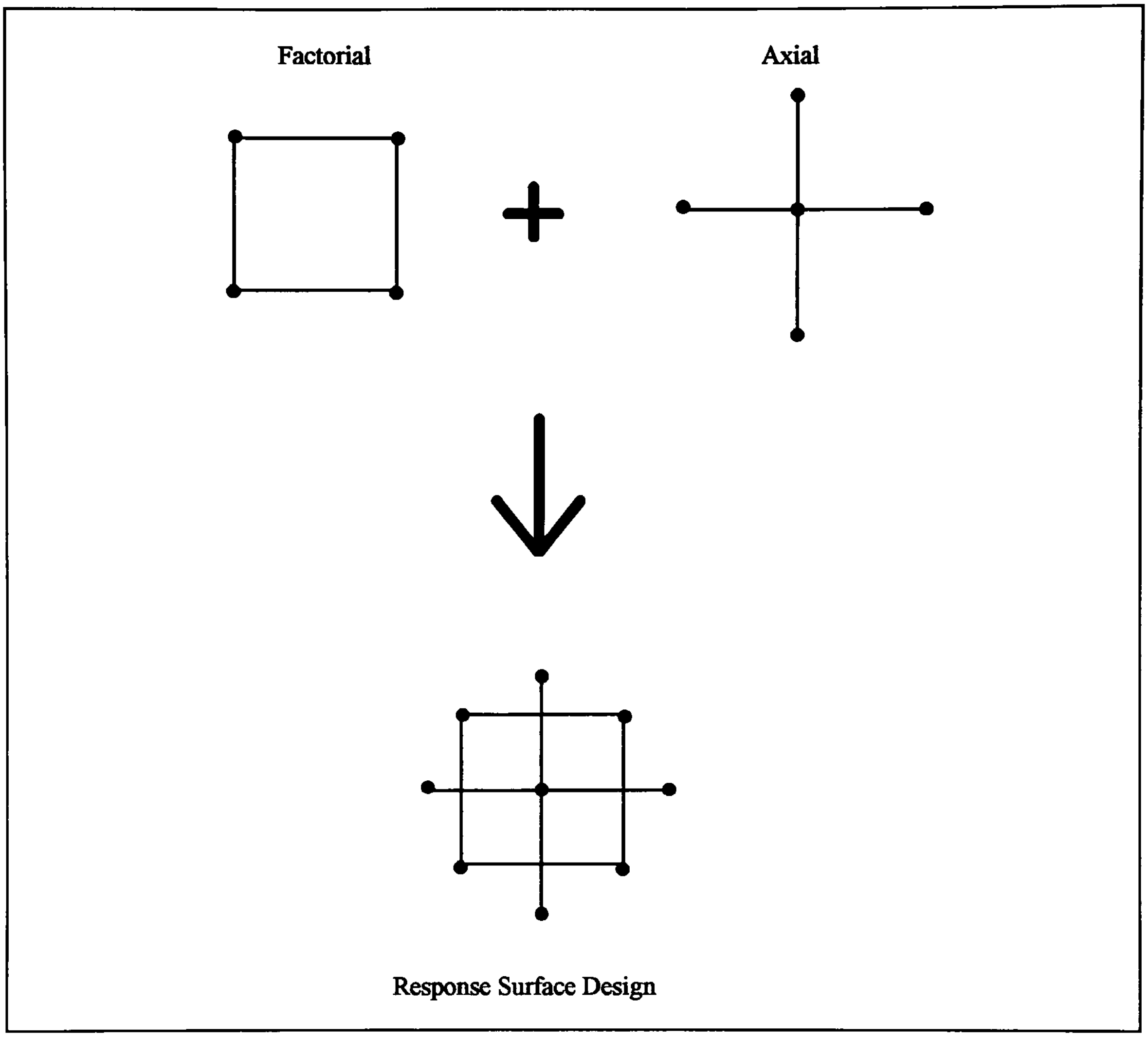


Figure B1. Composition of a Response Surface Design

In summary, many types of Design of Experiments exist. Depending on the objectives of the experiment, different designs need to be selected. If resources are tight, then a screening experiment may be appropriate, but if a better prediction model is desired, then a response surface design should be considered.

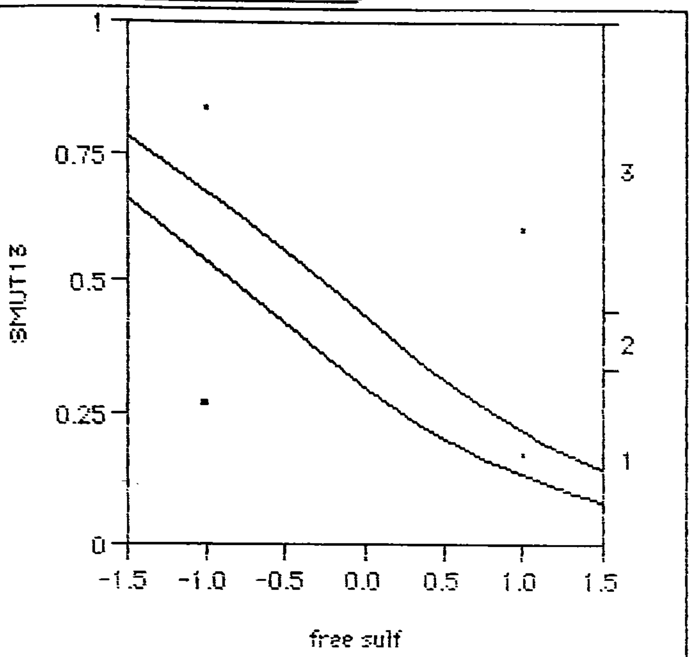
Appendix C

Table C1. Values of $t_{\alpha,\nu}$ Used for the t-Test [37]

ν	α = .10	α = .05	α = .025	α = .01	α = .005	ν
1	3.078	6.314	12.706	31.821	63.657	1
2	1.886	2.920	4.303	6.965	9.925	2
3	1.638	2.353	3.182	4.451	5.841	3
4	1.533	2.132	2.776	3.747	4.604	4
5	1.476	2.015	2.571	3.365	4.032	5
6	1.440	1.943	2.447	3.143	3.707	6
7	1.415	1.895	2.365	2.998	3.499	7
8	1.397	1.860	2.306	2.896	3.355	8
9	1.383	1.833	2.262	2.821	3.250	9
10	1.372	1.812	2.228	2.764	3.169	10
11	1.363	1.796	2.201	2.718	3.106	11
12	1.356	1.782	2.179	2.681	3.055	12
13	1.35	1.771	2.160	2.650	3.012	13
14	1.345	1.761	2.145	2.624	2.977	14
15	1.341	1.753	2.131	2.602	2.947	15
16	1.337	1.746	2.120	2.583	2.921	16
17	1.333	1.740	2.110	2.567	2.898	17
18	1.330	1.734	2.101	2.552	2.878	18
19	1.328	1.729	2.093	2.539	2.861	19
20	1.325	1.725	2.086	2.528	2.845	20
21	1.323	1.721	2.080	2.518	2.831	21
22	1.321	1.717	2.074	2.508	2.819	22
23	1.319	1.714	2.069	2.500	2.807	23
24	1.318	1.711	2.064	2.492	2.797	24
25	1.316	1.708	2.060	2.485	2.787	25
26	1.315	1.706	2.056	2.479	2.779	26
27	1.314	1.703	2.052	2.473	2.771	27
28	1.313	1.701	2.048	2.467	2.763	28
29	1.311	1.699	2.045	2.462	2.756	29
inf.	1.282	1.645	1.960	2.326	2.576	inf.

Ordinal Response

SMUT13 By free sulf



Converged by Objective

Whole-Model Test

Model	-LogLikelihood	DF	ChiSquare	Prob>ChiSq
Difference	1.0966366	1	2.193273	0.138614
Full	7.3353581			
Reduced	8.4319948			

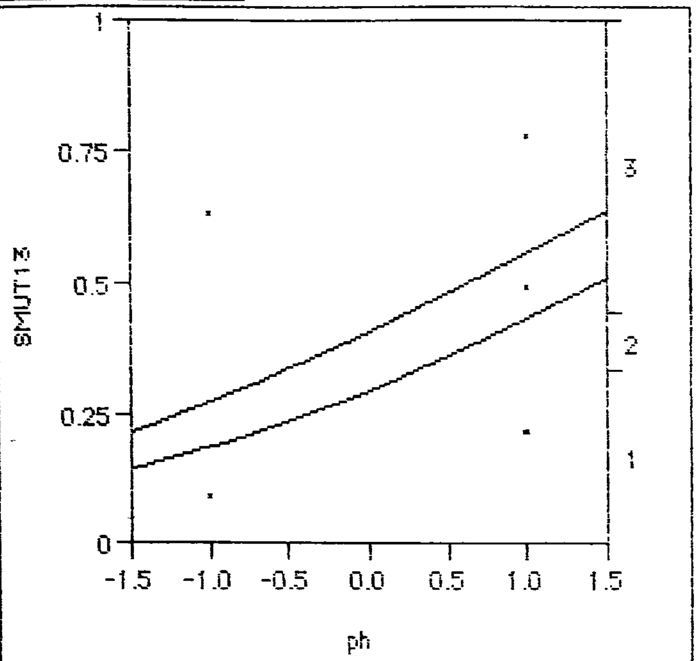
RSquare (U)	0.1301
Observations (or Sum Wgts)	9

Parameter Estimates

Term	Estimate	Std Error	ChiSquare	Prob>ChiSq
Intercept	-0.8501867	0.8150952	1.09	0.2969
Intercept	-0.2569031	0.771635	0.11	0.7393
free sulf	-1.0273971	0.7534136	1.86	0.1727

Ordinal Response

SMUT13 By ph



Converged by Gradient

Whole-Model Test

Model	-LogLikelihood	DF	ChiSquare	Prob>ChiSq
Difference	0.4026653	1	0.805331	0.369504
Full	8.0293295			
Reduced	8.4319948			

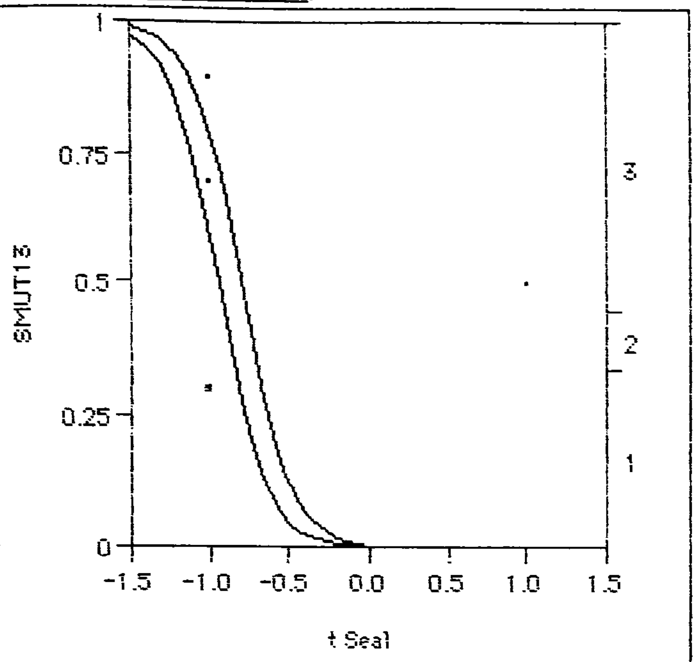
RSquare (U)	0.0478
Observations (or Sum Wgts)	9

Parameter Estimates

Term	Estimate	Std Error	ChiSquare	Prob>ChiSq
Intercept	-0.8736300	0.7676324	1.30	0.2551
Intercept	-0.3645040	0.7198989	0.26	0.6126
ph	0.61905392	0.7030748	0.78	0.3786

Ordinal Response

SMUT13 By t Seal



Converged by Objective

Whole-Model Test

Model	-LogLikelihood	DF	ChiSquare	Prob>ChiSq
Difference	3.6806197	1	7.361239	0.006664
Full	4.7513751			
Reduced	8.4319948			

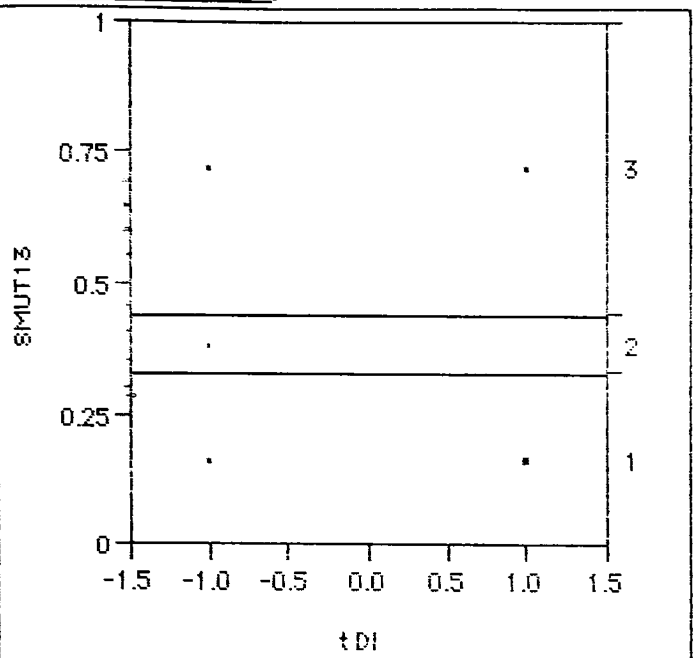
RSquare (U)	0.4365
Observations (or Sum Wgts)	9

Parameter Estimates

Term	Estimate	Std Error	ChiSquare	Prob>ChiSq
Intercept	-6.3346320	105.70841	0.00	0.9522
Intercept	-5.3538028	105.70743	0.00	0.9596
t Seal	-6.7400971	105.70743	0.00	0.9492

Ordinal Response

SMUT13 By t Di



Converged by Gradient

Whole-Model Test

Model	-LogLikelihood	DF	ChiSquare	Prob>ChiSq
Difference	0.0000000	1	0	1.000000
Full	8.4319948			
Reduced	8.4319948			

RSquare (U)	0.0000
Observations (or Sum Wgts)	9

Parameter Estimates

Term	Estimate	Std Error	ChiSquare	Prob>ChiSq
Intercept	-0.6931472	0.7108259	0.95	0.3295
intercept	-0.2231436	0.6747395	0.11	0.7409
t DI	0	0.6535659	0.00	1.0000

Analysis I

Response: Smut (Continuous)

Summary of Fit

RSquare	0.956833
RSquare Adj	0.80575
Root Mean Square Error	0.418121
Mean of Response	2.3
Observations (or Sum Wgts)	10

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	0.34965035	0.349650	?
Pure Error	1	0.00000000	0.000000	Prob>F
Total Error	2	0.34965035		?
				Max RSq
				1.0000

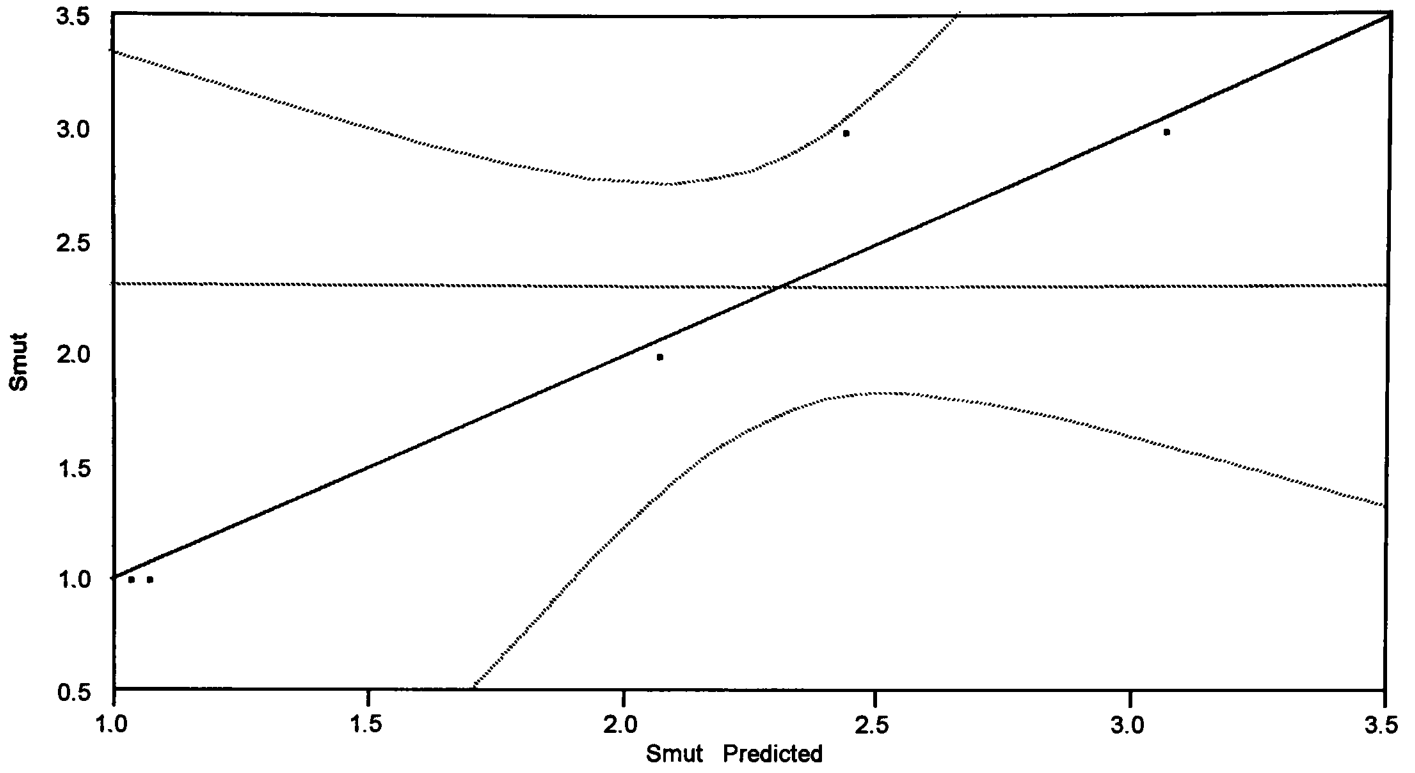
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	2.4405594	0.135419	18.02	0.0031
fs	0.3793706	0.143101	2.65	0.1177
pH	-0.129371	0.143101	-0.90	0.4614
t seal	0.6293706	0.143101	4.40	0.0480
t DI	0.1206294	0.143101	0.84	0.4880
fs*ts	-0.379371	0.143101	-2.65	0.1177
pH*ts	0.1293706	0.143101	0.90	0.4614
ts*tDI	-0.120629	0.143101	-0.84	0.4880

Effect Test

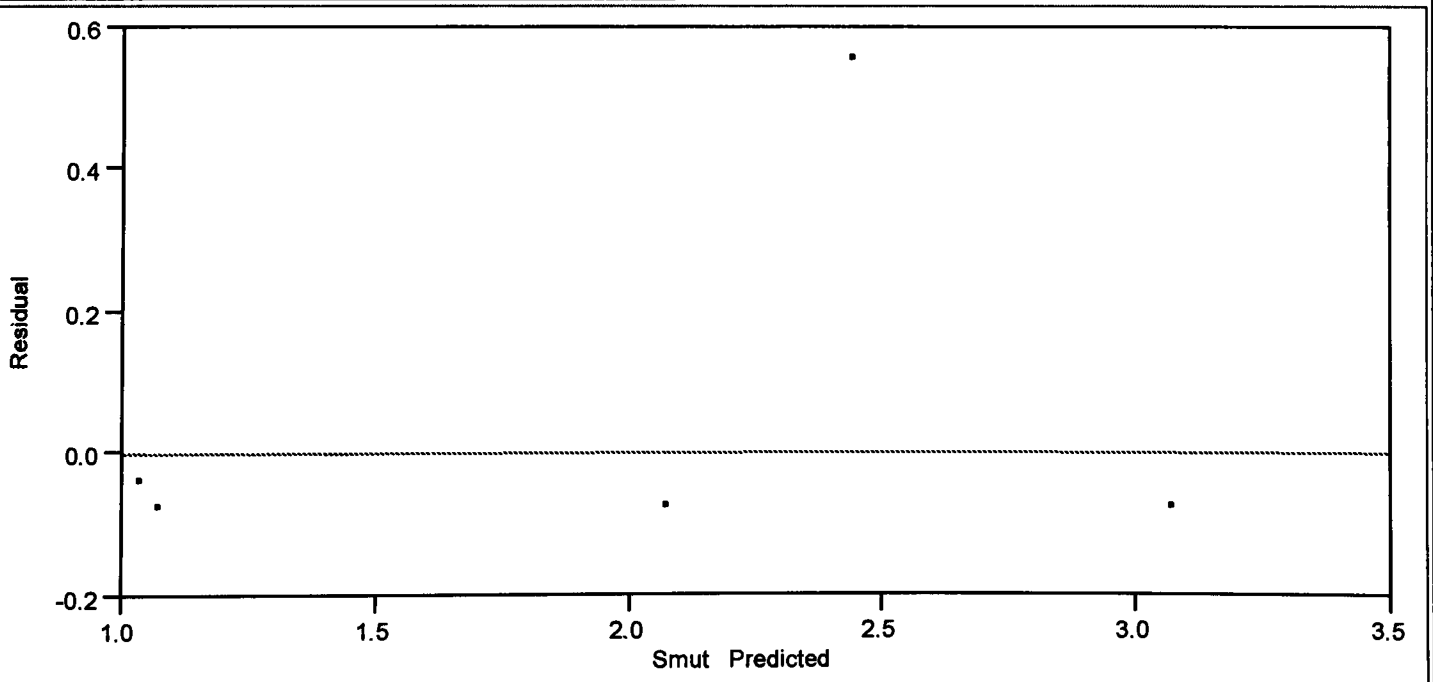
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
fs	1	1	1.2287079	7.0282	0.1177
pH	1	1	0.1428870	0.8173	0.4614
t seal	1	1	3.3816929	19.3433	0.0480
t DI	1	1	0.1242302	0.7106	0.4880
fs*ts	1	1	1.2287079	7.0282	0.1177
pH*ts	1	1	0.1428870	0.8173	0.4614
ts*tDI	1	1	0.1242302	0.7106	0.4880

Whole-Model Test

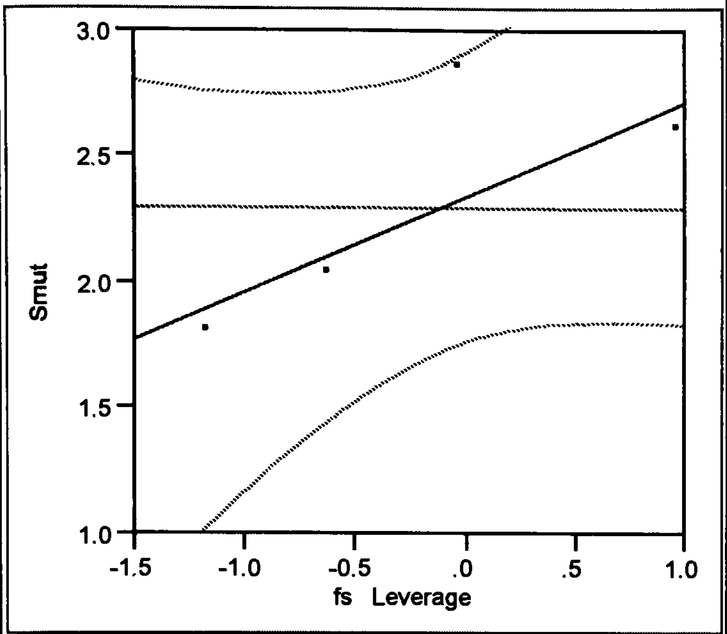


Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	7.7503497	1.10719	6.3331
Error	2	0.3496503	0.17483	Prob>F
C Total	9	8.1000000		0.1431



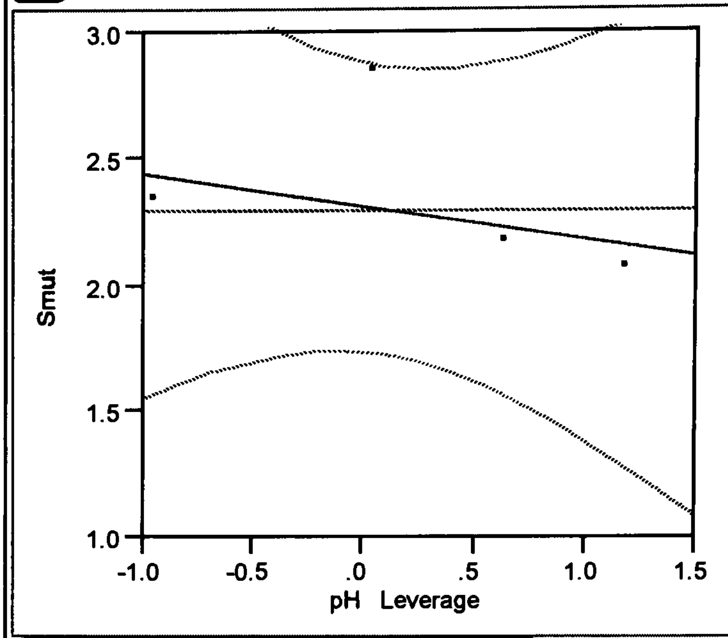
fs



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
1.2287079	7.0282	1	0.1177

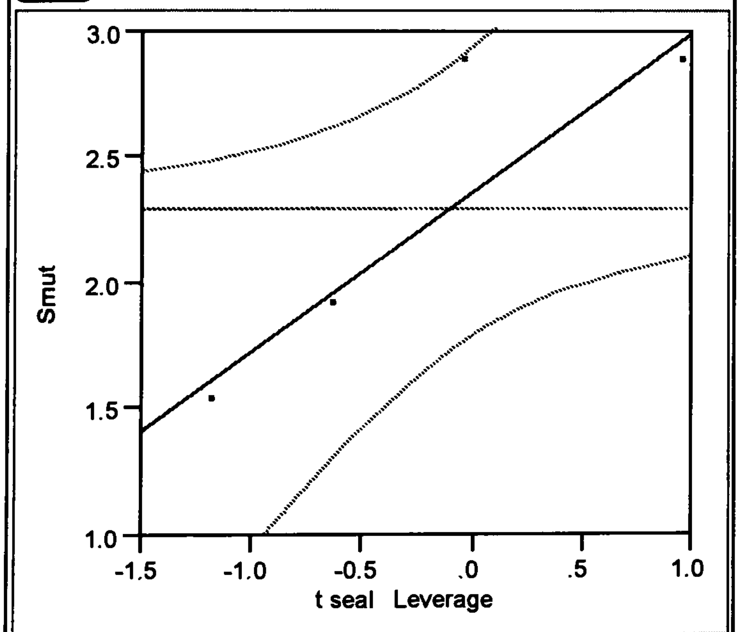
pH



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.14288696	0.8173	1	0.4614

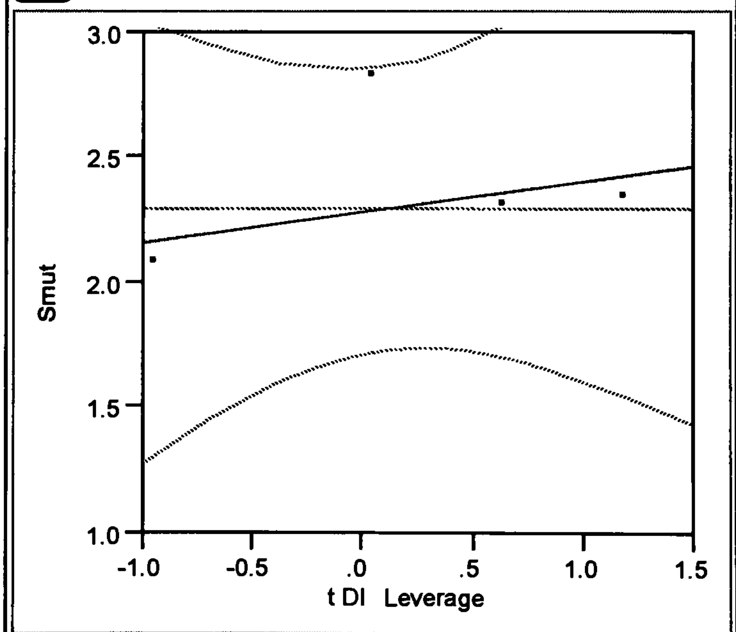
t seal



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
3.3816929	19.3433	1	0.0480

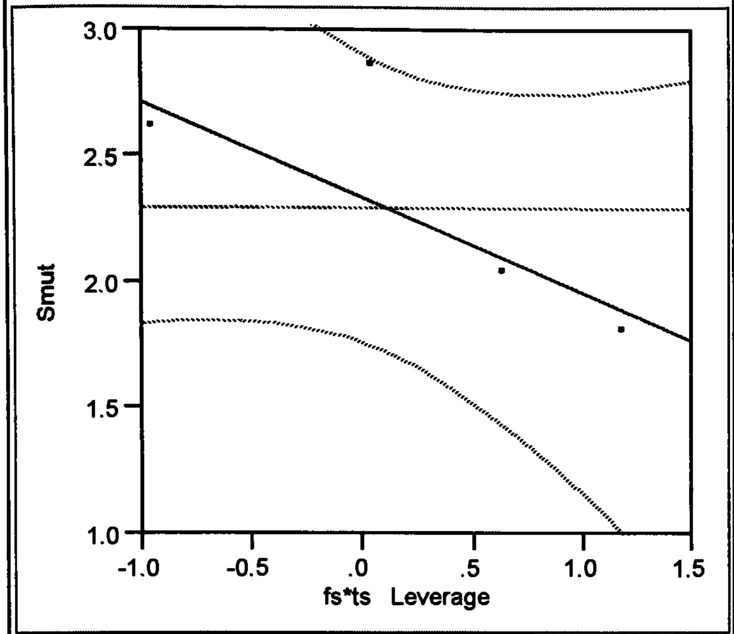
t DI



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.12423025	0.7106	1	0.4880

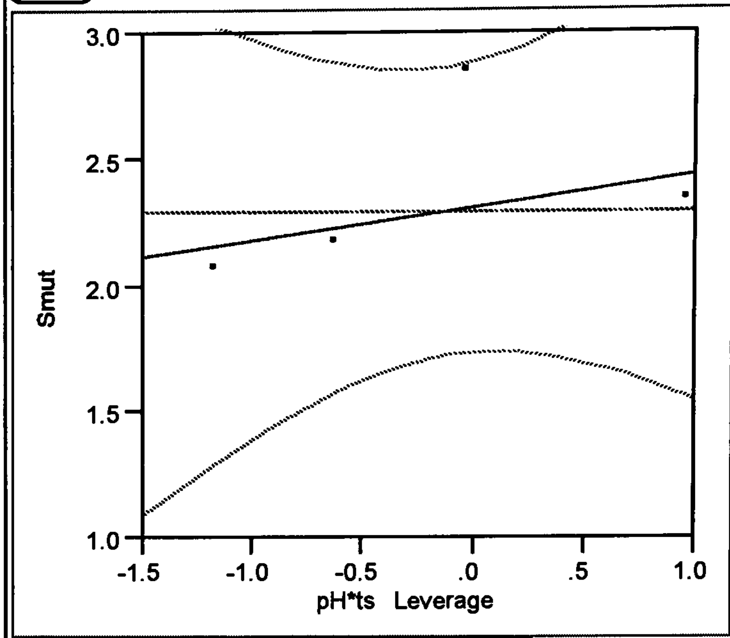
fs*ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
1.2287079	7.0282	1	0.1177

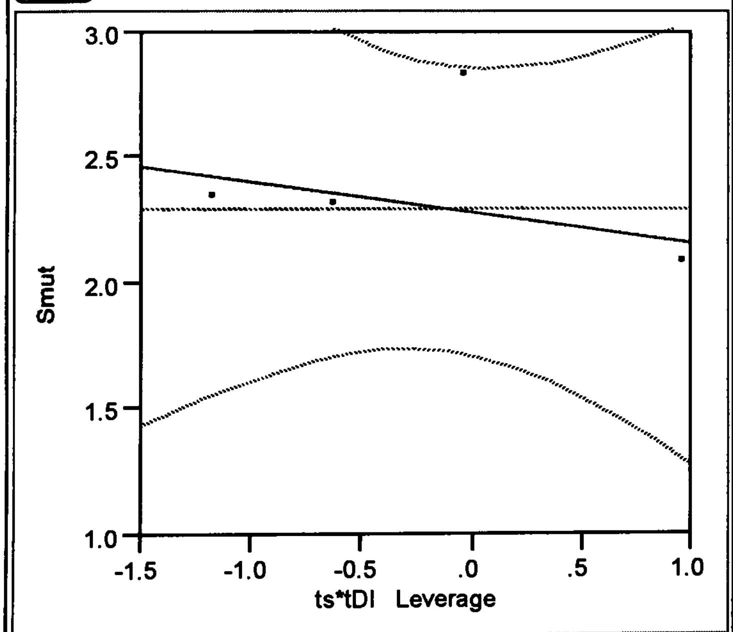
pH*ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.14288696	0.8173	1	0.4614

ts*tDI



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.12423025	0.7106	1	0.4880

Response: Smut (Continuous)

Analysis II

Summary of Fit

RSquare	0.982353
RSquare Adj	0.929412
Root Mean Square Error	0.258199
Mean of Response	2.222222
Observations (or Sum Wgts)	9

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	0.13333333	0.133333	?
Pure Error	1	0.00000000	0.000000	Prob>F
Total Error	2	0.13333333		?
				Max RSq
				1.0000

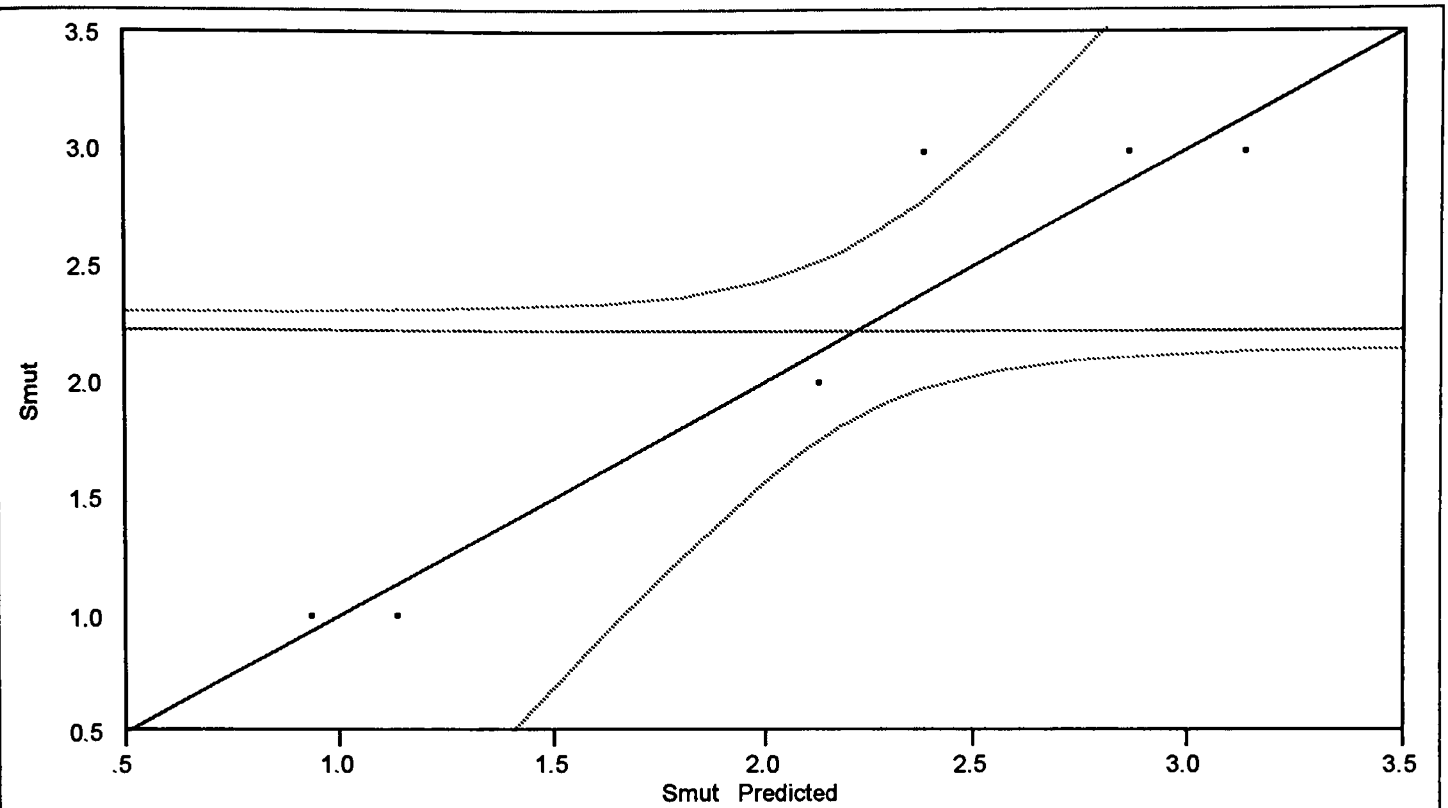
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	2.3833333	0.088192	27.02	0.0014
fs	0.3666667	0.088192	4.16	0.0533
pH	-0.116667	0.088192	-1.32	0.3169
t seal	0.6166667	0.088192	6.99	0.0198
t DI	0.1333333	0.088192	1.51	0.2697
fs*ts	-0.366667	0.088192	-4.16	0.0533
pH*ts	0.1166667	0.088192	1.32	0.3169

Effect Test

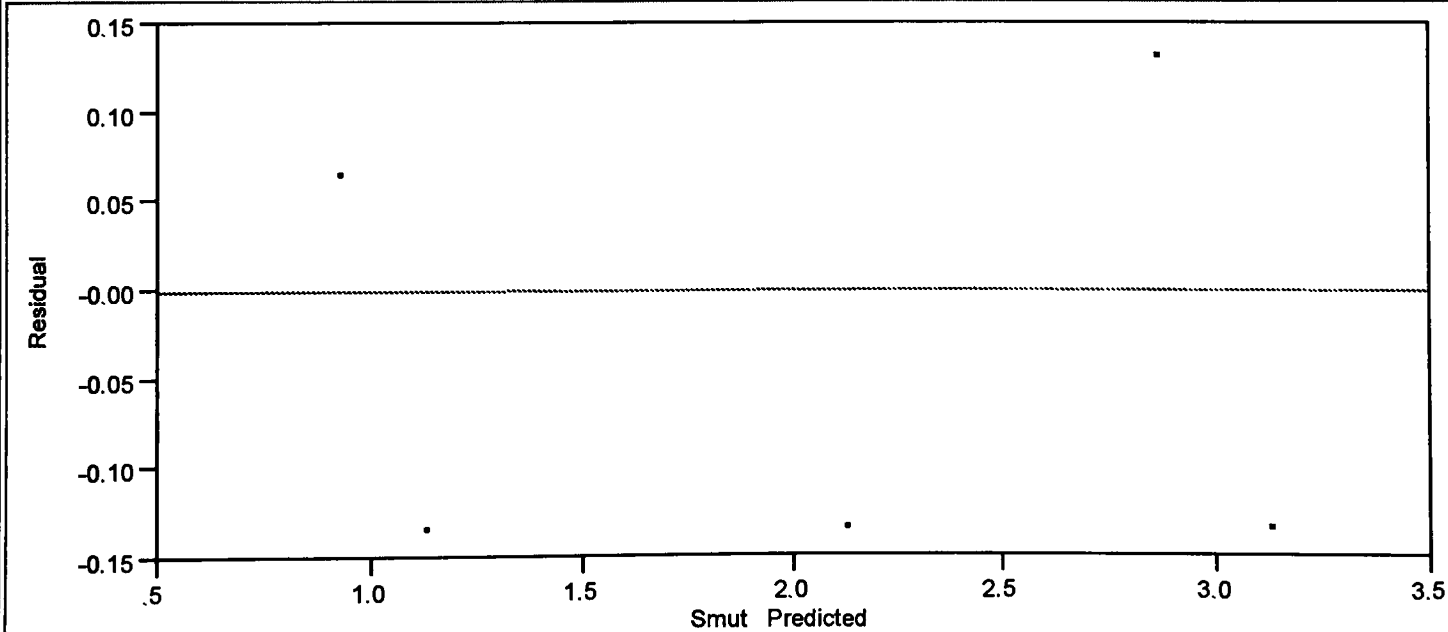
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
fs	1	1	1.1523810	17.2857	0.0533
pH	1	1	0.1166667	1.7500	0.3169
t seal	1	1	3.2595238	48.8929	0.0198
t DI	1	1	0.1523810	2.2857	0.2697
fs*ts	1	1	1.1523810	17.2857	0.0533
pH*ts	1	1	0.1166667	1.7500	0.3169

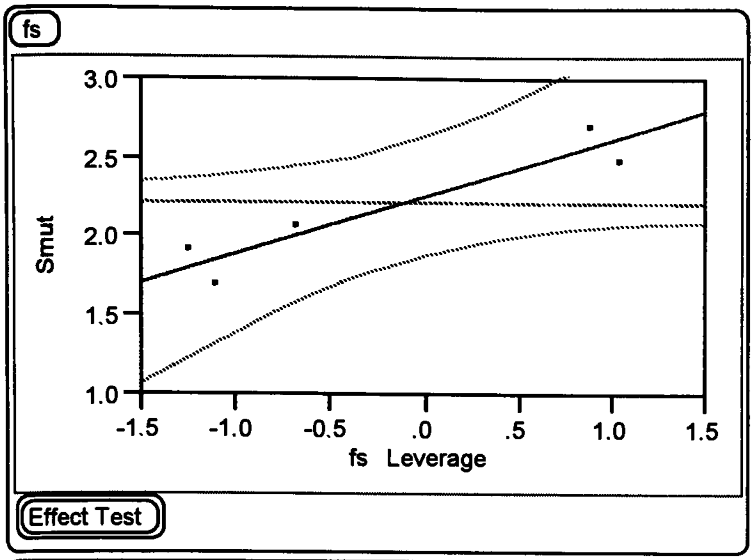
Whole-Model Test

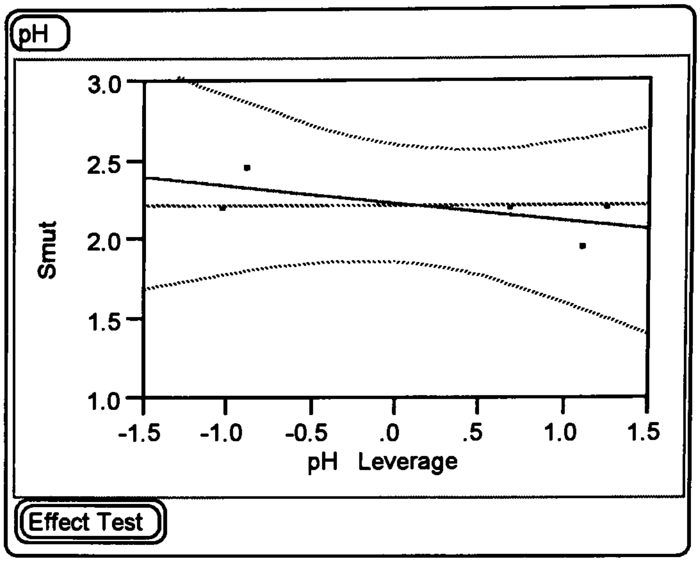


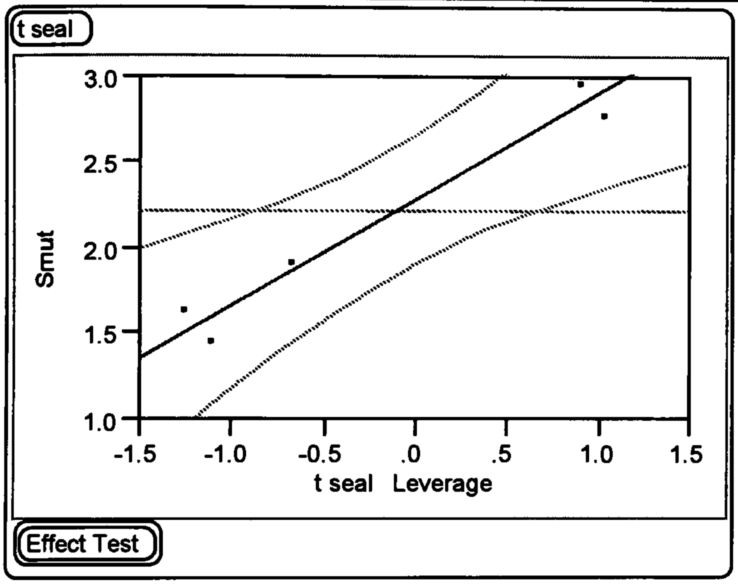
Analysis of Variance

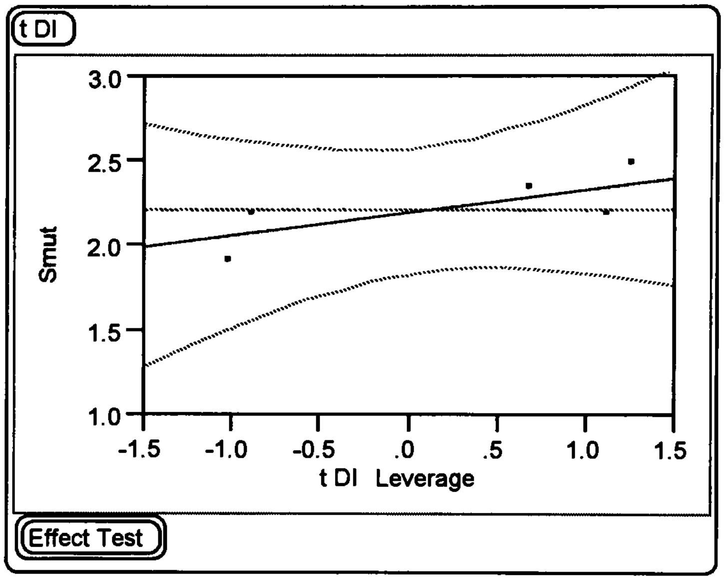
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	7.4222222	1.23704	18.5556
Error	2	0.1333333	0.06667	Prob>F
C Total	8	7.5555556		0.0520

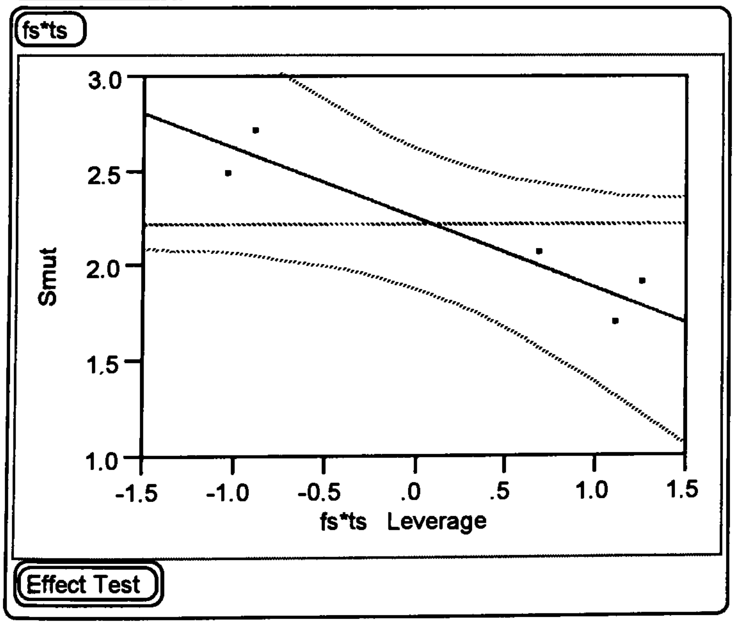


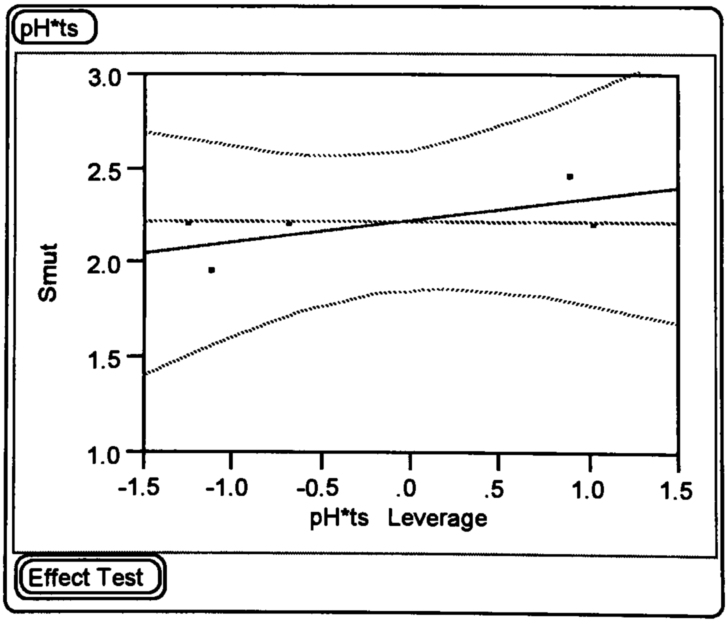

fs
Smut
3.0
2.5
2.0
1.5
1.0
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
fs Leverage
Effect Test


pH
Smut
3.0
2.5
2.0
1.5
1.0
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
pH Leverage
Effect Test


t seal
Smut
3.0
2.5
2.0
1.5
1.0
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
t seal Leverage
Effect Test


t DI
Smut
3.0
2.5
2.0
1.5
1.0
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
t DI Leverage
Effect Test


fs*ts
Smut
3.0
2.5
2.0
1.5
1.0
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
fs*ts Leverage
Effect Test


pH*ts
Smut
3.0
2.5
2.0
1.5
1.0
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
pH*ts Leverage
Effect Test

Appendix E

Response: Blue Analysis I

Summary of Fit

RSquare	0.992306
RSquare Adj	0.965376
Root Mean Square Error	0.62541
Mean of Response	1.342
Observations (or Sum Wgts)	10

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	0.33102413	0.331024	0.7336
Pure Error	1	0.45125000	0.451250	Prob>F
Total Error	2	0.78227413		0.5491
				Max RSq
				0.9956

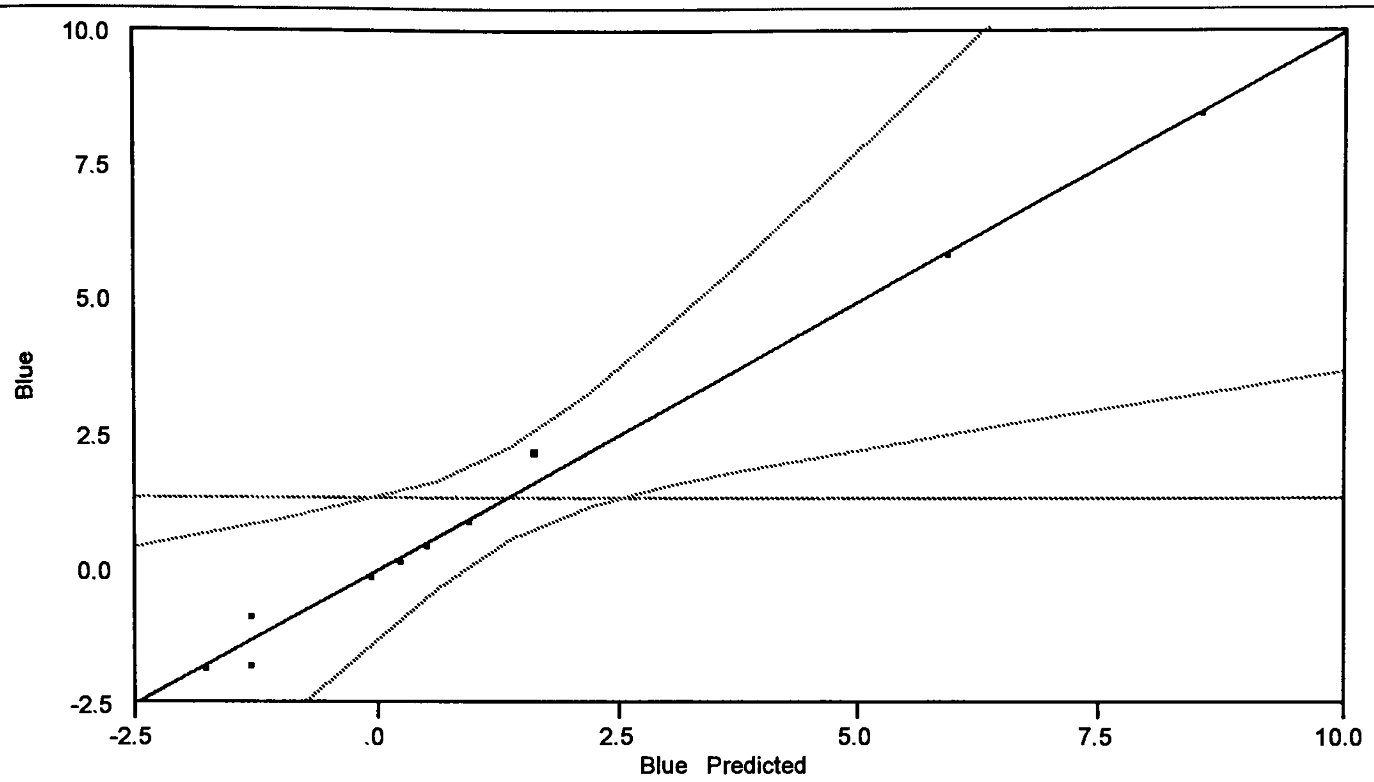
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.6356643	0.202555	8.08	0.0150
fs	0.4323776	0.214044	2.02	0.1808
pH	-2.102378	0.214044	-9.82	0.0102
ts	2.2898776	0.214044	10.70	0.0086
tDI	-0.104878	0.214044	-0.49	0.6726
fs*ts	0.4051224	0.214044	1.89	0.1989
pH*ts	-1.215122	0.214044	-5.68	0.0297
ts*tDI	-0.372622	0.214044	-1.74	0.2238

Effect Test

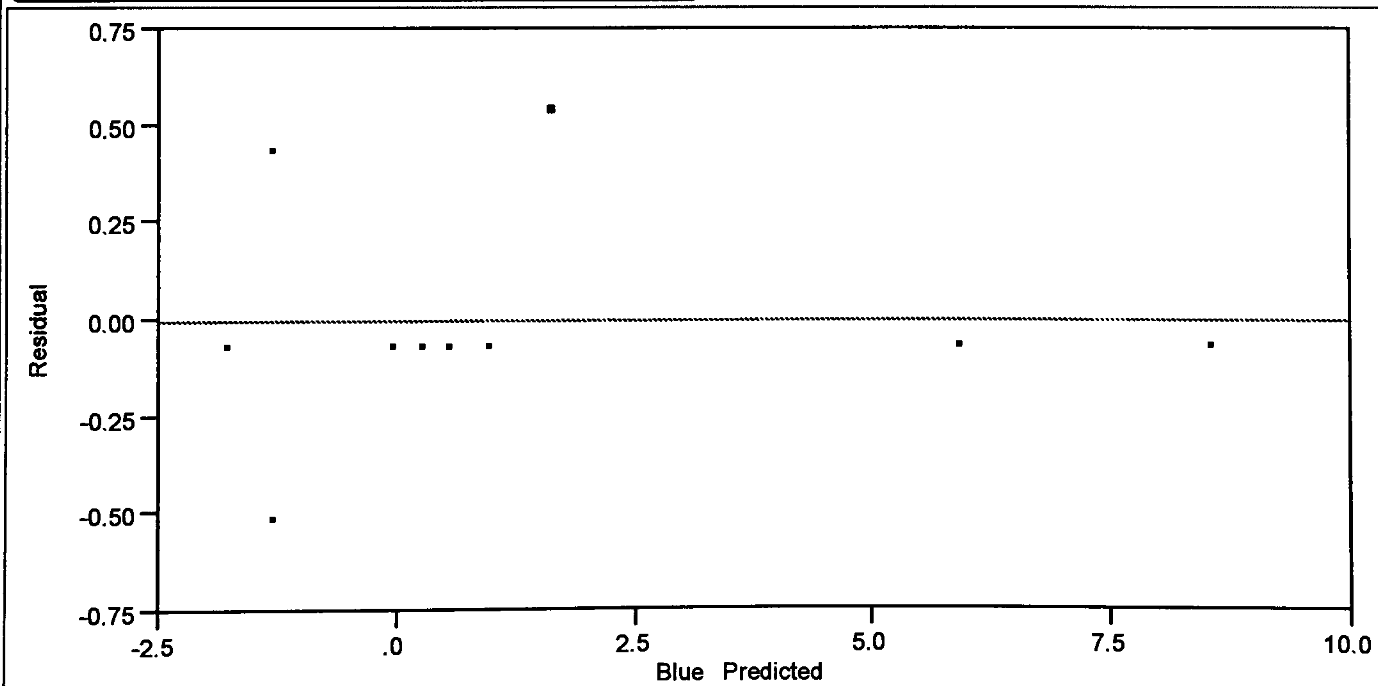
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
fs	1	1	1.596054	4.0805	0.1808
pH	1	1	37.734854	96.4748	0.0102
ts	1	1	44.765740	114.4503	0.0086
tDI	1	1	0.093905	0.2401	0.6726
fs*ts	1	1	1.401179	3.5823	0.1989
pH*ts	1	1	12.605534	32.2279	0.0297
ts*tDI	1	1	1.185384	3.0306	0.2238

Whole-Model Test

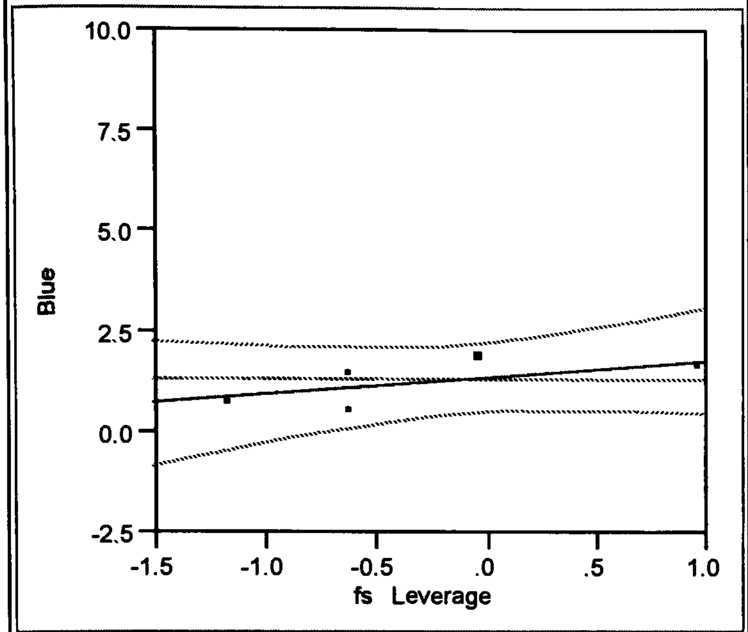


Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	100.88929	14.4128	36.8483
Error	2	0.78227	0.3911	Prob>F
C Total	9	101.67156		0.0267



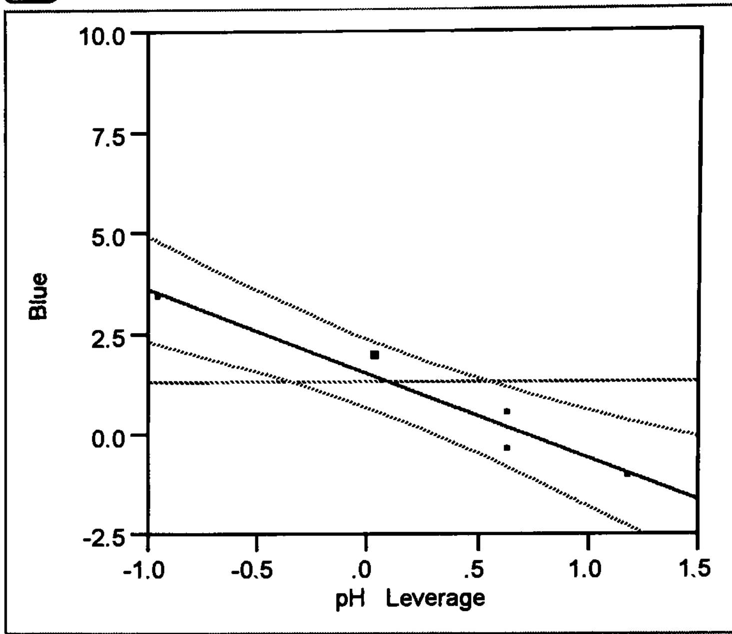
fs



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
1.5960542	4.0805	1	0.1808

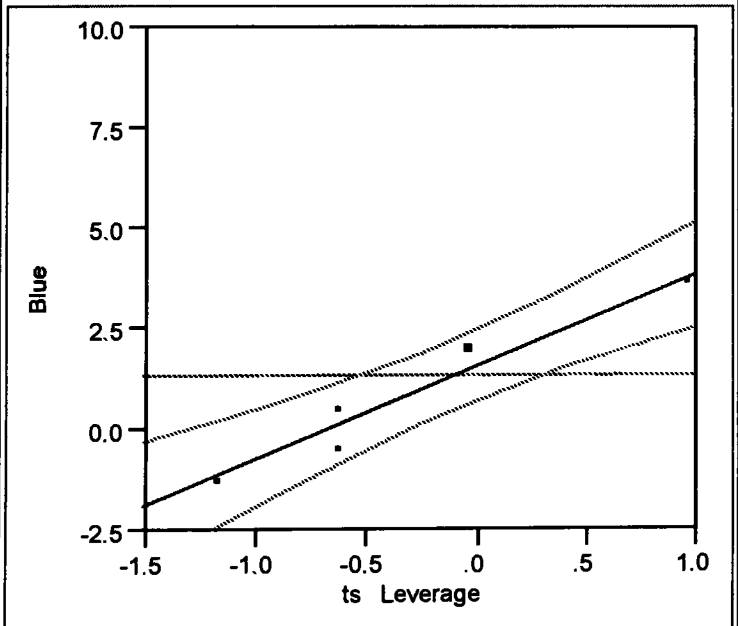
pH



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
37.734854	96.4748	1	0.0102

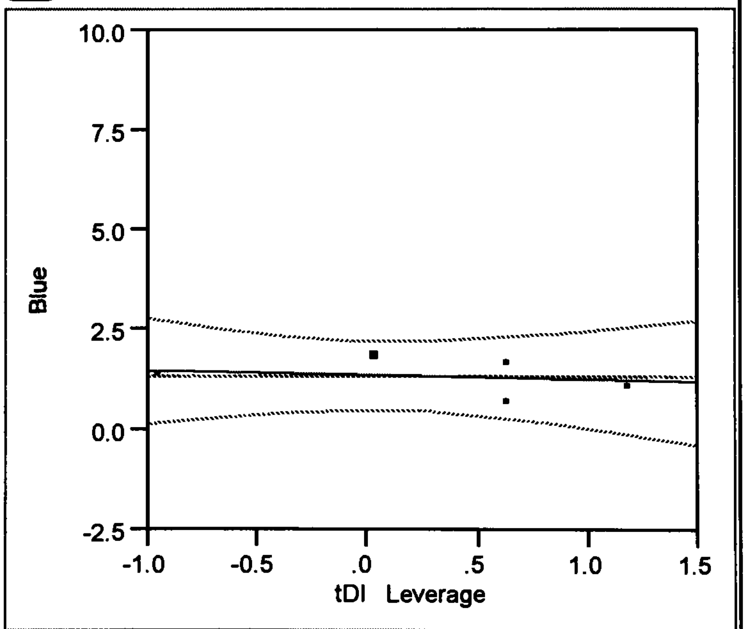
ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
44.765740	114.4503	1	0.0086

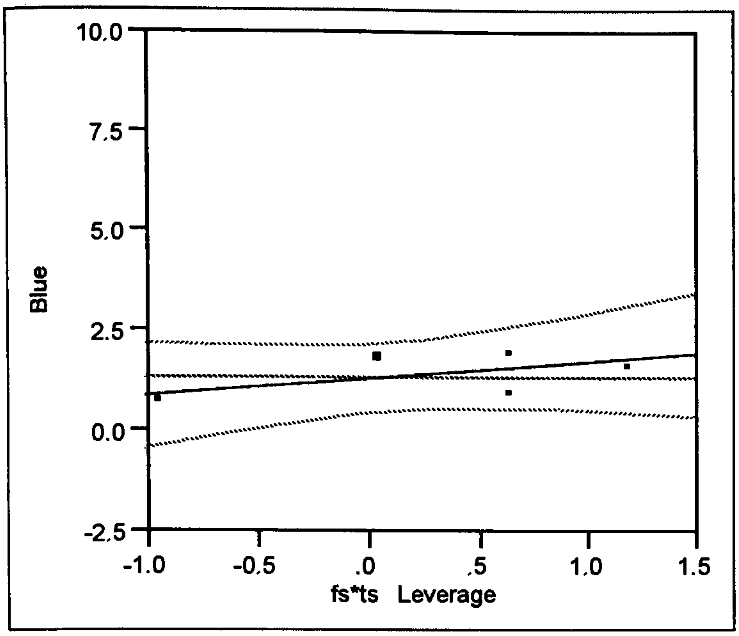
tDI



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.09390461	0.2401	1	0.6726

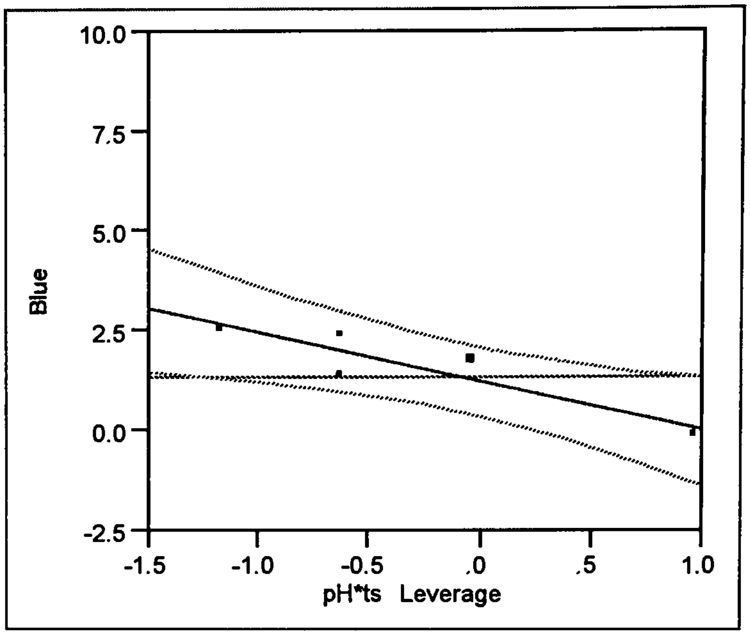
fs*ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
1.4011792	3.5823	1	0.1989

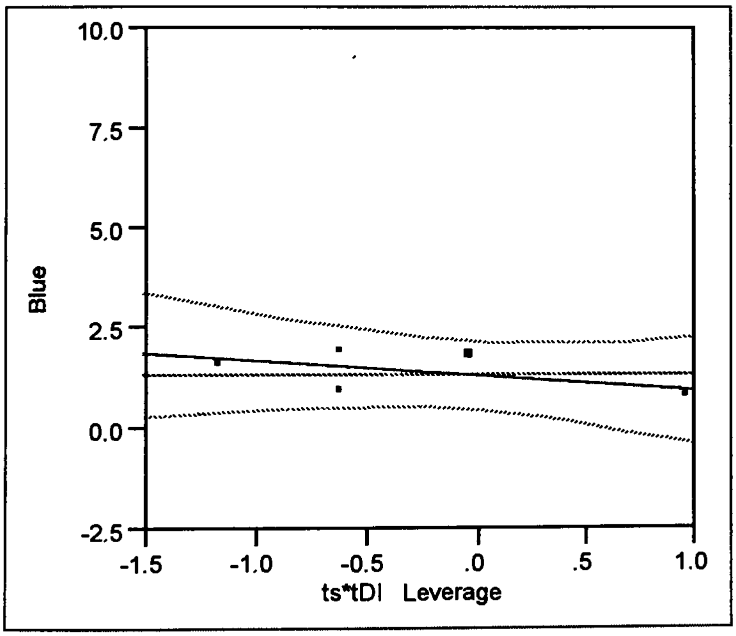
pH*ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
12.605534	32.2279	1	0.0297

ts*tDI



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
1.1853841	3.0306	1	0.2238

Response: Blue

Analysis II

Summary of Fit

RSquare	0.967475
RSquare Adj	0.913267
Root Mean Square Error	1.045862
Mean of Response	1.248889
Observations (or Sum Wgts)	9

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	2	2.8302321	1.41512	3.1360
Pure Error	1	0.4512500	0.45125	Prob>F
Total Error	3	3.2814821		0.3708
				Max RSq
				0.9955

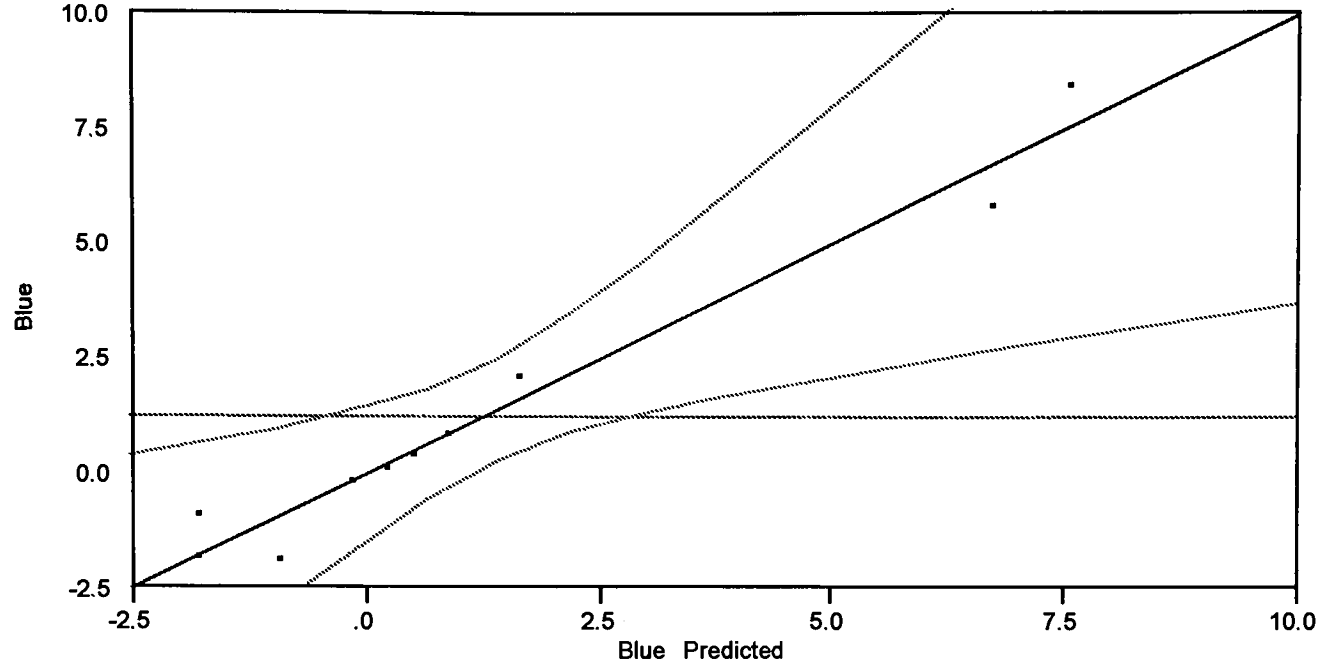
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.6280357	0.356317	4.57	0.0197
fs	0.3719643	0.356317	1.04	0.3732
pH	-2.041964	0.356317	-5.73	0.0105
ts	2.2294643	0.356317	6.26	0.0082
tDI	-0.044464	0.356317	-0.12	0.9086
pH*ts	-1.275536	0.356317	-3.58	0.0373

Effect Test

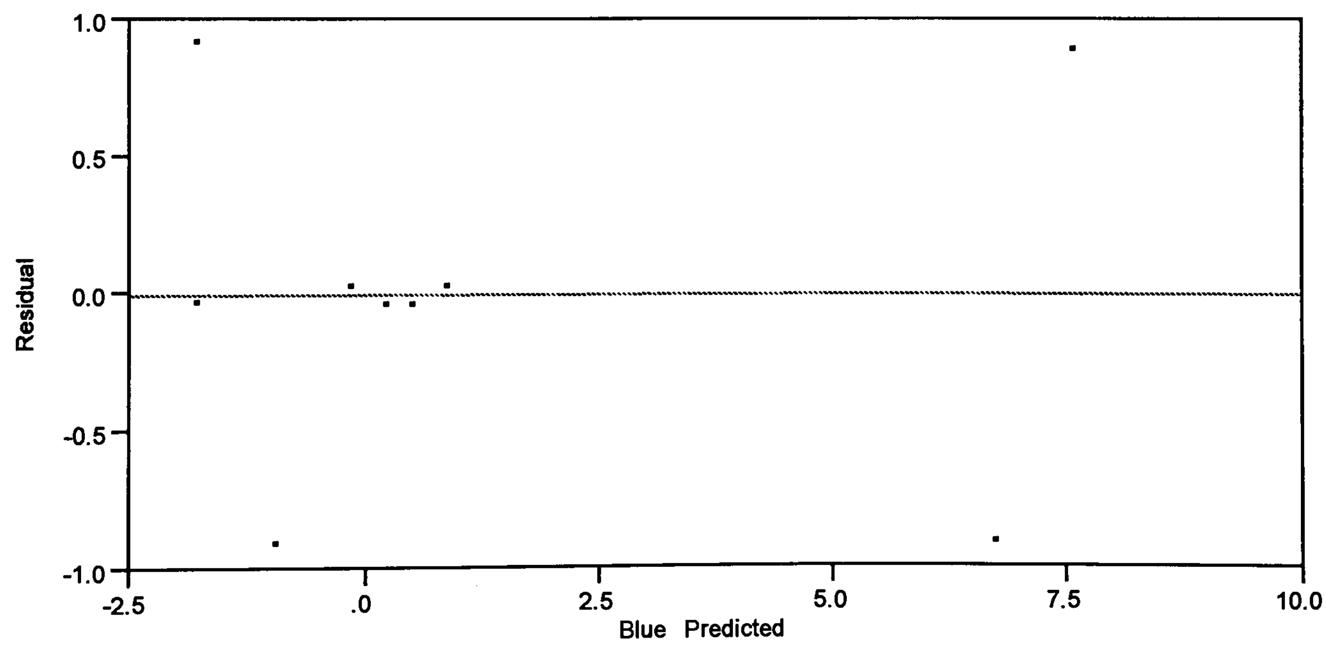
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
fs	1	1	1.192002	1.0898	0.3732
pH	1	1	35.922864	32.8414	0.0105
ts	1	1	42.822864	39.1496	0.0082
tDI	1	1	0.017033	0.0156	0.9086
pH*ts	1	1	14.017156	12.8148	0.0373

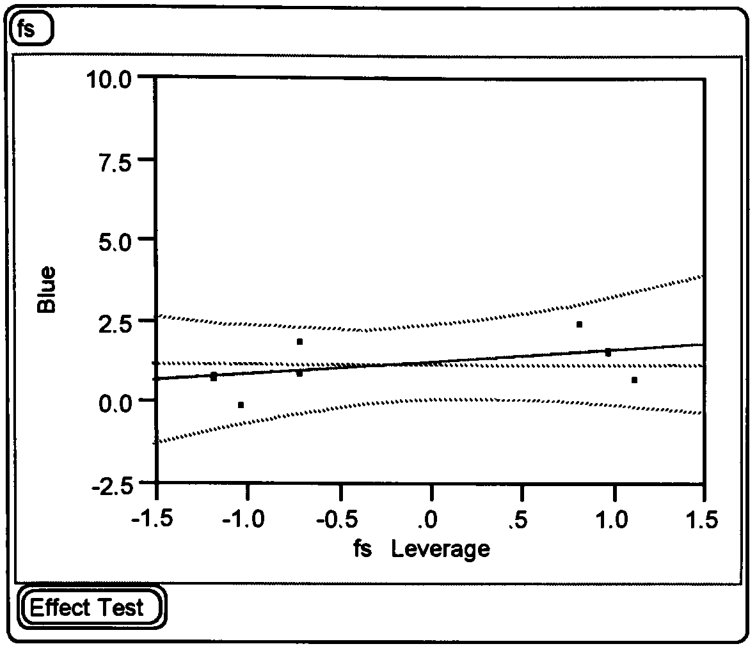
Whole-Model Test

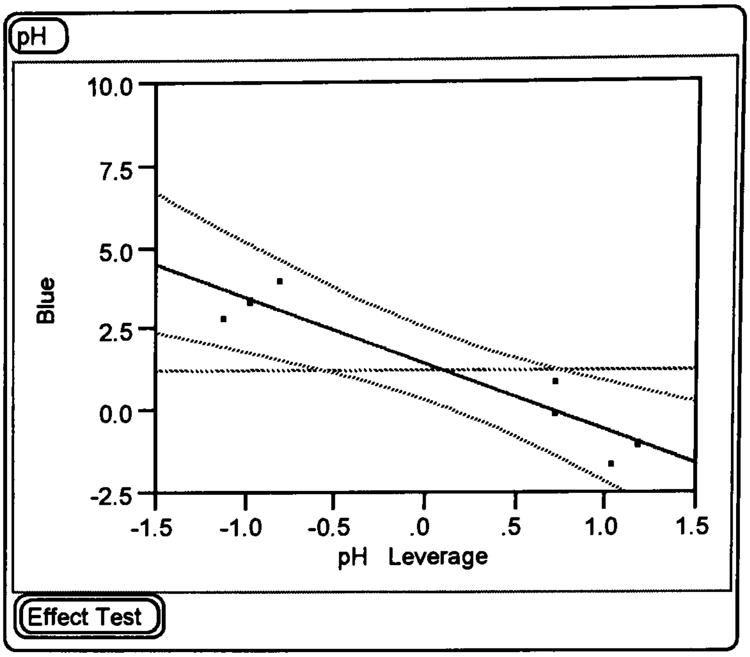


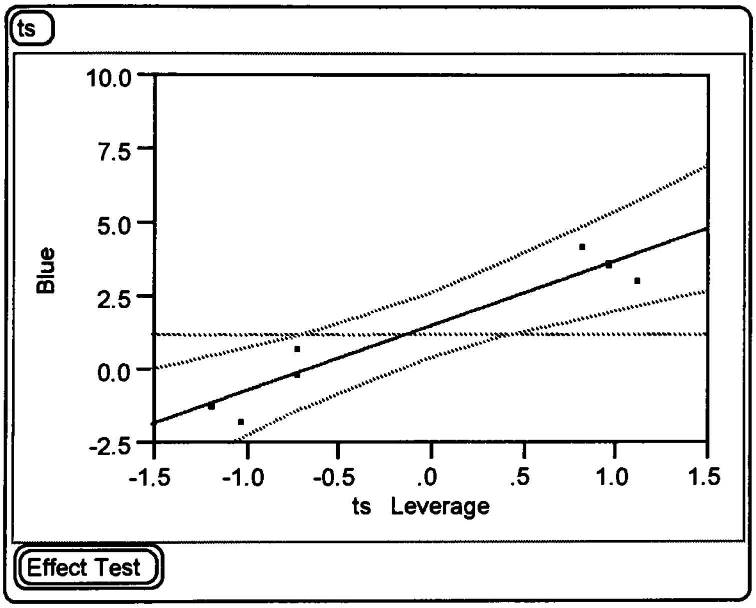
Analysis of Variance

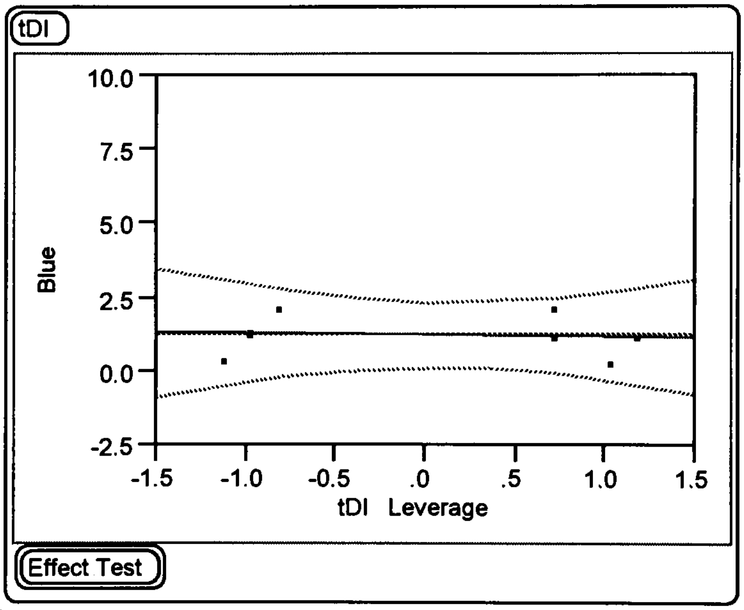
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	97.60981	19.5220	17.8474
Error	3	3.28148	1.0938	Prob>F
C Total	8	100.89129		0.0193

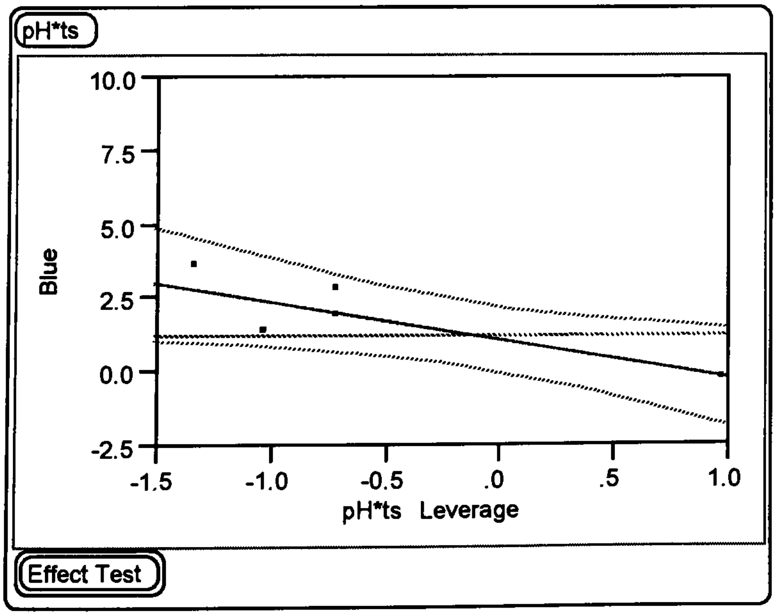



fs
Blue
10.0
7.5
5.0
2.5
0.0
-2.5
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
fs Leverage
Effect Test


pH
Blue
10.0
7.5
5.0
2.5
0.0
-2.5
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
pH Leverage
Effect Test


ts
Blue
10.0
7.5
5.0
2.5
0.0
-2.5
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
ts Leverage
Effect Test


tDI
Blue
10.0
7.5
5.0
2.5
0.0
-2.5
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
tDI Leverage
Effect Test


pH*ts
Blue
10.0
7.5
5.0
2.5
0.0
-2.5
-1.5
-1.0
-0.5
.0
.5
1.0
pH*ts Leverage
Effect Test

Response: Degree of Seal

Summary of Fit

RSquare	0.952841
RSquare Adj	0.787786
Root Mean Square Error	2.696649
Mean of Response	5.191
Observations (or Sum Wgts)	10

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	12.503636	12.5036	6.1286
Pure Error	1	2.040200	2.0402	Prob>F
Total Error	2	14.543836		0.2444
				Max RSq
				0.9934

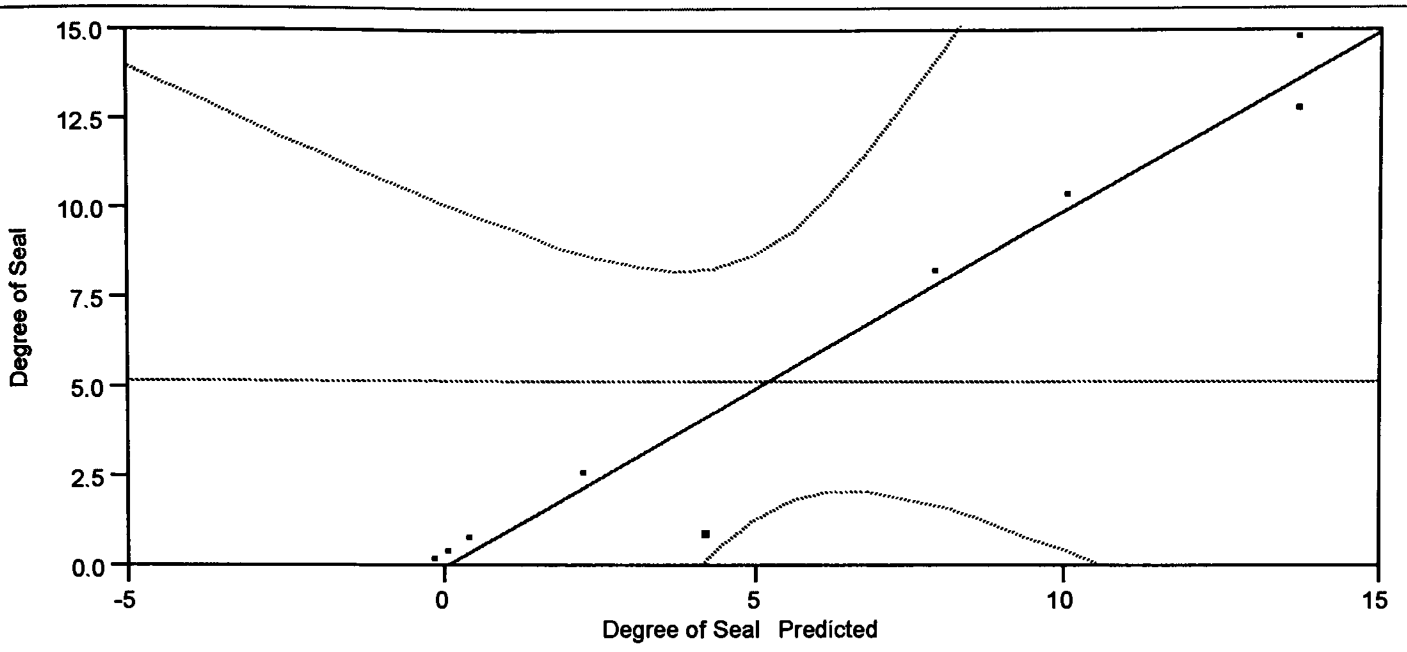
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	4.2454545	0.873378	4.86	0.0398
fs	-1.288636	0.92292	-1.40	0.2974
pH	-0.241364	0.92292	-0.26	0.8182
ts	-4.246136	0.92292	-4.60	0.0441
tDI	1.6211364	0.92292	1.76	0.2211
fs*pH	-1.768636	0.92292	-1.92	0.1954
fs*ts	1.0461364	0.92292	1.13	0.3746
fs*tDI	0.2738636	0.92292	0.30	0.7946

Effect Test

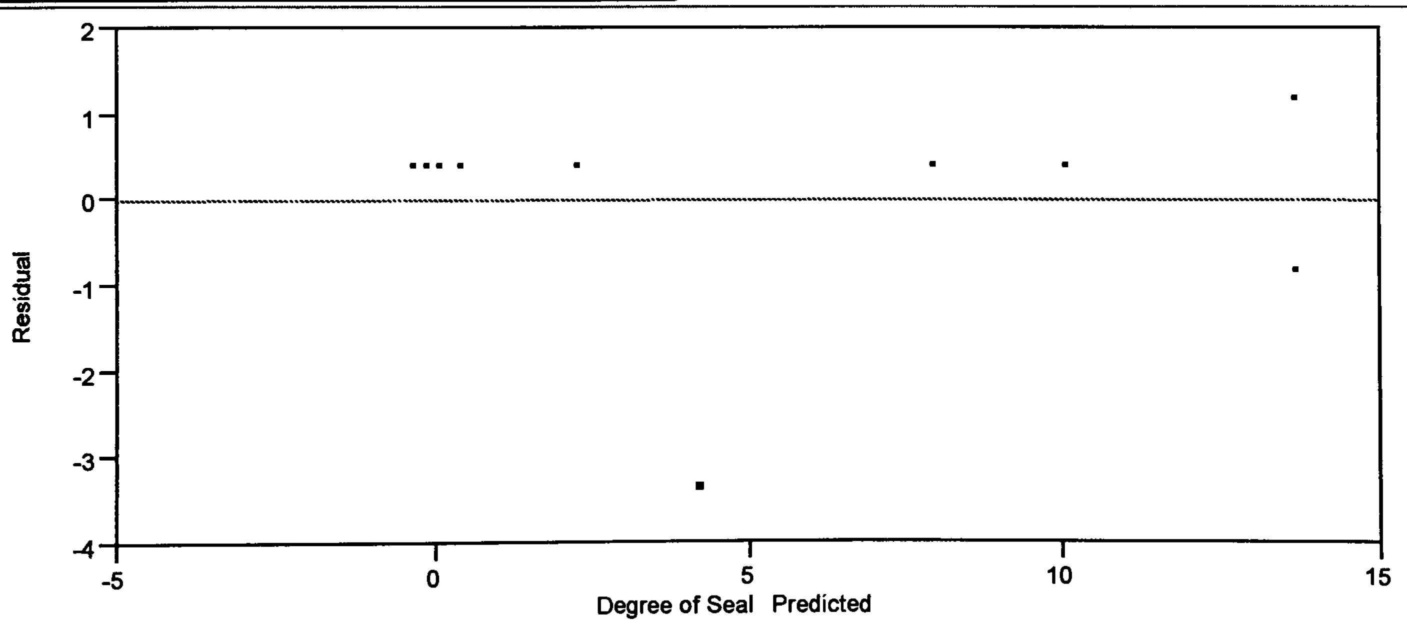
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
fs	1	1	14.17692	1.9495	0.2974
pH	1	1	0.49735	0.0684	0.8182
ts	1	1	153.92498	21.1670	0.0441
tDI	1	1	22.43677	3.0854	0.2211
fs*pH	1	1	26.70535	3.6724	0.1954
fs*ts	1	1	9.34325	1.2848	0.3746
fs*tDI	1	1	0.64031	0.0881	0.7946

Whole-Model Test

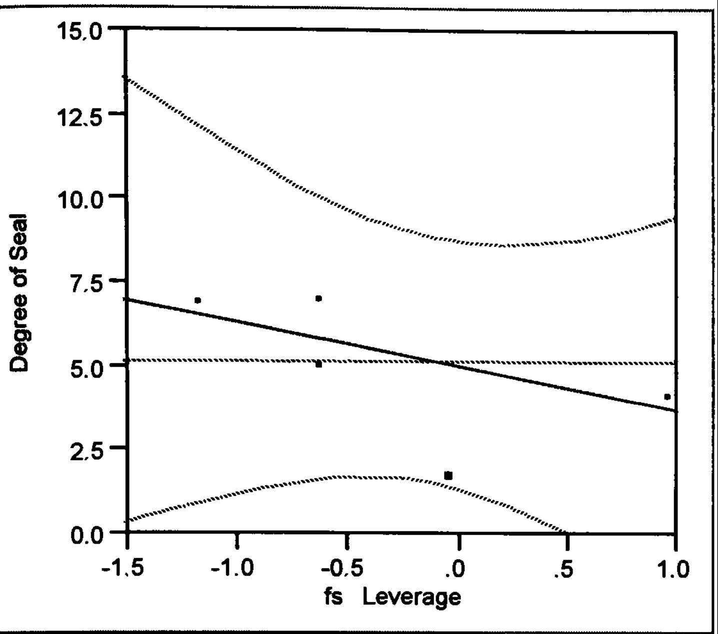


Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	293.85765	41.9797	5.7728
Error	2	14.54384	7.2719	Prob>F
C Total	9	308.40149		0.1556



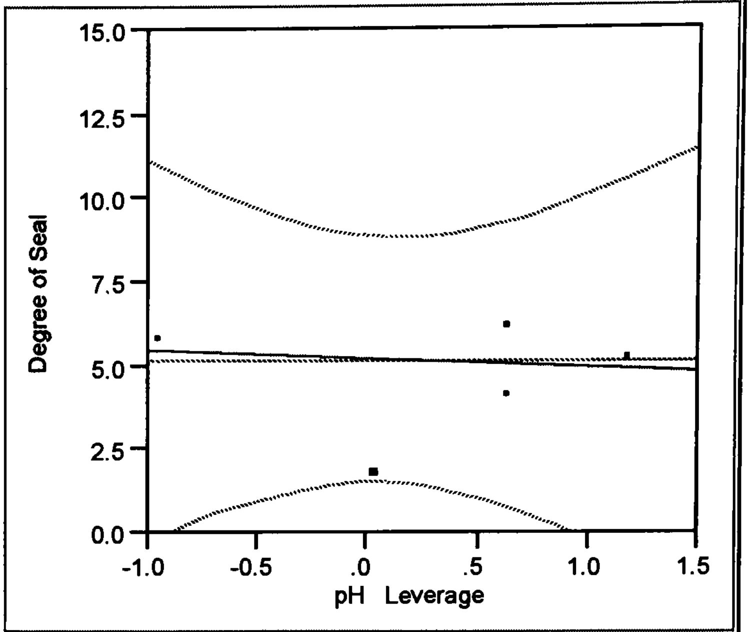
fs



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
14.176923	1.9495	1	0.2974

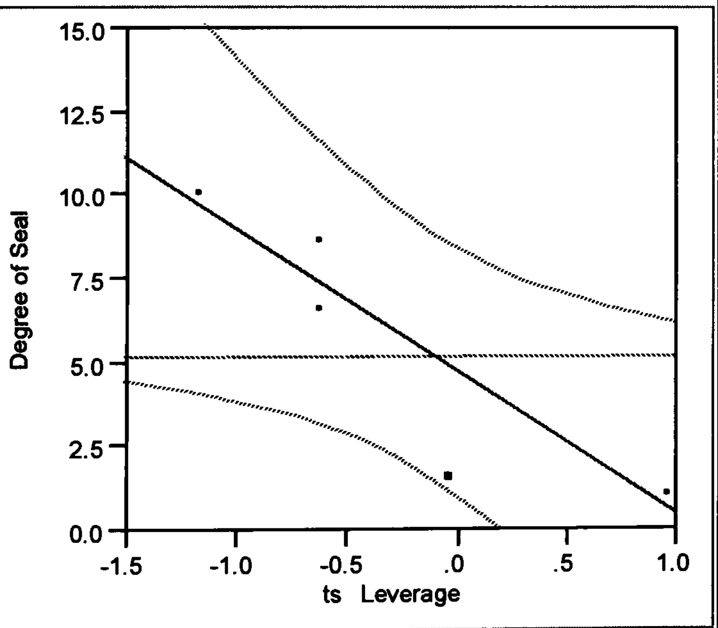
pH



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.49735319	0.0684	1	0.8182

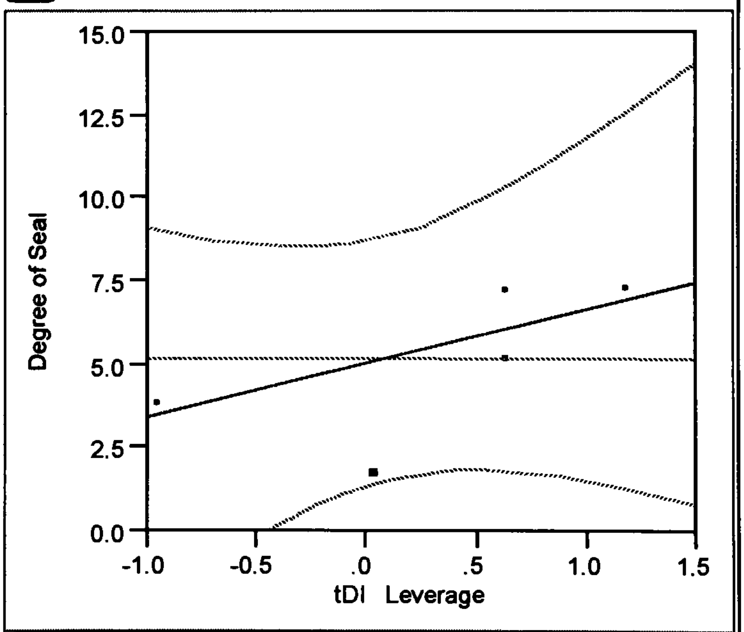
ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
153.92498	21.1670	1	0.0441

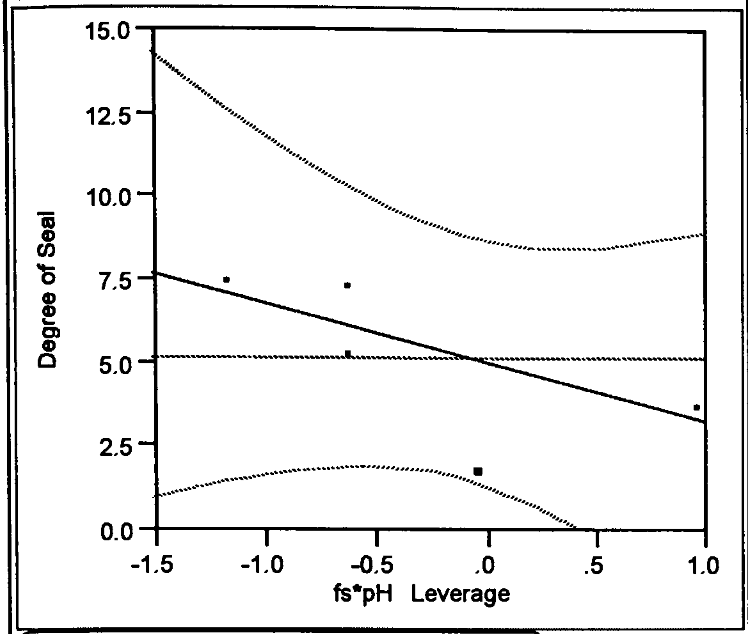
tDI



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
22.436769	3.0854	1	0.2211

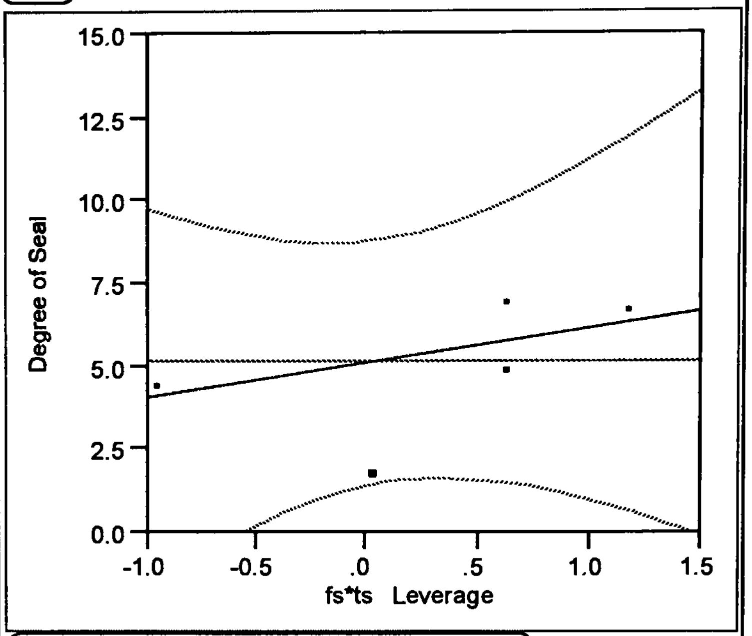
fs*pH



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
26.705353	3.6724	1	0.1954

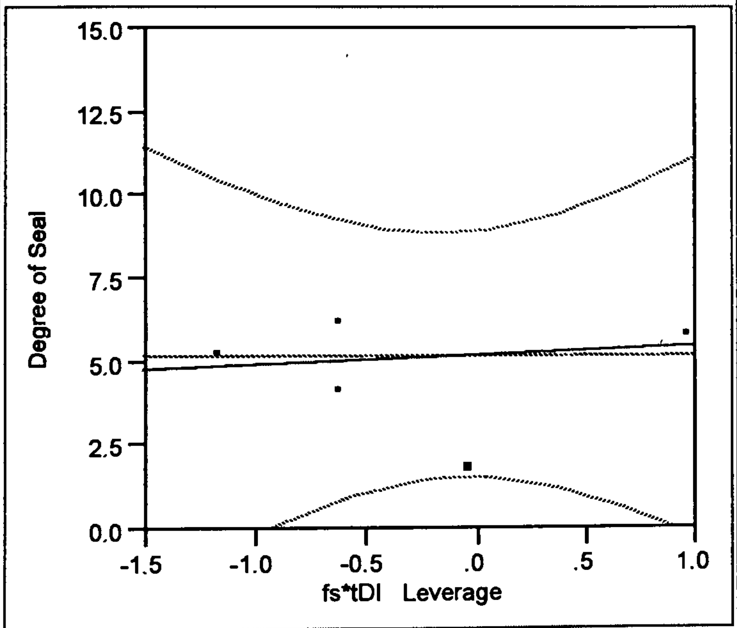
fs*ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
9.3432468	1.2848	1	0.3746

fs*tDI



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.64030953	0.0881	1	0.7946

Response: Degree of Seal

Summary of Fit

RSquare	0.990247
RSquare Adj	0.960989
Root Mean Square Error	1.184947
Mean of Response	5.667778
Observations (or Sum Wgts)	9

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	0.7680000	0.76800	0.3764
Pure Error	1	2.0402000	2.04020	Prob>F
Total Error	2	2.8082000		0.6497
				Max RSq
				0.9929

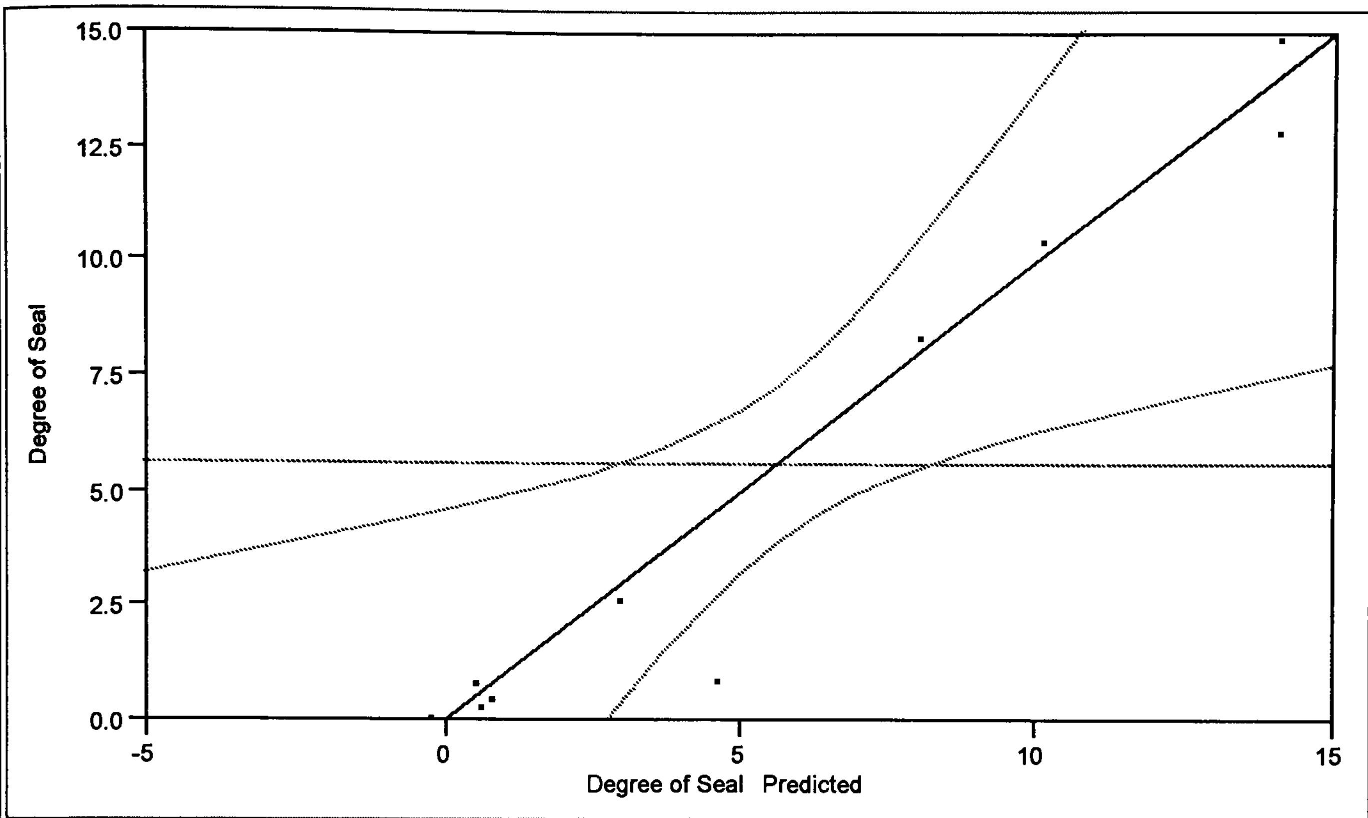
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	4.6175	0.404737	11.41	0.0076
fs	-1.2425	0.404737	-3.07	0.0917
pH	-0.2875	0.404737	-0.71	0.5512
ts	-4.2	0.404737	-10.38	0.0092
tDI	1.575	0.404737	3.89	0.0601
fs*ts	1	0.404737	2.47	0.1321
ts*tDI	-1.7225	0.404737	-4.26	0.0510

Effect Test

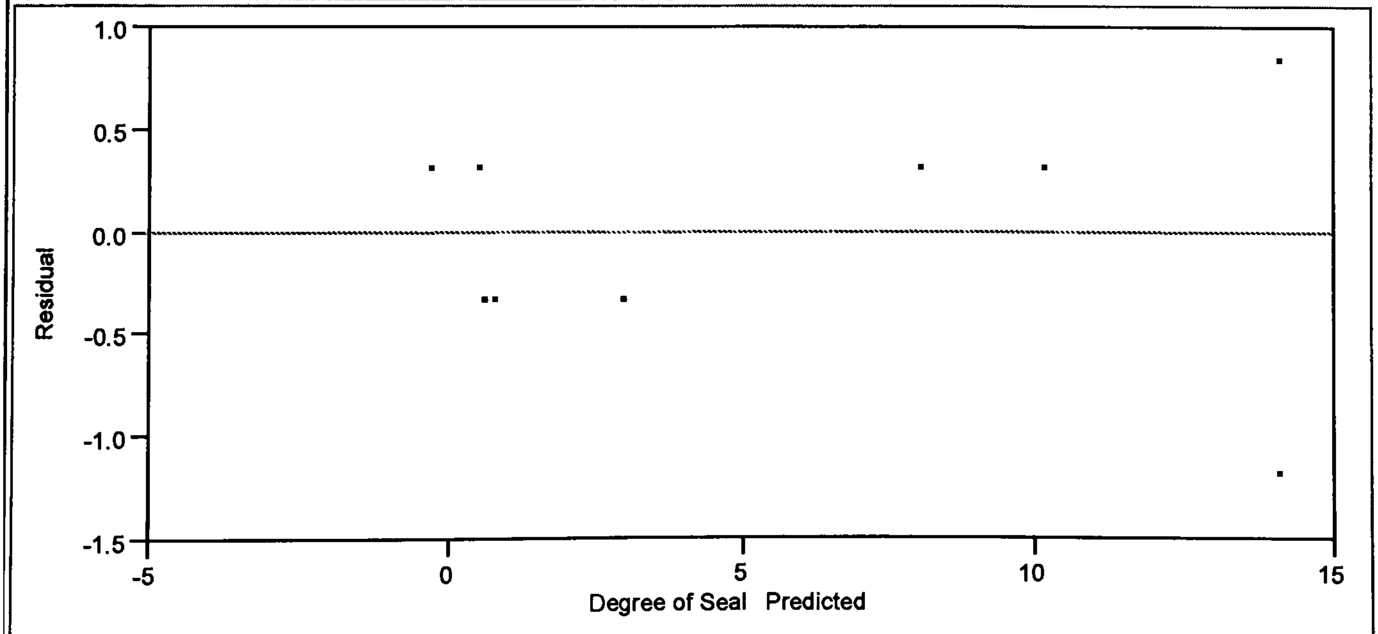
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
fs	1	1	13.23263	9.4243	0.0917
pH	1	1	0.70848	0.5046	0.5512
ts	1	1	151.20000	107.6846	0.0092
tDI	1	1	21.26250	15.1432	0.0601
fs*ts	1	1	8.57143	6.1046	0.1321
ts*tDI	1	1	25.43148	18.1123	0.0510

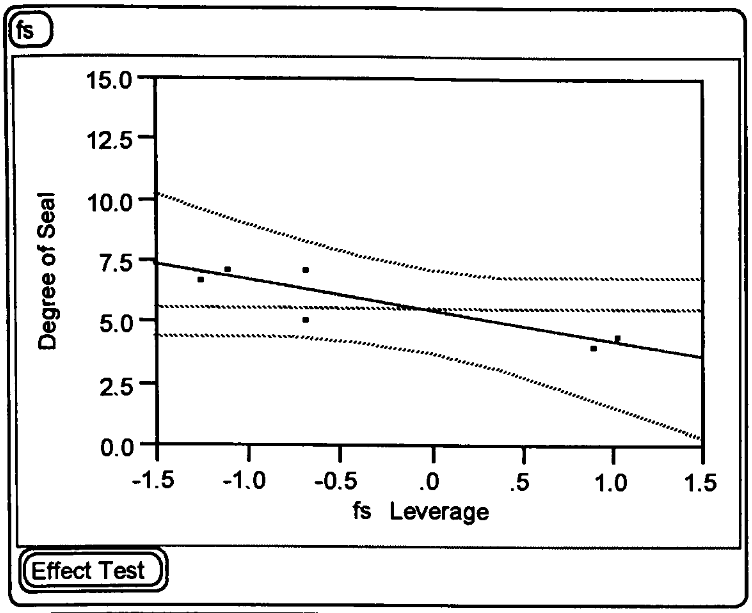
Whole-Model Test

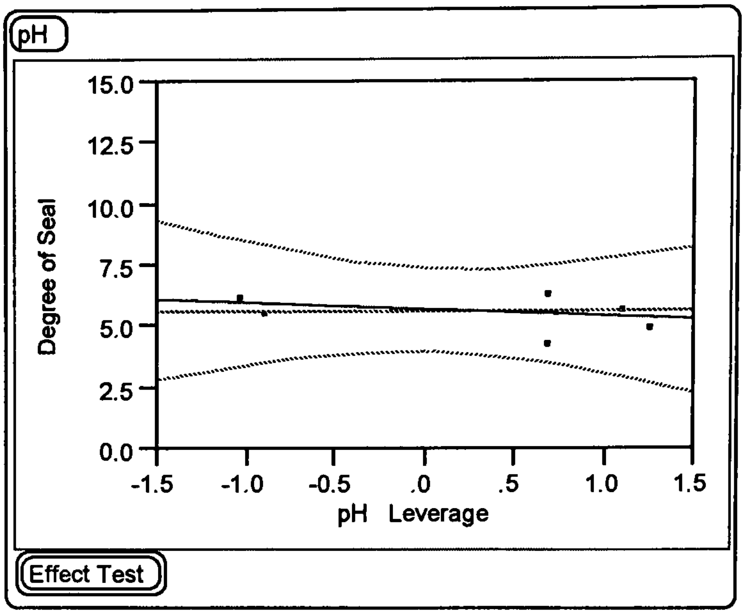


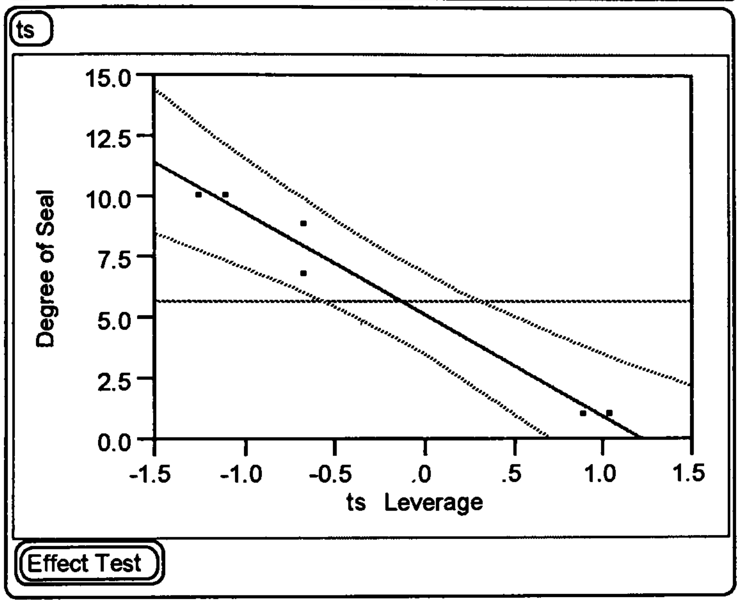
Analysis of Variance

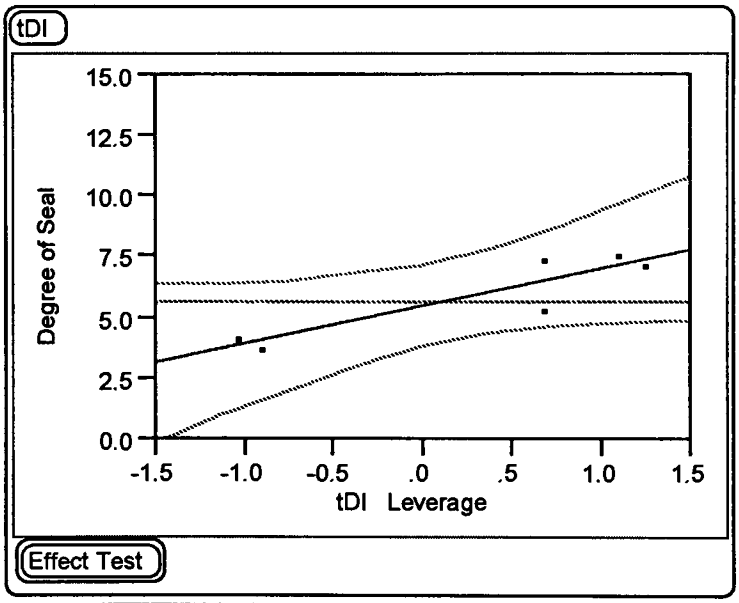
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	285.13476	47.5225	33.8455
Error	2	2.80820	1.4041	Prob>F
C Total	8	287.94296		0.0290

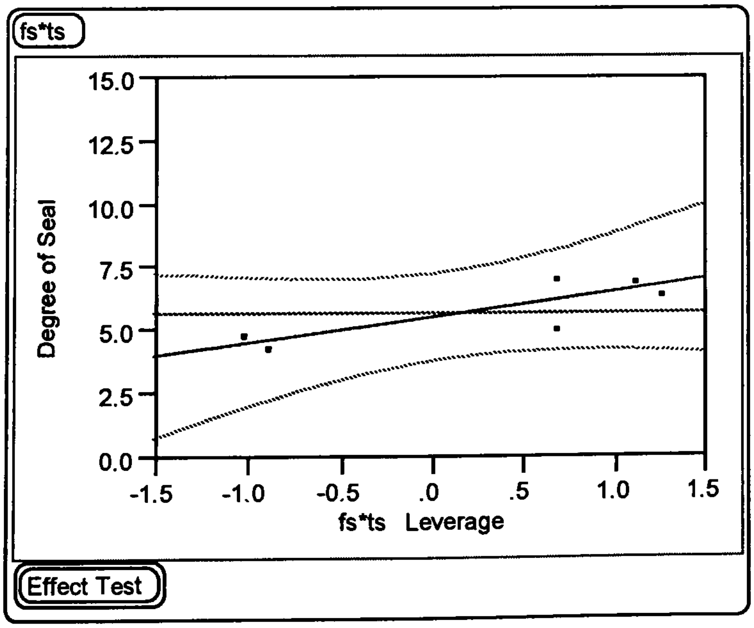


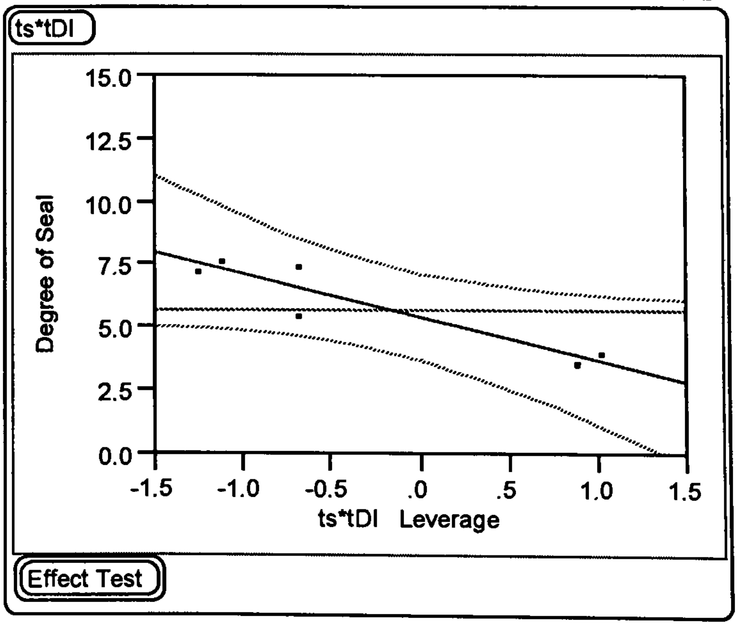

fs
Degree of Seal
15.0
12.5
10.0
7.5
5.0
2.5
0.0
-1.5 -1.0 -0.5 .0 .5 1.0 1.5
fs Leverage
Effect Test


pH
Degree of Seal
15.0
12.5
10.0
7.5
5.0
2.5
0.0
-1.5 -1.0 -0.5 .0 .5 1.0 1.5
pH Leverage
Effect Test


ts
Degree of Seal
15.0
12.5
10.0
7.5
5.0
2.5
0.0
-1.5 -1.0 -0.5 .0 .5 1.0 1.5
ts Leverage
Effect Test


tDI
Degree of Seal
15.0
12.5
10.0
7.5
5.0
2.5
0.0
-1.5 -1.0 -0.5 .0 .5 1.0 1.5
tDI Leverage
Effect Test


fs*ts
Degree of Seal
15.0
12.5
10.0
7.5
5.0
2.5
0.0
-1.5 -1.0 -0.5 .0 .5 1.0 1.5
fs*ts Leverage
Effect Test


ts*tDI
Degree of Seal
15.0
12.5
10.0
7.5
5.0
2.5
0.0
-1.5 -1.0 -0.5 .0 .5 1.0 1.5
ts*tDI Leverage
Effect Test

Response: Darkness

Summary of Fit

RSquare	0.95398
RSquare Adj	0.792911
Root Mean Square Error	0.872046
Mean of Response	20.261
Observations (or Sum Wgts)	10

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	1.3804787	1.38048	9.8290
Pure Error	1	0.1404500	0.14045	Prob>F
Total Error	2	1.5209287		0.1966
				Max RSq
				0.9958

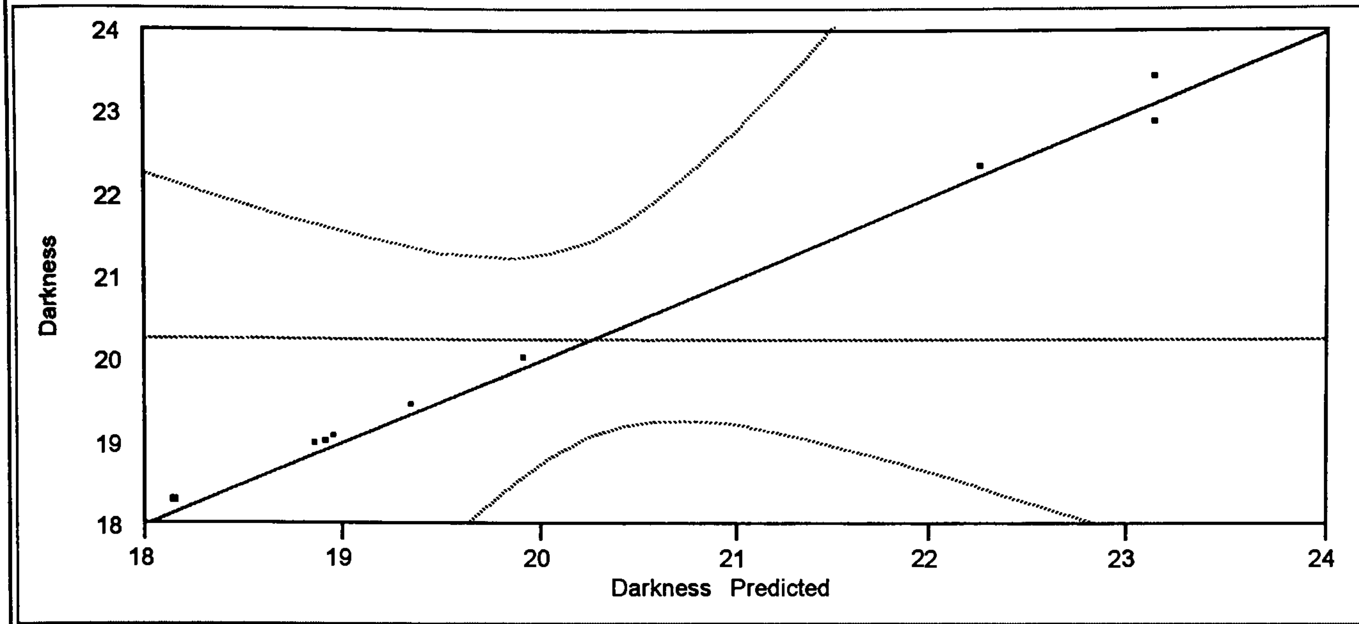
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	19.941608	0.282434	70.61	0.0002
fs	-0.273059	0.298455	-0.91	0.4568
pH	1.1130594	0.298455	3.73	0.0650
ts	-0.660559	0.298455	-2.21	0.1573
tDI	-0.154441	0.298455	-0.52	0.6564
fs*ts	0.1230594	0.298455	0.41	0.7201
pH*ts	-0.983059	0.298455	-3.29	0.0811
ts*tDI	-0.195559	0.298455	-0.66	0.5796

Effect Test

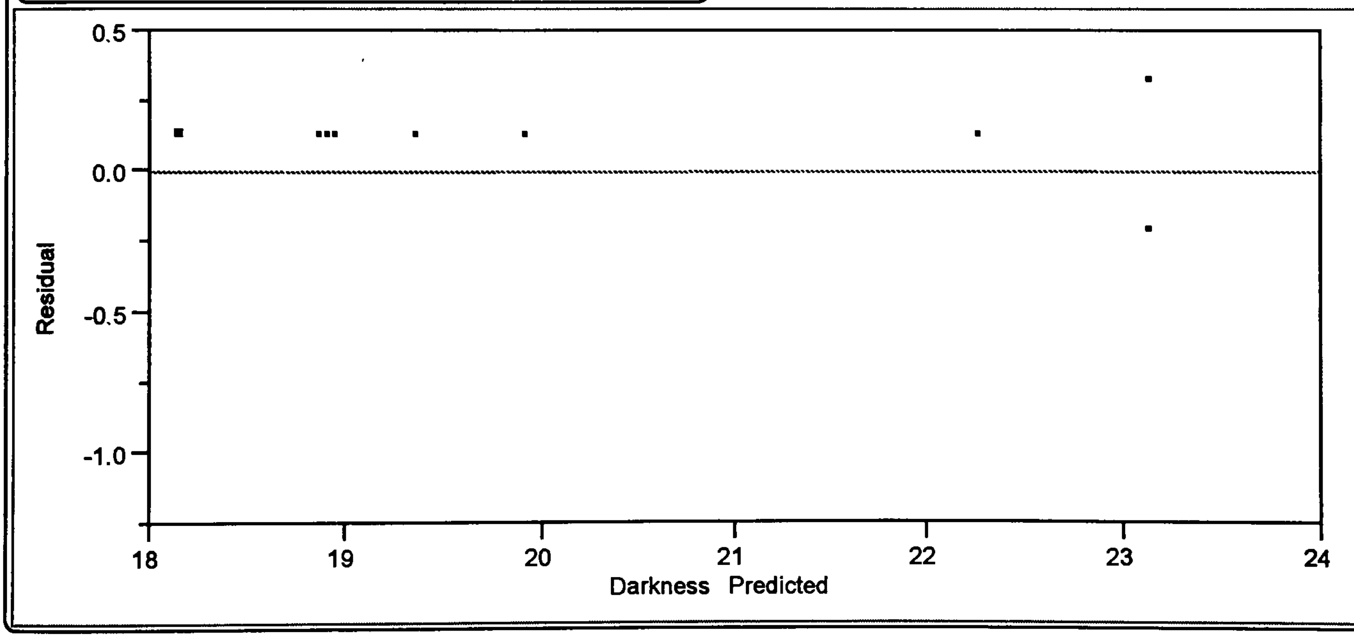
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
fs	1	1	0.636555	0.8371	0.4568
pH	1	1	10.576889	13.9085	0.0650
ts	1	1	3.725161	4.8985	0.1573
tDI	1	1	0.203631	0.2678	0.6564
fs*ts	1	1	0.129286	0.1700	0.7201
pH*ts	1	1	8.250510	10.8493	0.0811
ts*tDI	1	1	0.326497	0.4293	0.5796

Whole-Model Test

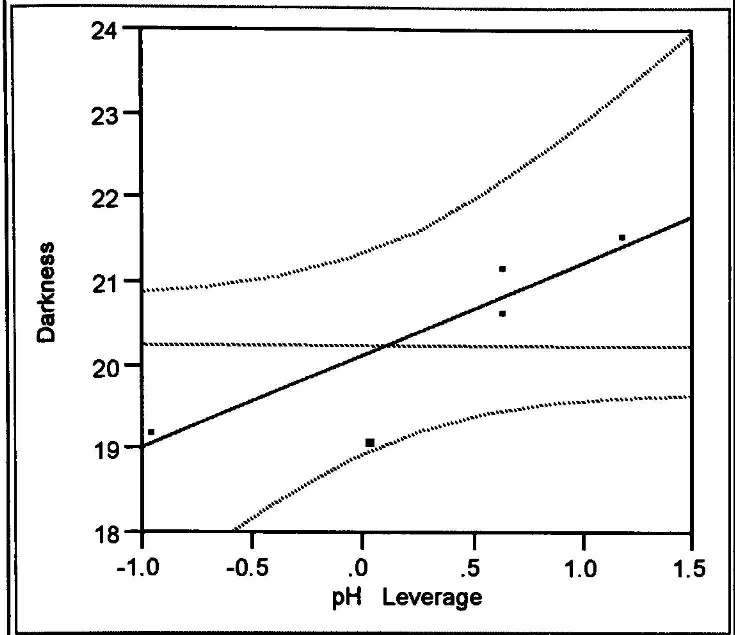


Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	31.528561	4.50408	5.9228
Error	2	1.520929	0.76046	Prob>F
C Total	9	33.049490		0.1520



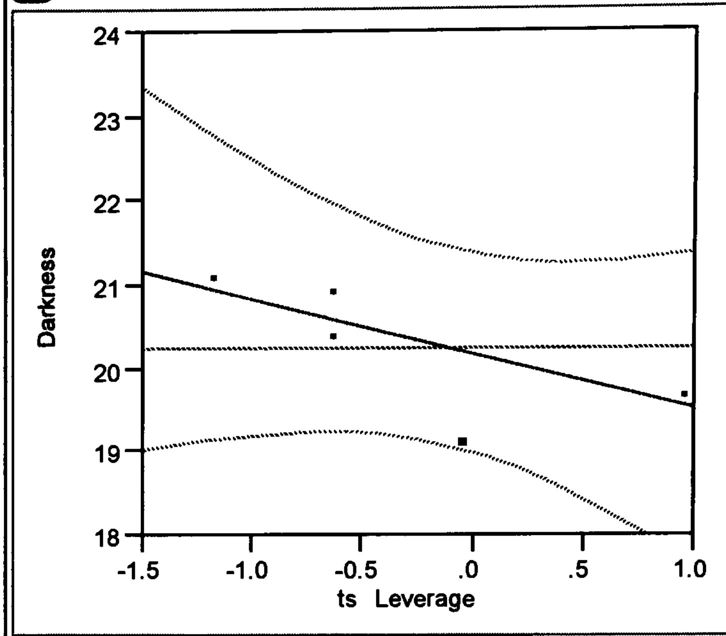
pH



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
10.576889	13.9085	1	0.0650

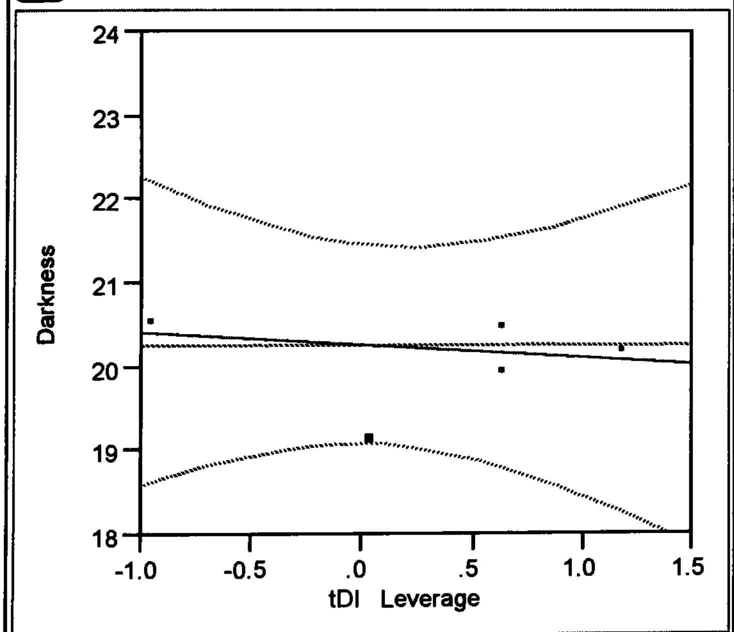
ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
3.7251609	4.8985	1	0.1573

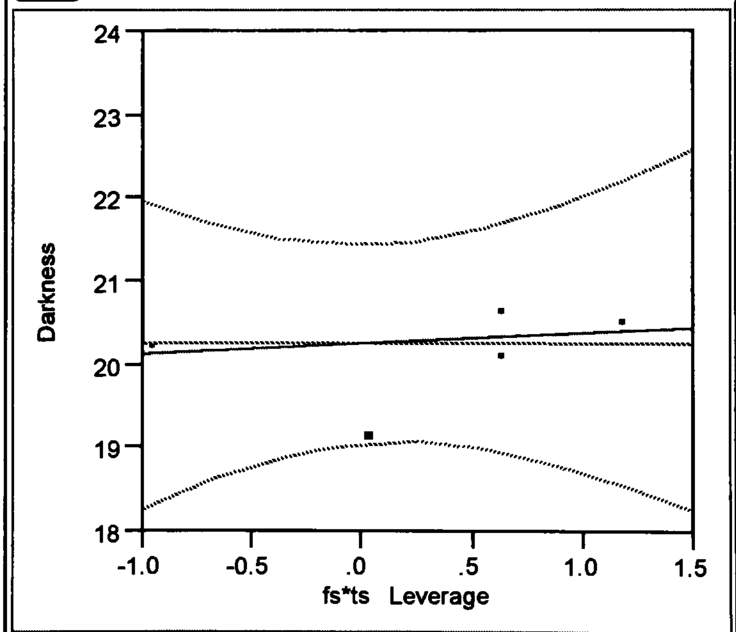
tDI



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.20363103	0.2678	1	0.6564

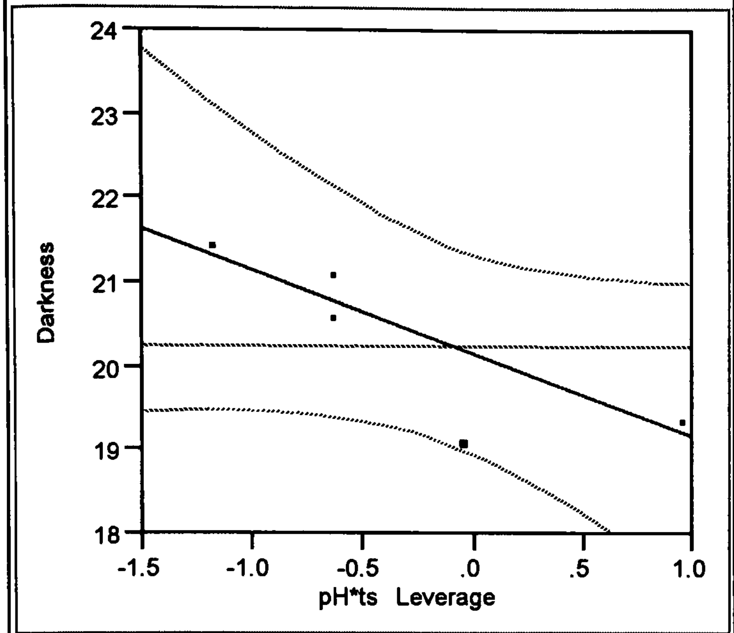
fs*ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.12928588	0.1700	1	0.7201

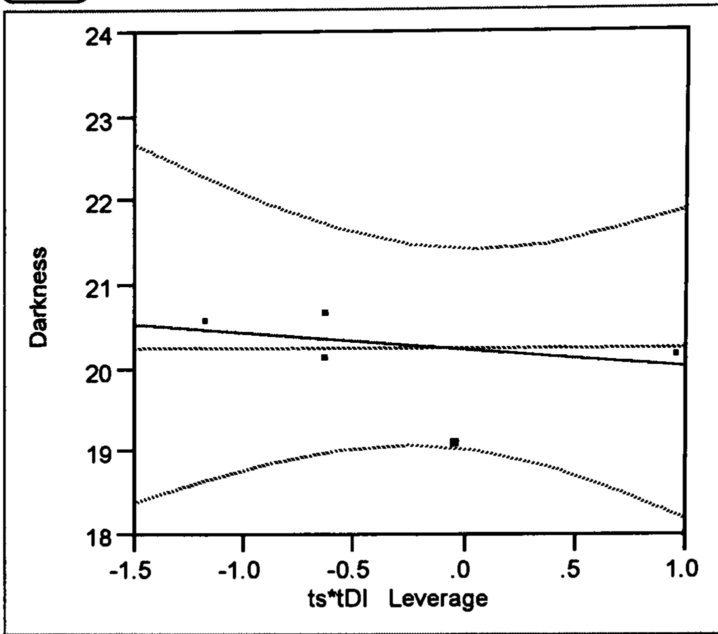
pH*ts



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
8.2505098	10.8493	1	0.0811

ts*tDI



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
0.32649670	0.4293	1	0.5796

Response: Darkness

Summary of Fit

RSquare	0.981272
RSquare Adj	0.950059
Root Mean Square Error	0.438307
Mean of Response	20.42
Observations (or Sum Wgts)	9

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	2	0.43588929	0.217945	1.5518
Pure Error	1	0.14045000	0.140450	Prob>F
Total Error	3	0.57633929		0.4937
				Max RSq
				0.9954

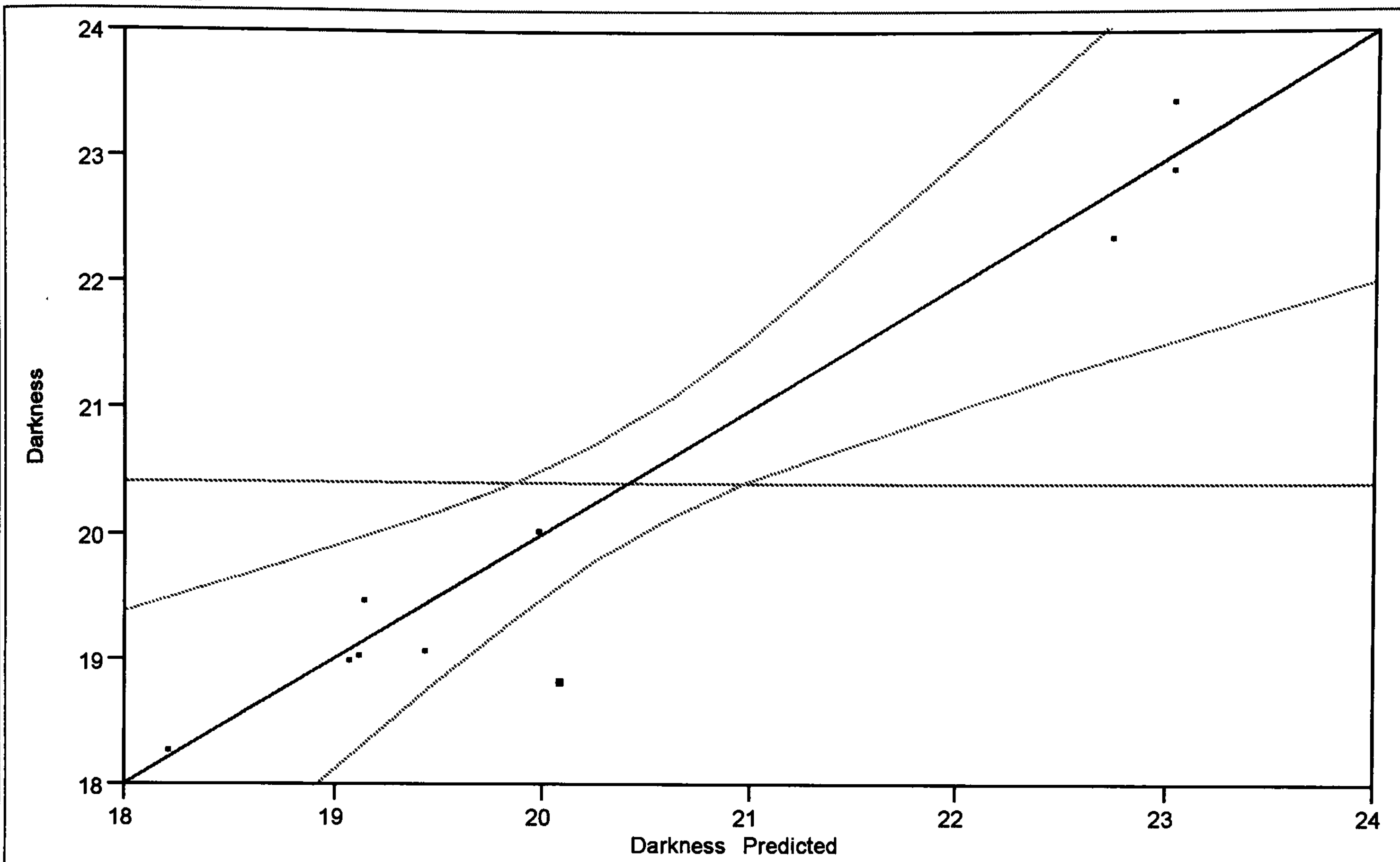
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	20.093393	0.149328	134.56	0.0000
fs	-0.285893	0.149328	-1.91	0.1514
pH	1.1258929	0.149328	7.54	0.0048
ts	-0.673393	0.149328	-4.51	0.0204
tDI	-0.141607	0.149328	-0.95	0.4129
pH*ts	-0.995893	0.149328	-6.67	0.0069

Effect Test

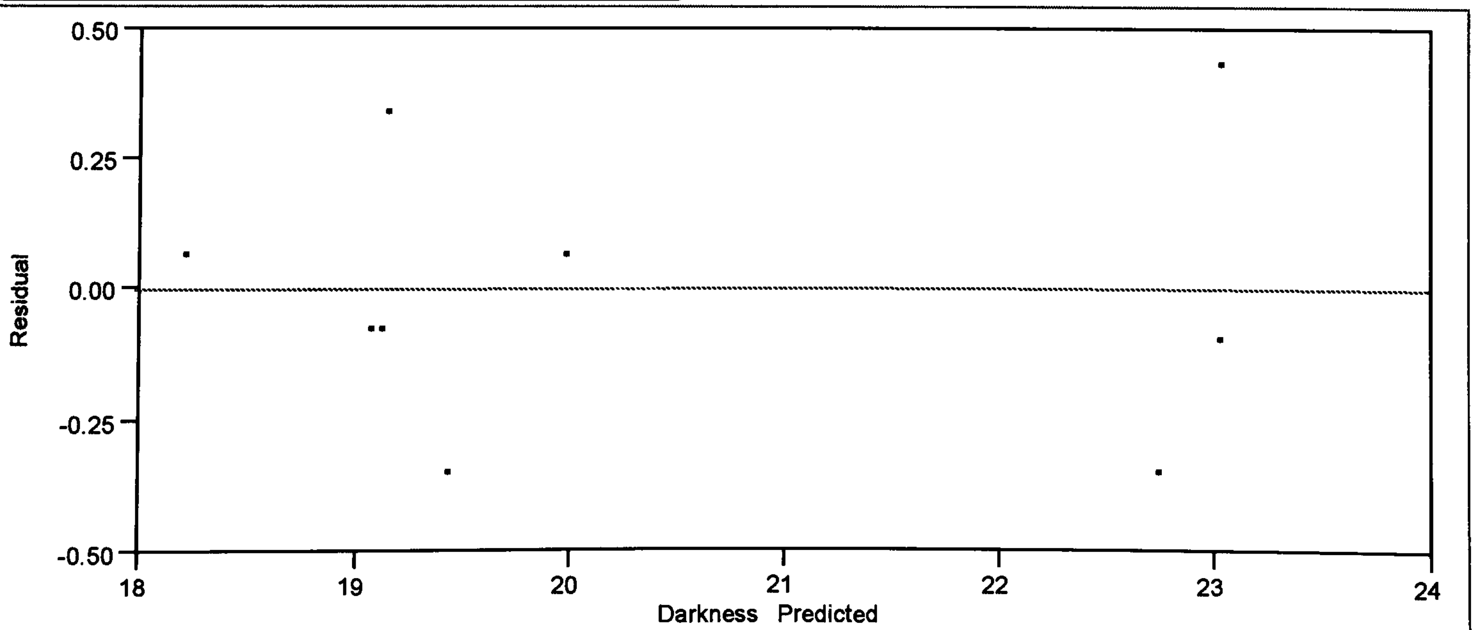
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
fs	1	1	0.704176	3.6654	0.1514
pH	1	1	10.921161	56.8476	0.0048
ts	1	1	3.906715	20.3355	0.0204
tDI	1	1	0.172761	0.8993	0.4129
pH*ts	1	1	8.544761	44.4778	0.0069

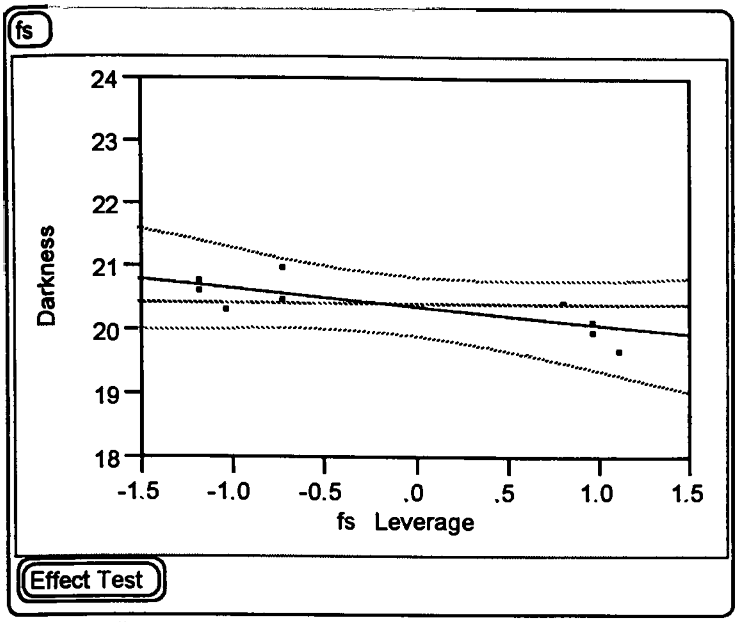
Whole-Model Test

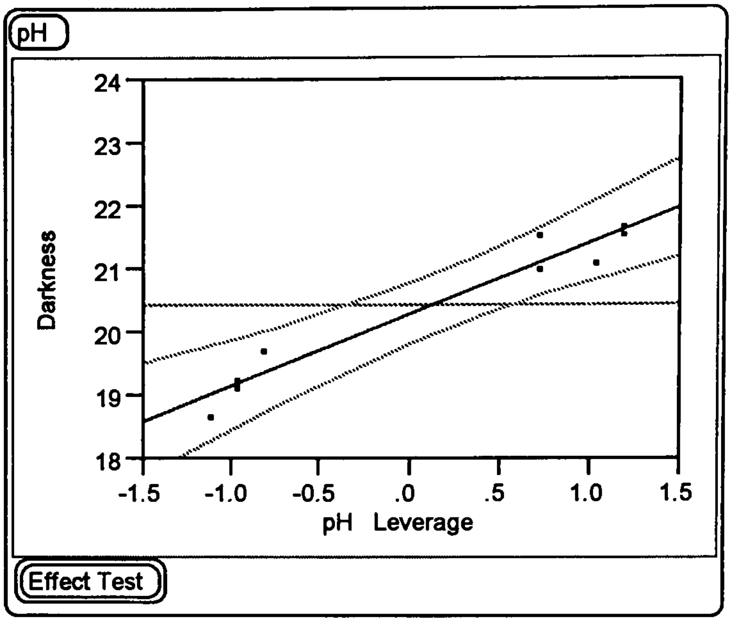


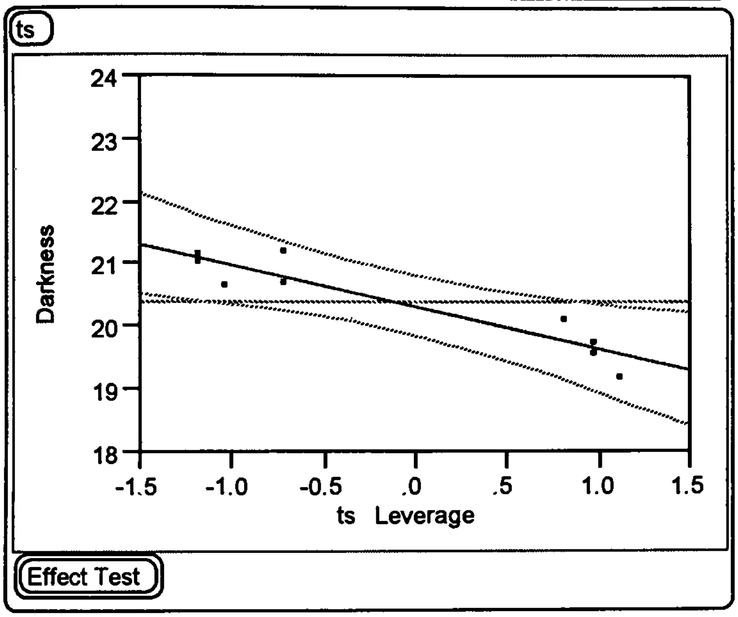
Analysis of Variance

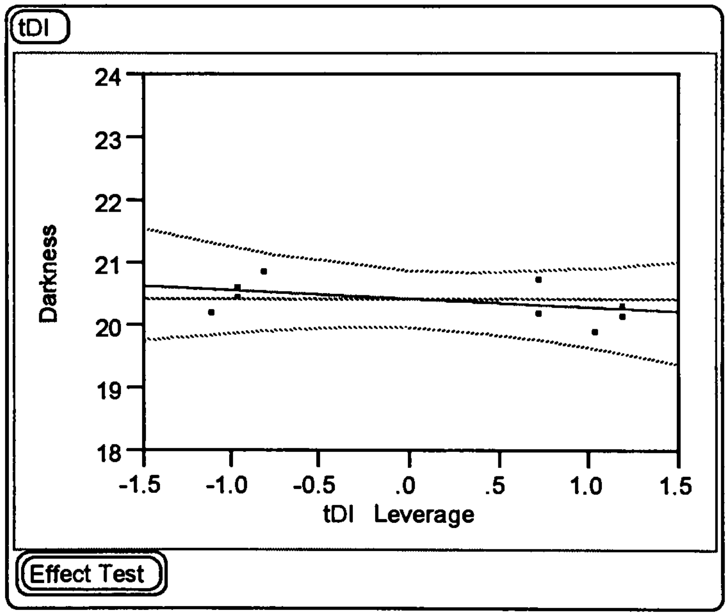
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	30.197861	6.03957	31.4376
Error	3	0.576339	0.19211	Prob>F
C Total	8	30.774200		0.0086

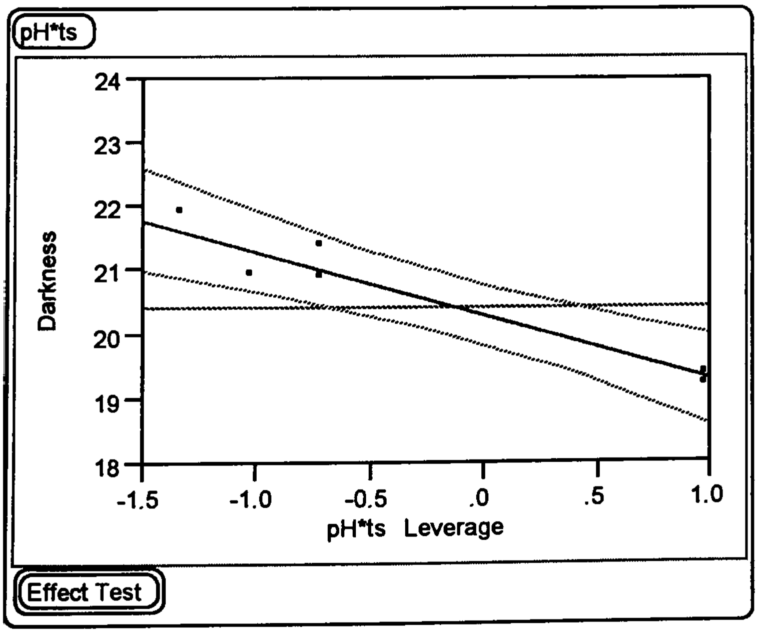



fs
Darkness
24
23
22
21
20
19
18
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
fs Leverage
Effect Test


pH
Darkness
24
23
22
21
20
19
18
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
pH Leverage
Effect Test


ts
Darkness
24
23
22
21
20
19
18
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
ts Leverage
Effect Test


tDI
Darkness
24
23
22
21
20
19
18
-1.5
-1.0
-0.5
.0
.5
1.0
1.5
tDI Leverage
Effect Test


pH*ts
Darkness
24
23
22
21
20
19
18
-1.5
-1.0
-0.5
.0
.5
1.0
pH*ts Leverage
Effect Test

Response: Darkness

Summary of Fit

RSquare	0.913343
RSquare Adj	0.870014
Root Mean Square Error	0.69089
Mean of Response	20.261
Observations (or Sum Wgts)	10

Lack of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack of Fit	1	1.4594581	1.45946	5.1956
Pure Error	5	1.4045167	0.28090	Prob>F
Total Error	6	2.8639748		0.0716
				Max RSq
				0.9575

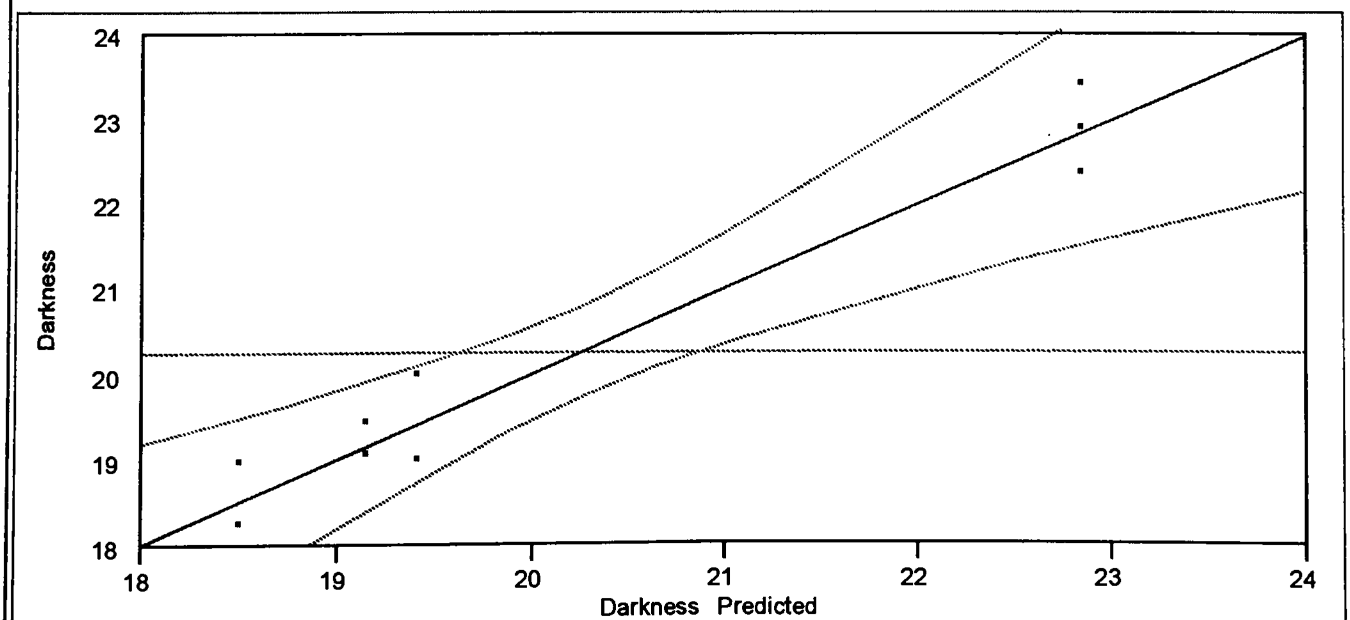
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	19.974299	0.22152	90.17	0.0000
pH	1.1498364	0.233768	4.92	0.0027
t seal	-0.697336	0.233768	-2.98	0.0245
pH*ts	-1.019836	0.233768	-4.36	0.0048

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
pH	1	1	11.548347	24.1937	0.0027
t seal	1	1	4.247491	8.8985	0.0245
pH*ts	1	1	9.084661	19.0323	0.0048

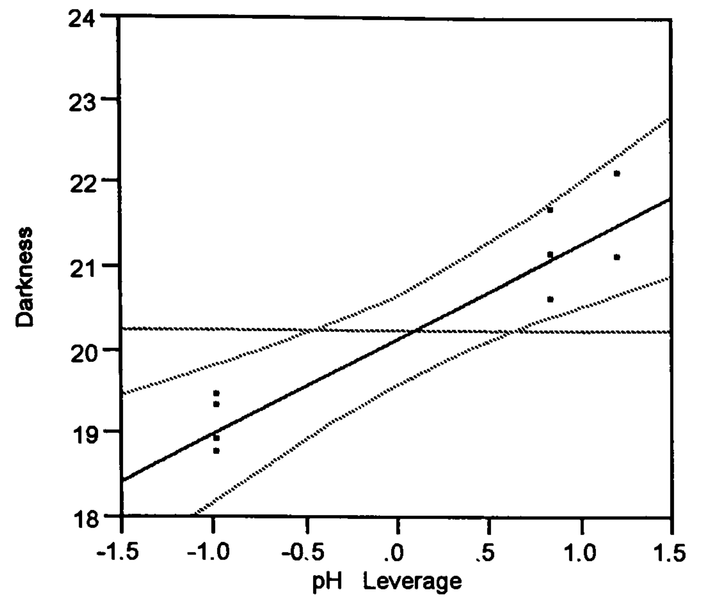
Whole-Model Test



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	30.185515	10.0618	21.0795
Error	6	2.863975	0.4773	Prob>F
C Total	9	33.049490		0.0014

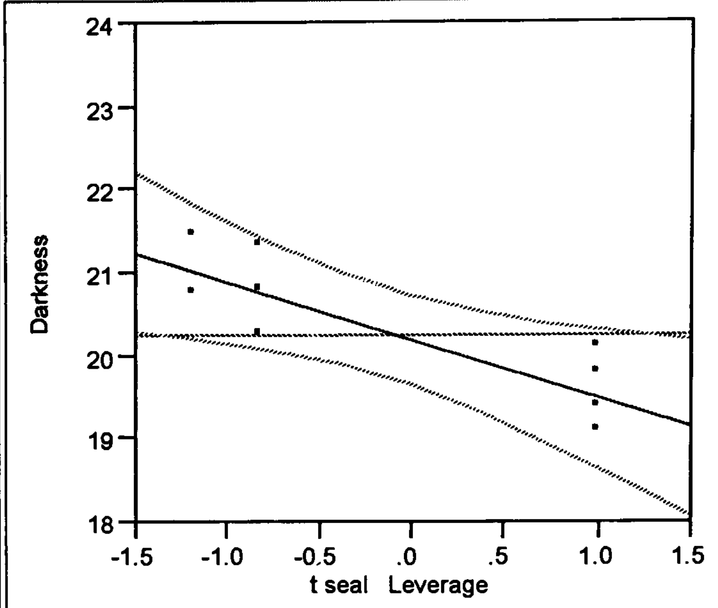
pH



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
11.548347	24.1937	1	0.0027

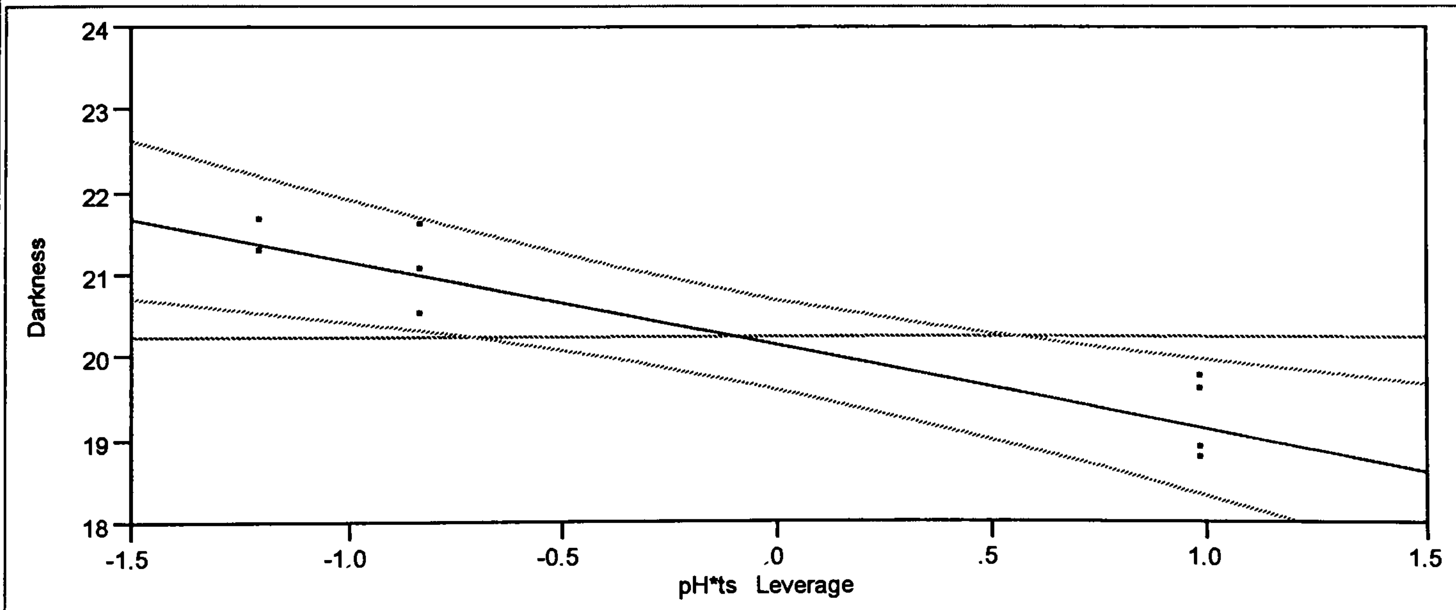
t seal



Effect Test

Sum of Squares	F Ratio	DF	Prob>F
4.2474905	8.8985	1	0.0245

pH*ts



Effect Test

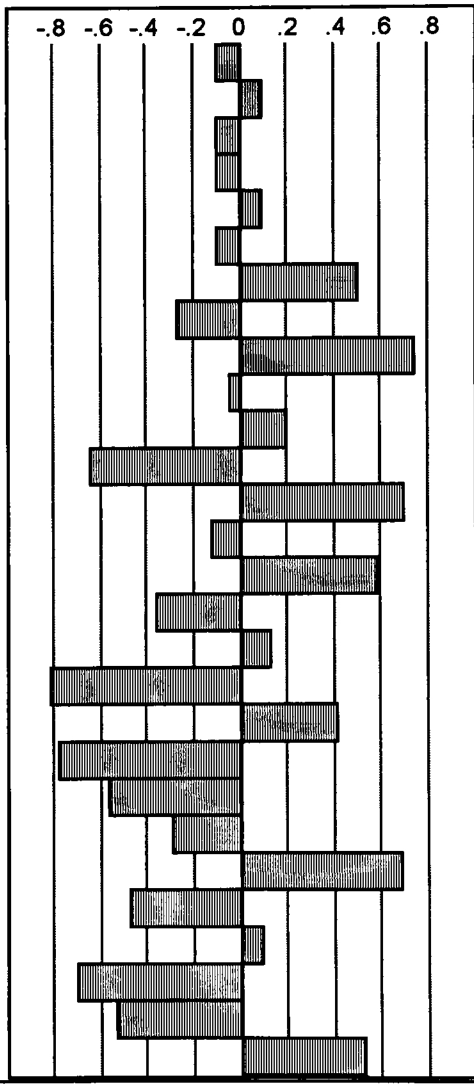
Sum of Squares	F Ratio	DF	Prob>F
9.0846615	19.0323	1	0.0048

Correlations

Variable	fs	pH	ts	tDI	Smut	Blue	Degree of Seal	Darkness
fs	1.0000	-0.1011	0.1011	-0.1011	0.5064	0.2029	-0.3592	-0.2950
pH	-0.1011	1.0000	-0.1011	0.1011	-0.2709	-0.6470	0.1256	0.6868
ts	0.1011	-0.1011	1.0000	-0.1011	0.7420	0.6968	-0.8108	-0.4757
tDI	-0.1011	0.1011	-0.1011	1.0000	-0.0353	-0.1158	0.4100	0.0956
Smut	0.5064	-0.2709	0.7420	-0.0353	1.0000	0.5870	-0.7746	-0.6963
Blue	0.2029	-0.6470	0.6968	-0.1158	0.5870	1.0000	-0.5625	-0.5256
Degree of Seal	-0.3592	0.1256	-0.8108	0.4100	-0.7746	-0.5625	1.0000	0.5304
Darkness	-0.2950	0.6868	-0.4757	0.0956	-0.6963	-0.5256	0.5304	1.0000

Pairwise Correlations

Variable	by Variable	Correlation	Count	Signif Prob
pH	fs	-0.1011	10	0.7810
ts	fs	0.1011	10	0.7810
ts	pH	-0.1011	10	0.7810
tDI	fs	-0.1011	10	0.7810
tDI	pH	0.1011	10	0.7810
tDI	ts	-0.1011	10	0.7810
Smut	fs	0.5064	10	0.1352
Smut	pH	-0.2709	10	0.4490
Smut	ts	0.7420	10	0.0140
Smut	tDI	-0.0353	10	0.9228
Blue	fs	0.2029	10	0.5741
Blue	pH	-0.6470	10	0.0432
Blue	ts	0.6968	10	0.0251
Blue	tDI	-0.1158	10	0.7502
Blue	Smut	0.5870	10	0.0744
Degree of Seal	fs	-0.3592	10	0.3080
Degree of Seal	pH	0.1256	10	0.7296
Degree of Seal	ts	-0.8108	10	0.0044
Degree of Seal	tDI	0.4100	10	0.2393
Degree of Seal	Smut	-0.7746	10	0.0085
Degree of Seal	Blue	-0.5625	10	0.0905
Darkness	fs	-0.2950	10	0.4080
Darkness	pH	0.6868	10	0.0282
Darkness	ts	-0.4757	10	0.1646
Darkness	tDI	0.0956	10	0.7928
Darkness	Smut	-0.6963	10	0.0253
Darkness	Blue	-0.5256	10	0.1187
Darkness	Degree of Seal	0.5304	10	0.1148



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