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The study of factors that affect cell depth during etching in direct transfer gravure

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THE STUDY OF FACTORS THAT AFFECT CELL DEPTH DURING ETCHING IN DIRECT TRANSFER GRAVURE

by

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ABSTRACT

This thesis is to study the four main factors which affect cell depth in copper cylinder etching in direct transfer gravure, and using ferric chloride as an etchant. The multivariate regression method was used to analyze the sample responses (cell depth) of nine different treatments of an etched-screened-tone scale. The regression equations for predicting cell depths from the four variable factors were calculated and the optimum condition which would give the best tonal gradation was predicted. Also the factors which cause uneven cell depth were detected.

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CERTIFICATE OF APPROVAL

MASTER'S THESIS

This is to certify that the Master's Thesis of

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TABLE OF CONTENTS

LIST OF TABLES

Table

LIST OF FIGURES

LIST OF PLATES

LIST OF CURVES

Curves

Chapter 1

INTRODUCTION

The Problem Proper

In gravure cylinder etching, there is one method called direct transfer which is a simplified and uncomplicated method of transfering a line or a halftone image onto a cylinder ready for etching. The etchant commonly used in this method is ferric chloride which in theory is supposed to etch all cells to the same depth in the copper surface of the cylinder. Tonal gradation can be created by varying the width of the cell.

In Practice, the midtone cells etch more deeply than the shadow cells. The deeper midtone cells contribute to an increase in ink volume making it about the same as that of the shadow areas. This problem has the effect of compressing the total tone range from highlight to shadow.

The aim of this thesis is to study the factors that affect the ferric chloride etching of the copper clad cylinder. Though this study will not include the solution to the problem. The results of this study will probably be useful for further study of gravure cylinder etching.

Justification of The Study

Gravure printing today is playing an important role in the printing industry and is becoming increasingly more important everyday. This is true not only in the packaging and specialties field but also in the publication field as well. The reason for this is its advantages over other printing processes, i.e., gravure has a recessed image of long image carrier life, it gives more color saturation, it has highest possible press speed, and has the widest range of press width and variable repeat length. As new techniques and automatic machines are invented, gravure receives more and more daily use.

Although new engraving methods have been introduced to gravure cylinder engraving, such as electro-mechanical engraving and laser engraving, chemical etching with ferric chloride still receives extensive use. One of the chemical etching methods is direct transfer which is used by packaging printers. Some of the advantages of this direct transfer cylinder making process[1] are:

1. Reduced production cost.
2. Greater production with fewer people.
3. A simplified etching procedure requiring less skill on the part of the cylinder maker.

4. Cost reduction for remaking of cylinders due to rapid and reliable processing.
5. Exact duplication of the dot structure can be achieved because of the reliability of the screened positive.
6. The possibility of attaining greater cell depth in order to provide greater ink volume which is sometimes needed for packaging printing.*

Present State of The Art

The current principle of gravure is to etch or engrave millions of image cells into the copper surface of the printing cylinder by one means or another. These cells will hold a different volume of ink, depending on the tone of a individual image area. This is accomplished by variation of cell depth and/or cell width.

There are many means for imaging gravure cylinders today. The oldest and most basic is chemical etching. In this process,

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*Personal conversation with Mr. Fred Voetsch, General Manager, North American Roto Engraving Inc., Wadsworth, Ohio, July 12, 1980.

the etchant that is usually used is ferric chloride ($FeCl_3$). Chemical etching can be used in three different ways in the variation of ink volume for each tone area.

The first method is conventional gravure, which is the method that uses a gravure screen and a continuous tone positive to expose a low contrast sensitive diffusion transfer resist. Tonal gradations are produced by varying only cell depth while the cell width remains constant.

The second method is lateral hard dot gravure which also uses a low contrast sensitive diffusion transfer resist but is exposed with a screened positive and continuous tone positive. The cell volume is varied by both the depth and the width of the cell.

The third method is direct transfer gravure, which employs the use of a resist-coated copper surfaced cylinder which is exposed with a screened positive. The cell depth is theoretically constant and thus the ink volume in this method is varied by the width of the cell.

The three chemical etching methods are briefly shown in Figure 1.

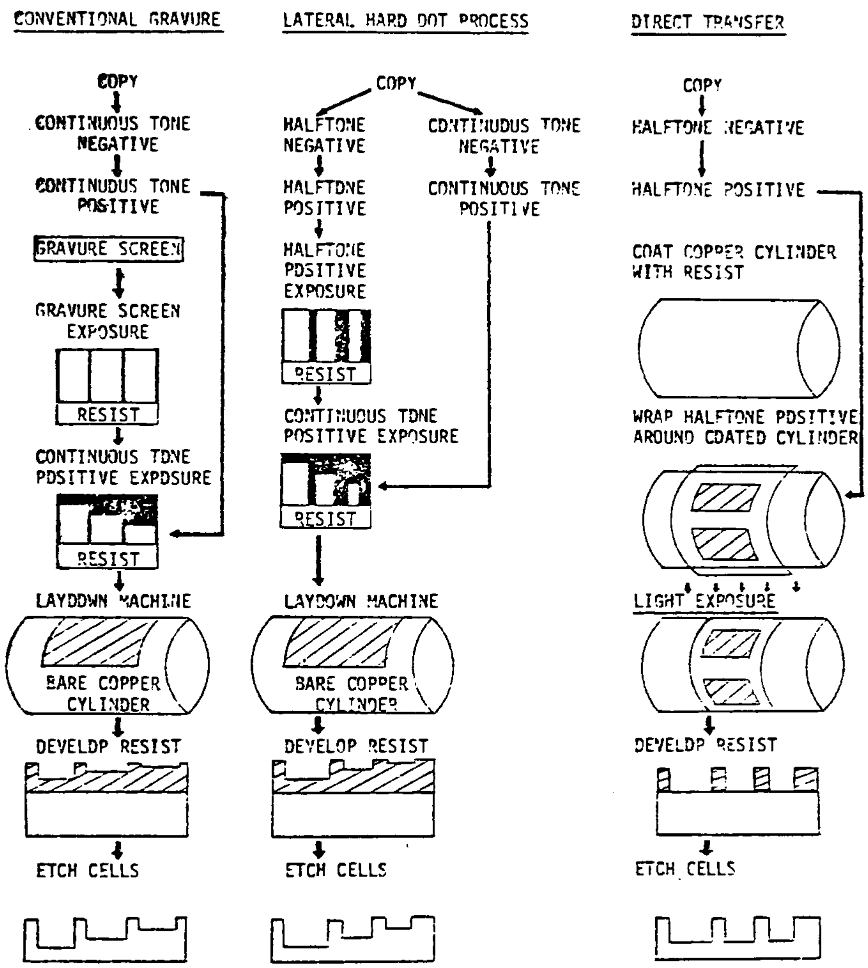

Figure 1.

Chemical Etching Process*

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*Taken from the handout for Gravure course at the Rochester Institute of Technology, Rochester, New York.

There is another etching method which was introduced by the Gravure Research Institute called the powderless etching method[2] (Figure 2), which is supposed to improved the tonal reproduction capabilities of direct transfer gravure. The principle of this is to coat the copper cylinder with a photoresist and expose it to a screened positive and then develop the cylinder in a solvent which removes the unhardened resist. Special chemicals are added to the ferric chloride prior to etching, one of which is thiourea. As etching proceeds, and as cells are formed by etching, the cells are filmed over by the special chemical substance which is called a banking agent. The banking agent at the bottom of the cells, where the force of the etchant is greatest, is broken through so that the cells are etched deeper without etching sideways. In this way, the cell shape is varied both in depth and width.

Another method of imaging a gravure cylinder is electromechanical engraving. The principle of this is to scan reflection copy and distinguish the light and dark areas. The information of density values is then converted to electronic signals and sent to a computer. Corresponding to these signals, the computer will control a diamond stylus (engraving head), to engrave cells into the copper surface of the cylinder (Figure 3).

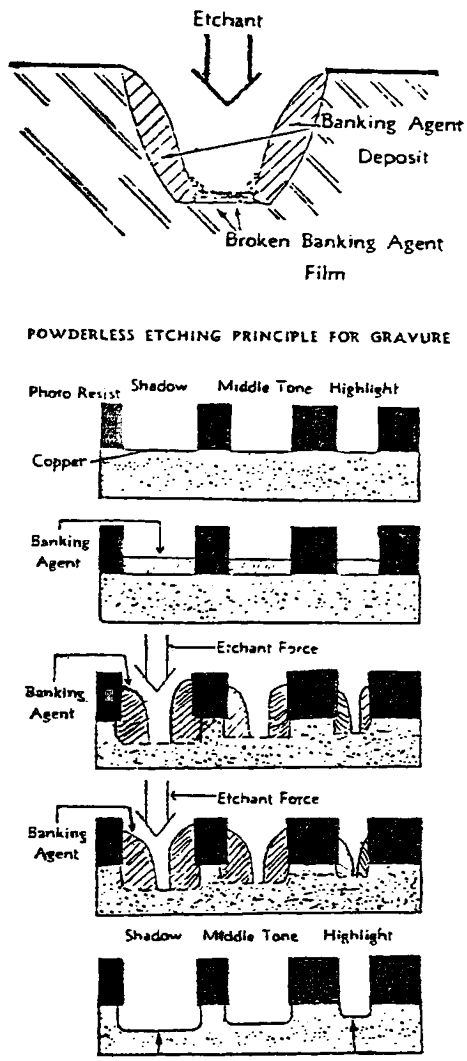

Figure 2.

Powderless Etching Principle*

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*Taken from: Steven A. Dzurik, "Powderless and Spray Etching Systems," Gravure Bulletin, Vol.21, No.4, Winter 1970, p. 41-42.

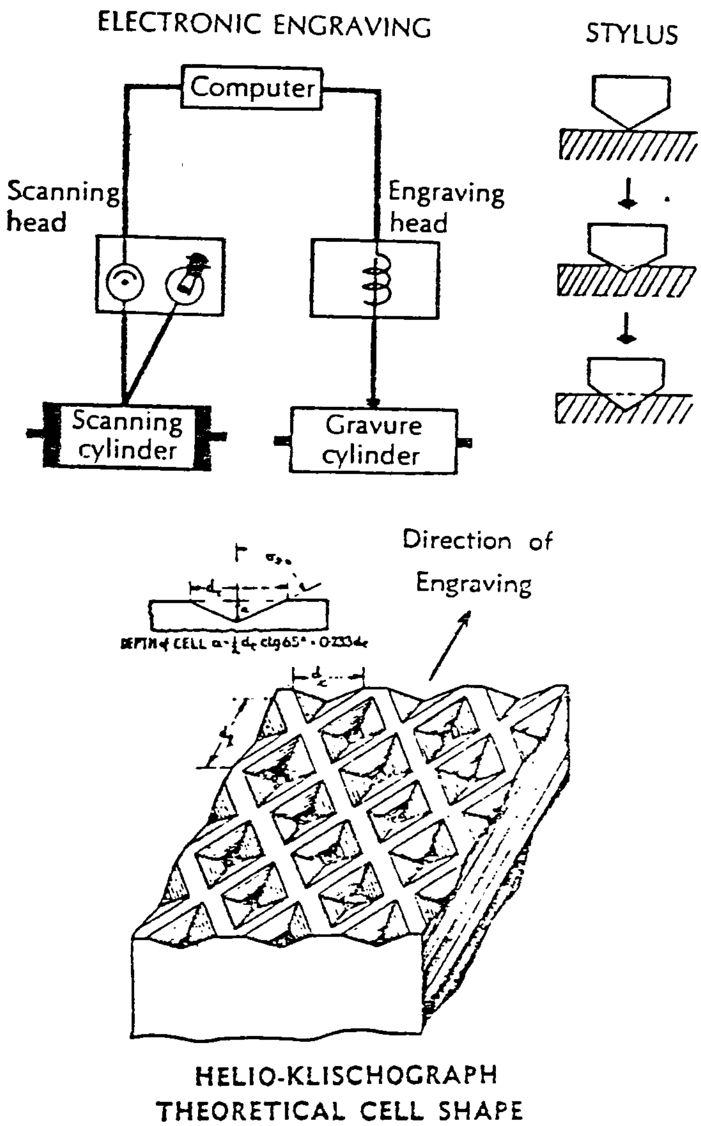

Figure 3.

Electro-mechanical Engraving*

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*Taken from: Oscar Smiel, Technical Guide for the Gravure Industry (New York: Gravure Technical Association, Inc., 1975), p. 107.

The latest engraving method uses a laser beam instead of a stylus to engrave the gravure cylinder. This approach uses a sheet of plastic that is butted and welded to form a cylindrical sleeve that is slipped over the steel cylinder, which is then heated to make the plastic shrink for a tight fit to the cylinder. A low energy laser is then used to engrave the plastic surface with grooves instead of cells. This approach is still under development in England.[3]

Endnotes for Chapter 1

[1]A.A. Scala, "Cylinder Engraving by Direct Transfer methods," Gravure Bulletin, Vol. 15, No. 1, February 1964, p. 84-85.

[2]Steven A. Dzurik, "Powderless and Spray Etching Systems," Gravure Bulletin, Vol. 21, No. 4, Winter 1970, p. 41-44.

[3]R. A. G. Dunkley, "Laser Gravure," GRI Newsletter, No. 4, March 1978, p. 25-31.

Chapter 2

REVIEW OF THE LITERATURE

The direct transfer method of gravure cylinder etching uses a single etch bath with the etching working directly on the copper cylinder. The principle of etching does not involve the absorption and penetration of a gelatin layer or tissue as in conventional or lateral hard dot gravure. The ferric chloride will react with and etch copper where there is no protection offered by the light hardened resist.

Pure ferric chloride for medicinal and laboratory use is made by passing chlorine gas over heated iron wire. Purple colored anhydrous ferric chloride is formed on cooling the vapor. For commercial use ferric chloride is made by dissolving scrap iron in hydrochloric acid or muriatic acid and oxidizing this with nitric acid. When the concentration is high, solid lumps of hydrated ferric chloride are formed, and these are colored yellow brown and are very hygroscopic.*

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*The ability of material to absorb or otherwise take up moisture from the surroundings.

Chemistry of Etching

The reaction of ferric chloride and copper in etching is

$$FeCl_3 + Cu \longrightarrow FeCl_2 + CuCl \quad (1)$$

Ferric Chloride undergoes what is known as a reductions,* and copper undergoes what is termed oxidation.** There are many other solutions besides ferric chloride, which are strong oxidizing agents and will oxidize copper, but they don't produce a smooth etch or retain their colors. Since ferric chloride meets all the requirements, it is almost universally used.

Cuprous chloride (CuCl), which is the result of the reaction in equation (1) is a white solid, soluble in an adequate amount of ferric chloride as shown in equation (2).

$$CuCl + FeCl_3 \longrightarrow CuCl_2 + FeCl_2 \quad (2)$$

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*A reaction in which the atoms in an element gain electrons and its valence is correspondingly dereased. In equation (1), Fe^{+3} is reduced to Fe^{+2}.

**An opposite reaction of reduction. In equation (1), Cu^{0} is oxidizes to Cu^{+1}.

The disintegration of the ferric chloride etching is due to the gradual reduction of ferric chloride to ferrous chloride. Ferrous chloride has no etching effect on the copper. It will only increase the copper content of the bath. The net result is usually a shallow etching.

When a fresh bath is made up, it is a reddish brown color. Then, after the copper has been etched the ferric chloride turns to a dark chocolate color which is the best atching solution. With further use, the solution takes on a dirty, greenish color, of cupric and ferrous chloride.[1] At this point it can no longer be used for etching.

The important factors that affect this reaction and cell depth are:

1. <u>Concentration of the etching solution</u>

In the printing trade, the concentration of a solution is often expressed in density unit 'Be' (degree Baume').* From the experiment in letterpress plates etching, copper plates were

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*Unit for measuring the density of specific gravity of solutions. Named for the scale on a graduated hydrometer invented by Antoine Baume', French chemist.

etched face down in a tray at 90°F (32.2°C) in various concentrations of ferric chloride. The more concentration of ferric chloride used the slower the etching rate, as shown in Table 1.[2]

Table 1

Effect of Ferric Chloride Concentration on Copper Etching*

Degrees Baumé	Minutes to Etch 0.001 Inch	Etch Factor	Quality of Etch
27	5.8	3.7	Rough
30	4.9	3.8	"
33	5.4	3.9	"
36	5.7	3.7	"
40	6.0	4.2	"
42	7.1	3.7	"
45	8.6	3.6	"

*Taken from: Roland M. Schaffert and others, The Ferric Chloride Etching of Copper for Photoengravings (Ohio: Photo-Engravers Research, Inc., 1949), p. 14.

2. Effect of free acid content

Free acid is defined as the acid present in excess of that produced by hydrolysis of the ferric chloride. Pure ferric chloride when dissolved in pure water will give a definite acid solution and cannot be made basic. A small proportion of ferric chloride will always undergo this reaction,

$$FeCl_3 + H_2O \longrightarrow Fe(OH)_3 + 3HCl \qquad (3)$$

The acid produced might be neutralized with a base, but this will only result in more ferric chloride undergoing the above reaction, so that the acidity is maintained. However, if hydrochloric acid is added to ferric chloride, such acid is called free acid. This free acid content has a marked effect on the rate of etching. It is found in the experiment using carbon tissue that, although a solution carrying less than .2% free acid by weight would probably not cause the tissue to lift from the engraving cylinder such a solution would etch so fast that etchers would lose control of the process and images would lack the required long tone scale.[3]

3. Effect of temperature

It has been found that temperature affects primarily the rate of etching and has a minor influence on the color -

loss* in letterpress plate etching. The results in Table 2 were obtained by etching halftones. The etching solution used was 40° Be' ferric chloride in which copper was dissolved until a concentration of two oz. per gallon had been reached. A slight up and down agitation of the plate was employed during the etching process. It was concluded that the higher the temperature the higher the etching rate and color loss. Also these results can be related to ferric chloride etching of copper surfaced gravure cylinders.[4]

4. Effect of dissolved copper

The role that dissolved copper plays in etching has been found by etching letterpress halftones in 40° Be' ferric chloride solution at 90°F (32.2°C) with various amounts of copper in the bath. Table 3 shows the time required to etch to about 0.0025 inch (0.0635 mm.) in the highlights.[5]

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*A letterpress plate term, the higher the etch factor (ratio of cell depth over cell width) the lower the color loss. This can be related to side etching in gravure cylinder etching, the increase in side etching may causes a problem of cell wall damage.

Table 2

Effect of Temperature of Ferric Chloride on

Copper Etching of Halftones (120 Lines)*

Temperature, Degrees F.	Minutes to Etch Highlights to 0.0025 Inch	Highlight Color Loss (Percent Dot Area Reduction)
70	27	19
90	26	24
110	23	28
130	16	25

*Roland M. Schaffert and others, p. 18.

Table 3

The Effect of Dissolved Copper on the Time of Etching for Halftones*

Dissolved Copper, Oz./Gallon	Time to Etch Highlights to 0.0025 Inch, Minutes
0.0	18
0.5	23
2.0	28
4.0	30
8.0	40

The results of this halftone etching in letterpress can be related to gravure cellular etching, because both involve small etching areas with shallow etching. Also, the reaction of ferric chloride and copper is the same.

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*Taken from: Roland M. Schaffert and others, The Ferric Chloride Etching of Copper for Photoengravings (Ohio: Photo-Engravers Research, Inc., 1949), p. 21.

5. <u>Effect of ferric sulfate content</u>

From the experiment using Rotofilm* and 0-4% of ferric sulfate content, the 0% iron was eliminated because of the difficulty of closing out the etch in 7.25 minutes. The 1% was also eliminated for the same reason. The field of choice was therefore narrowed down to the 2, 3 & 4% ferric sulfate.

For the differences in cell width, the 2 and 3% iron held the dot size better, limiting the normal spread of the cell size. It was also shown that the 2% iron was somewhat erratic in its behavior. It held cell size fairly well in the shadows and highlights but showed a great spread in the middle tones.[6]

6. <u>Effect of viscosity</u>

It was found that as the ferric chloride solution is diluted, its velocity is reduced, making it possible for the solution to get through the tissue** and out again more rapidly.[7]

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*A Du Pont product having similar characteristic to, and made to replace, carbon tissue. Note that this study didn't use Rotofilm.

**Carbon tissue is a light-sensitive, orange-colored, gelatin coated paper used for the transfer of an image from a glass or film positive to the copper cylinder or plate. Note that this paper is directed toward direct transfer gravure which uses no carbon tissue.

7. <u>Effect of surface tension</u>

From the result of the study of physical properties versus dilution of rotogravure solution in the Hunt Laboratory,* we can see that the surface tension goes up slowly from 48 to 45° Be', Figure 4. It then levels off until it reaches about 42° Be'. The surface tension then rises again as the solution is diluted down as far as 32° Be'. This peculiar behavior of the surface tension has not been correlated with its effect on the etching peocess.[8]

8. <u>Effect of trace metals</u>

In any etchant system, there are some trace metals such as Na, K, Ca, Mg, Zn, Ag, Au, Pa, Cu, Co, Mn, Ni, Pb, Sn, B, Va, Sb, Si, Zr, etc. as contaminants.

These can be detected and measured by using an atomic absorption spectrophotometer. The majority of these trace metals have no influence on the performance of ferric chloride as an etchant. Some however, have undesirable effects, while others have some beneficial characteristics.[9]

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*Philip A. Hunt Chemical Corporation, Palisades Park, New Jersey.

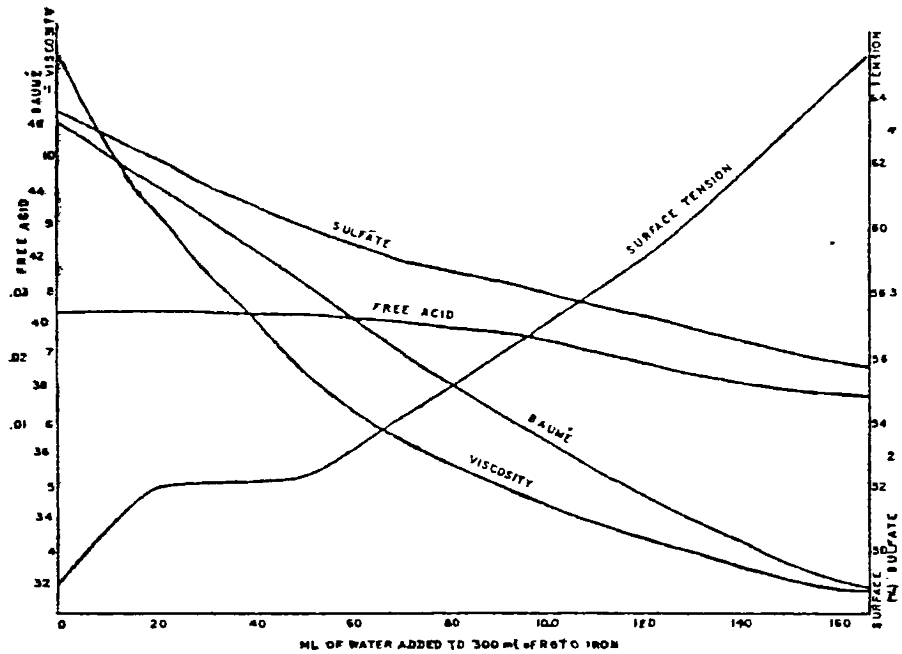

Figure 4

Physical Properties VS Dilution

of Ferric Chloride*

*Taken from: Donald Alnutt, "Rotogravure Iron Chloride Solution," Gravure, Vol. 2, No. 8, August 1956, p. 22.

9. <u>Effect of agitation</u>

An experiment done on the etching of width-variable gravure has proved its value under production conditions where the cylinder is rotated in a trough of etching solution while a light weight soft rubber roller rotates freely on the surface. The effect of roller agitation was found experimentally to be that the depth of cells in the darker tones increases as the speed rises, resulting in a fairly marked increase in contrast, Figure 5.[10]

10. <u>Effect of pressure of blanket roller on etching cylinder</u>

As shown in Figure 6, ferric chloride is supplied to the gravure cylinder by a blanket roller which gives the following advantages:

1. Breaks the small air bubbles or pockets which prevent ferric chloride from attacking copper.

2. Keeps ferric chloride circulating to provide a constant supply of fresh ferric chloride to etching the copper.

3. Reduces the need for a large volume inventory of ferric chloride.

4. Can reverse the direction of the rotating cylinder, to allow the blanket roller to attack both leading and trailing cell edges.

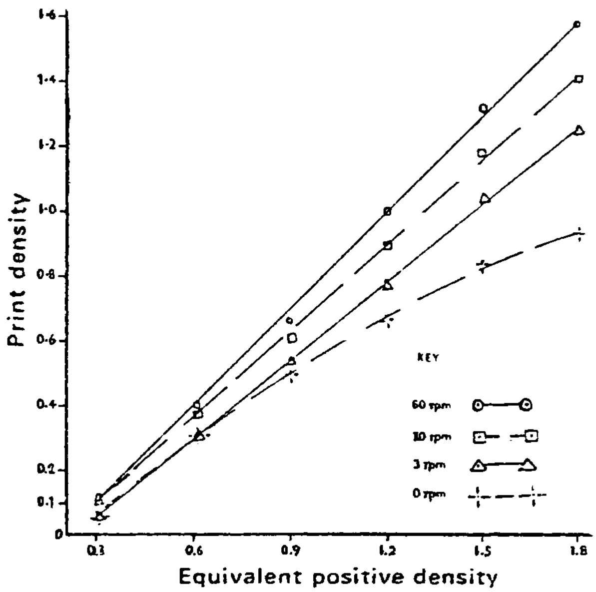

Figure 5

Effect of Agitation on Contrast*

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*Taken from: H.M. Cartwright and R. MacKay, "Gravure in the 'seventies," British Printer, Vol. 85, No. 4, April 1972, p.101.

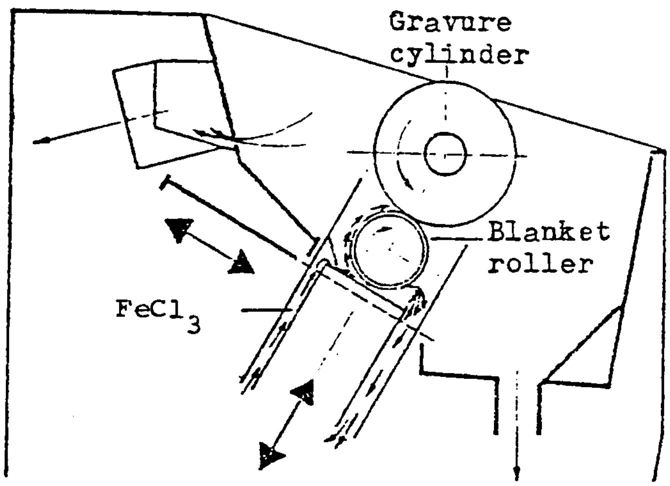

Figure 6

K. Walter Combi Etcher*

*Taken from printed material of K. Walter-Direct Etching System, Munich, West Germany.

By using the blanket roller, the rate of etching is increased and can be controlled by the pressure applied between the blanket roller and the gravure cylinder.[11]

All the above results have been reported from different experiments and different circumstances. Some of them used carbon tissue instead of photoengraver resist, and some were experiments in letterpress plate making. But all of these used ferric chloride as an etchant for copper, which is the same as this study.

Hypotheses

From the literature studied and from practical experience, there are four main factors that affect the reaction of ferric chloride etching of a copper cylinder in the direct transfer gravure cylinder imaging process. These factors are:

1. Baume' density of ferric chloride
2. Temperature of etching system (ferric chloride solution and copper clad cylinder)
3. Speed of cylinder rotation while etching
4. Pressure applied between the blanket roller and the gravure cylinder

It is hypothesized that the increase in value of these factors should increase the rate of etching (resulting in deeper cells) except for the Baume' density that should give the opposite result.

It is theorized that these factors' effects on cell depth may relate to the problem of unequal depth of the cells to some degree.

This study assumes that chemical and trace metals in ferric chloride etchant are the same in every experiment, and their effects on the etching are constant. This is also true for other parameters such as the characteristic of the photo resist, the thickness of coated-photo resist, the exposure time, etc.

Endnotes for Chapter 2

[1]Roland M. Schaffert and others, The Ferric Chloride Etching of Copper for Photoengravings (Ohio: Photo-Engravers Research, Inc., 1949), p. 10-11.

[2]Roland M. Schaffert and others., p. 14.

[3]Donald Alnutt, "Rotogravure Iron Chloride Solution," Gravure, Vol. 2, No. 8, August 1956, p. 22.

[4]Roland M. Schaffert and others, p. 17-18.

[5]Roland M. Schaffert and others, p. 19-22.

[6]Joseph W. Dragonetti, "Echants and Automatic Etching, Discussed by Intaglio Club," Gravure, Vol. 6, June 1960, p. 26, 54, 56.

[7]Donald Alnutt, p. 22.

[8]Donald Alnutt, p. 22, 52.

[9]D. L. Dzirr, "Ferric Chloride isn't simply $FeCl_3$," Gravure Bulletin, Vol. 29, No. 7, Spring 1978, p. 44-50.

[10]H. M. Cartwright and R. MacKay, "Gravure in the 'seventies," British Printer, Vol. 85, No. 4, April 1972,p.100-104.

[11]William L. Elwell, "Roller Blanket Etching," Gravure Bulletin, Vol. 21, No. 4, Winter 1970, p. 36-39.

Chapter 3

METHODOLQGY

Due to certain physical limitations which affect the experiment, the study of the four factors which affect cell depth concentrated on only the two extreme (high & low) and normal conditions of each factor. This resulted in nine data from the following design of the experiment.

The Design of Experiment

Eight experiments of the two extreme conditions were disigned by a half-repricated 2^4 experiment.[1] This means that only one half of all the combinations of four factors with two levels (two extreme, high & low) were used. And the procedures of choosing that half of all the combinations were:

1. All the combinations of factors with two levels (2^4 = 16 different runs) were written in a table (as shown in Table 4) by using "A", "B", "C" & "D" to represent the density in 'Be', the temperature, the speed of cylinder rotation and the pressure between blanket roller and gravure cylinder, respectively. For each combination, only the factor at high level was written down by using the alphabet that represents that factor, but in a lower case.

2. All the combinations in Table 4 were divided into two groups, by giving a positive sign to a factor with the high level and a negative sign to a low level factor (no alphabet written).

Table 4

All Combinations of 4 Factors with 2 Levels Each

		Baume′ Density, High (A)		Baume′ Density, Low (A)	
		Temp., High (B)	Temp., Low (B)	Temp., High (B)	Temp., Low (B)
Speed, High (C)	Pressure, High (D)	abcd	acd	bcd	cd
	Pressure, Low (D)	abc	ac	bc	c
Speed, Low (C)	Pressure High (D)	abd	ad	bd	d
	Pressure Low (D)	ab	a	b	1 (all Low)

The four factors in each combination were multiplied together by following the algebraic sign rule. The positive and negative sign of the product was the tool used to divide these combinations into two groups as follows,

Positive sign group	Nagative sign group
1 (all Low)	a
ab	b
ac	c
bc	d
ad	abc
bd	abd
cd	acd
abcd	bcd

3. The positive sign group was chosen, because the resultant data from this group would show more interaction between the factors that affected the etching than the other group.

For the ninth data, every factor was set at the normal condition as usual.

All conditions for the four factors in this experiment were set as shown in Table 5.

Table 5

The Conditions of 9 Tests in the Experiment*

Test No.	Density of $FeCl_3$, °Bé	Temperature, °C	Surface Speed, Cm/sec	Pressure, Kg/cm^2
1	38 (L)	13 (L)	27.4 (L)	2.04 (L)
2	38 (L)	13 (L)	76.2 (H)	3.06 (H)
3	38 (L)	29 (H)	27.4 (L)	3.06 (H)
4	38 (L)	29 (H)	76.2 (H)	2.04 (L)
5	41 (M)	21 (M)	51.8 (M)	2.55 (M)
6	44 (H)	13 (L)	76.2 (H)	2.04 (L)
7	44 (H)	13 (L)	27.4 (L)	3.06 (H)
8	44 (H)	29 (H)	27.4 (L)	2.04 (L)
9	44 (H)	29 (H)	76.2 (H)	3.06 (H)

*The sequence of tests should be in a random sequence, but for this experiment, the sequence was like in Table 5. This is because of some physical problems to control the conditions of independent variables (Be' density, temperature, surface speed and pressure).

Experimentation

Materials & Chemical Used

- A 220 mm. (diameter) copper surface gravure cylinder
- An 11 step screened tone scale (150 lines/in^2) with optical density of 0.03, 0.08, 0.11, 0.14, 0.18, 0.22 0.24, 0.27, 0.33 and 0.42.
- Agfa Gavaert Copyrex resist
- Developer for resist
- Toluol
- Pumice powder
- K. Walter staging solution
- Chalk slurry
- Alcohol
- 5% hydrochloric acid
- Ferric Chloride*

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*Ncte that ferric chloride used in this experiment had been used once, so there was some copper dissolved in it. But this is very little, as the cells are etched in microns, especially when compared to the amount of $FeCl_3$ used. This is what is usually used in the trade.

Equipment Used

- K. Walter ring coating machine
- K. Walter exposing machine
- K. Walter Combi Etcher
- Gravure microscope

Experimental Procedure[2]

1. Clean cylinder with powdered pumice and water, then rinse with water.
2. Rinse with 5% HCl and rinse with water.
3. Dry quickly with squeegee.
4. Load cylinder into K. Walter ring coating machine.
5. Coat cylinder with Agfa Gavaert copyrex resist at speed number 4 (353 mm./min.).
6. Expose the coated cylinder with the same positive screened tone scale on nine protions of the cylinder, using the same 16 mm. shutter opening and 18 speed (110 mm./min.).
7. Load exposed cylinder into K. Walter Combi Etcher.
8. Develop with 5% toluol dilution by these following steps:
   a. Pour developer over resist area of the rotating cylinder of 20 to 30 speed (51.8 to 74.9 cm./sec.).

develop for 1 minute.

b. Stop rotation and wait for 20 seconds.

c. Rotate again and develop another 1.5 minutes.

d. Stop rotation and wait for 20 seconds.

e. Rotate at 50 speed (126.2 cm./sec.) and rinse with water at room temperature (21°C) for 1 minute.

f. Increase speed to top speed (178 cm./sec.) and air dry.

g. Stop

9. Stage out cylinder in other portions except the first one with K. Walter staging solution.

10. Degrease with chalk slurry.

11. Water rinse.

12. Etch by hand with 15% diluted $FeCl_3$ for a half minute.

13. Etch at 13°C, 38°Be', 27.4 cm./sec. and 2.04 Kg./cm.^{2}, and etch for 4 minutes in both direction of rotation.

14. Rinse with water.

15. Dry cylinder with compressed air.

16. Stage the first etched portion out with K. Walter staging solution. Wash out the second portion with alcohol.

17. Etch the second portion with the designed condition, and repeat the etching procedure to the other portions respectively as designed.

18. Measure cell depths three times in each step of each etched-screened tone scale with gravure microscope. Find the average cell depths of each step.

Endnotes for Chapter 3

[1]Albert D. Rickmers and Hollis N. Todd, Statistics, An Introduction (New York: McGraw-Hill Book Company, 1967), p. 344-348.

[2]Printed materials for Printing Plate Methodology course, School of Printing, Rochester Institute of Technology, Rochester, New York.

Chapter 4

EXPERIMENTAL RESULTS

The cell depths of nine screened tone scales with 11 steps each, after being etched, were measured by a gravure microscope and came up with 99 results as shown in Table 6.

Vector Analysis*

By using a Characteristic Vector Analysis computer Program** written by Mr. Peborovsky of Graphic Arts Research Center, Rochester, New York, it was found that there was one vector or facter of the four factors that could explain 97.39% of the response. This means that by controlling the variation of this factor under the same conditions of this experiment and keeping the other three factors constant under normal condition, the results (as shown in Table 7) will be 97.39% close to those which resulted from the experiment. And if it is included with

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*An analysis which reduces the parameters that explain a set of responses to an intrinsic minimum.

**This computer program was written by based upon the article of J. L. Simonds.

another specific factor, the results will be 98.84% close to the results achieved under the experimental conditions. If another factor is added the results will be 99.35% as shown in Table 8. The general basis & computational results of characteristic vector analysis are shown in Appendix A.

Table 6

Experimental Results*

Test No.	Temp. °C	$FeCl_3$ °Bé	Speed Ft/sec	Press. Bar	Cell Depth (Microns) Step Number 1	2	3	4	5	6	7	8	9	10	11
1	13	38	54	2	22	22	22	22	22	18	16	20	16	16	18
2	13	38	150	3	32	36	36	36	38	38	36	38	32	32	32
3	29	38	54	3	22	24	26	24	24	24	24	22	18	20	20
4	29	38	150	2	38	44	44	46	46	44	46	44	44	44	42
5	21	41	102	2.5	30	38	38	38	34	34	34	34	34	32	30
6	13	44	150	2	30	44	44	44	42	44	44	42	42	40	40
7	13	44	54	3	18	24	24	22	22	22	20	20	20	20	20
8	29	44	54	2	18	30	34	34	30	30	28	28	26	26	26
9	29	44	150	3	30	46	48	50	50	48	52	52	48	48	48

*Note that the units used for surface speed and pressure are in ft./sec. and bar which give easier figures for the calculation. (1 Bar = 1.019716 Kg./cm^2., 1 Ft./min. = .508 Cm./sec.).

Table 7

The Artificial Responses Using One Vector,

Explain 97.39% of Experimental Responses*

Test No.	Cell Depth (Microns)										
	Step Number										
	1	2	3	4	5	6	7	8	9	10	11
1	19	22	23	22	21	20	18	19	16	16	17
2	28	36	37	38	37	36	36	36	34	33	33
3	21	25	26	25	24	23	21	22	20	20	20
4	33	44	45	46	45	45	46	46	44	43	42
5	27	36	36	37	36	35	35	35	33	32	32
6	32	42	43	44	43	43	44	43	41	41	40
7	21	24	25	24	23	22	20	21	18	18	19
8	25	30	31	31	30	29	28	29	26	26	26
9	35	48	49	50	49	49	51	50	48	47	46

*Note that these numbers are round up to two significant figures.

Table 8

The Artificial Responses Using Three Vectors,

Explain 99.35% of Experimental Responses*

Test No.	Cell Depth (Microns)										
	Step Number										
	1	2	3	4	5	6	7	8	9	10	11
1	22	22	22	21	21	20	18	19	16	16	17
2	32	36	36	36	38	36	36	37	33	33	33
3	22	24	25	24	25	23	22	23	19	20	21
4	38	45	44	46	46	45	46	45	44	43	41
5	30	38	38	38	34	35	34	33	34	32	30
6	30	43	44	45	43	43	44	42	42	41	39
7	18	23	25	24	23	22	20	21	18	19	19
8	18	31	33	33	29	29	28	28	27	27	27
9	30	46	48	50	50	49	52	51	48	48	49

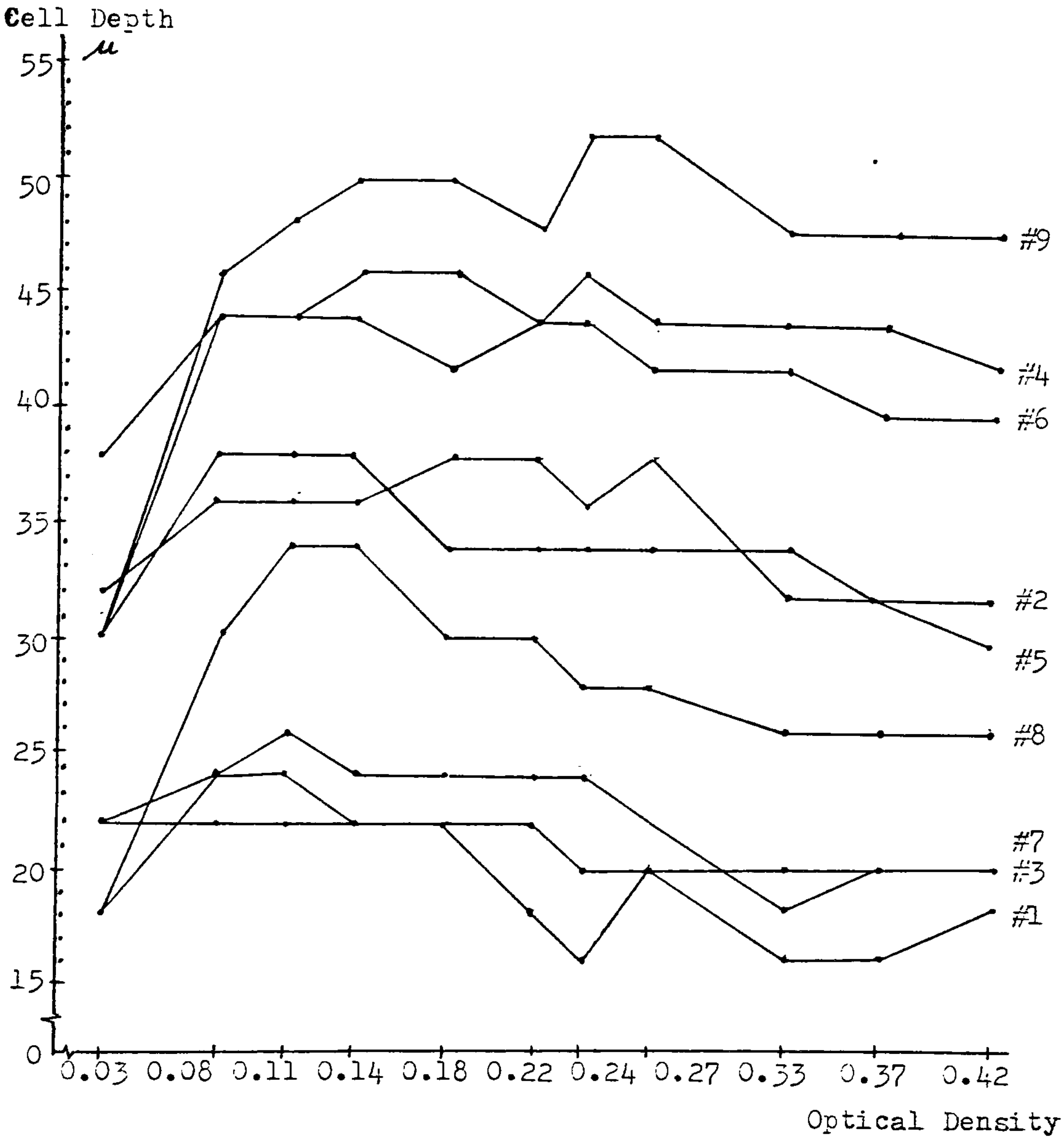
*Note that these number are round up to two significant figures.

Curve Analysis

The three unknown vectors which explain the responses up to 99.39% were found by ploting a graph of nine experimental conditions. The results between the original optical density of a positive screened tone scale and the cell depth are shown in Curve 1. From this curve, rank order was made according to the average cell depth, as follows:

Rank Order	Experimental Condition				
	Test No.	Temperature	Density	Speed	Pressure
1*	9	H	H	H	H
2	4	H	L	H	L
3	6	L	H	H	L
4	2	L	L	H	H
5	5	M	M	M	M
6	8	H	H	L	L
7	3	H	L	L	H
8	7	L	H	L	H
9	1	L	L	L	L

*The highest average cell depth.



Curve 1

Original Optical Density of Positive

Screened Tone Scale VS Cell Depth

This rank order shows that the factor which most conforms to the cell depth is "speed". Because, as the experimental conditions were set at high speed levels the responses resulted in deeper cell depth, and resulted in shallower cell depth as the speed was slowed down. This means that the first vector which explains 97.39% of the experimental responses is "speed". Also the second and third vectors were found in the same way, and these are "temperature" and "density" respectively.

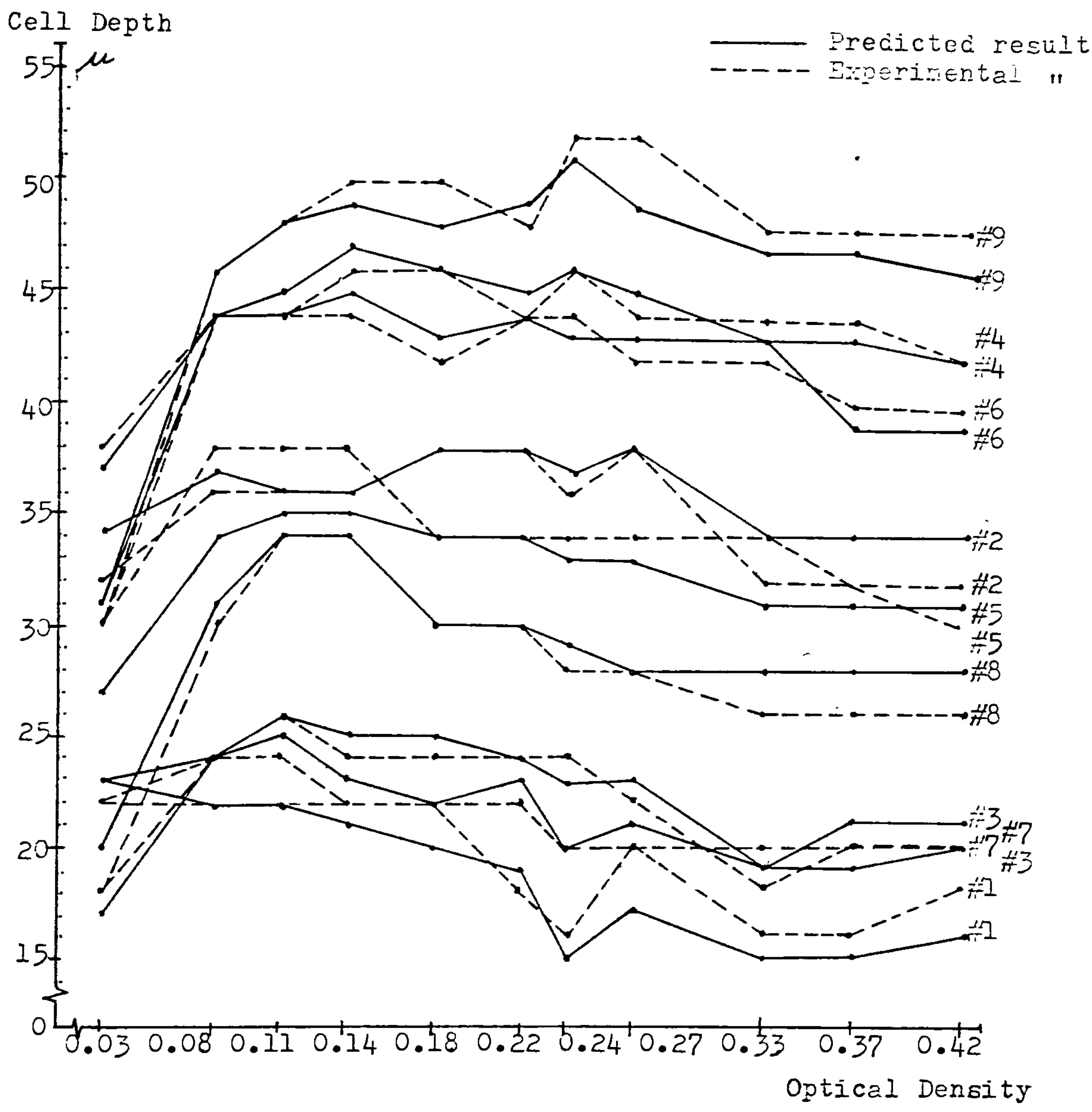
Multivariate Regression Analysis

Multivariate Regression Analysis was used to find the set of regression equations which estimate or predict the values of dependent variables (in this study this is cell depth) from observations of independent variables (these are temperature, density, speed, and pressure). By using Partial Correlation and Multivariate Regression computer program[2], the regression equation for each step of screened tone scale was calculated.

These regression equations are:

$$Y_1 = 45.9167 + .09375T - .75D + .130208S - 1.5P$$
$$Y_2 = -14.7778 + .28125T + .75D + .182292S - 2.5P$$
$$Y_3 = 122.2847 + .40625T + .916667D + .121875S - 2.5P$$
$$Y_4 = -23.2222 + .46875T + .916667D + .192708S - 3.5P$$
$$Y_5 = -15.1944 + .40625T + .583333D + .203125S - 1.5P$$
$$Y_6 = -27.2361 + .375T + .833333D + .208333S - 1.0P$$
$$Y_7 = -38.0625 + .53125T + .916667D + .234375S - 0.5P$$
$$Y_8 = -27.5417 + .40625T + .75D + .223958S - 0.5P$$
$$Y_9 = -38.4306 + .40625T + 1.08333D + .223958S - 2.5P$$
$$Y_{10} = -34.5694 + .46875T + .916667D + .213542S - 1.5P$$
$$Y_{11} = -32.4167 + .40625T + .916667D + .203125S - 1.5P$$

Curve 2 shows the comparison of cell depths predicted by these equations and the experimental results.



Curve 2

The Comparison Between the Predicted Cell

Depth And the Experimental Cell Depth

It was found from computer results that the average of squared multiple correlations (SMC) of dependent variables (cell depth) with the four dependent variables (four factors which controlled while etching) was 0.9813. This has found by dividing the sum of SMC by 11. This means that these four factors have 98.13% correlation to the cell depth. The risk to use these regression coefficients is less than 0.1% on the average, as shown in the significance column. More details of computational procedure and computer results of multivariate regression analysis are shown in Appendix B. Appendix C shows the computer results for predicted cell depth which were based upon the above regression equations and the treatment conditions used in the experiment.

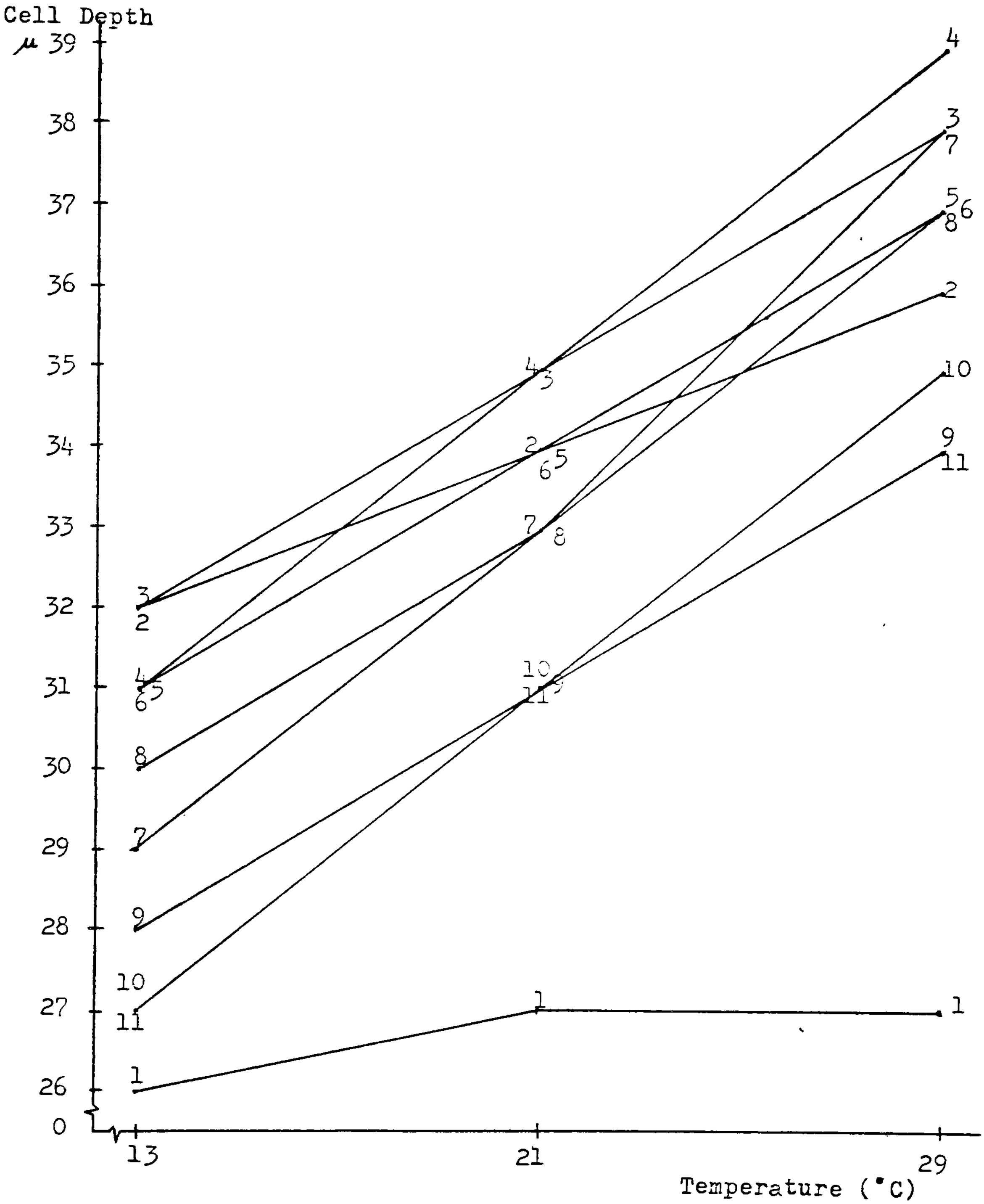
The Proofs of Hypotheses

To prove the hypotheses, four curves (Curve 3, 4, 5 & 6) of temperature, density, speed and pressure versus cell depth were plotted, by having 11 lines (representing each step of screened tone scale) for each curve. By setting the three factors at the normal or medium condition and altering the fourth factor, a predicted cell depth can be achieved by using Appendix C. The predicted cell depths for each variable factor, as the other three factors were kept constant, are shown in Table 9.

From Curve 3, 4, 5 & 6, one can see that as temperature, density and speed increase in value the cell depth increases. The rate of etching also increase because the time used in etching is kept constant. But as pressure increases in value the cell depth decreases.

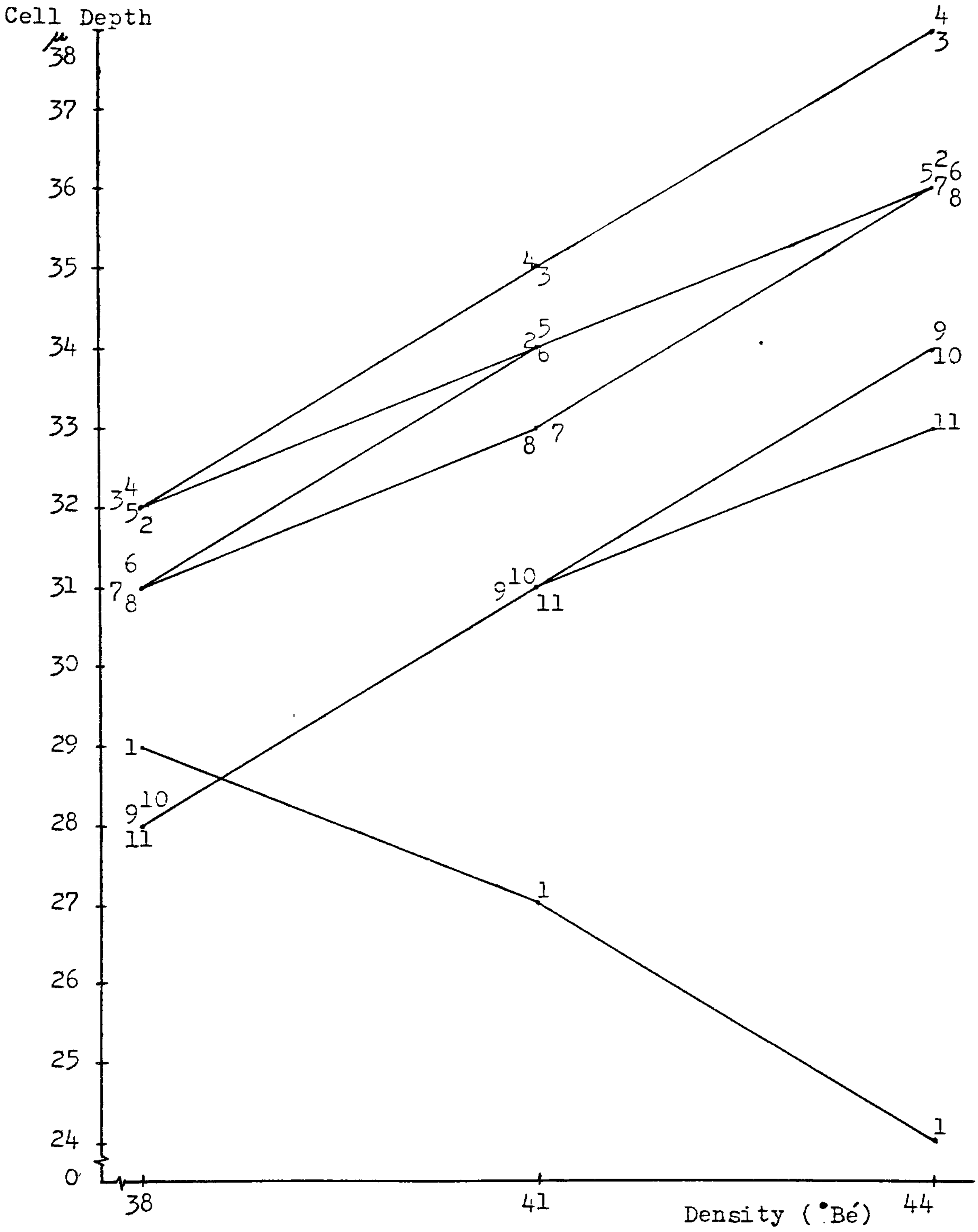
Optimum Condition

A curve of the mean value of cell depth (mean vector in Appendix A) was plotted against the original optical density of the screened tone scale, as shown in Curve 7. This curve shows uneven cell depths of cells between steps of screened tone scale. The cell depth increases in value instantly in the first three steps and gradually decreases until the end of the scale.



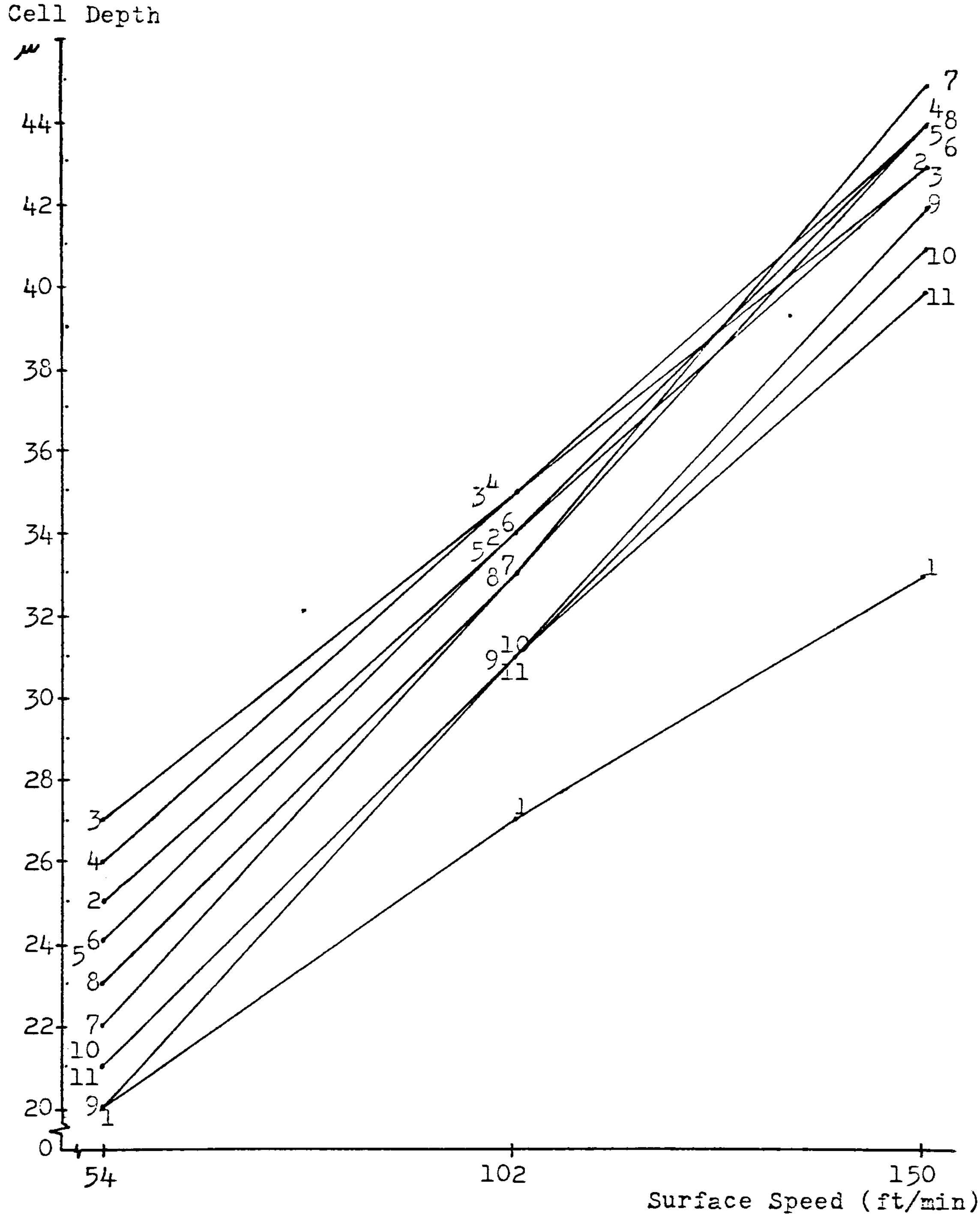
Curve 3

Temperature VS Cell Depth



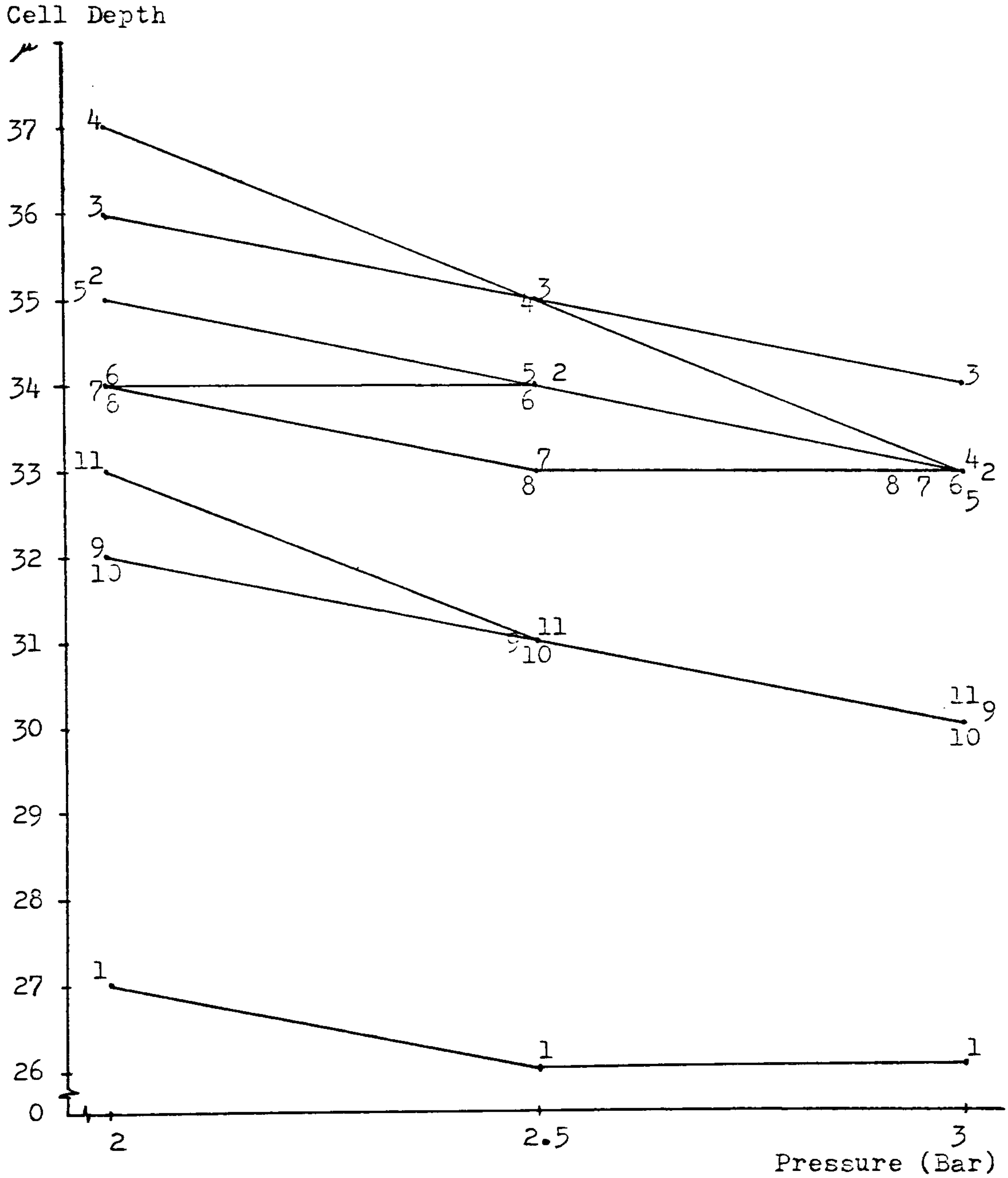
Curve 4

Density VS Cell Depth



Curve 5

Speed VS Cell Depth



Curve 6

Pressure VS Cell Depth

Table 9

The Predicted Cell Depths for Each Variable Factor
(As the Other Three Factors Are Fixed)

Temperature Variable

Temp. C	Density Be	Speed Ft/sec	Pressure Bar	Step Number 1	2	3	4	5	6	7	8	9	10	11
13	41	102	2.5	26	32	32	31	31	31	29	30	28	27	27
21	41	102	2.5	27	34	35	35	34	34	33	33	31	31	31
29	41	102	2.5	27	36	38	39	37	37	38	37	34	35	34

Density Variable

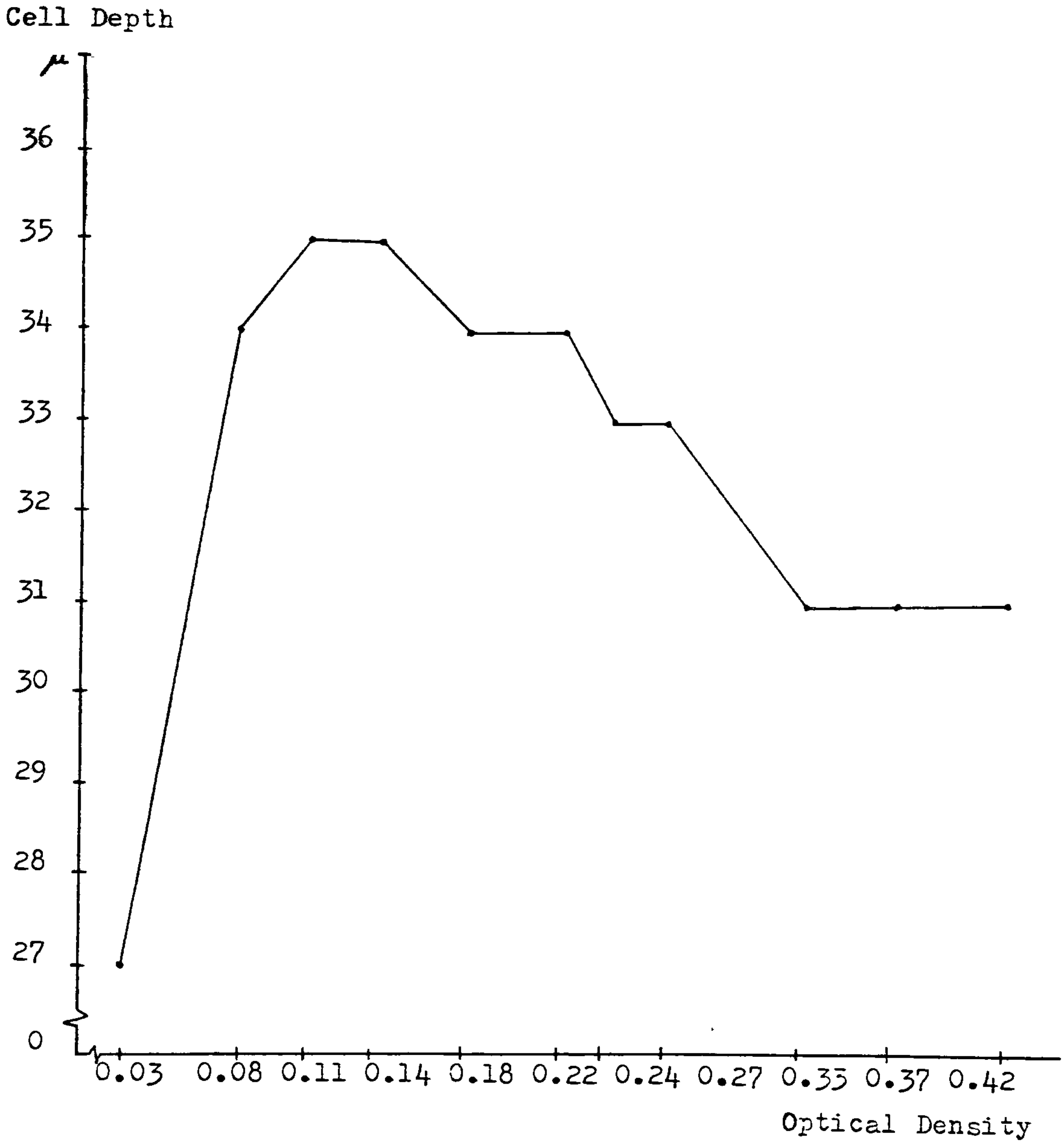
21	38	102	2.5	29	32	32	32	32	31	31	31	28	28	28
21	41	102	2.5	27	34	35	35	34	34	33	33	31	31	31
21	44	102	2.5	24	36	38	38	36	36	36	36	34	34	33

Speed Variable

21	41	54	2.5	20	25	27	26	24	24	22	23	20	21	21
21	41	102	2.5	27	34	35	35	34	34	33	33	31	31	31
21	41	150	2.5	33	43	43	44	44	44	45	44	42	41	40

Pressure Variable

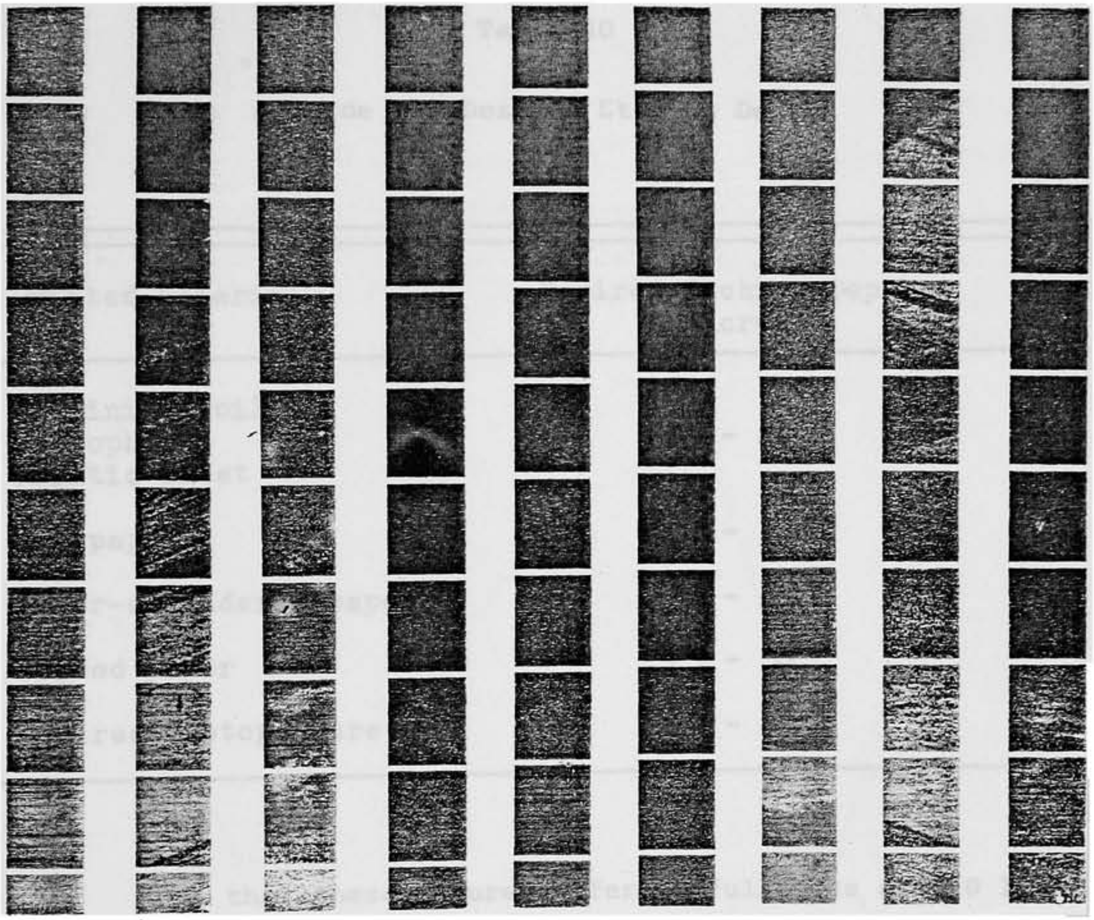
21	41	102	2.0	27	35	36	37	35	34	34	34	32	32	33
21	41	102	2.5	27	34	35	35	34	34	33	33	31	31	31
21	41	102	3.0	26	33	34	33	33	33	33	33	30	30	30



Curve 7

Average Cell Depth VS Original Optical Density

To find the optimum condition, Curve 1 and the nine proofs in Plate 1 were considered. From Curve 1, the cell depth response from test number 7 gave the most even cell depth when compared to the others. But from the reference in Table 10, the average of these cell depths in test number 7 (21 μ) is too shallow when compared to the desired etching depth of 23 to 28 μ (an average of 25 μ) for coated paper. This is revealed by the proof in Plate 1. The printed densities in the midtone area are close to the shadow and the shadow area densities are too low. Increasing the densities by the same amount at every step creates the optimum condition (in other words, to increase the cell depths). Curve 8 plots the regression coefficients of each independent factor against eleven original optical densities of screened tone scale. It shows that by increasing the value of speed gives the most even increment to cell depth in every area. The increment, which is needed to add to every cell depth from test number 7, is 4 μ (25-21 = 4). As shown in Curve 8, 1 ft./min. (.508 cm./sec.) increment of speed gives 0.2 μ increment of cell depth in almost every step except the first one. It is concluded that a 20 ft./min. (10.16 cm./sec.) in increment is needed to get 4 μ increment ($\frac{4\mu}{.2\mu}$ x 1 ft./min. or $\frac{4\mu}{.2\mu}$ x .508 cm./sec.). From conditional treatment of test number 7 (13°C temperature, 44°Be' density, 54 ft./min. (27.43 cm./sec.) speed and 3 bars pressure),



Test 1 Test 2 Test 3 Test 4 Test 5 Test 6 Test 7 Test 8 Test 9

Plate 1

Nine Proofs of Screened Tone Scales*

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*Note that only ten steps of each screened tone scales were printed.

Table 10

A Guide For Desired Etching Depth*

Printed Material	Desired Etching Depth, Micron
Aluminium foil Cellophane Plastic sheet	35 - 45
Raw paper	32 - 38
Super-calendered paper	30 - 35
Coated paper	25 - 30
Indirect rotogravure	20 - 25

Note that these figures refer to fulltones of 150 lines per inch (60 lines per mm.) screen, and are to be seen only as a guide. For half-tone work these figures also apply for the maximal tones. Half-tones should be between 2 and 5 microns less deeply etched otherwise the light shades will be too dark.

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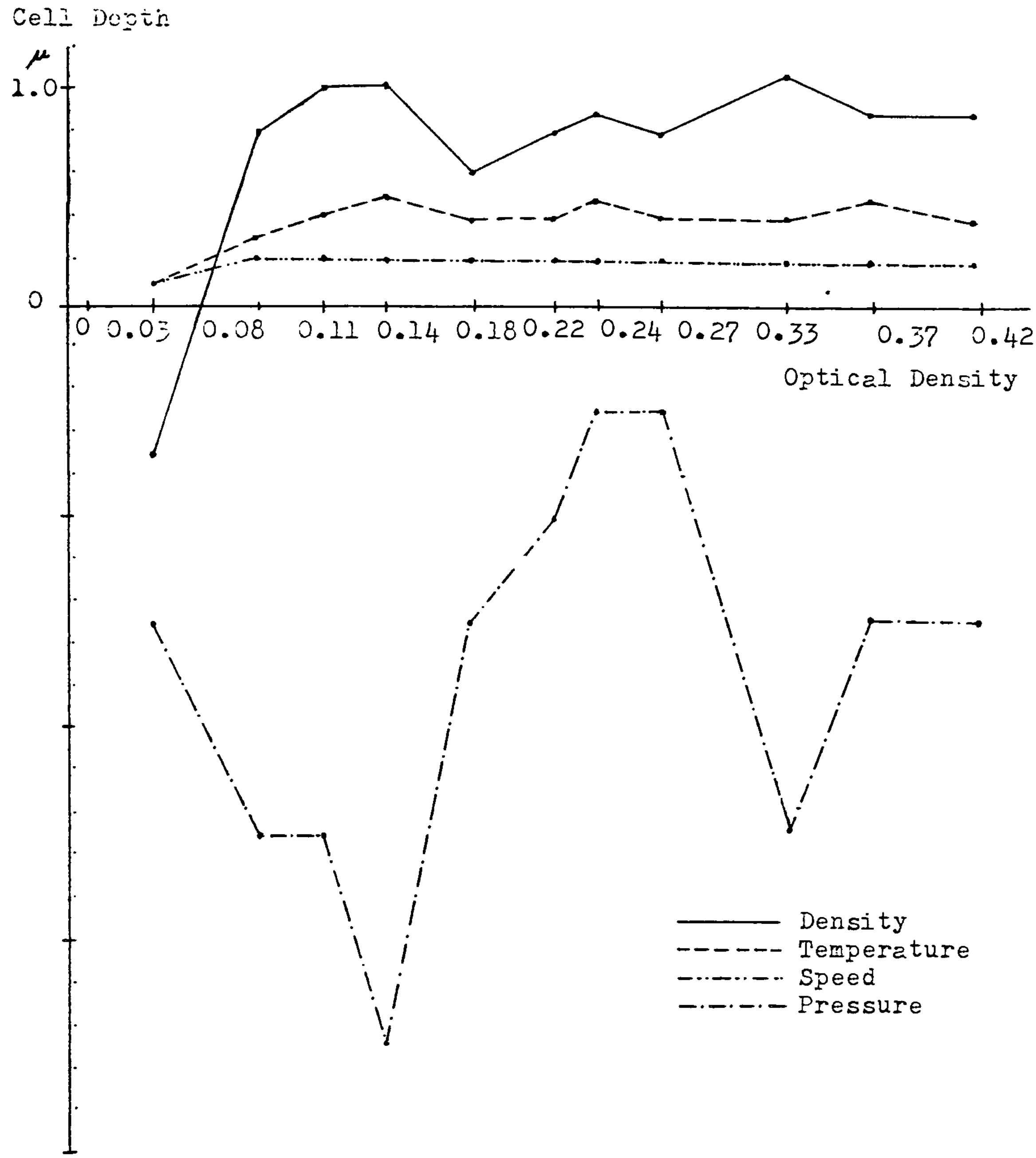
*Taken from K. Walter Etching Machine Manual, Maschinenfabrik K. Walter, Munich, West Germany.

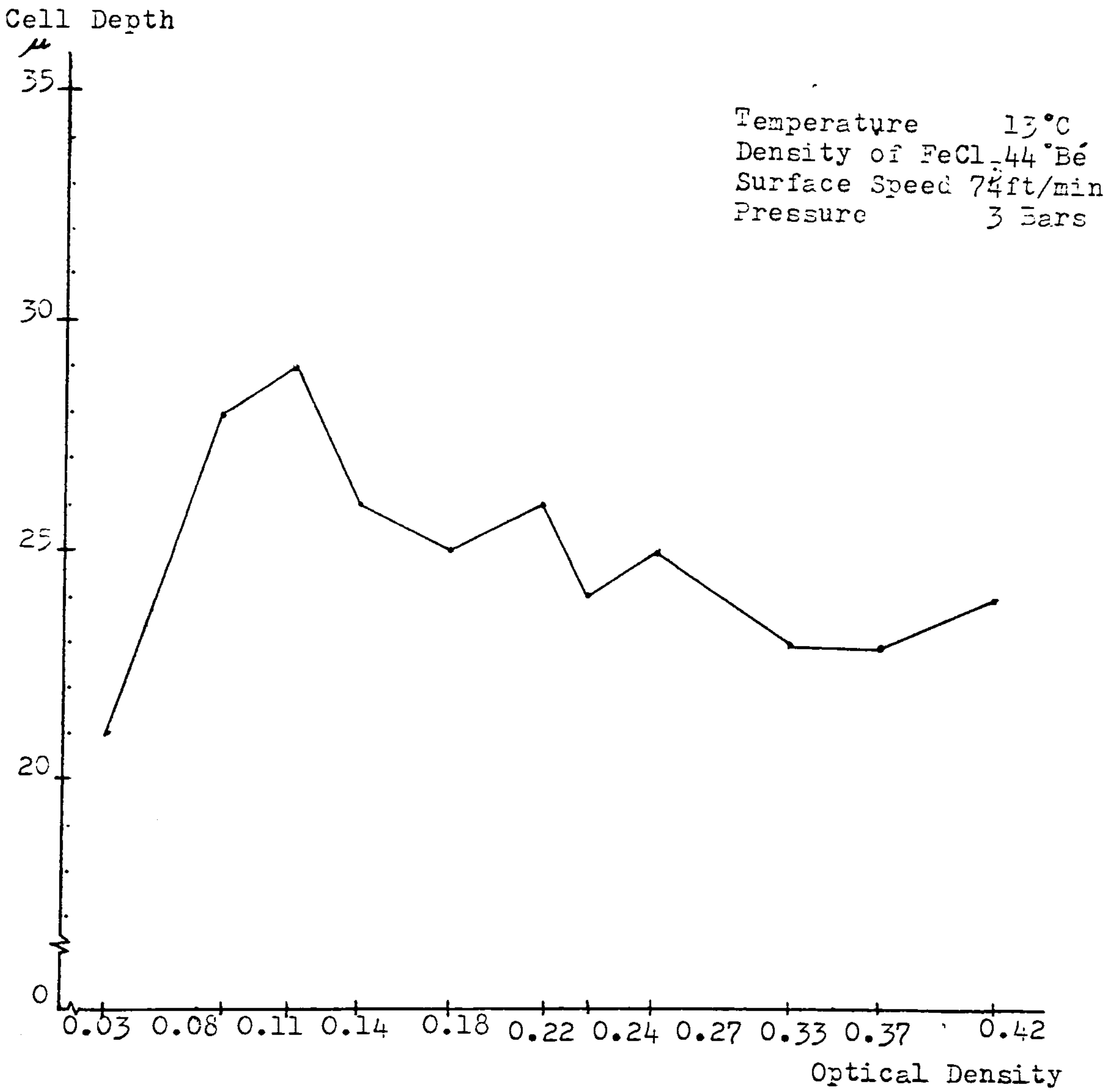


Curve 8

Regression Coefficient of Each Independent Variable

VS Original Optical Density of Tone Scale

the predicted optimum condition for the best tonal gradation of printed tone scale is found by increasing the surface speed of rotation to 74 ft./min. or 37.59 cm./sec. (54 + 20 ft./min., 27.43 + 10.16 cm./sec.). The other factors are also kept at the same conditions as test number 7. The plot of the predicted cell depths versus original optical densities of screened tone scale at the predicted optimum condition is shown in Curve 9.



Curve 9

Predicted Cell Depth VS Original Optical

Density At the Predicted Optimum Condition

Endnotes for Chapter 4

[1]J.L. Simonds, "Application of Characteristic Vector Analysis to Photographic and Optical Response Data," Journal of The Optical Society of America, Vol. 53, No. 8, August 1963, p. 968-974.

[2]James Frane, "Partial Correlation and Multivariate Regression," Biomedical Program (Los Angeles: University of California Press, 1975), p. 621-636.

Chapter 5

CONCLUSIONS AND RECOMMENDATIONS

General conclusions can't be made from this experiment because there were many physical problems which occured during the experimentation which affect the results. For the specific conclusion of this experiment, it was determined that:

1. Among the four factors with the conditions which were controlled during etching, "speed" was the factor which could explain the responses close to 97.39%, while 1.48%, 0.51% and 0.65% of the responses were explained by "temperature", "density" and "pressure" respectively.

2. The increase in value of "speed", "temperature" and "density" caused the increase in cell depth or etching rate as the etching time was constant.

3. At the same increment of the four independent variables, "pressure" was the factor which most affected cell depth or rate of etching. And "density", "temperature" and "speed" were factors which affected cell depth to the lesser degree.

4. The cell depth of any treatment condition of the four independent variables used in this experiment could be predicted

by regression equation of each known original optical density of each screened tone scale step. The risk of using these regression equations to explain the average of 98% of the experimental response was lesser than 0.01%.

5. The predicted optimum condition which would give the best tonal gradation on the tone scale (based on the observation with the proofs) was

a) 13°C (temperature of the system)

b) 44°Be' (density of $FeCl_3$)

c) 74 ft./min. (37.59 cm./sec.) (surface speed of rotation)

d) 3 Bars (pressure between the blanket roller and the gravure cylinder)

6. The increase of pressure by one unit caused the most uneven cell depth of the tone scale and the increase of one unit of speed caused the least.

In this experiment, there were many problems which occured; these are:

1. The etching machine used, which is usually used in the trade, is too large for easy control of the temperature of the system and density of $FeCl_3$. This caused a problem in running this experiment (couldn't be in random sequence), and

limited the levels of each independent variable to be tested.

2. The copper clad cylinder used is too big and too expensive to test for only one treatment, so the cylinder was divided into nine parts for nine treatments which caused the problem of staging cleaning after completion of each test. This is the reason for uncleaned surfaces before etching and resulted in the bad data of uneven and shallower cell depths in some steps of some tone scales. Also this problem of the large cylinder limited the number of tests in the experiment.

3. $FeCl_3$ used in the experiment was used once and the amount of copper dissolved in the solution was not identified. This amount of copper probably was enough to affect the response cell depth while etching.

Because of these problems, it is recommended that:

1. The etching machine and copper clad cylinder size should be small enough (and specific for experimentation) that it can be easily controlled to the required conditions.

2. More tests should be made and more levels of each independent variable should be tested. This would yield more precise regression equation of cell depth predictions. Also, there should be some tests which are run twice under the same conditions as repricates, this will give the error term in

multivariate analysis of variance table (MANOVA).

3. The sequence of experiments should be random, to make sure that all the sample data will disperse in a normal distribution curve, which represents the whole population.

4. The other variables, besides the four independent variables used in the experimentation, should be controlled accurately, especially the amount of copper dissolved in $FeCl_3$. Also, how these variables affect the etching results and the chemical reaction of $FeCl_3$ in copper etching itself should be studied more in detail.

5. Data accuracy should be improved to achieve in a more precise prediction under optimum conditions. This would yield results of greater accuracy based on the data analysis. It is believed that this would also lead to answer to the problem of deeper cells in the midtones than in the shadows.

Appendix A

The General Basis & Computational Results of Characteristic Vector Analysis Method

General Basis of the Method

The method of characteristic vector analysis determines the number and form of a set of basis curves, or characteristic vectors which, in linear combination, are capable of matching the curve-shape differences among the family of responses curves. Then each set of response data within the family can be represented simply by the scalar multiples of the characteristic vectors.*

This can be stated in mathematical equation like this:

$$
\begin{array}{lllllll}
Z_1 & = \bar{Z}_1 & + Y_1V_{1,1} & + Y_2V_{2,1} & + Y_3V_{3,1} & + \ldots Y_pV_{p,1} & \\
Z_2 & = \bar{Z}_2 & + Y_2V_{1,2} & + Y_2V_{2,2} & + Y_3V_{3,2} & + \ldots Y_pV_{p,2} & \\
\vdots & \vdots & \vdots & \vdots & \vdots & \vdots & p \leq r \\
Z_r & = \bar{Z}_r & + Y_rV_{1,r} & + Y_2V_{2,r} & + Y_3V_{3,r} & + \ldots Y_pV_{p,r} &
\end{array}
$$

Z = artificial response of each level.

$\bar{Z}$ = mean response of each level from all experimental conditions. In this case, $\bar{Z}$ is the mean of 9 responses of each step of screened tone scale.

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*J. L. Simonds, "Application of Characteristic Vector Analysis to Photographic and Optical Response Data," *Journal of The Optical Society of America*, Vol. 53, No. 8, August 1963, p. 969.

The Y's (scalar multiples) are the amounts of the characteristic vectors which must be added to the mean response vector in order to produce the sample response vector.

The characteristic vectors V are uniquely determined for a given family of response curves. The same characteristic vectors apply to all response vectors belonging to the original family from the vectors are derived.

"r" is the number of responses for each experimental condition, in this study "r" is the number of steps of screened tone scale.

"p" is the number of characteristic vectors explain all the differences among a family of response curves, each represented by r responses, will be equal to or less than r.

The Computational Results for Characteristic Vector Analysis*

From the computer results, the mean vector ($\bar{Z}$) is (start from $\bar{Z}_1$ for step number 1 to $\bar{Z}_{11}$ for step number 11 of screened tone scale)

Step no.		Mean Vector
1	Z_1	26.666667
2	Z_2	34.222222
3	Z_3	35.111111
4	Z_4	35.111111
5	Z_5	34.222222
6	Z_6	33.555556
7	Z_7	33.333333
8	Z_8	33.333333
9	Z_9	31.111111
10	Z_{10}	30.888889
11	Z_{11}	30.666667

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*Computational Procedure is shown in J. L. Simonds, "Application of Characteristic Vector Analysis to Photographic and Optical Response Data," Journal of The Optical Society of America, Vol. 53, No. 8, August 1963, p. 971-974.

The characteristic vector when consider just only one vector is ($V_{1,1}$ to $V_{1,11}$)

Step no.	Characteristic Vector 1	
1	$V_{1,1}$	15.902941
2	$V_{1,2}$	26.640582
3	$V_{1,3}$	26.410532
4	$V_{1,4}$	29.651220
5	$V_{1,5}$	29.571662
6	$V_{1,6}$	30.213150
7	$V_{1,7}$	34.820595
8	$V_{1,8}$	32.492018
9	$V_{1,9}$	33.425429
10	$V_{1,10}$	32.092413
11	$V_{1,11}$	30.270316

$V1 \rightarrow V1$ explains 97.39%

And the scalar multiples (Y_1) for nine experimental conditions are

Experimental condition number	Scalar multiple 1
1	-0.452488
2	0.080601
3	0.342930
4	0.377413
5	0.049647
6	0.305430
7	-0.387752
8	-0.142172
9	0.512249

So the mathematical equations to explain the set of response by using one characteristic vector are

$$Z_1 = \overline{Z}_1 + Y_1 V_{1,1}$$

$$Z_1 = 26.666667 + (-0.452488)(15.902941) = 19.470777$$

$$Z_2 = \overline{Z}_2 + Y_1 V_{1,2}$$

$$Z_3 = \overline{Z}_3 + Y_1 V_{1,3}$$

$$\vdots \qquad \vdots \qquad \vdots$$

$$Z_{11} = \overline{Z}_{11} + Y_1 V_{1,11}$$

Above are the equations for the set of artificial response of the first experimental condition. The others (8 experimental conditions) are almost the same as this set, except the scalar multiples (Y_1) are different in each experimental condition. The general equation can be written like this:

$$Z_r = \overline{Z}_r + Y_1 V_{1,r}$$

"r" is the number of responses from each experimental condition (11 steps of screened tone scale), and Y_1 value varies from one experimental condition to another.

In the same way the general equation for the artificial response when using 3 characteristic vectors is

$$Z_r = \overline{Z}_r + Y_1 V_{1,r} + Y_2 V_{2,r} + Y_3 V_{3,r}$$

The characteristic vector number 1 is the same as shown before.

The characteristic vector number 2 is

Step no.		Characteristic Vector 2
1	$V_{2,1}$	11.425873
2	$V_{2,2}$	0.857825
3	$V_{2,3}$	-2.161418
4	$V_{2,4}$	-1.789799
5	$V_{2,5}$	0.286544
6	$V_{2,6}$	-0.457517
7	$V_{2,7}$	-0.818144
8	$V_{2,8}$	0.126334
9	$V_{2,9}$	-0.585220
10	$V_{2,10}$	-0.580911
11	$V_{2,11}$	-1.403103

V1 → V2 explains 98.84%

The characteristic vector number 3 is

Step no.		Characteristic Vector 3
1	$V_{3,1}$	0.577331
2	$V_{3,2}$	3.230745
3	$V_{3,3}$	2.855210
4	$V_{3,4}$	2.609243
5	$V_{3,5}$	-2.267296
6	$V_{3,6}$	-0.278548
7	$V_{3,7}$	-1.562351
8	$V_{3,8}$	-2.978224
9	$V_{3,9}$	1.835979
10	$V_{3,10}$	-0.490915
11	$V_{3,11}$	-2.214568

V1 → V3 explains 99.35%

And the scalar multiples for nine experimental conditions when using 3 characteristic vectors are

Experimental condition number	Scalar Multiples		
1	-0.452488	0.228583	-0.145233
2	0.080601	0.386443	-0.316175
3	-0.342930	0.059849	-0.229552
4	0.377413	0.446753	0.056765
5	0.049647	0.192927	0.659823
6	0.305430	-0.135232	0.274071
7	-0.387752	-0.196027	-0.077291
8	-0.142172	-0.586175	0.264188
9	0.512249	-0.397121	-0.486592

Appendix B

Computational Procedure and Computer Results of Multivariate Regression Analysis

Multivariate Regression Analysis Procedure

In this case, the multivariate linear model* was used and the model is

$$\underset{(Nxp)}{\mathbf{Y}} = \underset{(Nxq)(qxp)(Nxp)}{\mathbf{XB} + \mathbf{E}_0}$$

or

$$\begin{matrix} Y_{11} & Y_{12} & \cdots & Y_{1p} \\ Y_{21} & Y_{22} & \cdots & Y_{2p} \\ \vdots & \vdots & & \vdots \\ Y_{N1} & Y_{N2} & \cdots & Y_{Np} \end{matrix} = \begin{matrix} 1 & X_{11} & X_{12} & \cdots & X_{1k} \\ 1 & X_{21} & X_{22} & \cdots & X_{2k} \\ \vdots & \vdots & & & \vdots \\ 1 & X_{N1} & X_{N2} & \cdots & X_{Nk} \end{matrix} \begin{matrix} B_{01} & B_{02} & \cdots & B_{0p} \\ B_{11} & B_{12} & \cdots & B_{1p} \\ \vdots & \vdots & & \vdots \\ B_{k1} & B_{k2} & \cdots & B_{kp} \end{matrix} + \begin{matrix} E_{11} & E_{12} & \cdots & E_{1p} \\ E_{21} & E_{22} & \cdots & E_{2p} \\ \vdots & \vdots & & \vdots \\ E_{N1} & E_{N2} & \cdots & E_{Np} \end{matrix}$$

Y = Nxp data matrix of N independent observations or p responses (this case N = 9, p = 11)

X = Nxq design matrix of fixed known independent variables ($q = k+1$, $N > q$, this case $k = 4$)

B = qxp matrix of parameters to be estimated (regression coefficient for predicting dependent variables from independent variables)

$\mathbf{E}_0$ = matrix of random errors.

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*Neil H. Timm, Multivariate Analysis with Application in Education and Psychology (California: Brooks/Cole Publishing Company, 1975), p. 307.

where $E(\mathbf{Y}) = \mathbf{XB}$

$E(\mathbf{Y})$ = the expected value of the random Nxp matrix $\mathbf{Y}$

Each expected dependent variable can be written in univariate model like this:

$$y_{ij} = b_{0j} + b_{1j}x_{ij} + b_{2j}x_{i2} + \ldots. b_{kj}x_{ik}$$

y_{ij} = the ij^{th} element of the matrix $\mathbf{Y}$

To solve for $\mathbf{B}$,

$$\mathbf{B} = [\mathbf{X'X}]^{-1} \mathbf{X'Y}$$

$\mathbf{X'}$ = the transpose of $\mathbf{X}$ matrix, obtained by interchanging the rows and columns of $\mathbf{X}$ matrix

$[\mathbf{X'X}]^{-1}$ = the inverse of $\mathbf{X'X}$ square metrix (a matrix of order (nxn) such that pre- or postmultiplication of $\mathbf{X'X}$ by $[\mathbf{X'X}]^{-1}$ yields the identity matrix

Identity matrix is a square matrix whose diagonal elements are all 1s and off-diagonal elements are all 0s.

Summary MANOVA* Table

Source	Sum of square	Degree of freedom	Mean square
Crude	$\mathbf{Y'Y}$	N	
Due to b_0	$(\Sigma \mathbf{Y})^2/N$	1	$(\Sigma \mathbf{Y})^2/N$
Due to regression	$\mathbf{Y'Y}-(\Sigma \mathbf{Y})^2/N$	k	$\mathbf{Y'Y}-(\Sigma \mathbf{Y})^2/Nk$
Residual	$\mathbf{Y'Y}-\mathbf{B'}\ \mathbf{X'XB}$	N-k-1	$\mathbf{Y'Y}-\mathbf{B'X'XB}/(N-k-1)$

From the summary MANOVA table, F-statistic is used to tested the significance of multiple regression coefficients against residual mean square, and the hypothesis are:

$$H_0 : \mathbf{B} = 0$$

$$H_1 : \mathbf{B} \neq 0$$

*Multivariate Analysis Of Variance

Also from the F-statistic value, significant level ($\hat{\alpha}$) is found by Critical Values of the F Distribution - Table, this $\hat{\alpha}$ tells how much risk it is to use the regression coefficients.

For squared multiple correlation, it is calculated by

$$SMC = \frac{SSregression - SSb_0}{Y^2 - (\Sigma Y)^2/N}$$

$$= \frac{B'X'XB - (\Sigma Y)^2/N}{Y'Y - (\Sigma Y)^2/N}$$

which tells that how much each predicted dependent variable correlates with the dependent variables.

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BMDP6R - PARTIAL CORRELATION AND MULTIVARIATE REGRESSION        PROGRAM REVISED FEBRUARY 1976
HEALTH SCIENCES COMPUTING FACILITY                              MANUAL DATE -- 1975
UNIVERSITY OF CALIFORNIA, LOS ANGELES

PROGRAM CONTROL INFORMATION

        PROBLEM     TITLE IS 'THESIS NO.1'./
        INPUT       VARIABLES ARE 16.
                    CASES ARE 9.
                    FORMAT IS '(A2,2F3.0,2F4.0,11F3.0)'./
        VARIABLE    LABEL IS EXP.
                    NAMES ARE EXP,TEMP,DENSITY,SPEED,PRESSURE,STEP1,STEP2,STEP3,
                              STEP4,STEP5,STEP6,STEP7,STEP8,STEP9,STEP10,STEP11./
        PRINT       MATRICES ARE CORR,RESI,COEF./
        REGRESSION  DEPENDENT = STEP1,STEP2,STEP3,STEP4,STEP5,STEP6,STEP7,STEP8,
                              STEP9,STEP10,STEP11.
                    INDEPENDENT = TEMP,DENSITY,SPEED,PRESSURE.
                    TOLERANCE = .01.
                    PRECISION = DOUBLE./
        END/

PROBLEM TITLE . . . . . . . .THESIS NO.1

NUMBER OF VARIABLES TO READ IN. . . . . . . . . .        16
NUMBER OF VARIABLES ADDED BY TRANSFORMATIONS. . .         0
TOTAL NUMBER OF VARIABLES . . . . . . . . . . . .        16
NUMBER OF CASES TO READ IN. . . . . . . . . . . .         9
CASE LABELING VARIABLES . . . . . . . . . . . . .       EXP
LIMITS AND MISSING VALUE CHECKED BEFORE TRANSFORMATIONS
BLANKS ARE. . . . . . . . . . . . . . . . . . . .     ZEROS
INPUT UNIT NUMBER . . . . . . . . . . . . . . . .       105
REWIND INPUT UNIT PRIOR TO READING. . DATA. . . .        NO

INPUT FORMAT
        (A2,2F3.0,2F4.0,11F3.0)

VARIABLES TO BE USED
             2  TEMP           3  DENSITY        4  SPEED           5  PRESSURE       6  STEP1
             7  STEP2          8  STEP3          9  STEP4          10  STEP5         11  STEP6
            12  STEP7         13  STEP8         14  STEP9          15  STEP10        16  STEP11

INDEPENDENT VARIABLES ARE
             2  TEMP           3  DENSITY        4  SPEED           5  PRESSURE

DEPENDENT VARIABLES ARE
             6  STEP1          7  STEP2          8  STEP3           9  STEP4         10  STEP5
            11  STEP6         12  STEP7         13  STEP8          14  STEP9         15  STEP10
            16  STEP11

NUMBER OF INDEPENDENT VARIABLES . . . . . . . . .         4
NUMBER OF DEPENDENT VARIABLES . . . . . . . . . .        11
TOTAL NUMBER OF VARIABLES USED. . . . . . . . . .        15
WEIGHT VARIABLE . . . . . . . . . . . . . . . . .
PRECISION . . . . . . . . . . . . . . . . . . . .    DOUBLE
TOLERANCE FOR MATRIX INVERSION. . . . . . . . . .  .0100000

APPROX. NUMBER OF VARIABLES WHICH CAN BE ANALYZED    118
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CORRELATIONS

		TEMP 2	DENSITY 3	SPEED 4	PRESSURE 5	STEP1 6	STEP2 7	STEP3 8	STEP4 9	STEP5 10	STEP6 11	STEP7 12	STEP8 13	STEP9 14
TEMP	2	1.000												
DENSITY	3	.000	1.000											
SPEED	4	.000	-.000	1.000										
PRESSURE	5	-.000	.000	.000	1.000									
STEP1	6	.108	-.325	.902	-.108	1.000								
STEP2	7	.237	.337	.920	-.131	.825	1.000							
STEP3	8	.144	.291	.574	-.132	.759	.908	1.000						
STEP4	9	.354	.260	.874	-.165	.772	.985	.997	1.000					
STEP5	10	.309	.167	.728	-.071	.822	.975	.975	.982	1.000				
STEP6	11	.279	.232	.929	-.046	.796	.983	.985	.979	.987	1.000			
STEP7	12	.344	.222	.910	-.020	.793	.979	.983	.980	.990	.993	1.000		
STEP8	13	.281	.194	.929	-.022	.804	.973	.973	.978	.996	.985	.998	1.000	
STEP9	14	.274	.274	.905	-.105	.798	.993	.986	.987	.993	.982	.986	.981	1.000
STEP10	15	.330	.242	.902	-.066	.798	.983	.983	.988	.990	.985	.994	.986	.996
STEP11	16	.302	.255	.985	-.070	.777	.975	.975	.979	.992	.982	.991	.991	.990

		STEP10 15	STEP11 16
STEP10	15	1.000	
STEP11	16	.996	1.000

SQUARED MULTIPLE CORRELATIONS OF EACH INDEPENDENT VARIABLE WITH ALL OTHER INDEPENDENT VARIABLES
(MEASURES OF MULTI-COLINEARITY OF PREDICTOR VARIABLES)
AND TESTS OF SIGNIFICANCE OF MULTIPLE REGRESSION
DEGREES OF FREEDOM FOR F-STATISTICS ARE 3 AND 5

VARIABLE NO.	NAME	SMC	F-STATISTIC	SIGNIFICANCE (F LESS THAN)
2	TEMP	.00000	.00	1.00000
3	DENSITY	.00000	.00	1.00000
4	SPEED	.00000	.00	1.00000
5	PRESSURE	.00000	.00	1.00000

SQUARED MULTIPLE CORRELATIONS(SMC) OF EACH DEPENDENT VARIABLE WITH THE INDEPENDENT VARIABLES
AND TESTS OF SIGNIFICANCE OF MULTIPLE REGRESSION
DEGREES OF FREEDOM FOR F-STATISTICS ARE 4 AND 4

VARIABLE NO.	NAME	SMC	F-STATISTIC	SIGNIFICANCE (F LESS THAN)
6	STEP1	.94271	16.45	.00947
7	STEP2	.97574	40.22	.00178
8	STEP3	.98473	64.47	.00069
9	STEP4	.98340	59.24	.00082
10	STEP5	.98919	91.46	.00035
11	STEP6	.99764	415.00	.00002
12	STEP7	.99510	203.08	.00007
13	STEP8	.97948	47.73	.00125
14	STEP9	.97972	48.32	.00122
15	STEP10	.98559	68.37	.00062
16	STEP11	.98060	50.56	.00111

REGRESSION COEFFICIENTS FOR PREDICTING DEPENDENT VARIABLES (COLUMNS) FROM INDEPENDENT VARIABLES (ROWS)

		STEP1 6	STEP2 7	STEP3 8	STEP4 9	STEP5 10	STEP6 11	STEP7 12	STEP8 13
TEMP	2	.937500E-01	.281250E 00	.406250E 00	.468750E 00	.406250E 00	.375000E 00	.531250E 00	.406250E 00
DENSITY	3	-.750000E 00	.750000E 00	.916667E 00	.916667E 00	.583333E 00	-.833333E 00	.916667E 00	.750000E 00
SPEED	4	.130208E 00	.182292E 00	.171875E 00	.192708E 00	.203125E 00	.208333E 00	.234375E 00	.223958E 00
PRESSURE	5	-.150000E 01	-.250000E 01	-.250000E 01	-.350000E 01	-.150000E 01	-.100000E 01	-.500000E 00	-.500000E 00

		STEP9 14	STEP10 15	STEP11 16
TEMP	2	.406250E 00	.468750E 00	.406250E 00
DENSITY	3	.108333E 01	.916667E 00	.916667E 00
SPEED	4	.223958E 00	.213542E 00	.203125E 00
PRESSURE	5	-.250000E 01	-.150000E 01	-.150000E 01

	STEP1 6	STEP2 7	STEP3 8	STEP4 9	STEP5 10	STEP6 11	STEP7 12	STEP8 13
INTERCEPT	.459167E 02	-.147778E 02	-.222847E 02	-.232222E 02	-.151944E 02	-.272361E 02	-.380625E 02	-.275417E 02

	STEP9 14	STEP10 15	STEP11 16
INTERCEPT	-.380306E 02	-.345694E 02	-.324161E 02

STANDARD ERRORS FOR REGRESSION COEFFICIENTS

		STEP1 6	STEP2 7	STEP3 8	STEP4 9	STEP5 10	STEP6 11	STEP7 12	STEP8 13
TEMP	2	.104E 00	.926E-01	.729E-01	.853E-01	.683E-01	.329E-01	.501E-01	.104E 00
DENSITY	3	.276E 00	.287E 00	.199E 00	.227E 00	.182E 00	.818E-01	.184E 00	.276E 00
SPEED	4	.173E-01	.154E-01	.122E-01	.142E-01	.114E-01	.549E-02	.902E-02	.173E-01
PRESSURE	5	.166E 01	.148E 01	.117E 01	.136E 01	.109E 01	.521E 00	.866E 00	.166E 01

		STEP9 14	STEP10 15	STEP11 16
TEMP	2	.106E 00	.853E-01	.937E-01
DENSITY	3	.282E 00	.227E 00	.250E 00
SPEED	4	.116E-01	.142E-01	.156E-01
PRESSURE	5	.169E 01	.136E 01	.150E 01

	STEP1 6	STEP2 7	STEP3 8	STEP4 9	STEP5 10	STEP6 11	STEP7 12	STEP8 13
INTERCEPT	.129E 02	.111E 02	.873E 01	.102E 02	.818E 01	.394E 01	.648E 01	.129E 02

	STEP9 14	STEP10 15	STEP11 16
INTERCEPT	.127E 02	.102E 02	.112E 02

NAMES FOR RESIDUALS ARE CREATED BY THE PROGRAM USING R FOLLOWED BY THE FIRST SEVEN CHARACTERS OF THE CORRESPONDING VARIABLE NAME.

RESIDUALS (CASE NUMBERS REFER TO DATA BEFORE DELETION OF CASES)

CASE LABEL	NO.	WEIGHT	RSTEP1 / RSTEP11 (17 / 27)	RSTEP2 (18)	RSTEP3 (19)	RSTEP4 (20)	RSTEP5 (21)	RSTEP6 (22)	RSTEP7 (23)	RSTEP8 (24)	RSTEP9 (25)	RSTEP10 (26)
1	1	1.000	-.6667 / 2.3333	-.2222	-.1111	.8889	1.7778	-.5556	.6667	2.6667	.8889	1.1111
2	2	1.000	-1.6667 / -1.6667	-1.2222	-.1111	-.1111	-.2222	.4444	-1.3333	-.3333	-2.1111	-1.8889
3	3	1.000	-.6667 / -.6667	-.2222	-.1111	-1.1111	-1.2222	.4444	.6667	-1.3333	-1.1111	-.8889
4	4	1.000	1.3333 / .3333	-.2222	-1.1111	-1.1111	-.2222	-.5556	-.3333	-1.3333	.8889	1.1111
5	5	1.000	3.3333 / -.6667	3.7778	2.8889	2.8889	-.2222	.4444	.6667	.6667	2.8889	1.1111
6	6	1.000	-.6667 / -.6667	-.2222	-.1111	-1.1111	-1.2222	.4444	.6667	-1.3333	-1.1111	-.8889
7	7	1.000	1.3333 / .3333	-.2222	-1.1111	-1.1111	-.2222	-.5556	-.3333	-1.3333	.8889	1.1111
8	8	1.000	-1.6667 / -1.6667	-1.2222	-.1111	-.1111	-.2222	.4444	-1.3333	-.3333	-2.1111	-1.8889
9	9	1.000	-.6667 / 2.3333	-.2222	-.1111	.8889	1.7778	-.5556	.6667	2.6667	.8889	1.1111

NUMERICAL CONSISTENCY CHECK

RESIDUAL MEAN SQUARES ARE COMPUTED FROM BOTH COVARIANCE MATRIX AND RESIDUALS, AND RELATIVE DIFFERENCE (DIFFERENCE DIVIDED BY SMALLER OF TWO ESTIMATES) IS COMPUTED.

RESIDUAL MEAN SQUARES COMPUTED FROM

	COVARIANCE MATRIX	RESIDUALS	RELATIVE DIFFERENCE
RSTEP1	.550000E 01	.550000E 01	-.808461E-15
RSTEP2	.438889E 01	.438889E 01	-.242804E-14
RSTEP3	.272222E 01	.272222E 01	.261016E-14
RSTEP4	.372222E 01	.372222E 01	.900772E-14
RSTEP5	.238889E 01	.238889E 01	-.390385E-14
RSTEP6	.555556E 00	.555556E 00	-.266787E-13
RSTEP7	.150000E 01	.150000E 01	-.187998E-13
RSTEP8	.550000E 01	.550000E 01	-.122730E-13
RSTEP9	.572222E 01	.572222E 01	-.125725E-13
RSTEP10	.372222E 01	.372222E 01	-.775499E-15
RSTEP11	.450000E 01	.450000E 01	.908065E-15

PROBLEM NUMBER 1 COMPLETED.

BMDP6R - PARTIAL CORRELATION AND MULTIVARIATE REGRESSION
HEALTH SCIENCES COMPUTING FACILITY
UNIVERSITY OF CALIFORNIA, LOS ANGELES

PROGRAM REVISED FEBRUARY 1976
MANUAL DATE -- 1975

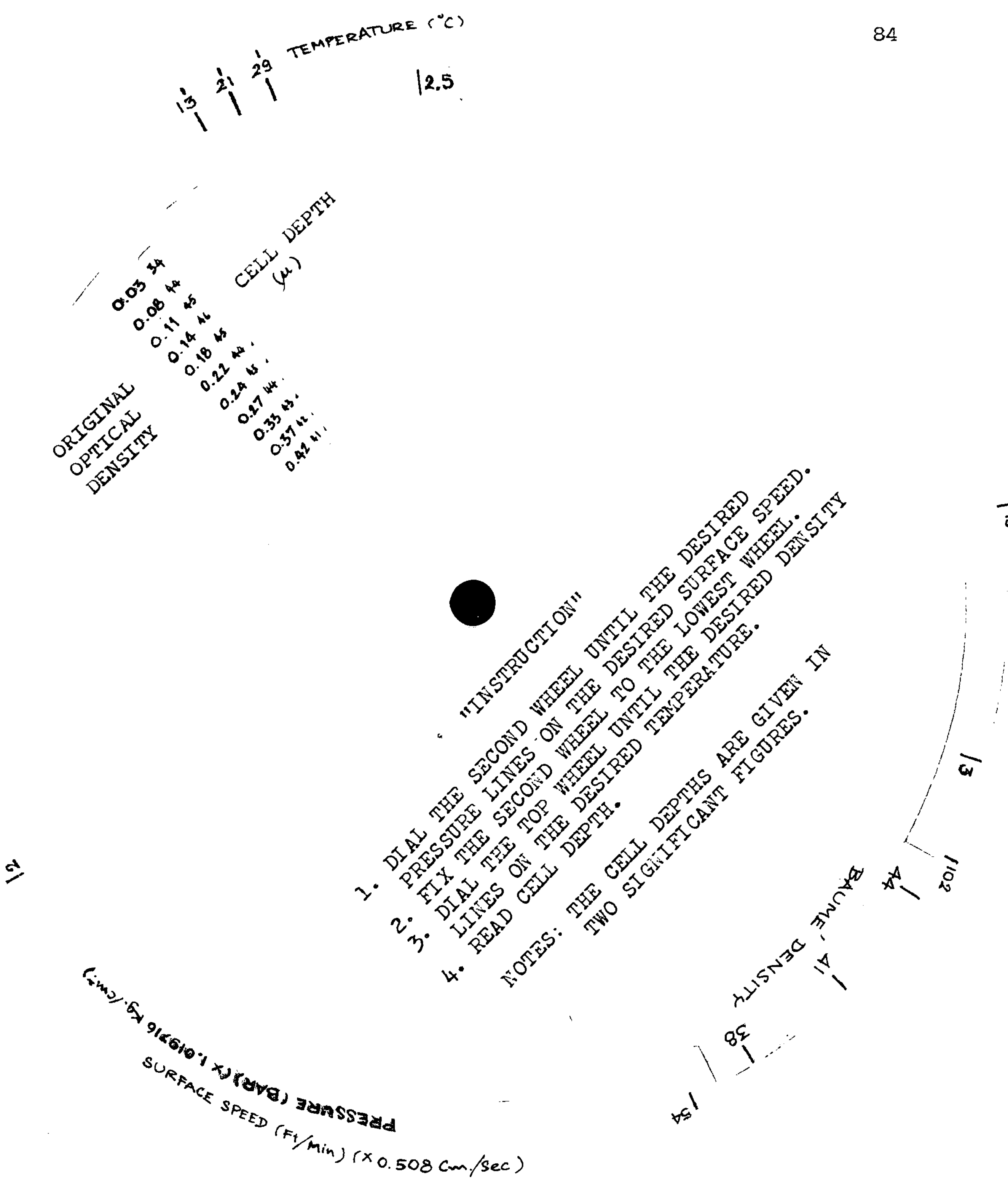
PROGRAM CONTROL INFORMATION

FINISH/

PROGRAM TERMINATED NORMALLY.
STOP 0

Appendix C

A Computer for Predicting Cell Depth, Concerning Only the Conditions Used in the Experiment


TEMPERATURE (°C)
13
21
29
2.5
CELL DEPTH (μ)
ORIGINAL OPTICAL DENSITY
0.03 34
0.08 44
0.11 45
0.14 46
0.18 45
0.22 44
0.24 45
0.27 44
0.33 43
0.37 42
0.42 41
"INSTRUCTION"
1. DIAL THE SECOND WHEEL UNTIL THE DESIRED PRESSURE LINES ON THE DESIRED SURFACE SPEED.
2. FIX THE SECOND WHEEL TO THE LOWEST WHEEL.
3. DIAL THE TOP WHEEL UNTIL THE DESIRED DENSITY LINES ON THE DESIRED TEMPERATURE.
4. READ CELL DEPTH.
NOTES: THE CELL DEPTHS ARE GIVEN IN TWO SIGNIFICANT FIGURES.
BAUME DENSITY
38
41
44
102
154
13
12
PRESSURE (BAR) (x 1.019716 Kg./cm²)
SURFACE SPEED (Ft/Min) (x 0.508 Cm./Sec)

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