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THE REGRESSION ANALYSIS OF ORE TREATMENT TEST RESULTS

PART 2: THE DEVELOPMENT AND ASSESSMENT OF SECOND-ORDER REGRESSION EQUATIONS

by

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EXTRACTION METALLURGY DIVISION

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PART 2: The Development and Assessment of Second-Order Regression Equations.

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SUMMARY

This report presents examples of the output provided by a computer program recently developed by the staff of the Extraction Metallurgy Division for conducting regression analyses of test data to produce empirical models relating, quantitatively, the levels of the significant variables and the values of important test responses. The meaning of this output information and how it may be used to assess the value of the calculated models are discussed. Because the methods used and the discussion are applicable to any set of test data, this report may be used as a guide in interpreting any regression analyses resulting from the Extraction Metallurgy Division's stepwise-regression computer program.

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INTRODUCTION

It is well known that the amount of information that can be obtained from a series of experiments can often be greatly increased and be more valuable if the results are analysed by regression techniques. This is particularly true if the experiments have been designed with subsequent regression analysis in mind [1-5].

The purpose of conducting a regression analysis of experimental data is to find an equation which by relating the test variables to the test response makes possible the prediction of the response that may be expected from a given set of test conditions. The successful development of such an equation can provide the investigator with a broad, quantitative understanding of the process under study.

The two chief drawbacks to the general use of the methods of regression analysis are the complexity of the calculations and a lack of understanding among experimentalists of the statistical concepts involved. Within the past year, the staff of the Extraction Metallurgy Division has developed a computer program for doing the calculations required in conducting a regression analysis of experimental data. This program is now available for general use; indeed, it has already been used to a considerable extent. A previous report (Part 1) described how the output from this program can be used on a relatively simple experimental design--involving a few tests--to show the relative importance of the independent variables involved and how to assess the first-order models derived from the data. The present report, Part 2, deals with the assessment of the results of the regression analysis of a more complex experimental design that permits the development of second-order models.

PROCEDURE

The statistical analyses described in this report were based on data obtained from a laboratory investigation that was conducted in the Extraction Metallurgy Division to study the acid leaching of an Elliot Lake uranium ore. In that investigation four independent variables were studied: initial acid addition, temperature, initial oxidizer addition, and particle size of the ore. Each variable was controlled at least once to one of five different levels, as shown in the four columns headed "Independent Variables" in Table 1. The variables and variable levels chosen for the present work were based on an earlier study, the results of which were analysed statistically to show the significant variables involved.[7]

Table 1 shows that the levels chosen for the independent variables in the design of the complete experiment were balanced around an average or central level. Because of this the experimental design used in the experimental work is called a central composite design. Other experimental designs that would allow the development of second-order models could have been used, and these can be found in standard statistical tests (1-5).

The complete acid leaching test program consisted of thirty-two tests run in random order. Twenty-four tests (Tests 1 to 8 and 12 to 27) were required to complete the experimental design. Eight replicate tests (Tests 9 to 11 and 28 to 32) were made to provide an independent estimate of the experimental error involved. The experimental error includes all errors attributable to physical measurements, experimental techniques and unknown random variables. Four system responses (first-hour U_3O_8 extraction, 48-hour U_3O_8 extraction, acid consumption, and the final electromotive-force value of the leaching solution) were measured during each test.

The design used in the experimental work provided sufficient data for a full second-order relationship between the four variables and the measured responses to be calculated. Assuming that all the variables are significant and that the full second-order equation is needed to express the relationship between the four independent variables and the response under consideration, the resulting statistical model would have the following form:

$$\begin{aligned} \text{Response} = B_0 &+ B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 + B_5X_1X_2 \\ &+ B_6X_1X_3 + B_7X_1X_4 + B_8X_2X_3 + B_9X_2X_4 \\ &+ B_{10}X_3X_4 + B_{11}X_1^2 + B_{12}X_2^2 + B_{13}X_3^2 + B_{14}X_4^2 \\ &+ \text{Error} \end{aligned}$$

In this model, B_0 is a constant term, B_1 to B_{14} are the regression coefficients or parameters to be determined, and X_1 to X_4 are the independent variables.

The specific values for the coefficients shown in the above general model for each of the four responses studied were calculated on the Department's CDC-3100 computer, using the stepwise multivariable regression program DRMEML. This program was developed by the staff of the Extraction Metallurgy Division. A FORTRAN listing of DRMEML is available and with minor modifications it can also be used on either UNIVAC-1108 or IBM/360 system computers.

RESULTS OF REGRESSION ANALYSIS AND DISCUSSION

The levels used for the four independent variables investigated in this work are shown in Table 1, in the columns headed X_1, X_2, X_3 and X_4. The measured and predicted values for each of the four responses observed are also given in this table. The predicted values are those obtained by inserting the tabulated values of the independent variables into the regression equations finally developed and shown in Tables 2 to 5.

The results shown in Tables 2 to 5 were obtained by first calculating first-order or linear models. Where it was shown that the first-order models did not fit the data, second-order models were developed. Further assessment of the model's value was obtained from a study of (a) the sources of over-all variation in response, (b) the percent of variation in response due to each significant term, and (c) the standard error of the estimate of the response mean due to the model's lack of precision. All of these statistics are calculated simultaneously with the derivation of the regression model and are produced by the computer program used.

Table 2 gives the results of the regression analysis, the relevant response being the extraction of uranium at the end of the first hour of leaching. The empirical model shown (Table 2 (a)) is a second-order model containing terms X_1^2 and X_3^2 and the cross product term X_2X_3, along with the first-order term in X_3. The second-order model is shown because the first-order one, which was calculated first, did not fit the observed data. Actually, the second-order model shown in Table 2 is not as good a fit to the data as we would like; but more complex models could not be calculated with the experimental design used.

The lack-of-fit test used here is based on the deviations between the observed values of the response (first hour's extraction in Table 1) and those values of the response that can be predicted by the model (Table 2). These deviations have two causes: the inadequacy with which the model fits the data, and the inherent experimental error. The lack-of-fit test compares the observed variances associated with these two causes of deviation. If there is no significant difference between these two variances, we accept the model as fitting the test data to within the limits of experimental error.

With this introduction, the statistical meaning of the lack-of-fit variance test in Table 2(d) becomes clear. For the model in Table 2(a) to be an acceptable fit to the test data, the ratio of lack-of-fit variance to the experimental error variance must <u>not</u> be statistically significant. With the results in Table 2(d), this ratio is significant at the 95 per cent confidence level with a numerical value of 3.48*. However, it would have been insignificant if the value had been 3.44*. Consequently, the data, although significant at a

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* See any table of variance ratios or F-test (see Reference 8).

95 per cent confidence level, are not significant at a 94 per cent level. With these data, then, we could with reservations accept the model in 2(a) as being an acceptable fit, since the relevant variance test is very close to being not significant.

The other variance test shown in 2 (d), the regression variance over the residual variance, must be significant if the model is to be useful for predictive purposes. The regression variance is the fraction of the total variance in the response (first hour's extraction) that is accounted for by the terms in the model in 2 (a). The residual variance is the difference between the total variance and the regression variance. With these definitions in mind it will be apparent that, since a good model should account for a very large proportion of the total response variance, the ratio of regression variance to residual variance should be not only significant but also, as has been stated by Draper [4], at least four times the tabulated value given in the Table of variance ratios[8]. In the example in Table 2 (d), the numerical value of the variance ratio is 16.26, which is highly significant at the 95 per cent confidence level since this value is almost six times the lowest value(2.73, from Table of Variance Ratios) needed for statistical significance.

Section (b) of Table 2 shows the experimental error observed in this test work, and also the error that may be expected between a response estimated from the equation for specific conditions and the observed value of the response in a test run under the same conditions. On the basis of the statistical theory of normal distribution [3], the results in Table 2 (b) show that the experimental error is such that, if the true extraction for the given set of variables is 83.7 per cent (the response mean shown in 2 (a)), about 68 per cent of the responses obtained in a series of replicate tests would fall within the range of 83.7 ± 1.33 per cent, while about 95 per cent of the responses would be in the range of 83.7 ± 2.73 per cent. Also, if a set of conditions were chosen* so that when these conditions were substituted in the model an extraction of 83.7 per cent was predicted, the observed responses from a series of replicate tests run under these conditions would be in the range of 83.7 ± 2.24 per cent 68 per cent of the time and 83.7 ± 4.60 per cent 95 per cent of the time.

Sections (c) and (e) of Table 2 are generally self-explanatory. Section (e) shows that, while 70.6 per cent of the variation observed in the response can be explained by the significant independent variables given in the model, 26.6 per cent of the variation is unexplained. This unexplained variation is due to the size of the experimental error, which in turn can be due to operator error, or to the effect of an important variable which was not controlled in the test work because its importance was not recognized. Because

* No regression model should be used to predict a response by substituting values of the independent variables outside of the ranges used in the test work.

of the size of the unexplained variation, careful consideration should be given to the possibility that a significant variable has been overlooked. Section 2 (c) shows the relative importance of the significant terms on the basis of how much of the total regression variation (70.6 per cent) is attributable to each significant term.

To sum up the results in Table 2, the best model we can derive from the test data is:

$$\text{First-hour } U_3O_8 \text{ extraction} = 59.35 + 15.6\,X_3 + 0.11\,X_2X_3 + 0.0014\,X_1^2 - 6.06\,X_3^2,$$

where X_1 is acid addition (lb/ton), X_2 is temperature (°C), and X_3 is the oxidizer addition (lb/ton). A study of this model shows that the initial uranium extraction rate is influenced mainly by the amount of oxidizer added (X_3), since this variable appears in three of the five terms in the model. The model also shows that, because of the negative term associated with the X_3^2 term, the rate of increase in the uranium extraction rate decreases with increased oxidizer addition. Increases in the acid addition (X_1) and in the temperature (X_2) result in minor (as compared to the effect of oxidizer) but significant increases in the extraction rate. The grind (X_4) had no significant effect on the initial extraction rate, since it did not appear in any of the terms of the model. The model, therefore, has given us considerable insight into the way the process can be controlled.

Opposed to this, the border-line significance of the lack-of-fit test in 2 (d), the 26 per cent unexplained variation in 2 (e), and the fact that the response predicted from the model could deviate from the observed response more than could be expected due to experimental error about one third of the time (2 (b)), all show that the model's predictive power is limited. If it is thought that the predicted response must be closer than $\pm$ 4.6 per cent (2 (b)) to the response observed from an actual experiment, then further study and experiments would be needed to improve the precision of the model.

Detailed analyses similar to that done with the results of Table 2 (fully described above) can be done for Tables 3, 4 and 5. Table 3 shows that the second-order model for the 48-hour uranium extraction contains the same variables as were contained in the model for the first hour's extraction (Table 2). However, the 48-hour extraction is more dependent on acid addition and temperature, and less dependent on oxidizer addition, than was the first hour's extraction. Neither the fit of the 48-hour-extraction model to the data, nor its predictive power, is as good as were those of the model with the first hour's extraction as the response.

Table 4 relates the acid consumption to the operating variables. As might be expected, the second-order model shows that the consumption of acid increases with increasing acid addition and temperature. Table 4 also shows that this model fits the data more closely than do the models in Tables 2 and 3 and can predict acid consumption from the operating variables with a relatively high degree of precision.

Table 5 shows a first-order model relating final e.m.f. of the leaching solution to leaching temperature. Although only 30 per cent of the variation in the observed e.m.f. values is accounted for by this model, the very high experimental error observed here makes it meaningless to try to develop a more complex model. This conclusion is valid because the ratio of lack-of-fit variance to experimental error variance is not significant (5 (d)). The model shows only that the 48-hour e.m.f. decreases as the leaching temperature increases. However, the reason for the high experimental error must be found and eliminated, if the effect of the operating variables on final e.m.f. is to be clarified further.

The discussion of the results given in this report is applicable to the results of any regression analysis done with the computer program that was used in this work. The program produces results similar to that shown in the tables reproduced here, regardless of the source of the data or of the experimental design used. Consequently, this report can be used as a guide in interpreting the results obtained from regression analyses run on any set of experimental results.

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TABLE I

Regression Input Data and Predicted Responses

Run No.	INDEPENDENT VARIABLES X1 Acid (lb/ton)	X2 Temp (°C)	X3 Oxidant (lb/ton)	X4 Grind (%-200M)	RESPONSES 1-hr U3O8 Extr. Measured (%)	1-hr U3O8 Extr. Predicted (%)	48-hr U3O8 Extr. Measured (%)	48-hr U3O8 Extr. Predicted (%)	Acid Consumption Measured (lb/ton)	Acid Consumption Predicted (lb/ton)	E.M.F. Measured (mv)	E.M.F. Predicted (mv)
1	50.0	55.0	1.0	50.5	79.0	78.3	87.1	85.6	39.9	40.9	370	375
2	50.0	55.0	2.0	71.1	84.9	81.7	89.1	87.4	40.3	40.9	374	375
3	70.0	55.0	1.0	49.5	86.4	81.6	89.8	90.0	43.6	45.7	375	375
4	70.0	55.0	2.0	71.2	84.7	85.0	90.8	92.5	49.3	45.7	382	375
5	50.0	75.0	1.0	71.9	82.8	80.5	87.7	88.9	42.8	42.0	378	361
6	50.0	75.0	2.0	49.6	85.5	86.1	93.9	90.7	43.0	42.0	364	361
7	70.0	75.0	1.0	72.0	87.4	83.8	97.6	94.6	51.7	53.2	362	361
8	70.0	75.0	2.0	54.3	89.2	89.4	97.5	97.1	51.9	53.2	369	361
9*	60.0	65.0	1.5	62.9	84.8	84.7	89.6	90.9	45.4	46.5	364	368
10*	60.0	65.0	1.5	60.4	83.5	84.7	90.1	90.9	48.6	46.5	365	368
11*	60.0	65.0	1.5	59.1	84.0	84.7	89.9	90.9	47.9	46.5	376	368
12	70.0	75.0	2.0	71.8	87.5	89.4	97.5	97.1	52.7	53.2	361	361
13	70.0	75.0	1.0	52.1	85.7	83.8	96.8	94.6	52.3	53.2	373	361
14	50.0	75.0	2.0	72.3	86.4	86.1	92.0	90.7	40.4	42.0	361	361
15	50.0	75.0	1.0	54.3	82.6	80.5	88.7	88.9	43.7	42.0	360	361
16	70.0	55.0	2.0	52.6	83.7	85.0	95.9	92.5	40.6	45.7	397	375
17	70.0	55.0	1.0	73.0	80.6	81.6	90.7	90.0	47.5	45.7	375	375
18	50.0	55.0	2.0	53.7	81.3	81.7	89.1	87.4	38.6	40.9	379	375
19	50.0	55.0	1.0	73.0	79.5	78.3	84.3	85.6	39.6	40.9	366	375
20	60.0	65.0	0.5	65.3	68.1	74.1	88.6	88.7	44.6	46.5	357	368
21	60.0	65.0	2.5	62.1	85.6	83.1	90.7	93.0	45.4	46.5	365	368
22	40.1	65.0	1.5	61.5	81.8	82.0	87.1	85.7	35.3	36.4	366	368
23	80.0	65.0	1.5	62.1	89.2	88.5	97.7	96.2	53.1	52.4	373	368
24	60.0	45.0	1.5	61.7	79.7	81.4	91.0	86.9	40.9	39.9	398	382
25	60.0	85.0	1.5	62.5	87.2	88.0	96.0	94.8	47.5	48.5	359	354
26	60.0	65.0	1.5	54.6	84.8	84.7	88.8	90.9	47.4	46.5	374	368
27	60.0	65.0	1.5	86.9	84.0	84.7	89.0	90.9	45.8	46.5	366	368
28*	60.0	65.0	1.5	62.9	82.6	84.7	89.2	90.9	49.8	46.5	353	368
29*	60.0	65.0	1.5	61.6	86.1	84.7	89.2	90.9	48.4	46.5	360	368
30*	60.0	65.0	1.5	61.0	83.0	84.7	87.7	90.9	47.6	46.5	352	368
31*	60.0	65.0	1.5	61.8	83.6	84.7	87.3	90.9	48.4	46.5	351	368
32*	60.0	65.0	1.5	61.8	81.8	84.7	87.3	90.9	48.5	46.5	354	368

* Duplicate runs.

TABLE 2

Regression Results for First-Hour U_3O_8 Extraction

(a) Empirical Model

$$U_3O_8 \text{ Ext 1 hr } (\%) = 59.35 + 15.6\,X_3 + 0.11\,X_2\,X_3 + 0.0014\,X_1^2 - 6.06\,X_3^2$$

Response Mean = 83.7% Deviation in Response = ± 3.9%

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 95%.

(b) Standard Error of Estimate for Response Mean

Source	Confidence Level of 95%	
	Standard Error	Interval
Empirical Model	± 2.24	± 4.60
System or Exp. Error	± 1.33	± 2.73

(c) Variation in Response Due to Significant Terms

Variables	Percent of Variation	Coefficients
X 3	25.4	15.59095
X 2 X 3	15.8	0.1094828
X 1 X 1	3.9	0.001366662
X 3 X 3	25.5	- 6.063556
Total	70.6	
Constant Term in Empirical Model		59.352685

(d) Variance Tests

Source	Deg Freedom	F-Calculated
Regression Variance / Residual Variance	4, 27	16.26*
Lack-Fit Variance / Exp. Error Variance	20, 7	3.48*

* Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation in Response

Source	Amount (%)
Significant Independent Variables	70.6
Unexplained Sources or Lack of Fit	26.6
System or Experimental Error	2.7
Total	100.0

TABLE 3

Regression Results for 48-Hour U3O8 Extraction

(a) Empirical Model

U_3O_8 Ext 48 hr (%) = 74.79 + 0.0033 X_1X_2 + 0.036 X_1X_3

Response Mean = 90.9% Deviation in Response = ± 3.7 %

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 95 %.

(b) Standard Error of Estimate for Response Mean

Source	Confidence Level of 95%	
	Standard Error	Interval
Empirical Model	± 2.13	± 4.36
System or Exp. Error	± 1.17	± 2.39

(c) Variation in Response Due to Significant Terms

Variables	Per cent of Variation	Coefficients
X1 X2	54.3	0.00328743
X1 X3	14.9	0.03615844
Total	69.2	
Constant Term in Empirical Model		74.790388

(d) Variance Tests

Source	Deg Freedom	F-Calculated
Regression Variance / Residual Variance	2, 29	32.53 *
Lack-Fit Variance / Exp. Error Variance	22, 7	4.07 *

* Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation in Response

Source	Amount (%)
Significant Independent Variables	69.2
Unexplained Sources or Lack of Fit	28.6
System or Experimental Error	2.2
Total	100.0

TABLE 4

Regression Results for Acid Consumption

(a) Empirical Model

$$\text{Acid Consumption (lb/t)} = 27.55 + 0.016\, X_1X_2 - 0.0054\, X_1^2 - 0.0058\, X_2^2$$

Response Mean = 45.7 lb/ton Deviation in Response = ± 4.6 lb/ton

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 95%.

(b) Standard Error of Estimate for Response Mean

Source	Confidence Level of 95%	
	Standard Error	Interval
Empirical Model	± 1.95	± 4.00
System or Exp. Error	± 1.26	± 2.57

(c) Variation in Response Due to Significant Terms

Variables	Percent of Variation	Coefficients
X1 X2	51.0	0.01615577
X1 X1	12.5	-0.005416226
X2 X2	20.3	-0.005802543
Total	83.8	
Constant Term in Empirical Model		27.551173

(d) Variance Tests

Source	Deg Freedom	F-Calculated
Regression Variance / Residual Variance	3, 25	48.52*
Lack-Fit Variance / Exp. Error Variance	21, 7	2.88

* Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation in Response

Source	Amount (%)
Significant Independent Variables	83.8
Unexplained Sources of Lack of Fit	14.4
System or Experimental Error	1.8
Total	100.0

TABLE 5

Regression Results for Electromotive Force

(a) Empirical Model

E.M.F. (mv) = 413.6 - 0.7 X_2

Response Mean = 368 mv Deviation in Response = $\pm$ 11.3 mv

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 95%.

(b) Standard Error of Estimate for Response Mean

Source	Confidence Level of 95%	
	Standard Error	Interval
Empirical Model	$\pm$ 9.60	$\pm$ 19.6
System or Exp. Error	$\pm$ 8.50	$\pm$ 17.4

(c) Variation in Response Due to Significant Terms

Variables	Percent of Variation	Coefficients
X2	29.9	-0.700000
Total	29.9	
Constant Term in Empirical Model		413.59375

(d) Variance Tests

Source	Deg Freedom	F-Calculated
Regression Variance / Residual Variance	1, 30	12.77*
Lack-Fit Variance / Exp.Error Variance	23, 7	1.30

* Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation in Response

Source	Amount (%)
Significant Independent Variables	29.9
Unexplained Sources or Lack of Fit	56.8
System or Experimental Error	13.3
Total	100.0