

**UNCLASSIFIED**

~~CONFIDENTIAL~~

NLCO-697

Metallurgy and Ceramics

INGOT MOLD HEATING AS A METHOD  
OF  
CONTROLLING METAL QUALITY  
BY

EARL B. BLASCH  
GALEN F. JENNINGS  
GEORGE E. WULLER, JR.

AEC RESEARCH AND DEVELOPMENT REPORT



~~CONFIDENTIAL~~  
THIS DOCUMENT CONTAINS CONFIDENTIAL - RESTRICTED DATA  
RELATING TO CIVILIAN APPLICATION OF ATOMIC ENERGY.

FEED MATERIALS PRODUCTION CENTER  
NATIONAL LEAD COMPANY OF OHIO

~~CONFIDENTIAL~~  
**RESTRICTED DATA**

This document contains restricted data as defined in the Atomic Energy Act of 1946. The information relates to the civilian applications of atomic energy. Its transmittal or the disclosure of its contents in any manner to an unauthorized person is prohibited.

**UNCLASSIFIED**

~~CONFIDENTIAL~~

2439



## LEGAL NOTICE

THIS REPORT WAS PREPARED AS AN ACCOUNT OF GOVERNMENT SPONSORED WORK. NEITHER THE UNITED STATES, NOR THE COMMISSION, NOR ANY PERSON ACTING ON BEHALF OF THE COMMISSION:

- A. MAKES ANY WARRANTY OR REPRESENTATION, EXPRESS OR IMPLIED, WITH RESPECT TO THE ACCURACY, COMPLETENESS, OR USEFULNESS OF THE INFORMATION CONTAINED IN THIS REPORT, OR THAT THE USE OF ANY INFORMATION, APPARATUS, METHOD, OR PROCESS DISCLOSED IN THIS REPORT MAY NOT INFRINGE PRIVATELY OWNED RIGHTS; OR
- B. ASSUMES ANY LIABILITIES WITH RESPECT TO THE USE OF, OR FOR DAMAGES RESULTING FROM THE USE OF ANY INFORMATION, APPARATUS, METHOD, OR PROCESS DISCLOSED IN THIS REPORT.

AS USED IN THE ABOVE, "PERSON ACTING ON BEHALF OF THE COMMISSION" INCLUDES ANY EMPLOYEE OR CONTRACTOR OF THE COMMISSION TO THE EXTENT THAT SUCH EMPLOYEE OR CONTRACTOR PREPARES, HANDLES OR DISTRIBUTES, OR PROVIDES ACCESS TO, ANY INFORMATION PURSUANT TO HIS EMPLOYMENT OR CONTRACT WITH THE COMMISSION.

PRINTED IN U.S.A. CHARGE 95 CENTS

Available from the

U.S. ATOMIC ENERGY COMMISSION,  
TECHNICAL INFORMATION SERVICE EXTENSION,  
P. O. BOX 1001,  
OAK RIDGE, TENNESSEE.

Please direct to the same address inquiries covering the procurement of other classified AEC reports.

037201030

## **DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

---

## **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

**UNCLASSIFIED**

~~CONFIDENTIAL~~

NLCO-697

Metallurgy and Ceramics

(M-3679, 20th Ed., Rev. 1)

INGOT MOLD HEATING AS A METHOD OF  
CONTROLLING METAL QUALITY

by

Earl B. Blasch  
Galen F. Jennings  
George E. Wuller, Jr.

of

TECHNICAL DIVISION  
NATIONAL LEAD COMPANY OF OHIO  
September 16, 1957

Approved By:

Approved By:

Approved By:

*H. A. Wieneke*

Head, Accountability-Quality  
Control Department

*Carl E. Polson*

Head, Metallurgical Department

*FL Cuthbert*

Technical Director

Classification cancelled (or changed to **UNCLASSIFIED**)  
by authority of *Dec 3-30-60*  
by *[Signature]* TIR, date *4-2-60*

NATIONAL LEAD COMPANY OF OHIO  
Cincinnati, Ohio  
Contract No. AT(30-1)-1156

~~CONFIDENTIAL~~

**UNCLASSIFIED**

**UNCLASSIFIED**

0370091030

## CONTENTS

	Page No.
ABSTRACT . . . . .	5
INTRODUCTION . . . . .	5
BACKGROUND . . . . .	7
DESIGN OF THE EXPERIMENT . . . . .	9
EQUIPMENT . . . . .	11
Mold Coil . . . . .	13
Mold . . . . .	14
EXPERIMENTAL PROCEDURE . . . . .	15
EXPERIMENTAL DATA AND STATISTICAL RESULTS . . . . .	15
Metal Quality . . . . .	16
Carbon . . . . .	16
Nitrogen . . . . .	18
Density . . . . .	18
Casting Data and Process Yields . . . . .	19
CONCLUSIONS . . . . .	21
ACKNOWLEDGMENTS . . . . .	21
REFERENCES . . . . .	21
APPENDIX - DETAILED CALCULATIONS OF BALANCED BLOCK EXPERIMENT FOR STATISTICAL EVALUATION OF HEATED MOLDS . . . . .	23
Introduction . . . . .	23
Analysis of Data for Carbon Content . . . . .	25
Explanation of Data Table . . . . .	25
Explanation of the Analysis of Variance Table . . . . .	26
Sum of Squares Calculations . . . . .	26
Total Sum of Squares . . . . .	26
"Between Blocks" Sum of Squares . . . . .	27
"Between Conditions" Sum of Squares . . . . .	28
"Residual" Sum of Squares . . . . .	28
"Block and Condition Interaction" Sum of Squares . . . . .	28
"Between Top and Bottom Within Each Cell" Sum of Squares . . . . .	29
"Between Ingots Within Each Cell" Sum of Squares . . . . .	29
"Unexplained Variation" Sum of Squares . . . . .	29
Mean Square Calculations . . . . .	30
"Between Blocks" Mean Square . . . . .	30
"Between Conditions" Mean Square . . . . .	30
"Block and Condition Interaction" Mean Square . . . . .	30

~~CONFIDENTIAL~~

## CONTENTS (Cont'd)

	Page No.
"Residual" Mean Square . . . . .	31
"Between Top and Bottom Within Each Cell" Mean Square . . . . .	31
"Between Ingots Within Each Cell" Mean Square . . . . .	31
"Unexplained Variation" Mean Square . . . . .	32
Evaluation of Data . . . . .	32
Effect of Interaction Between Blocks and Conditions . . . . .	33
Effect of Blocks . . . . .	33
Effect of Conditions . . . . .	33
Effect of Top and Bottom Samples Within Each Cell . . . . .	34
Effect Between Ingots Within Each Cell . . . . .	34
Computation of Means and Confidence Limits . . . . .	34
Analysis of Data for Density . . . . .	37
Evaluation of Data . . . . .	38
Effect of Interaction Between Blocks and Conditions . . . . .	38
Effect of Blocks . . . . .	39
Effect of Conditions . . . . .	39
Effect Between Top and Bottom Samples Within Each Cell . . . . .	39
Effect Between Ingots Within Each Cell . . . . .	40
Means and Confidence Limits . . . . .	40
Analysis of Data for Nitrogen Content . . . . .	41
Evaluation of Data . . . . .	42
Effect of Interaction Between Blocks and Conditions . . . . .	42
Effect of Blocks . . . . .	43
Effect of Conditions . . . . .	43
Effect Between Top and Bottom Samples Within Each Cell . . . . .	43
Effect Between Ingots Within Each Cell . . . . .	44
Means and Confidence Limits . . . . .	44
Analysis of Data for Metal Quality . . . . .	45
Evaluation of Data . . . . .	47
Effect of Interaction Between Blocks and Conditions . . . . .	47
Effect of Blocks . . . . .	48
Effect of Conditions . . . . .	48
Means and Confidence Limits . . . . .	48

~~CONFIDENTIAL~~

0370291030

## ABSTRACT

A process has been developed on a pilot plant scale for vacuum-casting uranium ingots, using liquation techniques with heated molds. In an experiment designed to statistically evaluate the effects of mold heating and associated melting techniques, marked increases in metal quality were achieved. The improved methods of vacuum-casting uranium ingots were demonstrated to give more economical production of metallic fuel elements.

## INTRODUCTION

Uranium fuel elements (slugs) are fabricated at the National Lead Company of Ohio from vacuum-cast ingots. A rolling operation reduces the uranium ingots to rods which are machined to fuel element size and inspected for surface defects.

The current practice for vacuum-casting uranium ingots is to melt the uranium in an induction furnace and to pour molten uranium at about 2550°F into magnesium oxide-coated graphite molds (vertically split molds) which have a temperature at the bottom of approximately 300°F. The molds are heated by radiant heat from the bottom of the melt crucibles.

Rejection or acceptance of fuel elements (slugs) was based on their surface condition. The effects of melting and casting techniques on metal quality (slug to good slug yield) have been of vital interest since the startup of the Fernald plant. Results of investigations<sup>1,2</sup> showed that the remelt cycle affected slug yield.



~~CONFIDENTIAL~~

03170201030

~~CONFIDENTIAL~~

## BACKGROUND

Previous work at Mallinckrodt Chemical Works<sup>3,4,5</sup> on the use of a supplementary heating source for ingot molds in the vacuum-casting of uranium was pointed primarily to improvements in ingot soundness. Uranium ingots weighing approximately 600 pounds each were cast in graphite molds with the tops of the molds at temperatures ranging from 1500°F to 2200°F. The "hot top" mold was heated by an induction coil. The molds were brought to temperature before pour and were held at temperature for periods of 6 to 20 minutes after pour. Results indicated that 7 and 8-inch-diameter ingots free of large axial cavities could be cast by this method. However, certain operational difficulties and process detriments were encountered by Mallinckrodt during this work. Prominent among these were:

1. Graphite seizure in the "hot top" section of the mold reduced the mold life and caused poor surfaces on the upper part of the ingot.
2. The horizontally split mold had a tendency to separate at the parting line.
3. Problems were met in measuring the temperature of the mold in the induction field.
4. Surface cracks or "hot tears" occurred on some of the ingots cast in the heated molds.
5. The cooling cycle for the ingots cast in the induction-heated molds was considerably lengthened.

The work at Mallinckrodt showed that higher casting temperatures of the metal improved ingot surface, while lower casting temperatures improved ingot chemical quality. It was reported<sup>6</sup> that "as long as [uranium] ingots are cast into unheated molds, a compromise must be made between ingot surfaces and chemical quality." The current production practice at Mallinckrodt for casting uranium ingots basically differs from National Lead Company of Ohio production practice in that Mallinckrodt uses a Fiberfrax insulator around the top half of the ingot mold to promote directional solidification.<sup>7</sup>

In the past, other AEC sites, notably Los Alamos Laboratories and Argonne National Laboratory, have given advice that is pertinent to the vacuum-casting of uranium ingots. Their findings which have a bearing on the work reported here may be summarized as follows:

1. Undesirable surface reactions occur upon the pouring of molten uranium into an unheated, MgO-coated mold.
2. Holding the metal in the crucible with the power on may mix inclusions by causing inductive stirring.
3. It is possible to control the metal purity by the use of proper melting techniques to effect the liquation of impurities from the molten uranium.

~~CONFIDENTIAL~~

Although the ingot molds at National Lead Company of Ohio are subjected to an outgassing operation (800° to 1100°F) prior to use in the remelt furnaces, it is believed that adsorbed gases are present in the pores of the graphite and in the mold coating at the time of pour, due to (1) insufficient outgassing and (2) exposure to atmospheric conditions prior to assembly in the vacuum furnace. The release of these gases when the hot metal comes into contact with the mold at pour can have deleterious effects on surface or subsurface quality. Impurities in the ingot, such as nitrogen and hydrogen, may also be increased. Observed furnace pressure increases of 50 to 400 microns during pour is indicative of this outgassing phenomenon. In addition, the present practice of using superheat pour temperatures (required in order to obtain ingots having good surfaces) and a relatively cold mold does not allow liquation of certain impurities, notably oxides, hydrides, nitrides, carbides, and slag from the melt. (The liquation mechanism is not fully understood.)

Cleaning of the charge material to minimize the impurities and available reactants by leaching and pickling<sup>8</sup> has been a costly process. Therefore, the use of untreated charge material was chosen for this experiment.

~~CONFIDENTIAL~~

0371229.030

## DESIGN OF THE EXPERIMENT

The ultimate goal of the heated mold development using liquation techniques was to establish a vacuum-casting process which would give a significantly higher metal quality than that obtained by present practice. To achieve the metal quality increase desired, a thorough degassing of the mold prior to casting and the use of an appropriate melt cycle to liquate the impurities were needed. In essence, the objectives of heated mold development were techniques capable of "controlling" the temperatures of the metal and the mold at pour. A program to investigate mold heating in conjunction with liquation techniques was initiated in the National Lead Company of Ohio Pilot Plant. A single vacuum furnace was used. Two series of scoping tests were necessary before a statistically controlled evaluation of conditions could be made. The initial mold coil design was tested in the first scoping series, and improvements in metal quality were indicated. The second scoping series of heats established design criteria for a permanent mold heating coil and casting conditions for a controlled evaluation; metal quality results for the various test conditions were promising.

A balanced block experiment was then designed. In a block experiment, the major variables likely to be affected by uncontrollable or random variables associated with time are considered during the course of the complete experiment. A balanced experiment refers to the obtaining of an equal amount of data for each variable being studied within each block. The same number of ingots were cast for each condition. Three selected "heated mold" conditions (A, B, and C) and a "standard" condition (representative of production practice) were used to statistically evaluate the combined effect of liquation melt cycles and heated molds on ingot purity and metal quality. The casting conditions for the designed experiment are described in Table I.

TABLE I  
CASTING CONDITIONS\*

Condition	Heat Cycle
Standard	120 kw to 2600°F; hold at 50 kw for 20 minutes; power off; pour at about 2600°F; no supplementary mold heating.
A	Mold: 40 kw; Crucible: 120 kw to 2600°F; power off; pour at 2400°F; 900° to 1000°F temperature at the bottom of the mold.
B	Mold: 40 kw; Crucible: 120 kw to 2600°F; power off; pour at 2250°F; 900° to 1000°F temperature at the bottom of the mold.
C	Mold: 32 kw; Crucible: 120 kw to 2600°F; power off; pour at 2400°F; 700° to 800°F temperature at the bottom of the mold.

\* See also Table V.

The melt cycles used in the heated mold experiment are practical for production use, particularly because the existing equipment (slightly modified) can be used and because there is little difference in the cycle time from charge to charge.

~~CONFIDENTIAL~~

0377291030

~~CONFIDENTIAL~~



## EQUIPMENT

The FMPC Pilot Plant vacuum furnace was modified to provide mold heating. The design of the original Pilot Plant furnace is shown in Figure 1. The modified furnace with an induction coil for mold heating is shown in Figure 2. The upper portion of the mold in the modified furnace is induction heated while the crucible charge is melted. (Separate induction coils are used.) The mold bottom is brought to temperature primarily by conduction heating.

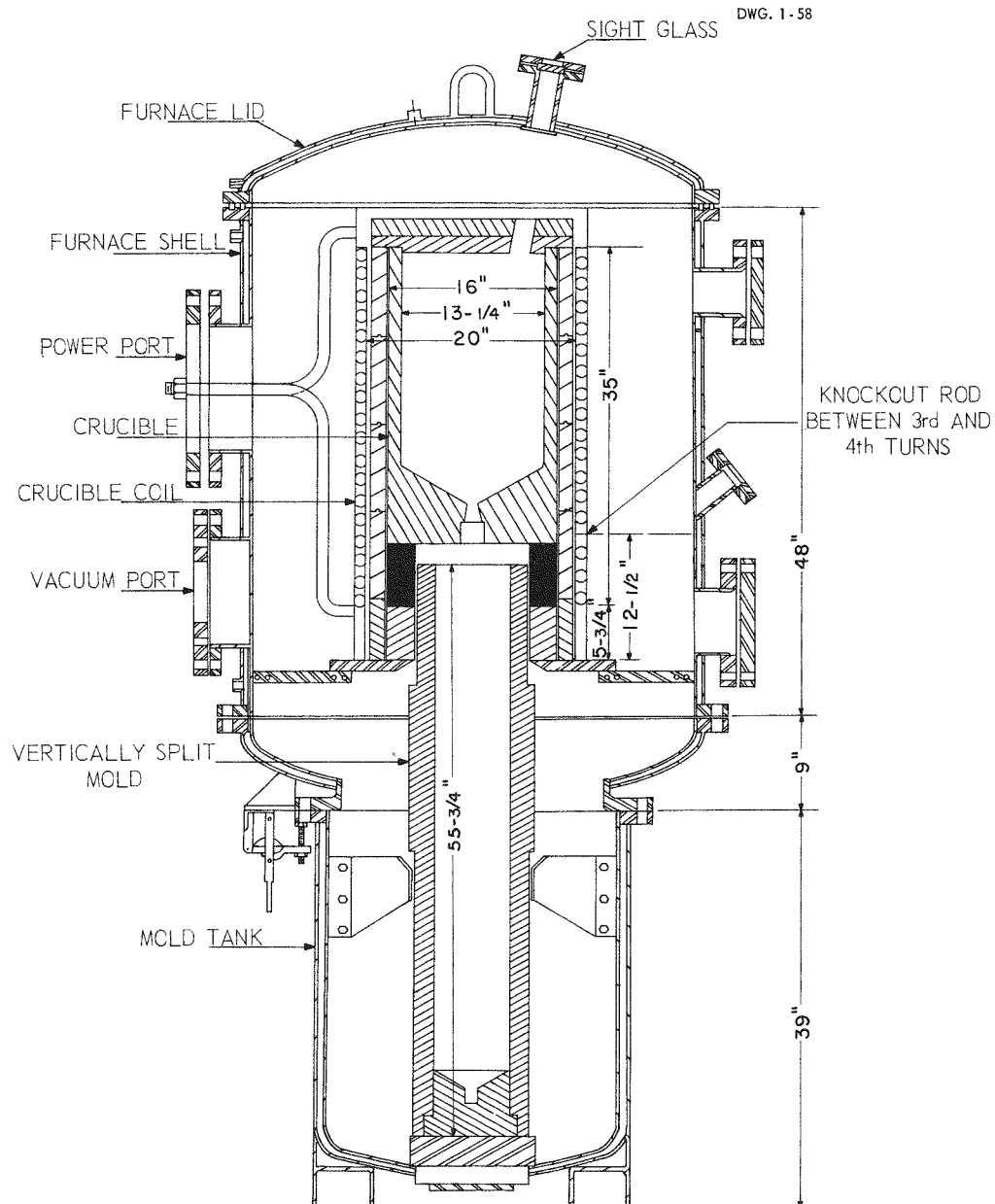


FIGURE 1 Original Pilot Plant Remelt Furnace

~~CONFIDENTIAL~~

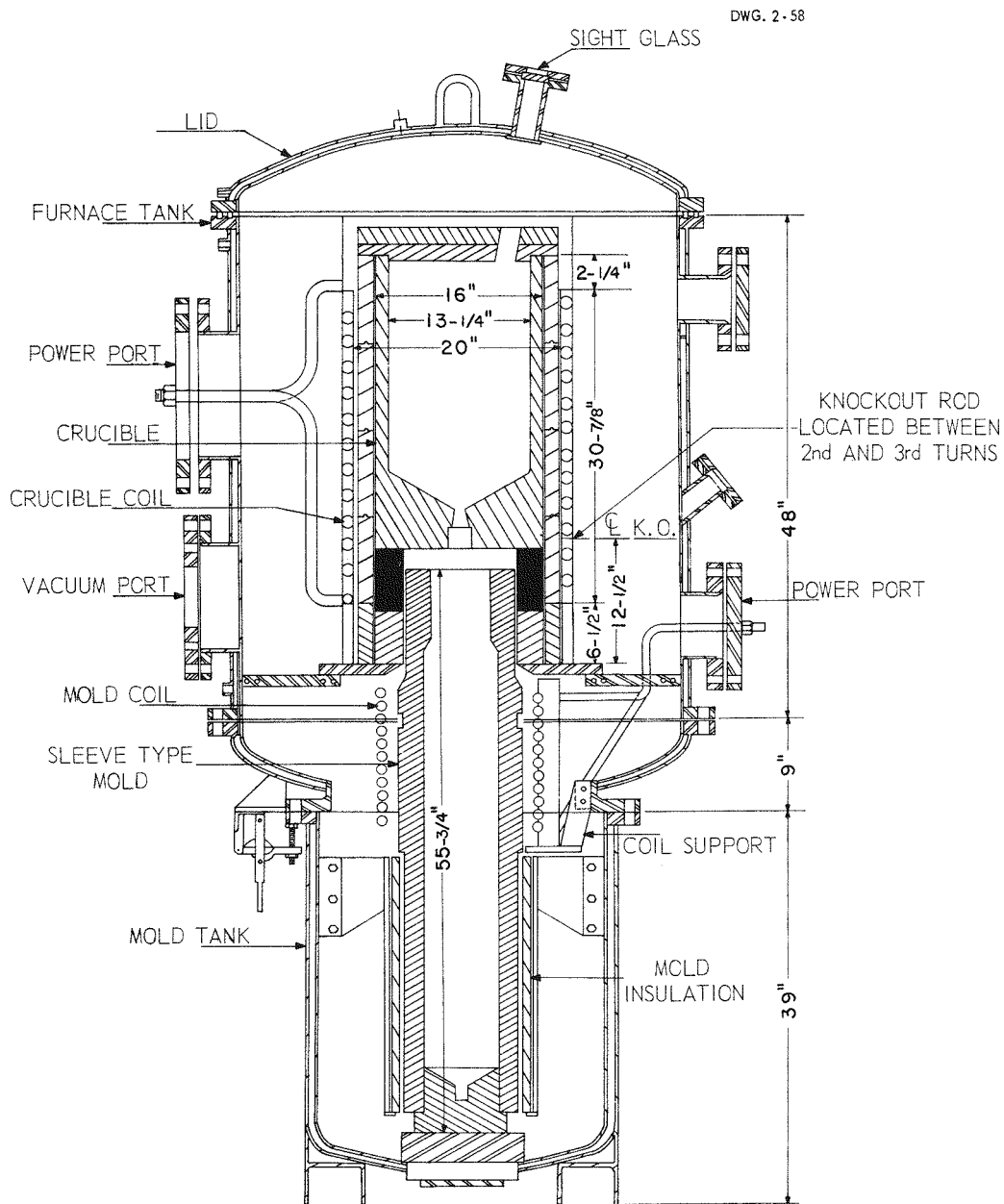


FIGURE 2 Pilot Plant Remelt Furnace as Modified for Mold Heating

A study of the design data and a review of the previous work<sup>3,4,5</sup> on heated ingot molds led to the following conclusions:

1. Heated mold designs used in previous work handicapped the handling of the mold and mold chamber.
2. In certain cases where two induction coils were used along the length of the mold, only the upper coil was found necessary.

~~CONFIDENTIAL~~

0310201030

3. Mold temperatures which exceeded the melting point of uranium were not only unnecessary but actually undesirable, as they caused mold seizure.
4. A process using heated molds would not be acceptable if it greatly lengthened the ingot cooling cycle of the production remelt operation.
5. A mold heating coil which utilized available power from the 200-kw motor-generator would be desirable. (Sufficient capacity was available to heat both the crucible and the mold in parallel.)

Heating of the top section of the mold during melting to achieve (1) a more uniform temperature gradient in the mold and (2) a temperature at the mold bottom of about 1000°F was believed to be an improved casting technique. Therefore, an induction coil was mounted on the stationary part of the furnace in the section between the crucible coil and the mold chamber parting line.

#### MOLD COIL

First, a coil was designed that heated a 13-inch segment of the mold 10 inches from the top, with power from a separate 50-kw generator. This coil operated successfully on the first attempt and showed the power requirements for producing temperatures up to 1000°F at the mold bottom. It was determined that a 40-kw power input with ceramic insulation around the segment of the mold in the mold chamber (Fig. 2) would give the desired temperatures in 2 hours. Also, the power applied to the mold was shown to have a direct relationship to the crucible coil power. Therefore, a simple parallel connection of the two coils could be made to the 200-kw power source. The correct power ratio of the two coils was yet to be determined.

In this experiment, the mold temperatures were controlled by varying the ratio of the power inputs to the crucible and mold coils. Different power ratios were obtained simply by varying the outside diameter of the mold section in the coil field. The power inputs obtained by this technique and used in the casting experiment are given in Table II.

TABLE II  
EFFECT OF MOLD SIZE ON POWER INPUT

Graphite Mold OD (in.)	Coil Voltage (v)	Crucible Coil Power (kw)	Mold Coil Power (kw)
11	352	120	45
12	352	120	60

The mold coil used in the designed experiment was made of thick-walled copper tubing of  $\frac{5}{8}$  inch diameter. It consisted of 12 turns wound on 3 transite legs with an approximate inside diameter of 15 inches. The coil was mounted to heat a 15-inch section of the mold about 15 inches from the mold top.

~~CONFIDENTIAL~~

Thus, a mold coil design was developed that had the following characteristics:

1. The coil does not interfere with the loading or removal of the mold.
2. A mold bottom temperature of about 1000°F can be attained during the normal heat cycle.
3. Cooling of the mold after pour is aided by the water-cooled coil. As a result, the normal cooling cycle need not be lengthened.
4. No special controls are needed for the mold induction coil. A simple switch allows connection to the power circuit.
5. Mold coil power is controlled by using molds having different outside diameters.

## MOLD

It became evident during the early phases of the work that holding together the two halves of the vertically split mold (production mold) at the top with steel bands would not be satisfactory for a heated mold, due to increased expansion of the metal bands at the higher temperatures: Excessive fins appeared on the ingots due to a separation of the mold sections during casting. The need for a one-piece "sleeve type" ingot mold was apparent. A sleeve mold was designed (Fig. 3) and was used throughout the casting test. This mold proved satisfactory; ingot surfaces were improved by the elimination of fins, and mold life was lengthened.

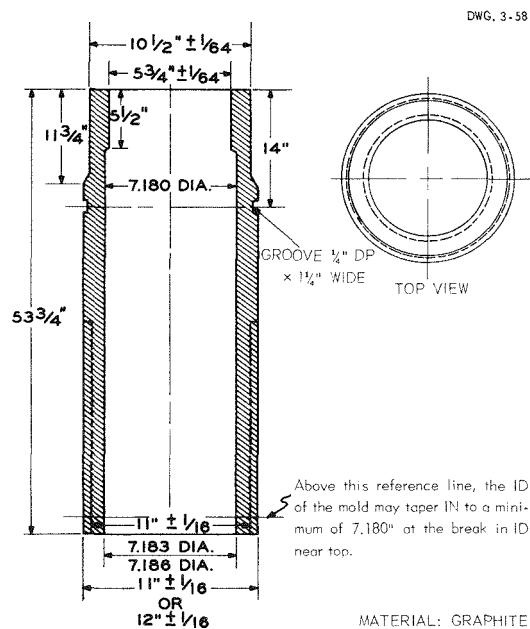


FIGURE 3 One - Piece "Sleeve" Type 7-inch-Diameter Ingot Mold

~~CONFIDENTIAL~~

0376281030

## EXPERIMENTAL PROCEDURE

Forty-eight normal uranium ingots were cast for the balanced block experiment. The composite metal charge used consisted of two chipped derbies, approximately 250 pounds of briquetted machine turnings, and 300 to 350 pounds of solid scrap. (Chipped derbies are derbies that are cleaned by means of an air hammer to remove excess  $MgF_2$  slag. Solid scrap includes ingot crops, sheared billet ends, cropped reject rods, and reject slugs.) The derbies taken from the production stream had been produced from several different lots of  $UF_4$ .

Normal operating pressure was below 500 microns. Metal temperatures were determined by the use of an optical pyrometer. Mold temperatures during the melting and casting were measured by thermocouples inserted in the mold wall. Three thermocouples were located approximately 10, 30, and 50 inches from the mold bottom. Average peak mold temperatures recorded for the various casting conditions are shown in Table III.

TABLE III  
AVERAGE PEAK MOLD TEMPERATURES

Casting Condition	Temp. Before Pour (°F)			Temp. After Pour (°F)		
	Mold Top	Mold Center	Mold Bottom	Mold Top	Mold Center	Mold Bottom
Standard	1520	495	330	1805	1430	1375
A	1895	1300	830	1935	1715	1550
B	1830	1240	845	1865	1640	1440
C	1835	1130	735	1895	1680	1515

To compensate for a possible time dependence, blocks of 12 ingots each were cast during as small a time interval as possible. Each block of heats was cast in the following order:

- 3 heats - Standard Condition (similar to production procedure)
- 3 heats - Condition A (high pour temperature and high mold temperature)
- 3 heats - Condition B (low pour temperature and high mold temperature)
- 3 heats - Condition C (high pour temperature and low mold temperature)

Four such blocks completed the designed experiment. The rolled rods produced from the ingots were machined to Hanford (HGE) solid slugs, and the chemical analyses and slug inspection results were statistically evaluated.

## EXPERIMENTAL DATA AND STATISTICAL RESULTS

The statistical investigation for the evaluation of carbon, nitrogen, density, and metal quality is based on analysis - of - variance<sup>9</sup> of a balanced block design. (See the Appendix for detailed data



~~CONFIDENTIAL~~

tables and calculations.) Confidence limits at the 95 per cent level are used throughout the statistical analysis.

In the heated mold experiment, the blocks (periods of time) were just as significant in producing changing of levels as were the major variables (conditions) being studied. The over-all effects of the casting conditions on metal quality and purity are seen in Table IV.

TABLE IV

SUMMARY OF RESULTS OF HEATED MOLD EXPERIMENT

Condition	Slug Reject (%)	Carbon (ppm)	Nitrogen (ppm)	Density (g/cc)
Standard	$20.1 \begin{smallmatrix} +5.4 \\ -4.5 \end{smallmatrix}$	$554 \pm 32$	$71.25 \pm 4.80$	18.902
A	$6.3 \begin{smallmatrix} +3.2 \\ -2.6 \end{smallmatrix}$	$513 \pm 32$	$32.50 \pm 4.80$	18.924
B	$11.9 \begin{smallmatrix} +4.2 \\ -3.5 \end{smallmatrix}$	$531 \pm 32$	$38.33 \pm 4.80$	18.923
C	$7.5 \begin{smallmatrix} +3.5 \\ -2.8 \end{smallmatrix}$	$518 \pm 32$	$43.54 \pm 4.80$	18.925

Results of the heated mold experiment are discussed below.

#### METAL QUALITY

A significant difference in metal quality was detected between conditions. The standard condition ingots had the highest average per cent of metal quality rejects, with the ingots of test conditions A, B, and C grouped at a lower level (Fig. 4). While the over-all variation between blocks was not significant, it was of interest to note that block 1 (Fig. 4) had a considerably lower reject rate than the other three blocks.

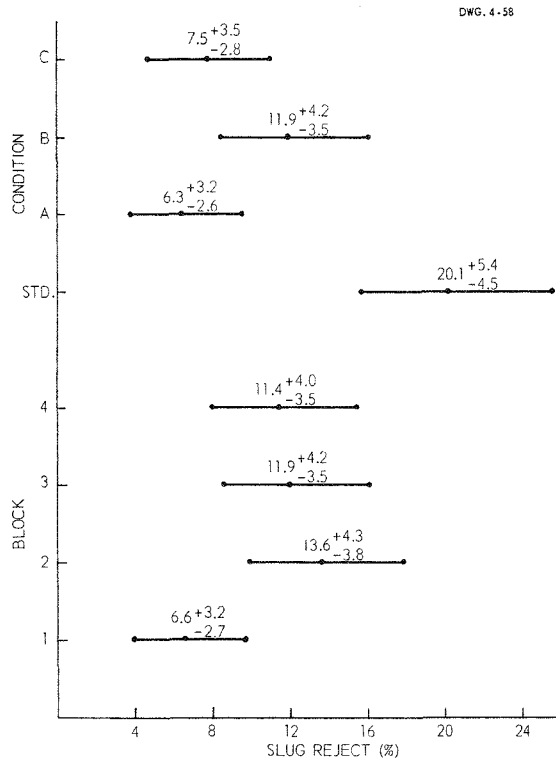
#### CARBON

No detectable difference in carbon contamination was found between conditions, between blocks, between top and bottom samples, or from condition-block interaction. The average carbon contaminations of the ingot metal in parts per million (ppm) for the various conditions and blocks of the experiment are shown in the line graphs of Figure 5.

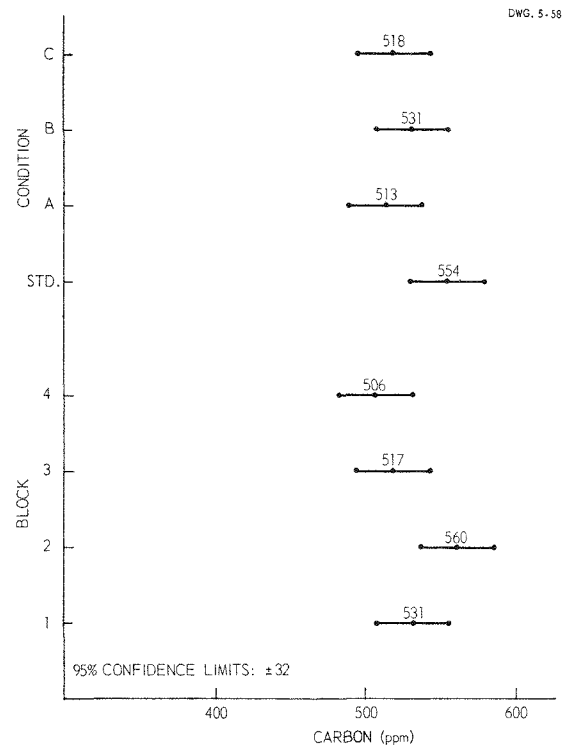
A significant difference was detected between ingots within each cell. (A cell consists of all of the samples for any one condition in one block.) This fact is important in that the variation due to ingots is removed from the data and a more sensitive test for differences between top and bottom samples is available. Thus, a real difference between top and bottom samples can be more easily detected. For this experiment, the difference was significant, with bottom samples having higher carbon values than the top samples.

~~CONFIDENTIAL~~

0371229.0330



**FIGURE 4** Metal Quality vs. Block and Condition Effects



**FIGURE 5** Carbon Contamination vs. Block and Condition Effects

~~CONFIDENTIAL~~

## NITROGEN

Significant differences in nitrogen contamination were detected between conditions, between blocks, and between top and bottom samples within cells. Ingots cast by the standard condition had a significantly higher average nitrogen contamination than did the ingots cast by the heated mold conditions. Figure 6 contains line graphs depicting the condition and block effect on nitrogen contamination. Examination of the data for differences between top and bottom samples within cells reveals that neither sample has a consistently higher average value than the other. No explanation for the significant difference is readily available.

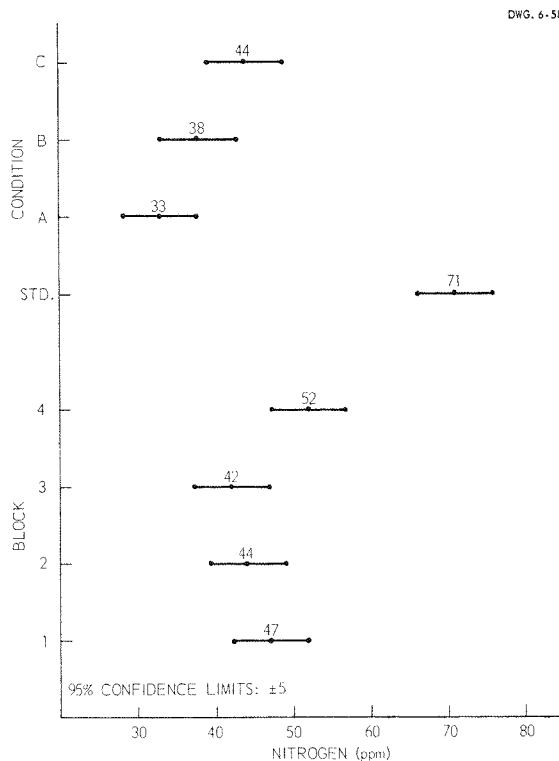


FIGURE 6 Nitrogen Contamination vs. Block and Condition Effects

## DENSITY

Significant differences in density of ingot metal were detected between conditions, between blocks, and from condition-block interaction. Ingots cast by the standard condition had significantly lower density than did ingots cast into heated molds. Block 1 ingot metal was of significantly lower density than that produced during the other blocks of castings. Figure 7 bar graphs show the average densities obtained under the various conditions and blocks. No confidence limits were attached to these values due to the significant interaction effect between conditions and blocks. The interaction effect indicated that the density of an ingot is highly dependent on the particular time period in which the ingot was cast, regardless of the casting conditions used. Density values for different conditions should not be compared unless the ingots are within the

~~CONFIDENTIAL~~

037259J34

same time period. It will be noted from the bar graph for block effects that block one had a lower average density than did the standard condition.

Figure 8 shows the average density values under each condition within each block. Block one results are completely different from results for the other three blocks. This emphasizes the necessity of "blocking" in future experimental work.

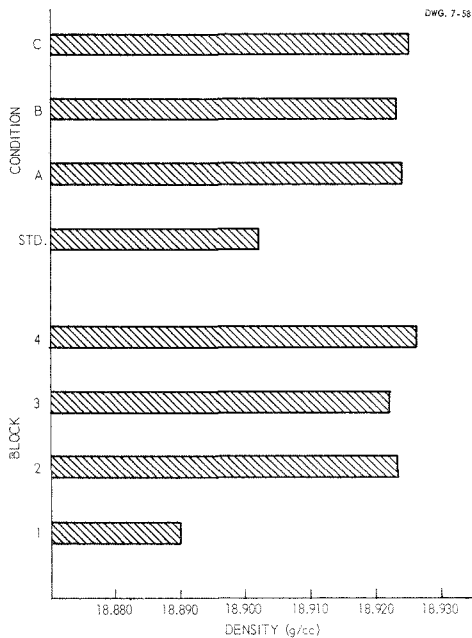


FIGURE 7 Density vs. Block and Condition Effects

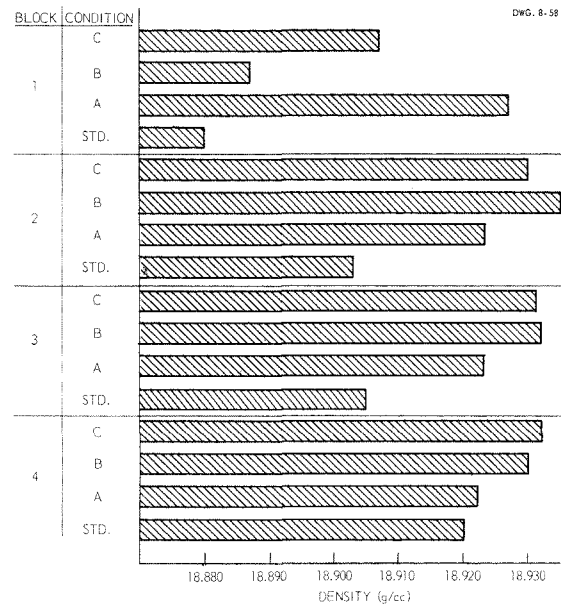


FIGURE 8 Density by Conditions Within Block

## CASTING DATA AND PROCESS YIELDS

The average melt cycles, hold times, and metal pour temperatures for the various casting conditions are given in Table V.

TABLE V

### CASTING CONDITIONS

Casting Condition	Total Time* to 2600°F (min)	Hold Time** (min)	Pour Temp. (°F)
Standard	105	20	2600
A	126	25	2400
B	114	47	2250
C	114	25	2400

\* Average times for conditions A, B, and C were somewhat lengthened due to reductions in power inputs to eliminate occasional arcing of the mold coil.

\*\* Standard Condition: 50 kw. Conditions A, B, and C: No power input.

~~CONFIDENTIAL~~

Figure 9 is a plot of average metal quality of the various casting conditions against rod number. Rod 1 is the rod fabricated from the bottom of the ingot, while rod 4 is fabricated from the ingot top. (The ingot is rolled bottom-first.) For ingots cast by the standard condition, maximum slug rejection for surface defects occurred in the third rod. For ingots cast using liquation techniques with heated molds, the fourth rod, on the average, had the highest reject per cent.

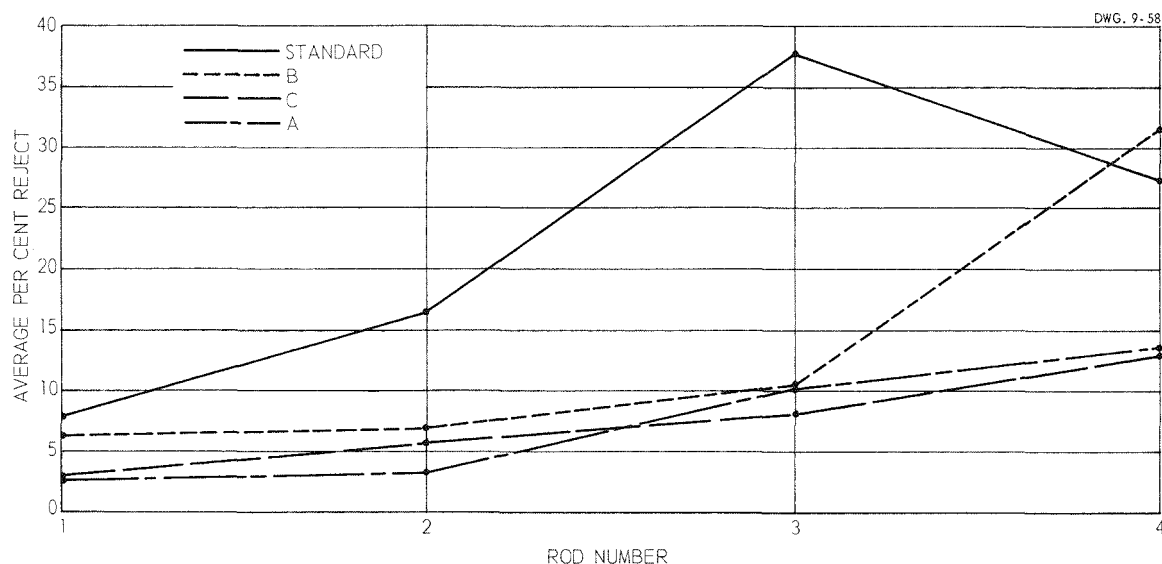


FIGURE 9 Slug Yield vs. Rod Number

The process yields of the standard heats and the various heated-mold conditions are compared in Table VI, which includes data from twelve ingots produced under each condition. An increase (over the standard condition) of better than 9 per cent in the over-all yield of the casting and fabrication operations is shown by the heated mold condition, C. Heated mold conditions were selected on the basis of scoping test results, however, and do not represent the optimum conditions for uranium ingot casting. Further evaluation of liquation techniques using heated molds will establish the optimum conditions. Additional increases in metal quality may be realized.

TABLE VI

COMPARISON OF PROCESS EFFICIENCY

Condition	Total Charge Weight (lb)	No. of Acceptable Slugs Produced	Over-all Yields, Slug Wt*/Charge Wt (%)
Standard	14,838	1036	55.5
A	14,826	1188	63.7
B	14,824	1124	60.7
C	14,834	1208	64.7

\* 7.948 lb/ HGE solid slug.

~~CONFIDENTIAL~~

03172281030



## CONCLUSIONS

The designed experiment in which 48 ingots were cast in the FMPC Pilot Plant has statistically shown that the use of heated ingot molds and associated melting techniques gives marked improvement in metal quality over that obtained by the present casting practice. The standard condition gave approximately a 20 per cent slug reject level, while the reject level was decreased to approximately 6 per cent by the use of condition A. Also, the heated-mold ingots had significantly lower nitrogen contamination and higher density values, on the average, than did the standard ingots. No real differences in levels of carbon contamination were detected, due to variability of results. However, the average carbon values of the heated-mold ingots were lower than the carbon values of the standard ingots.

## ACKNOWLEDGMENTS

The authors wish to thank the Pilot Plant personnel under the supervision of J. O. Davis for their assistance in this project. Thanks is also extended to E. Morris of the National Lead Company of Ohio Maintenance Department, for his advice and help in the design and fabrication of the mold heating coil. The work of J. H. Smith on the calculations included in the Appendix is also acknowledged.

## REFERENCES

- 1 Eikenberry, H. M., Summary Tech. Rpt., pp. 91-93, FMPC-540, April 15, 1955.
- 2 Bauer, R. F. and Eikenberry, H. M., Summary Tech. Rpt., pp. 110-111, NLCO-565, July 15, 1955.
- 3 Buntz, B. J., Janka, R. C., and Weber, L. G., Process Development Quarterly Rpt., Part I, pp. 92-119, NYO-1353, April 15, 1953.
- 4 Buntz, B. J., Janka, R. C., and Weber, L. G., Process Development Quarterly Rpt., Part II, pp. 38-70, NYO-1356, July 15, 1953.
- 5 Buntz, B. J., Janka, R. C., and Weber, L. G., Process Development Quarterly Rpt., Part II, pp. 85-101, NYO-1358, October 15, 1953.
- 6 Powers, W. O., Process Development Quarterly Rpt., Part I, p. 145, NYO-1353, April 15, 1953.
- 7 Buntz, B. J. and Janka, R. C., Process Development Quarterly Rpt., Part II, pp. 175-181, MCW-1383, February 1, 1956.
- 8 Torok, E. W., Bauer, R. F., and Eikenberry, H. M., Summary Tech. Rpt., pp. 77-89, NLCO-640, July 18, 1956.
- 9 Davies, O. L., Design and Analysis of Industrial Experiments, Hafner Publishing Co., N. Y., N. Y., 1954.

~~CONFIDENTIAL~~

0377201030

~~CONFIDENTIAL~~

## APPENDIX

## Detailed Calculations of Balanced Block Experiment For Statistical Evaluation Of Heated Molds

## INTRODUCTION

The purpose of this appendix is twofold:

1. To support the main report by showing the method whereby the conclusions are drawn from the statistical analysis of the data.
2. To familiarize personnel with the type of statistical design used and the calculations necessary for completing an analysis-of-variance table and making the tests that determine the significance of the conclusions.

It is not anticipated that each report having a statistical analysis should be reported in as much detail as this one is. All that is necessary in a routine report is the analysis-of-variance table and the statistical conclusions, supported with line graphs of averages and confidence limits, where applicable.

This experiment is known as a balanced block type. A block experiment is used when the major variables being studied are considered likely to be affected during the experiment by uncontrollable or random variables associated with time. Within a block of an experiment, the complete experiment is run at least once. The block must be as small a unit of time as practical in order to eliminate the extraneous effects associated with time. The blocks are replicated as many times as needed in order to obtain the amount of experimental data desired. Block designs are somewhat like insurance in that they may or may not be needed. However, since the cost and the evaluation of the experiment are not affected by the use of blocks, it is good practice to use them. During this experiment, we had classic examples of blocks that were just as significant in producing changes of levels as were the major variables (conditions) being studied.

A balanced experiment refers to obtaining within each block an equal amount of data for each variable being studied. That is, the same number of ingots were cast for each variable (condition).<sup>\*</sup> Since our experiment is designed to compare the average values obtained under each condition, this assures us that logical comparisons can be made within a block or over several blocks. For any one block, all conditions are equally represented and affected.

The balanced block type of experiment, as described above, then may be readily analyzed by the statistical technique of analysis-of-variance. Analysis-of-variance is a method by which the total variance of an experiment can be broken down into its component factors. For example, consider the metal quality reject data. The standard ingot casting procedure is an operation not perfectly reproducible. This gives rise to a variance,  $\sigma_E^2$ , in our data. It is necessary to conduct

<sup>\*</sup> The major variable being studied is the ingot casting procedure

~~CONFIDENTIAL~~

an experiment using three additional ingot casting conditions. If there is a difference between the four conditions, there will be an additional variance introduced into the data,  $\sigma_C^2$ . In evaluating the effects of the four conditions, the effect,  $\sigma_E^2 + n \sigma_C^2$ , is calculated (n being a constant that is dependent on the number of replications).  $\sigma_E^2$  is then estimated. For  $\sigma_C^2$  to exist, therefore,  $\sigma_E^2 + \sigma_C^2$  must be greater than  $\sigma_E^2$ . This is tested by the F-test with the appropriate degrees of freedom at the desired confidence level.

Example of F-test:

$$F_c = \frac{\sigma_E^2 + n \sigma_C^2}{\sigma_E^2}$$

If the new procedures introduced an additional variance to the data,  $\sigma_C^2$ , then  $F_c$  is larger than one. In actual practice,  $F_c$  is compared with a value,  $F_t$ , from the F-distribution at the desired confidence level and appropriate degrees of freedom.  $F_c$  must exceed  $F_t$  before it can be said that  $\sigma_C^2$  has a high degree of probability of being a real rather than a chance effect.

This is the basic test that analysis-of-variance provides. If results indicate that there is an additional variance effect, the next question to answer is, how are the four procedures different? Average values with confidence limits answer this question if the confidence limits do not overlap. When confidence limits do overlap, the difference between any two average values ( $\bar{x}_1 - \bar{x}_2$ ) with appropriate confidence limits inform the experimenter whether or not there is a significant difference between the two average values.

The use of the data table and the analysis of variance table is explained for carbon only. The statistical calculations for density, nitrogen content, and metal quality are similar to those for carbon.

~~CONFIDENTIAL~~

00712281030

## ANALYSIS OF DATA FOR CARBON CONTENT

## EXPLANATION OF DATA TABLE (Table VII)

The data table for carbon shows the chemical analysis for each sample taken from the forty-eight ingots. The samples are listed according to location (top or bottom of ingot), the block (cycle) in which the ingot was produced, and the condition used in producing the ingot.

The sums of the chemical analyses to be used in subsequent calculations are shown. The sum for each ingot, top and bottom samples, is shown as the ingot total. The sum of all the top and all the bottom samples is shown as the subtotal for each block and condition. The sum of all of the chemical analyses in a block is shown as the block total.

In addition to being divided by block and condition, the data table is divided into cells. A cell consists of all the samples for any one condition in one block. The cell consists of the six values shown under top and bottom samples for one block.

TABLE VII

DATA TABLE FOR CARBON\*

Block	Block Total		Condition											
			Standard			A			B			C		
			Top	Bottom	Ingot Total	Top	Bottom	Ingot Total	Top	Bottom	Ingot Total	Top	Bottom	Ingot Total
1			505	530	1035	460	510	970	600	644	1244	560	585	1145
			515	580	1095	440	500	940	560	570	1130	545	685	1230
			575	545	1120	395	490	885	490	475	965	445	530	975
		Sub Total	1595	1655	x x x	1295	1500	x x x	1650	1689	x x x	1550	1800	x x x
	12,734	Cell Total	x x x	x x x	3250	x x x	x x x	2795	x x x	x x x	3339	x x x	x x x	3350
2			620	615	1235	515	575	1090	540	510	1050	580	620	1200
			670	700	1370	580	590	1170	550	480	1030	440	500	940
			665	415	1080	490	545	1035	650	600	1250	495	505	1000
		Sub Total	1955	1730	x x x	1585	1710	x x x	1740	1590	x x x	1515	1625	x x x
	13,450	Cell Total	x x x	x x x	3685	x x x	x x x	3295	x x x	x x x	3330	x x x	x x x	3140
3			510	600	1110	435	490	925	465	505	970	415	395	810
			585	615	1200	475	575	1050	525	575	1100	475	530	1005
			565	580	1145	600	595	1195	460	435	895	465	535	1000
		Sub Total	1660	1795	x x x	1510	1660	x x x	1450	1515	x x x	1355	1460	x x x
	12,405	Cell Total	x x x	x x x	3455	x x x	x x x	3170	x x x	x x x	2965	x x x	x x x	2815
4			485	555	1040	545	585	1130	500	505	1005	455	510	965
			515	535	1050	515	475	990	555	590	1145	525	540	1065
			395	430	825	475	460	935	485	465	950	595	510	1105
		Sub Total	1395	1520	x x x	1535	1520	x x x	1540	1560	x x x	1575	1560	x x x
	12,205	Cell Total	x x x	x x x	2915	x x x	x x x	3055	x x x	x x x	3100	x x x	x x x	3135
TOTALS 50,794			6605	6700	13,305	5925	6390	12,315	6380	6354	12,734	5995	6445	12,440

\* All values are ppm



~~CONFIDENTIAL~~

## EXPLANATION OF THE ANALYSIS OF VARIANCE TABLE (Table VIII)

The analysis of variance table summarizes the results of the calculations made from data (Table VII). These are the numbers used to perform the statistical F-test and to compute the mean and confidence limits for the experiment.

TABLE VIII

## ANALYSIS OF VARIANCE TABLE FOR CARBON

Source of Variation	Sum of Squares, S.S.	Degrees of Freedom d.f.	Mean Square, M.S.	Expected M.S.
Between Blocks (Y)	37,320	$Y - 1 = 3$	12,440	$\sigma_E^2 + 2\sigma_I^2 + 24\sigma_Y^{2*}$
Between Conditions (C)	24,291	$C - 1 = 3$	8,097	$\sigma_E^2 + 2\sigma_I^2 + 24\sigma_C^2$
Block and Condition Interaction (YC)	79,830	$(Y - 1)(C - 1) = 9$	8,870	$\sigma_E^2 + 2\sigma_I^2 + 6\sigma_{YC}^2$
Residual	280,178	$(N - 1) - (Y - 1) - (C - 1) - (Y - 1)(C - 1) = 80$	3,502	
Between Top and Bottom Samples Within Each Cell (S)	47,157	$16(S - 1) = 16$	2,947	$\sigma_E^2 + 3\sigma_S^2$
Between Ingots Within Each Cell (I)	184,310	$16(I - 1) = 32$	5,760	$\sigma_E^2 + 2\sigma_I^2$
Unexplained Variation (E)	48,711	$80 - 16(S - 1) - 16(I - 1) = 32$	1,522	$\sigma_E^2$
TOTAL	421,619	$N - 1 = 95$		

\*  $\sigma^2$  - Variation component with the subscripts E, I, and Y referring to source of variation.

## SUM OF SQUARES CALCULATIONS

Total Sum of Squares

S. S. = Sum of Squares

$$\begin{aligned}
 S. S. &= \sum_{i=1}^N X_i^2 - \frac{(\sum_{i=1}^N X_i)^2}{N} \\
 &= 27,296,936 - 26,875,317 \\
 &= 421,619
 \end{aligned}$$

~~CONFIDENTIAL~~

03712281030

Where

$X_i$  = individual analysis (shown in data table)

$$\sum_{i=1}^N X_i^2 = 27,296,936 \text{ (by machine calculation, using values from data table)}$$

$$\sum_{i=1}^N X_i = 50,794$$

$$\left(\sum_{i=1}^N X_i\right)^2 = 2,580,030,436$$

$N$  = total number of samples

$$= 96$$

$$(C.T.)_1 = \frac{\left(\sum_{i=1}^N X_i\right)^2}{N}$$

$$= 26,875,317$$

The term,  $\frac{\left(\sum_{i=1}^N X_i\right)^2}{N}$ , is called the correction term  $(C.T.)_1$ . This is the same for "between blocks" and "between conditions" sums of squares.

#### "Between Blocks" Sum of Squares

$$S.S. = \frac{Y_1^2 + Y_2^2 + Y_3^2 + Y_4^2}{N} - (C.T.)_1$$

$$= 26,912,637 - 26,875,317$$

$$= 37,320$$

Where

$N_y$  = number of samples in block

$$= 24$$

$Y_1$  = 12,734 (sum of 1st block)

$Y_2$  = 13,450 (sum of 2nd block)

$Y_3$  = 12,450 (sum of 3rd block)

$Y_4$  = 12,205 (sum of 4th block)

~~CONFIDENTIAL~~

"Between Conditions" Sum of Squares

$$\begin{aligned} \text{S. S.} &= \frac{C_{\text{std}}^2 + C_A^2 + C_B^2 + C_C^2}{N} - (C. T.)_1 \\ &= 26,899,608 - 26,875,317 \\ &= 24,291 \end{aligned}$$

Where

$$\begin{aligned} N_C &= \text{number of samples in a condition} \\ &= 24 \\ C_{\text{std}} &= 13,305 \text{ (Sum of all standard values)} \\ C_A &= 12,315 \text{ (Sum of all A values)} \\ C_B &= 12,734 \text{ (Sum of all B values)} \\ C_C &= 12,440 \text{ (Sum of all C values)} \end{aligned}$$

"Residual" Sum of Squares

$$\begin{aligned} \text{S. S.} &= \sum_{i=1}^N X_i^2 - (C. T.)_2 \\ &= 27,296,936 - 27,016,758 \\ &= 280,178 \end{aligned}$$

Where

$$\begin{aligned} (C. T.)_2 &= \frac{(\text{cell 1})^2 + (\text{cell 2})^2 + \dots + (\text{cell 16})^2}{6} \\ &= \frac{(3250)^2 + (2795)^2 + \dots + (3135)^2}{6} \\ &= 27,016,758 \end{aligned}$$

"Block and Condition Interaction" Sum of Squares

Found by difference:

$$\begin{aligned} \text{S. S.} &= \text{Total S. S.} - \text{"Between Blocks" S. S.} - \text{"Between Conditions"} \\ &\quad \text{S. S.} - \text{"Residual" S.S.} \\ &= 421,619 - 37,320 - 24,291 - 280,178 \\ &= 79,830 \end{aligned}$$

~~CONFIDENTIAL~~

0377291030

The residual sum of squares is divided into three parts: the "between top and bottom samples within each cell" sum of squares, the "between ingots within each cell" sum of squares, and the "unexplained variation" sum of squares.

"Between Top and Bottom Within Each Cell" Sum of Squares

$$\begin{aligned}
 \text{S. S.} &= \frac{(\text{Subtotals})^2}{3} - (\text{C. T.})_2 \\
 &= \frac{\sum (1595)^2 + (1655)^2 + \dots + (1560)^2}{3} - 26,016,758 \\
 &= 47,157
 \end{aligned}$$

"Between Ingots Within Each Cell" Sum of Squares

$$\begin{aligned}
 \text{S. S.} &= \frac{\sum I_1^2 + I_2^2 + \dots + I_{48}^2}{2} - (\text{C. T.})_2 \\
 &= \frac{\sum (1035)^2 + (1095)^2 + \dots + (1105)^2}{2} - (\text{C. T.})_2 \\
 &= 27,016,758 - (\text{C. T.})_2 \\
 &= 184,310
 \end{aligned}$$

Where

I = Total for each ingot

"Unexplained Variation" Sum of Squares

Found by difference:

$$\begin{aligned}
 \text{S. S.} &= \text{"residual" S. S.} - \text{"between top and bottom within each cell"} \\
 &\quad \text{S. S.} - \text{"between ingots within each cell" S. S.} \\
 &= 280,178 - 47,157 - 184,310 \\
 &= 48,711
 \end{aligned}$$

~~CONFIDENTIAL~~

# MEAN SQUARE CALCULATIONS

## "Between Blocks" Mean Square

$$\begin{aligned}\text{Mean Square (M. S.)} &= \frac{\text{"between blocks" S. S.}}{\text{d.f.}} \\ &= \frac{37,300}{3} \\ &= 12,440\end{aligned}$$

Where

$$\begin{aligned}\text{Degrees of freedom (d.f.)} &= \text{No. of blocks(Y)} - 1 \\ &= 4-1 \\ &= 3\end{aligned}$$

## "Between Conditions" Mean Square

$$\begin{aligned}\text{M. S.} &= \frac{\text{"between conditions" S. S.}}{\text{d.f.}} \\ &= \frac{24,291}{3} \\ &= 8,097\end{aligned}$$

Where

$$\begin{aligned}\text{d.f.} &= \text{No. of Conditions(C)} - 1 \\ &= 4-1 \\ &= 3\end{aligned}$$

## "Block and Condition Interaction" Mean Square

$$\begin{aligned}\text{M. S.} &= \frac{\text{"block and condition interaction" S. S.}}{\text{d.f.}} \\ &= \frac{79,830}{9} \\ &= 8,870\end{aligned}$$

Where

$$\begin{aligned}\text{d.f.} &= (Y-1) \times (C-1) \\ &= (3) \times (3) \\ &= 9\end{aligned}$$

~~CONFIDENTIAL~~

0376281030

"Residual" Mean Square

$$\begin{aligned} \text{M. S.} &= \frac{\text{"residual" S. S.}}{\text{d.f.}} \\ &= \frac{280,178}{80} \\ &= 3502 \end{aligned}$$

Where

$$\begin{aligned} \text{d.f.} &= (N-1) - (Y-1) - (C-1) - (C-1)(Y-1) \\ &= 95 - 3 - 3 - 9 \\ &= 80 \\ N &= \text{total number of samples} \\ &= 96 \end{aligned}$$

"Between Top and Bottom Within Each Cell" Mean Square

$$\begin{aligned} \text{M. S.} &= \frac{\text{"between top and bottom in each cell" S. S.}}{\text{d.f.}} \\ &= \frac{47,157}{16} \\ &= 2,947 \end{aligned}$$

Where

$$\begin{aligned} \text{d.f.} &= [\text{No. of cells (16)}] [S-1] \\ &= 16 \\ S &= \text{number of sample locations in each ingot} \\ &= 2 \end{aligned}$$

"Between Ingots Within Each Cell" Mean Square

$$\begin{aligned} \text{M. S.} &= \frac{\text{"between ingots within each cell" S. S.}}{\text{d.f.}} \\ &= \frac{184,310}{32} \\ &= 5760 \end{aligned}$$

~~CONFIDENTIAL~~

Where

$$\begin{aligned} \text{d.f.} &= [\text{No. of cells (16)}] [I-1] \\ &= 32 \\ I &= \text{number of ingots in cell} \\ &= 3 \end{aligned}$$

"Unexplained Variation" Mean Square

$$\begin{aligned} \text{M. S.} &= \frac{\text{"experimental error" S. S.}}{\text{d.f.}} \\ &= \frac{48,711}{32} \\ &= 1522 \end{aligned}$$

Where

$$\begin{aligned} \text{d.f.} &= 80 - 16(S-1) - 16(I-1) \\ &= 32 \end{aligned}$$

EVALUATION OF DATA

The data in the analysis of variance table are used to determine the significance of each of the variables — time, condition, and interaction of time and condition — on the carbon content of the ingots. To test the significance of each of these variables, a null hypothesis is set up and tested by using the statistical F-test. The F value computed is the ratio of the mean square of the variable to be tested to a mean square value that includes all variation except the variable being tested.

From the analysis of variance table, it is shown that the mean square values are actual estimates of some combination of components of variation. For example, the "between blocks" mean square is an estimate of  $\sigma_E^2 + 2\sigma_I^2 + 24\sigma_Y^2$ , and the "between ingots within each cell" mean square is an estimate of  $\sigma_E^2 + 2\sigma_I^2$ . The  $\sigma^2$ 's are variation components, with Y referring to blocks, I to ingots, and E to unexplained variation. The coefficients with these components may be thought of as constants for this design. The ratio of M. S. between blocks and M. S. between ingots within each cell is computed.

$$\frac{\text{M. S. between blocks}}{\text{M. S. between ingots within each cell}} \xrightarrow{\text{Estimates}} \frac{\sigma_E^2 + 2\sigma_I^2 + 24\sigma_Y^2}{\sigma_E^2 + 2\sigma_I^2}$$

If there is no effect due to blocks, then  $\sigma_Y^2$  is equal to zero and the ratio is approximately 1. On the other hand, if there is an effect due to blocks, then  $\sigma_Y^2$  has a numerical value and the ratio is

~~CONFIDENTIAL~~

0377291030

equal to some number larger than 1. What the statistical F-test does, then, is to determine whether the computed F value is enough larger than 1 to be a real effect caused by the block variation component rather than a random variation within the experiment.

To perform the statistical F-test, the computed F value is compared with an F value obtained from the F table for 95 per cent confidence at the desired number of degrees of freedom. If the computed ratio exceeds the F value from that table, there is shown to be a significant effect. If the computed value is less than the value from the table, it is shown that the experiment was unable to detect a significant effect.

#### Effect of Interaction Between Blocks and Conditions

Hypothesis: There is no interaction between blocks and conditions.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. for interaction}}{\text{M. S. between ingots within each cell}} \\ &= \frac{8870}{5760} \\ &= 1.54 \end{aligned}$$

F (table) = 2.19 for a ratio which has a numerator with 9 degrees of freedom and a denominator with 32 degrees of freedom

Since  $1.54 < 2.19$ , the hypothesis is not rejected. That is, no interaction effect could be detected.

#### Effect of Blocks

Hypothesis: There is no difference between blocks.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between blocks}}{\text{M. S. between ingots within each cell}} \\ &= \frac{12,440}{5760} \\ &= 2.16 \end{aligned}$$

F (table) = 2.90

Since  $2.16 < 2.90$ , the hypothesis is not rejected. That is, no difference between blocks could be detected.

#### Effect of Conditions

Hypothesis: There is no difference between conditions

$$F \text{ (computed)} = \frac{\text{M. S. between conditions}}{\text{M. S. between ingots within each cell}}$$



~~CONFIDENTIAL~~

$$\begin{aligned}
 &= \frac{8,097}{5,760} \\
 &= 1.41 \\
 F \text{ (table)} &= 2.90
 \end{aligned}$$

Since  $1.41 < 2.90$ , the hypothesis is not rejected. That is, no difference between conditions could be detected.

#### Effect of Top and Bottom Samples Within Each Cell

$$\begin{aligned}
 \text{Hypothesis:} & \quad \text{There is no difference between top and bottom samples within each cell.} \\
 F \text{ (computed)} &= \frac{\text{M. S. between top and bottom samples within each cell}}{\text{M. S. unexplained variation}} \\
 &= \frac{2947}{1522} \\
 &= 1.94 \\
 F \text{ (table)} &= 1.97
 \end{aligned}$$

Since  $1.94 < 1.97$ , the hypothesis is not rejected. That is, no difference between top and bottom samples within each cell could be detected.

#### Effect Between Ingots Within Each Cell

$$\begin{aligned}
 \text{Hypothesis:} & \quad \text{There is no difference between ingots within each cell.} \\
 F \text{ (computed)} &= \frac{\text{M. S. between ingots within each cell}}{\text{M. S. unexplained variation}} \\
 &= \frac{5760}{1522} \\
 &= 3.78 \\
 F \text{ (table)} &= 1.81
 \end{aligned}$$

Since  $3.78 > 1.81$ , the hypothesis is rejected. That is, there is a significant difference between ingots within each cell.

#### COMPUTATION OF MEANS AND CONFIDENCE LIMITS

In general, whenever a statistical F-test is found to give a significant result, the only question this answers is that there is a significant effect due to the variable being tested. The next step is to provide the means with their respective confidence limits in order to further evaluate the

~~CONFIDENTIAL~~

0370001001

data and determine how the variable affects the experiment. The equations used to calculate the means and confidence limits for conditions and cycles are given below.

$$\bar{z} = \text{Mean}$$

$$= \frac{\text{Total of samples}}{\text{Number of samples}}$$

$$\text{Confidence Limits on } \bar{z} \text{ are } \bar{z} \pm \frac{t \cdot s}{\sqrt{N_i}}$$

Where

$$\begin{aligned} \frac{t \cdot s}{\sqrt{N_i}} &= \frac{(2.04) (75.9)}{4.9} \\ &= 31.6 \end{aligned}$$

and  $\bar{z}$  = Mean of individual cycle or condition

$$\begin{aligned} t &= \text{Value from "t" distribution table for 95\% confidence limits} \\ &\quad \text{and 32 degrees of freedom} \\ &= 2.04 \end{aligned}$$

$$\begin{aligned} N_i &= \text{number of values in mean} \\ &= 24 \end{aligned}$$

$$\begin{aligned} s &= \sqrt{\text{"Between ingots within each cell" M. S.}} \\ &= \sqrt{5760}^* \\ &= 75.9 \end{aligned}$$

\* Numbered value applies only to carbon data.

~~CONFIDENTIAL~~

03712281030

~~CONFIDENTIAL~~

## ANALYSIS OF DATA FOR DENSITY

All calculations are similar to those for carbon. Only the results of calculations, the data table (Table IX), and the analysis of variance table (Table X) are shown in this report section. For calculations in this part of the experiment, the data are coded. This is done by dropping the the first two numbers of the density values. Since all of the densities are between 18 and 19 cc, only the figures to the right of the decimal are used. This does not affect the conclusions and simplifies calculations.

TABLE IX

DATA TABLE FOR DENSITY\*

Block	Block Total		Condition											
			Std			A			B			C		
			Top	Bottom	Ingot Total	Top	Bottom	Ingot Total	Top	Bottom	Ingot Total	Top	Bottom	Ingot Total
1			18.91	18.93	1.84	18.94	19.02	1.96	18.90	18.86	1.76	18.87	18.93	1.80
			18.84	18.80	1.64	18.91	18.86	1.77	18.86	18.93	1.79	18.91	18.90	1.81
			18.89	18.91	1.80	18.95	18.88	1.83	18.89	18.88	1.77	18.91	18.92	1.83
		Sub Total	2.64	2.64		2.80	2.76		2.65	2.67		2.69	2.75	
	21.60	Cell Total			5.28			5.56			5.32			5.44
2			18.90	18.90	1.80	18.93	18.88	1.81	18.92	18.94	1.86	18.93	18.93	1.86
			18.91	18.91	1.82	18.92	18.92	1.84	18.94	18.93	1.87	18.94	18.94	1.88
			18.91	18.89	1.80	18.95	18.94	1.89	18.97	18.91	1.88	18.92	18.92	1.84
		Sub Total	2.72	2.70		2.80	2.74		2.83	2.78		2.79	2.79	
	22.15	Cell Total			5.42			5.54			5.61			5.58
3			18.91	18.89	1.80	18.94	18.94	1.88	18.94	18.95	1.89	18.93	18.93	1.86
			18.91	18.92	1.83	18.88	18.91	1.79	18.91	18.94	1.85	18.95	18.93	1.88
			18.89	18.91	1.80	18.92	18.95	1.87	18.94	18.97	1.91	18.92	18.93	1.85
		Sub Total	2.71	2.72		2.74	2.80		2.79	2.86		2.80	2.79	
	22.21	Cell Total			5.43			5.54			5.65			5.59
			18.91	18.92	1.83	18.92	18.92	1.84	18.94	18.92	1.86	18.93	18.93	1.86
			18.91	18.90	1.81	18.93	18.91	1.84	18.93	18.91	1.84	18.95	18.95	1.90
			18.95	18.93	1.88	18.92	18.93	1.85	18.94	18.94	1.88	18.89	18.94	1.93
		Sub Total	2.77	2.75		2.77	2.76		2.81	2.77		2.77	2.82	
	22.22	Cell Total			5.52			5.53			5.58			5.59
TOTALS 88.18			10.84	10.81	21.65	11.11	11.05	22.17	11.08	11.08	22.16	11.05	11.15	22.20

\* All values are g/cc

NOTE: All totals are coded. A constant 18 g/cc is subtracted from each analysis.

~~CONFIDENTIAL~~

TABLE X  
ANALYSIS OF VARIANCE TABLE FOR DENSITY

Source of Variation	Sum of Squares, S.S.	Degrees of Freedom, d. f.	Mean Square, M. S.	Expected M. S.
Between Blocks (Y)	0.0111	3	0.0037	$\sigma_E^2 + 2\sigma_I^2 + 24\sigma_Y^2$
Between Conditions (C)	0.0087	3	0.0029	$\sigma_E^2 + 2\sigma_I^2 + 24\sigma_C^2$
Block and Condition Interaction (YC)	0.0320	9	0.00355	$\sigma_E^2 + 2\sigma_I^2 + 6\sigma_{YC}^2$
Residual	0.0518	80	0.0006475	
Between Top and Bottom Samples Within Cell (S)	0.0042	16	0.000246	$\sigma_E^2 + 3\sigma_S^2$
Between Ingots Within Cell (I)	0.0302	32	0.000944	$\sigma_E^2 + 2\sigma_I^2$
Unexplained Variation (E)	0.0174	32	0.00544	$\sigma_E^2$
TOTAL	0.1036	95		

### EVALUATION OF DATA

#### Effect of Interaction Between Blocks and Conditions

Hypothesis: There is no interaction between blocks and conditions.

$$\begin{aligned}
 F \text{ (computed)} &= \frac{\text{M. S. for interaction}}{\text{M. S. between ingots within each cell}} \\
 &= \frac{0.0355}{0.000944} \\
 &= 3.76
 \end{aligned}$$

$$F \text{ (table)} = 2.19$$

Since  $3.76 > 2.19$ , the hypothesis is rejected. That is, there is a significant interaction effect.

~~CONFIDENTIAL~~

00712291000

Effect of Blocks

Hypothesis: There is no difference between blocks.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between blocks}}{\text{M. S. between ingots within each cell}} \\ &= \frac{0.0037}{0.000944} \\ &= 3.92 \end{aligned}$$

$$F \text{ (table)} = 2.90$$

Since  $3.92 > 2.90$ , the hypothesis is rejected. That is, there is a significant difference between blocks.

Effect of Conditions

Hypothesis: There is no difference between conditions.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between conditions}}{\text{M. S. between ingots within each cell}} \\ &= \frac{0.0029}{0.000944} \\ &= 3.07 \end{aligned}$$

$$F \text{ (table)} = 2.90$$

Since  $3.07 > 2.90$ , the hypothesis is rejected. That is, there is a significant difference between conditions.

Effect Between Top and Bottom Samples Within Each Cell

Hypothesis: There is no difference between top and bottom samples within each cell.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between top and bottom samples within each cell}}{\text{M. S. unexplained variation}} \\ &= \frac{0.000246}{0.000544} \\ &= 0.453 \end{aligned}$$

$$F \text{ (table)} = 1.97$$

Since  $0.453 < 1.97$ , the hypothesis is not rejected. That is, no difference between top and bottom samples could be detected.

~~CONFIDENTIAL~~

Effect Between Ingots Within Each Cell

Hypothesis:        There is no difference between ingots within each cell.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between ingots within each cell}}{\text{M. S. unexplained variation}} \\ &= \frac{0.000944}{0.000544} \\ &= 1.74 \end{aligned}$$

F (table)        = 1.82

Since  $1.74 < 1.82$ , the hypothesis is not rejected. That is, no difference between ingots could be detected.

MEANS AND CONFIDENCE LIMITS

Since conditions, blocks, and interaction between conditions and blocks are all significant, any density values obtained for a condition are dependent on the time cycle in which the ingots are cast. Therefore, the means are reported without confidence limits. The means are comparable, but confidence limits are only appropriate if the block in which the ingots are cast is specified.

~~CONFIDENTIAL~~

0077229.030

## ANALYSIS OF DATA FOR NITROGEN CONTENT

The calculations for nitrogen content are not shown in detail because they are similar to those for carbon content. Table XI is the data table; Table XII is the analysis of variance table.

TABLE XI

DATA TABLE FOR NITROGEN\*

Block	Block Total		Condition											
			Standard			A			B			C		
			Top	Bottom	Ingot Total	Top	Bottom	Ingot Total	Top	Bottom	Ingot Total	Top	Bottom	Ingot Total
1			65	75	140	20	45	65	35	45	80	35	55	90
			70	75	145	20	45	65	65	40	105	30	40	70
			65	70	135	20	50	70	50	20	70	55	55	100
		Sub Total	200	220	xxx	60	140	xxx	150	105	xxx	120	140	xxx
	1135	Cell Total	xxx	xxx	420	xxx	xxx	200	xxx	xxx	255	xxx	xxx	260
2			50	70	120	30	30	60	30	35	65	30	35	65
			65	75	140	25	30	55	80	30	110	50	40	90
			65	75	140	30	50	80	45	20	65	40	35	75
		Sub Total	180	220	xxx	85	110	xxx	155	85	xxx	120	110	xxx
	1065	Cell Total	xxx	xxx	400	xxx	xxx	195	xxx	xxx	240	xxx	xxx	230
3			65	60	125	25	40	65	35	35	70	50	50	100
			50	60	110	10	35	45	20	50	70	45	50	95
			65	75	140	35	25	60	20	25	45	45	35	80
		Sub Total	180	195	xxx	70	100	xxx	75	110	xxx	140	135	xxx
	1005	Cell Total	xxx	xxx	375	xxx	xxx	170	xxx	xxx	185	xxx	xxx	275
4			65	75	140	30	35	65	65	20	85	30	55	85
			80	85	165	45	30	75	45	35	80	65	55	120
			120	90	210	40	35	75	45	30	75	35	40	75
		Sub Total	265	250	xxx	115	100	xxx	155	85	xxx	130	150	xxx
	1250	Cell Total	xxx	xxx	515	xxx	xxx	215	xxx	xxx	240	xxx	xxx	280
TOTALS	4455		825	885	1710	330	450	780	535	385	920	510	535	1045

\* All values are ppm.



~~CONFIDENTIAL~~

TABLE XII  
ANALYSIS OF VARIANCE TABLE FOR NITROGEN

Source of Variation	Sum of Squares, S. S.	Degrees of Freedom, d. f.	Mean Square, M. S.	Expected Mean Square
Between Blocks (Y)	1,384	3	461	$\sigma_E^2 + 2\sigma_I^2 + 24\sigma_Y^2$
Between Conditions (C)	21,215	3	7072	$\sigma_E^2 + 2\sigma_I^2 + 24\sigma_C^2$
Block and Condition Interaction (YC)	1,390	9	154.4	$\sigma_E^2 + 2\sigma_I^2 + 6\sigma_{YC}^2$
Residual	11,996	80	150	
Between Top and Bottom Samples Within Cell (S)	4,096	16	256	$\sigma_E^2 + 3\sigma_S^2$
Between Ingots Within Cell (I)	4,259	32	133	$\sigma_E^2 + 2\sigma_I^2$
Unexplained Variation (E)	3,641	32	114	$\sigma_E^2$
TOTAL	35,985	95		

### EVALUATION OF DATA

#### Effect of Interaction Between Blocks and Conditions

Hypothesis: There is no interaction between blocks and conditions.

$$\begin{aligned}
 F \text{ (computed)} &= \frac{\text{M. S. for interaction}}{\text{M. S. between ingots within each cell}} \\
 &= \frac{154}{133} \\
 &= 1.16
 \end{aligned}$$

$$F \text{ (table)} = 2.19$$

Since  $1.16 < 2.19$ , the hypothesis is not rejected. That is, no interaction effect could be detected.

~~CONFIDENTIAL~~

00712281030

Effect of Blocks

Hypothesis: There is no difference between blocks.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between blocks}}{\text{M. S. between ingots within each cell}} \\ &= \frac{461}{133} \\ &= 3.47 \\ F \text{ (table)} &= 2.90 \end{aligned}$$

Since  $3.47 > 2.90$ , the hypothesis is rejected. That is, there is a significant difference between blocks.

Effect of Conditions

Hypothesis: There is no difference between conditions.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between conditions}}{\text{M. S. between ingots within each cell}} \\ &= \frac{7072}{133} \\ &= 53.2 \\ F \text{ (table)} &= 2.90 \end{aligned}$$

Since  $53.2 > 2.90$ , the hypothesis is rejected. That is, there is a significant difference between conditions.

Effect Between Top and Bottom Samples Within Each Cell

Hypothesis: There is no difference between samples within each cell.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between top and bottom samples within each cell}}{\text{M. S. unexplained variation}} \\ &= \frac{256}{114} \\ &= 2.24 \\ F \text{ (table)} &= 1.97 \end{aligned}$$

Since  $2.24 > 1.97$ , the hypothesis is rejected. That is, there is a significant difference between top and bottom samples within each cell.

~~CONFIDENTIAL~~

Effect Between Ingots Within Each Cell

Hypothesis: There is no difference between ingots within each cell.

$$F \text{ (computed)} = \frac{\text{M. S. between ingots within each cell}}{\text{M. S. unexplained variation}}$$

$$= \frac{133}{114}$$

$$= 1.17$$

$$F \text{ (table)} = 1.81$$

Since  $1.17 < 1.81$ , the hypothesis is not rejected. That is, no difference between ingots could be detected.

MEANS AND CONFIDENCE LIMITS FOR CONDITIONS

$$\frac{t \cdot s}{\sqrt{N_i}} = \frac{(2.04) (11.53)}{4.90}$$

$$= 4.80$$

Where

$$s = \sqrt{133}$$

$$= 11.53$$

$$\sqrt{N_i} = \sqrt{24}$$

$$= 4.90$$

$$t = 2.04$$

~~CONFIDENTIAL~~

0370291031

ANALYSIS OF DATA FOR METAL QUALITY

The metal quality is measured as the per cent of the slugs rejected for seams and striations on the surface of the slugs. The data are shown in Table XIII. The original data collected do not show a normal distribution. (See distribution of the original data in Figure 10.) In order to apply the techniques of variance analysis, it is necessary to transform the data to a more nearly normal distribution. This is done by the arc sin transformation. (See distribution of the transformed data in Figure 11.) The transformation is:  $Y = \text{arc sin } \sqrt{X}$ , where X is the original value in per cent and Y is the transformed value in degrees.

TABLE XIII

DATA TABLE FOR METAL QUALITY  
(% Rejection)

Block	Transformed Block Total		Condition							
			Std		A		B		C	
			Actual	Transform.	Actual	Transform.	Actual	Transform.	Actual	Transform.
1			10.9	19.2	1.8	7.7	8.2	16.6	6.8	15.1
			2.8	9.6	3.7	11.1	17.4	24.6	3.7	11.1
			11.8	20.1	.91	5.4	12.7	20.8	8.2	16.6
	177.9	Cell Total	25.5	48.9	6.4	24.2	38.3	62.0	18.7	42.8
2			27.9	31.9	12.7	20.8	9.9	18.3	8.3	16.7
			25.9	30.6	7.3	15.6	10.8	19.2	13.9	21.9
			29.1	32.6	5.7	13.8	11.7	20.0	9.6	18.0
	259.4	Cell Total	82.9	95.1	25.7	50.2	32.4	57.5	31.8	56.6
3			34.9	36.2	2.8	9.6	7.3	15.6	3.6	10.9
			25.5	30.3	4.5	12.2	15.7	23.3	5.5	13.5
			43.1	41.0	5.6	13.7	4.9	12.8	15.6	23.2
	242.3	Cell Total	103.5	107.5	12.9	35.5	27.9	51.7	24.7	47.6
4			8.7	17.1	10.9	19.2	9.3	17.7	1.9	7.9
			25.2	30.1	15.1	22.8	20.4	26.8	3.6	10.9
			12.6	20.7	13.8	21.8	21.1	27.3	6.3	14.5
	236.8	Cell Total	46.5	67.9	39.8	63.8	50.8	71.8	11.8	33.3
TOTAL	916.4		258.4	319.4	84.8	173.7	149.4	243.0	87.0	180.3

~~CONFIDENTIAL~~

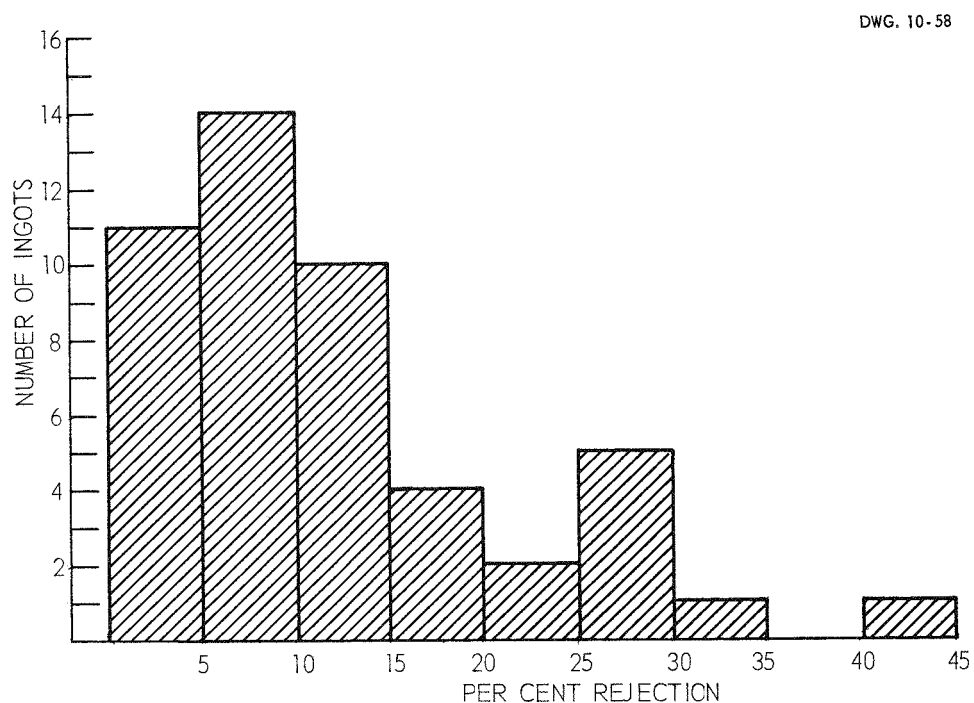


FIGURE 10 Original Data - Frequency Distribution

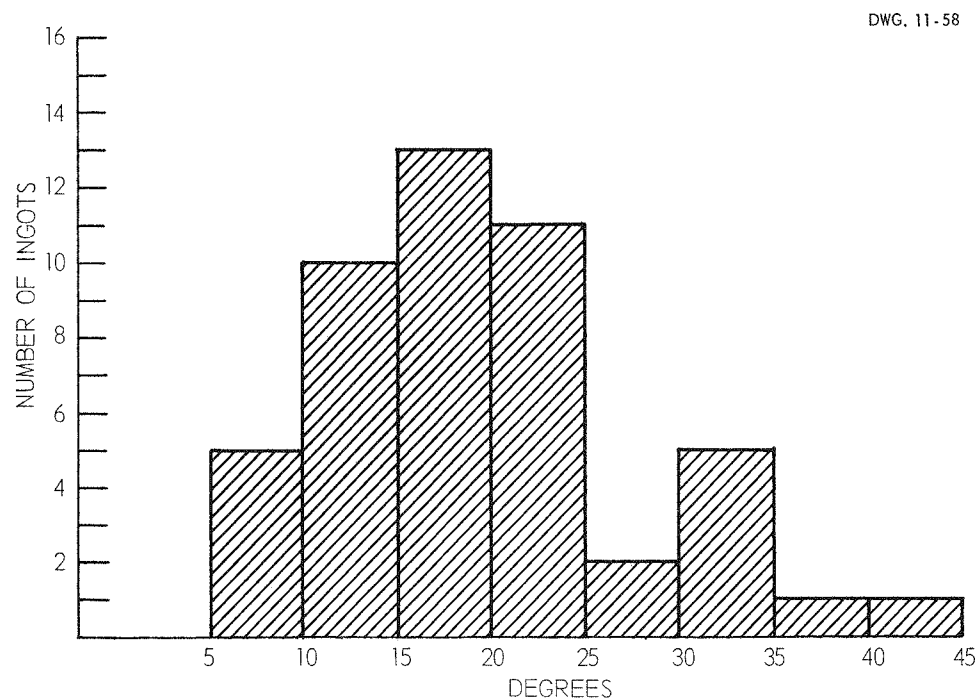


FIGURE 11 Transformed Data - Frequency Distribution

~~CONFIDENTIAL~~

037029.031

The calculations of the sum of squares and mean squares for the analysis of variance table (Table XIV) are similar to those for carbon. However, there is only one sample from each ingot, which simplifies some of the calculations.

TABLE XIV  
TRANSFORMED ANALYSIS OF VARIANCE TABLE FOR METAL QUALITY  
(% Rejection)

Source of Variation	Sum of Squares, S.S.	Degrees of Freedom, d. f.	Mean Square, M.S.	Expected Mean Square
Between Blocks (Y)	314.7	3	104.9	$\sigma_I^2 + 12\sigma_Y^2$
Between Conditions (C)	1149.8	3	383.3	$\sigma_I^2 + 12\sigma_C^2$
Block and Condition Interaction (YC)	174.1	9	19.3	$\sigma_I^2 + 3\sigma_{YC}^2$
Between Ingots Within Cell (I)	1238.1	32	38.7	$\sigma_I^2$
TOTAL	2876.7	47		

## EVALUATION OF DATA

### Effect of Interaction Between Blocks and Conditions

Hypothesis: There is no interaction between blocks and conditions.

$$\begin{aligned}
 F \text{ (computed)} &= \frac{\text{M. S. for interaction}}{\text{M. S. between ingots within each cell}} \\
 &= \frac{19.3}{38.7} \\
 &= 0.498
 \end{aligned}$$

$$F \text{ (table)} = 2.19$$

Since  $0.498 < 2.19$ , the hypothesis is not rejected. That is, no interaction effect could be detected.

~~CONFIDENTIAL~~

### Effect of Blocks

Hypothesis: There is no difference between blocks.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between blocks}}{\text{M. S. between ingots within each cell}} \\ &= \frac{104.9}{38.7} \\ &= 2.71 \end{aligned}$$

$$F \text{ (table)} = 2.90$$

Since  $2.71 < 2.90$ , the hypothesis is not rejected. That is, no significant difference between blocks could be detected.

### Effect of Conditions

Hypothesis: There is no difference between conditions.

$$\begin{aligned} F \text{ (computed)} &= \frac{\text{M. S. between conditions}}{\text{M. S. between ingots within each cell}} \\ &= \frac{383.3}{38.7} \\ &= 9.90 \end{aligned}$$

$$F \text{ (table)} = 2.90$$

Since  $9.90 > 2.90$ , the hypothesis is rejected. That is, there is a significant difference between conditions.

### MEANS AND CONFIDENCE LIMITS

95% Confidence Limits (using transformed data):

$$\begin{aligned} \frac{t \cdot s}{\sqrt{N_i}} &= \frac{(2.04) (5.87)}{3.46} \\ &= 3.46 \end{aligned}$$

Where  $t = 2.04$  for 32 degrees of freedom

$$\begin{aligned} s &= \sqrt{34.4} \\ &= 5.87 \end{aligned}$$

$$\begin{aligned} \sqrt{N_i} &= \sqrt{12} \\ &= 3.46 \end{aligned}$$

The above confidence limits are applied to the means of the transformed data. The means and confidence limits in degrees are then converted back to percent rejects by the relationship of  $Y_{\text{degrees}} = \arcsin \sqrt{X_{\text{percent reject}}}$ .

~~CONFIDENTIAL~~

0372281030