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ORNL-3310
UC-80 – Reactor Technology
TID-4500 (17th ed.)

THE EXPERIMENTAL DESIGN FOR BeO IRRADIATION EXPERIMENTS ORNL 41-8 AND ORNL 41-9

D. A. Gardiner

OAK RIDGE NATIONAL LABORATORY
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UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

Printed in USA. Price $0.75. Available from the

Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

ORNL-3310

Contract No. W-7405-eng-26

MATHEMATICS PANEL
Statistics Section

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Donald A. Gardiner

DATE ISSUED

JUL 18 1962

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ABSTRACT

The experimental plan for irradiating BeO pellets in Experiments ORNL 41-8 and ORNL 41-9 was choser in accordance with the principles of experimental design. The design is known by statisticians as a 2^5 factorial experiment "confounded" in six replications.

Five variables--size, density, grain size, temperature and time--are controlled at two levels to form the basic 2^5 factorial experiment. The sixth variable, neutron flux, is introduced by confounding on higher-order interactions.

This report explains in a nontechnical language the means by which the aims of the experimenters and the physical conditions affecting the experiment were utilized in constructing the experimental design.

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I. INTRODUCTION

BeO Irradiation Experiments ORNL 41-8 and ORNL 41-9 will be performed in the ETR in accordance with a plan chosen by personnel of the Reactor Chemistry Division and of the Statistics Section of the Mathematics Panel at ORNL. This report is a description of the details of this plan.

The purpose of the experiment is to study the effects of six variables on BeO pellets by means of 12 different examinations. 288 pellets of different sizes and compositions will be irradiated under different conditions of neutron flux and temperature and the results will be assessed through the following 12 tests:

(1) chemical analysis
(2) crushing strength
(3) electron microscopy
(4) fabrication history
(5) gas analysis
(6) metallography
(7) physical measurement
(8) petrographic analysis
(9) photography
(10) radiography
(11) thermal conductivity
(12) X-ray analysis.

Available for the experiment is a 3" square lattice position in the ETR. This will be fitted with a core filler piece drilled to accommodate four multicapsule assemblies of four capsules each. (See Figure 1.) The 288 pellets will be stacked within the 16 capsules according to the plan to be described. Figures 2 and 3 are photographs of typical capsules.

Some of the detail of the construction of a multicapsule assembly may be seen in Figure 4, which includes an exploded view of an assembly. The holes in the assembly are for gas tubes and thermocouples which are shown welded into place in Figure 5.

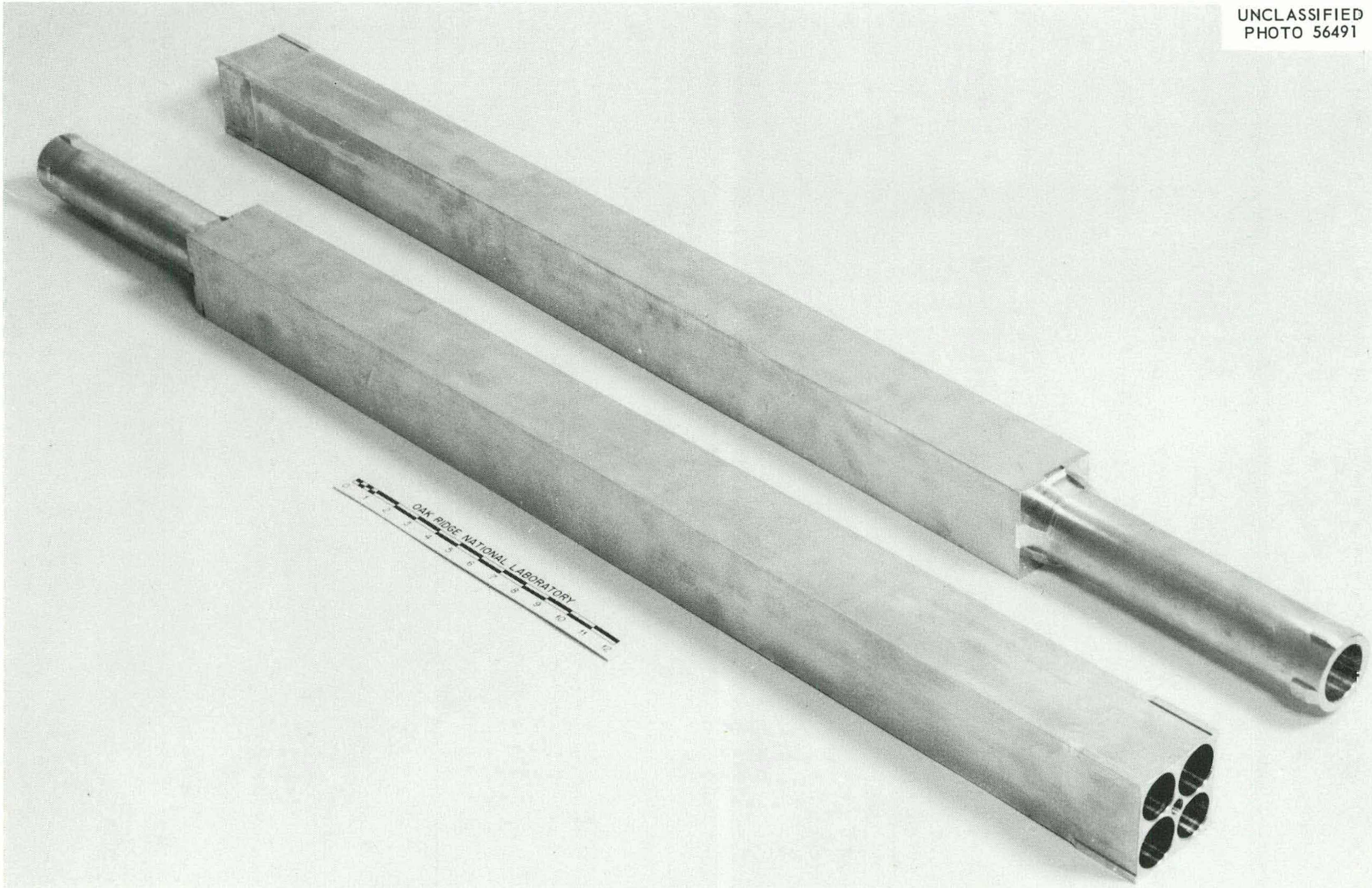

Fig. 1. ORNL 41-8; Core Filler Pieces.

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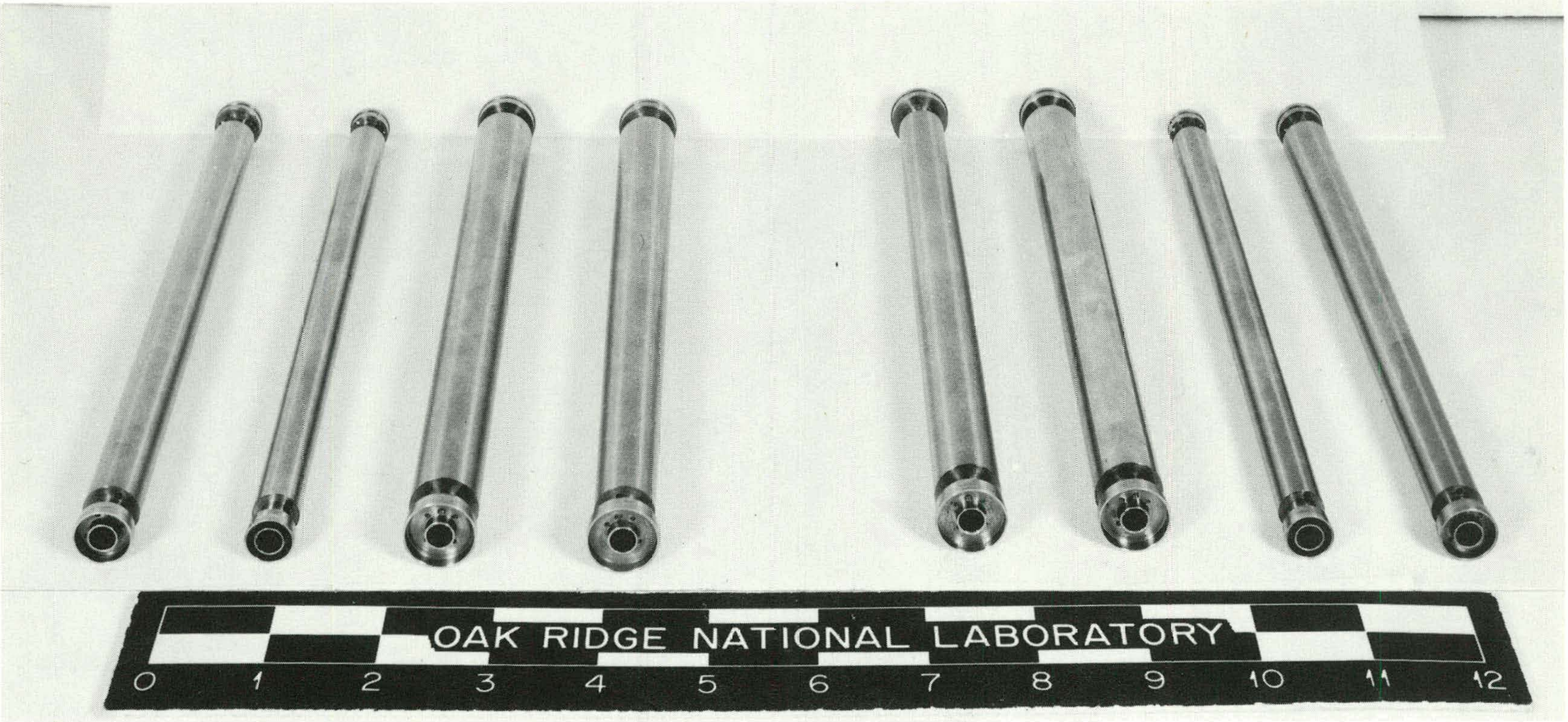
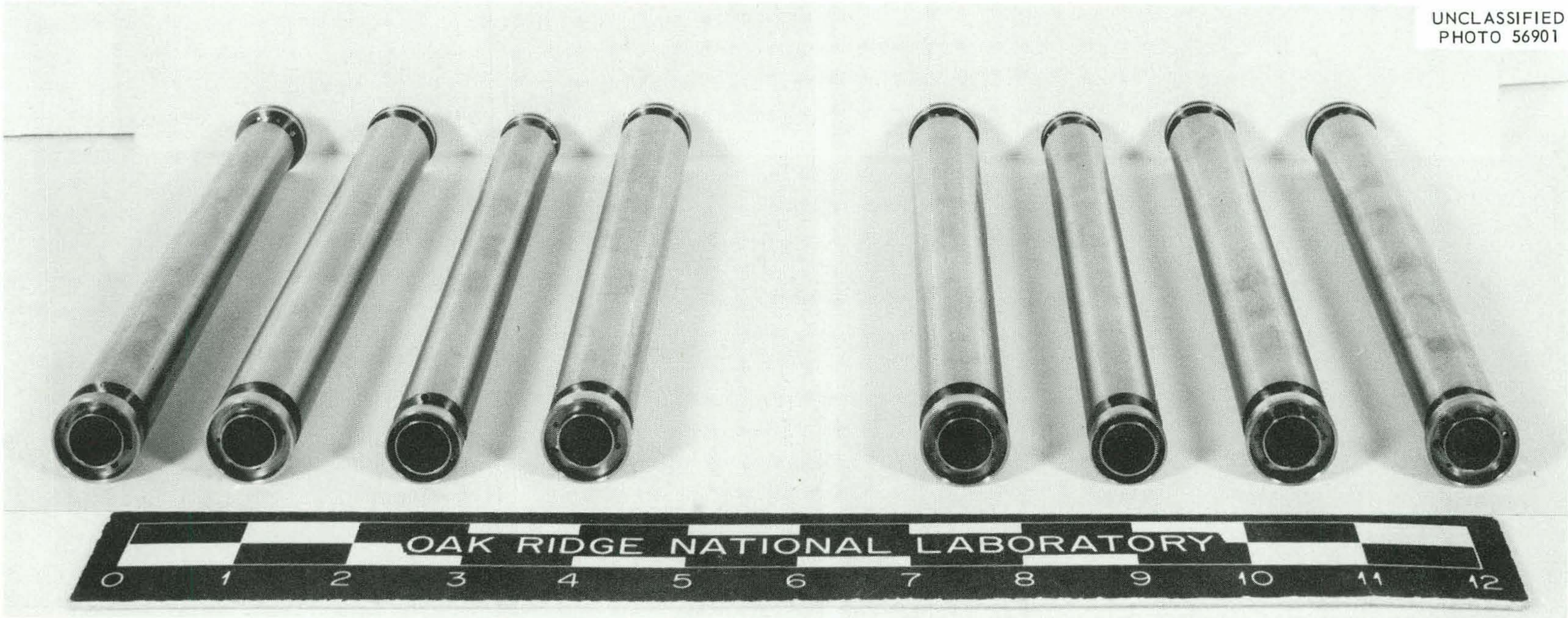


Fig. 2. Capsules for 1/4-in. BeO Samples ORNL 41-8 and -9.



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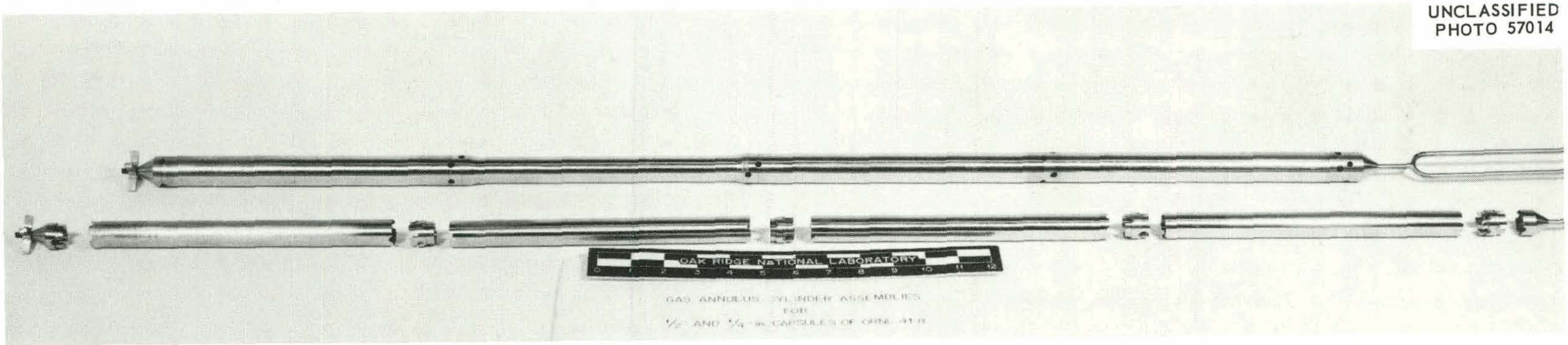

Fig. 4. Typical Multicapsule Assembly Before Fitting with Gas Tubes and Thermocouples.

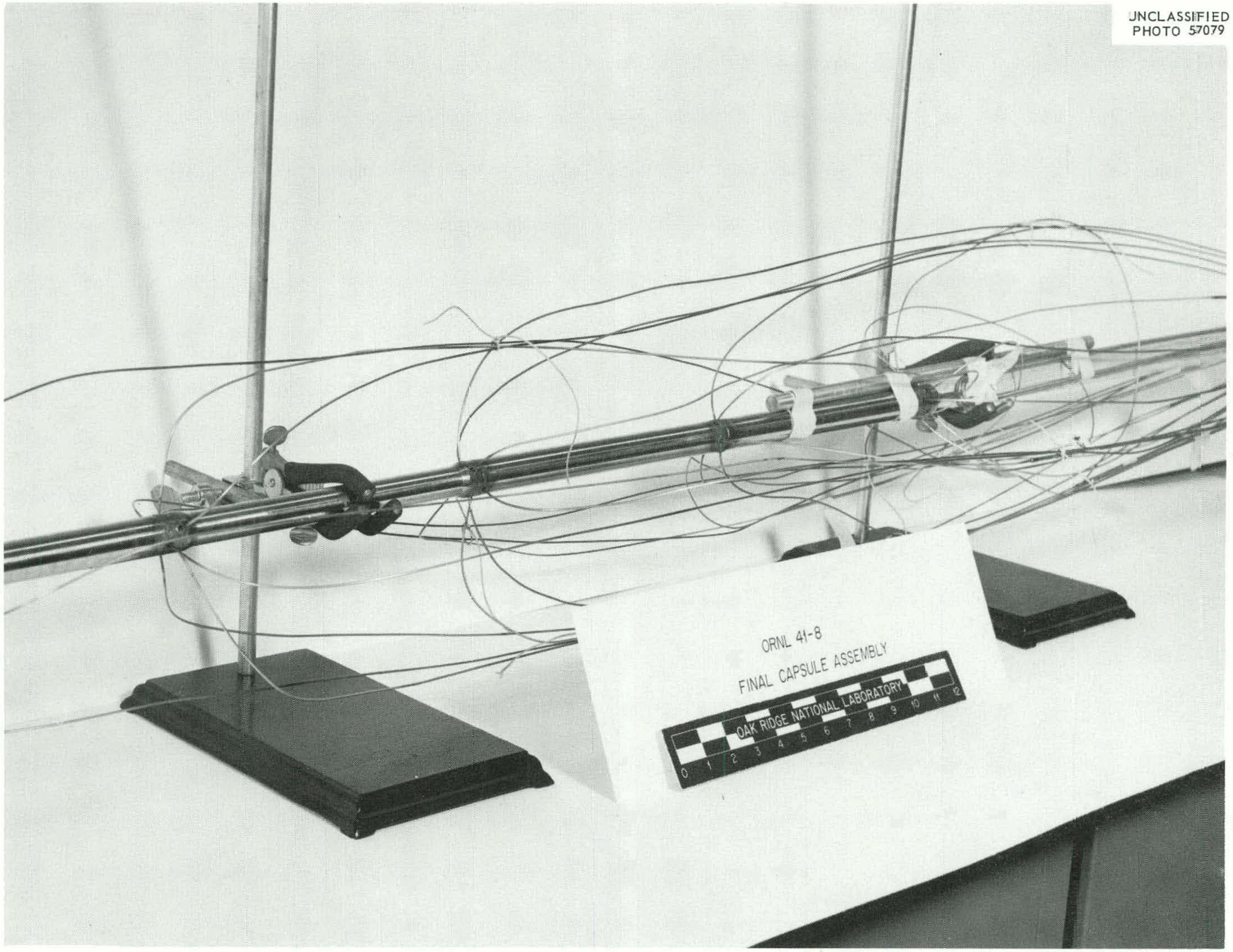

Fig. 5. Typical Multicapsule Assembly.

II. THE DESIGN VARIABLES

The six variables which are to be controlled during the experiment fall naturally into two classes: Fabrication variables, which have to do with how the pellets are made, and treatment variables, which have to do with the treatment the pellets receive during irradiation.

A. Fabrication Variables

There are three fabrication variables: Size of pellet, density of pellet, and size of grain of BeO. They are discussed separately and briefly below.

1. Size

By the name "size" we mean the dimension of the pellet. The pellets are all cylindrical in shape with diameters equal to their heights. Ninety-six of the pellets measure 1/2" by 1/2" (see Figure 6) and 192 pellets measure 1/4" by 1/4" (see Figure 7). The variable, size, is said to be controlled at two levels which we will call 1/4" and 1/2".

2. Density

The density variable will also be controlled at two levels. Some of the pellets are constructed so that their densities are 90 per cent of the theoretical density for BeO. The others are formed so that their densities are 96 per cent of the theoretical density for BeO. We shall use the terms "low" and "high" when referring to the two levels of this variable.

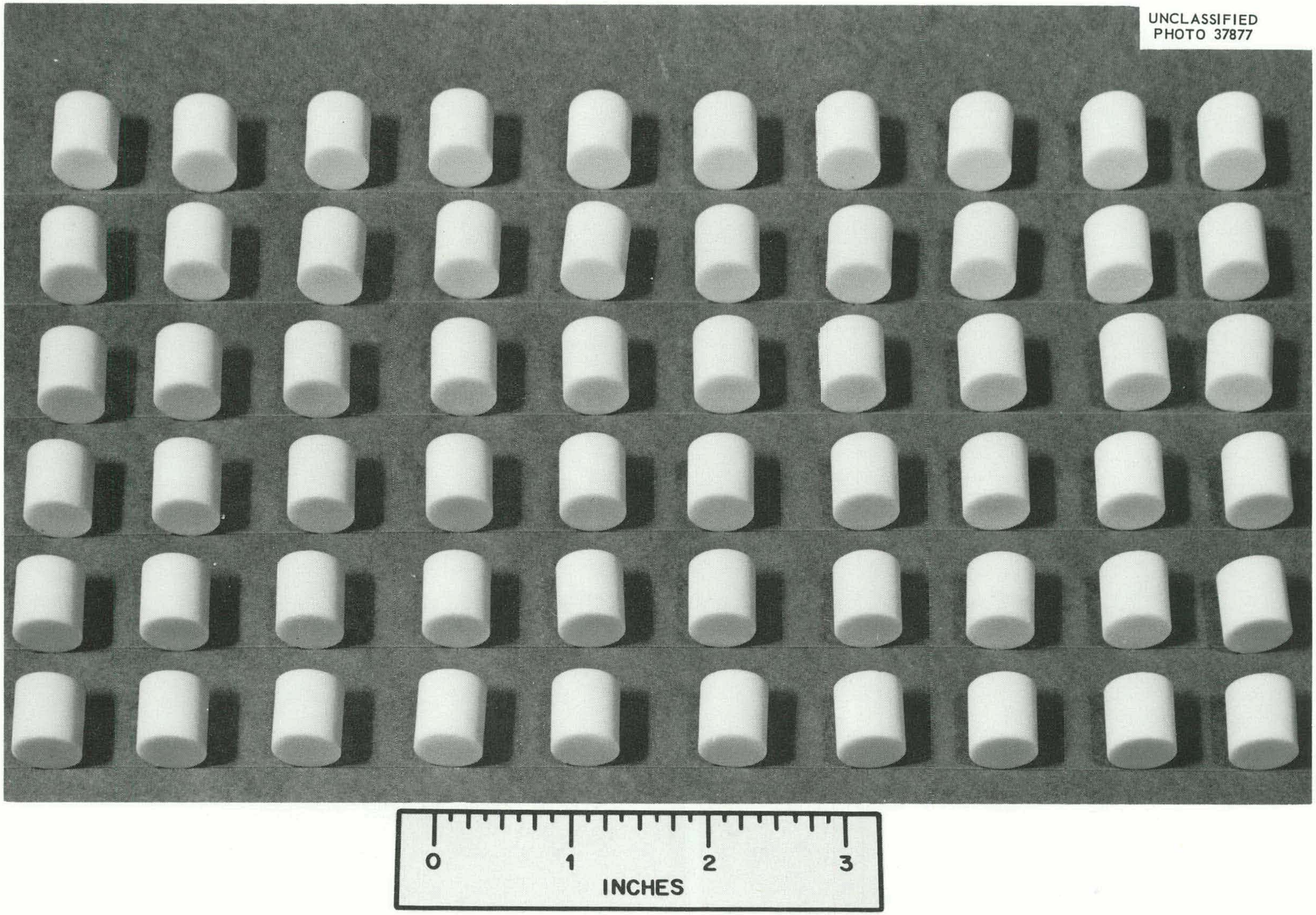

Fig. 6. One-Half-Inch BeO Pellets.

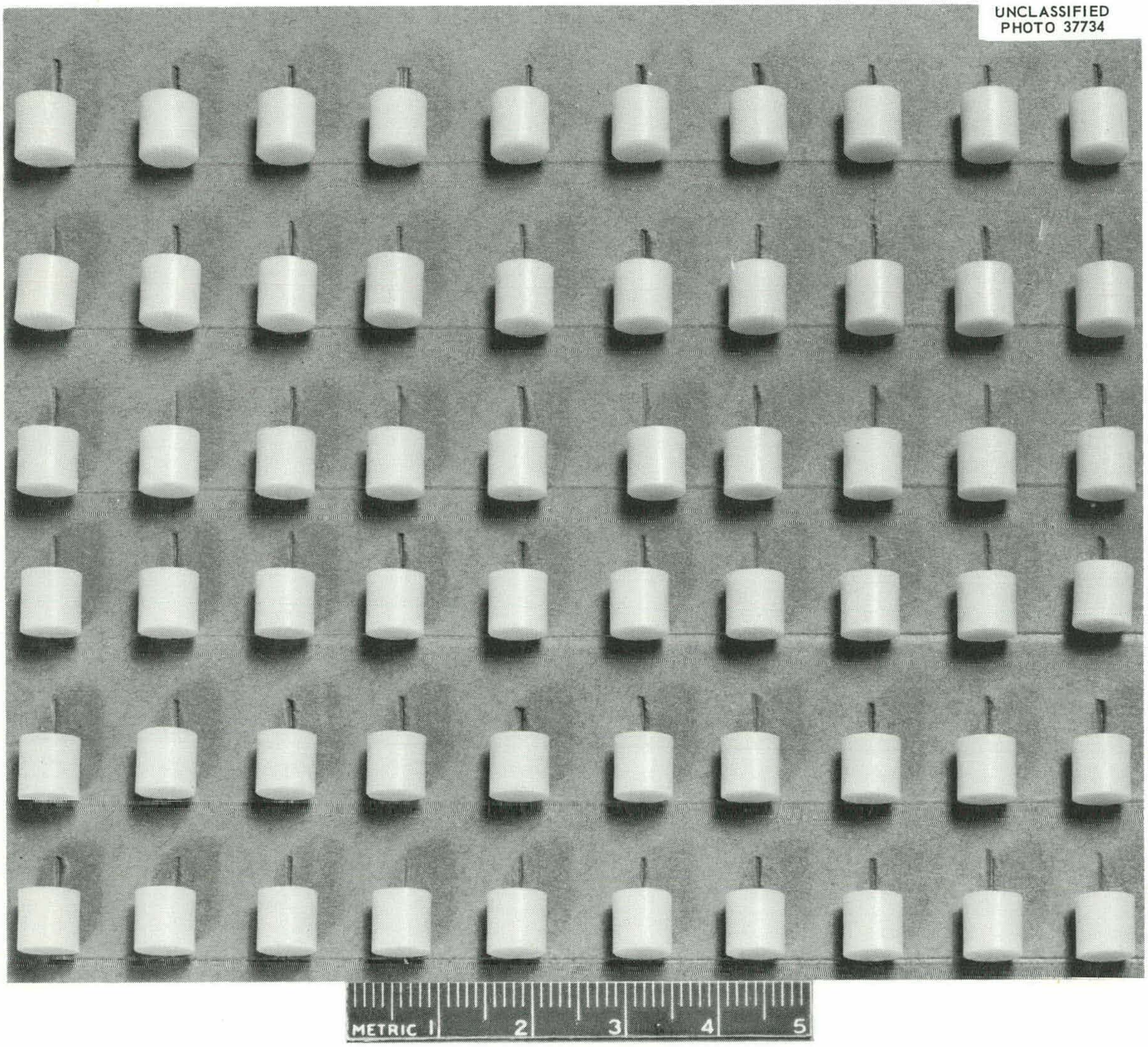

Fig. 7. One-Fourth-Inch BeO Pellets.

3. Grain Size

Some of the pellets used in the experiment will be fabricated with BeO of grain sizes between 15 and 25 microns. The remainder will be formed of BeO of grain sizes between 40 and 60 microns. In referring to the levels of this two-level factor we shall use the words small grain size and large grain size.

B. Treatment Variables

While the pellets are being irradiated their temperatures will be controlled. The pellets will be irradiated for different lengths of time, and they will be subjected to different amounts of neutron flux. Temperature, irradiation time, and neutron flux are the three treatment variables.

1. Temperature

The temperature of the pellets will be controlled by controlling the temperatures of the capsules. This is accomplished by using capsules of different wall thicknesses and by controlling the composition of the helium-argon gas mixture which flows around each capsule. Of the sixteen capsules, eight will be maintained at 650^{o} C and eight will be maintained at 1100^{o} C. Thus, temperature is also a two-level variable. The levels will be denoted by "low" and "high."

2. Time

It is desired that some of the pellets be subjected to a fast neutron dose in the magnitude of 8×10^{20} nvt and that others receive a dose in the order of 30×10^{20} nvt. This will be accomplished by varying the amount of time the pellets remain in the reactor. Two of the four multicapsule assemblies will be removed from the ETR after a number of cycles of operation calculated to yield 8×10^{20} nvt. The other two assemblies will remain in the reactor until their pellets have received a dose of 30×10^{20} nvt.

For the purpose of designing the experiment the controllable variable is time. This, too, will be a two-level variable and we will call the levels short and long.

The 144 pellets which receive the smaller dose comprise Experiment ORNL 41-8. The other 144 pellets comprise Experiment ORNL 41-9. The experimental design, however, treats the combined experiment as one.

3. Neutron Flux

Strictly speaking, neutron flux is not a controllable variable. A certain flux profile will exist for the lattice position assigned to this experiment and the experimental design must be planned with this in mind. However, by exercising control over the placement of the pellets within the capsules, a certain amount of control over neutron flux may be obtained. It is this method which was used in devising the experimental design. The result is that, in effect, neutron flux is controlled at 24 discrete levels.

III. PHYSICAL CONDITIONS AFFECTING THE DESIGN

A. Multicapsule Assemblies

The pellets which are stacked in a capsule of a given multicapsule assembly must be all of the same size. Also the four capsules stacked in a given multicapsule assembly must contain pellets all of the same size. Thus two of the multicapsule assemblies will contain pellets of only the 1/4" size and two will contain pellets of only the 1/2" size.

As was mentioned earlier, the temperature of a capsule may be controlled but the temperatures of pellets may not be controlled individually. The experimental design specifies that the two capsules at the top of a multicapsule assembly be controlled at one temperature and that the two capsules at the bottom be controlled at the other temperature. This pattern of temperatures will alternate from one assembly to the next.

B. Capsules

Each capsule has six inches of effective length. That is, pellets may be stacked in a capsule so that there are twelve 1/2" pellets in a capsule or so that there are twenty-four 1/4" pellets in a capsule. Because the diameter of a pellet varies with its height, pellets may not be intermixed in a capsule. Neither may capsules be intermixed in an assembly.

We define as a <u>basic unit</u> 1/2 inches of the effective length of a capsule. Thus there are 12 basic units to a capsule, 48 basic units to an assembly, and 192 basic units in the complete experiment. It is clear that <u>one</u> 1/2" pellet may be contained in one basic unit and that two 1/4" pellets may be contained in one basic unit. The experimental design is planned in terms of the basic unit.

C. Neutron Flux Gradient

The neutron flux a pellet receives is dependent upon its position in the reactor. For this experiment it has been requested that the flux profile along the length of the multicapsule assembly be approximately symmetrical, with the pellets at the center of the assemblies receiving about twice the flux received by the pellets at the extremes of the assemblies. The profile is expected to be smooth and monotonic from either end toward the center.

In planning the design, the profile is assumed to be symmetric. We may consider the profile to be characterized by 48 discrete amounts of flux, one for each of the basic units in an assembly. With the assumption of symmetry this reduces to 24 distinct fluxes since one half of the profile is a mirror image of the other half.

D. Postirradiation Examinations

It is physically impossible to examine every pellet irradiated, by all of the 12 examinations. Even were it physically possible it would be economically unfeasible. However, it is possible to divide the examinations into six groups, called batteries of tests, and to preassign certain pellets to be examined by a specific battery of tests.

The tests included in a battery will be compatible with one another. That is, it will be possible to examine a pellet by every examination in a battery. The batteries need not be mutually exclusive but they must be exhaustive of the 12 examinations in the set. Hence a given examination may be included in more than one battery of tests and every examination will be included in at least one battery.

IV. THE EXPERIMENTAL DESIGN

Consider for the moment that neutron flux is not a design variable. Then there remain five variables, each to be controlled at two levels. The total number of combinations of two levels of five variables is 2^5. An experimental design which utilizes all these combinations is called a 2^5 factorial experiment. Without replication such an experiment requires 32 basic units.

A. Replication

Since there are six batteries of tests to provide for, each of the 32 factorial combinations must be replicated six times. Then $6 \times 32 = 192$ basic units are required which, fortuitously, is exactly the number of basic units in the complete experiment. The experimental design chosen for this irradiation experiment is a 2^5 factorial experiment, replicated six times and modified as explained below.

B. Blocking

The neutron flux variable is handled by blocking or, more properly, by confounding. Confounding is a device by which confusion is deliberately introduced into an experimental arrangement in order to obtain information on additional factors or variables. Some of the effects which are estimable from an unconfounded factorial experiment may be expected to be negligible. These effects, such as interactions among two, three, or four variables, may be deliberately confused with the effect of an additional variable. Then the estimates obtained from the confused effect are attributed to the new variable.

In ORNL 41-8 and 41-9 neutron flux plays the role of the new variable. It is introduced by dividing the 32 basic units of each replicate into four blocks of eight basic units and assigning a level of neutron flux to each block. This is explained in more detail below.

The 32 basic units in a replicate must be distributed over the four multicapsule assemblies, since each multicapsule assembly accommodates only one of the four size-time combinations. This may be seen in the following table in which the letters A, B, C, D designate the four assemblies

	Size	
Time	1/4"	1/2"
Short	A	B
Long	C	D

Each assembly must therefore contain eight basic units of a replicate. That is, eight of the 32 basic units of a replicate must be put into assembly A, eight others must be put into assembly B, and so on.

Now each position in a multicapsule assembly is subjected to its own degree of neutron flux. A pellet placed in one position will receive a different amount of flux from that received by a pellet placed in another position, unless the positions are equidistant from the center. But even with the assumed symmetric flux pattern it is impossible to distribute the 32 basic units of a replicate over the four multicapsule assemblies (with eight basic units to the assembly) so that each of the 24 different fluxes is represented the same number of times.

However, by using the same positions in all four of the multicapsule assemblies, four distinct fluxes may be represented eight times in a replicate. This is equivalent to dividing the 32 basic units of a replicate into four blocks of eight units each, and assigning a different flux to each block.

When blocking is employed some information is lost. The scheme of blocking chosen for ORNL 41-8 and ORNL 41-9 sacrifices information on some two-variable and three-variable interactions. The information that is lost is not the same information in all the replicates, however. In every replicate, information on the main effects and on the most important two-variable interaction (flux by time, which measures neutron dose) is preserved.

C. Experimental Error

The number of 1/4" pellets in the experiment is twice the number of 1/2" pellets since each basic unit of the 1/4" multicapsule assemblies (A and C) will hold two 1/4" pellets. The design requires that the two 1/4" pellets in a basic unit be identical with respect to grain size and density. Therefore, they will be identical in every respect. These 16 pairs of identically treated 1/4" pellets in each replicate will furnish the measure of experimental error. In addition, the three-variable interactions which have not been lost through blocking may be used as measures of experimental error.

D. The Design Table

The experimental design selected for ORNL 41-8 and ORNL 41-9 is shown in detail in Table I. Each row in Table I represents four basic

TABLE I. THE EXPERIMENTAL DESIGN

Capsule	Position	Multi-Capsule Assembly: A* Time	A* Temp.	A* Size	A* Density	A* Grain Size	B Time	B Temp.	B Size	B Density	B Grain Size	C* Time	C* Temp.	C* Size	C* Density	C* Grain Size	D Time	D Temp.	D Size	D Density	D Grain Size	Replicate	Block in Replicate
1	1	–	–	–	–	+	–	+	+	+	+	+	+	–	–	–	+	–	+	+	–	I	a
	2	–	–	–	–	–	–	+	+	–	+	+	+	–	+	+	+	–	+	+	–	II	a
	3	–	–	–	+	–	–	+	+	–	–	+	+	–	–	–	+	–	+	+	–	III	a
	4	–	–	–	+	+	–	+	+	+	–	+	+	–	+	+	+	–	+	+	–	IV	a
	5	–	–	–	+	–	–	+	+	+	+	+	+	–	–	+	+	–	+	–	–	V	a
	6	–	–	–	–	–	–	+	+	+	–	+	+	–	–	+	+	–	+	+	+	VI	a
	7	–	–	–	+	–	–	+	+	–	–	+	+	–	+	+	+	–	+	–	+	I	b
	8	–	–	–	+	–	–	+	+	+	+	+	+	–	–	+	+	–	+	–	–	II	b
	9	–	–	–	+	+	–	+	+	–	+	+	+	–	–	+	+	–	+	+	+	III	b
	10	–	–	–	–	–	–	+	+	–	+	+	+	–	–	–	+	–	+	–	+	IV	b
	11	–	–	–	+	+	–	+	+	+	–	+	+	–	–	–	+	–	+	–	+	V	b
	12	–	–	–	–	+	–	+	+	+	+	+	+	–	–	–	+	–	+	+	–	VI	b
2	1	–	–	–	+	+	–	+	+	–	+	+	+	–	+	–	+	–	+	–	–	I	c
	2	–	–	–	–	+	–	+	+	–	–	+	+	–	+	–	+	–	+	+	+	II	c
	3	–	–	–	–	+	–	+	+	+	+	+	+	–	+	+	+	–	+	–	+	III	c
	4	–	–	–	–	+	–	+	+	–	–	+	+	–	–	+	+	–	+	–	–	IV	c
	5	–	–	–	–	–	–	+	+	–	–	+	+	–	+	–	+	–	+	+	+	V	c
	6	–	–	–	+	–	–	+	+	–	–	+	+	–	+	+	+	–	+	–	+	VI	c
	7	–	–	–	–	–	–	+	+	+	–	+	+	–	–	+	+	–	+	+	+	I	d
	8	–	–	–	+	+	–	+	+	+	–	+	+	–	–	–	+	–	+	–	+	II	d
	9	–	–	–	–	–	–	+	+	+	–	+	+	–	+	–	+	–	+	–	–	III	d
	10	–	–	–	+	–	–	+	+	+	+	+	+	–	+	–	+	–	+	+	+	IV	d
	11	–	–	–	–	–	–	+	+	–	+	+	+	–	+	+	+	–	+	+	–	V	d
	12	–	–	–	+	+	–	+	+	–	+	+	+	–	+	–	+	–	+	–	–	VI	d
3	1	–	+	–	–	–	–	–	+	+	–	+	–	–	–	+	+	+	+	+	+	VI	d
	2	–	+	–	–	–	–	–	+	+	+	+	–	–	–	+	+	+	+	–	–	V	d
	3	–	+	–	–	+	–	–	+	–	–	+	–	–	–	+	+	+	+	–	–	IV	d
	4	–	+	–	–	+	–	–	+	+	+	+	–	–	+	+	+	+	+	–	+	III	d
	5	–	+	–	–	+	–	–	+	–	–	+	–	–	+	–	+	+	+	+	+	II	d
	6	–	+	–	+	+	–	–	+	–	+	+	–	–	+	–	+	+	+	–	–	I	d
	7	–	+	–	–	+	–	–	+	+	+	+	–	–	–	–	+	+	+	+	–	VI	c
	8	–	+	–	+	+	–	–	+	+	–	+	–	–	–	–	+	+	+	–	+	V	c
	9	–	+	–	+	–	–	–	+	+	+	+	–	–	+	–	+	+	+	+	+	IV	c
	10	–	+	–	–	–	–	–	+	+	–	+	–	–	+	–	+	+	+	–	–	III	c
	11	–	+	–	+	+	–	–	+	+	–	+	–	–	–	–	+	+	+	–	+	II	c
	12	–	+	–	–	–	–	–	+	+	–	+	–	–	–	+	+	+	+	+	+	I	c
4	1	–	+	–	+	–	–	–	+	–	–	+	–	–	+	+	+	+	+	–	+	VI	b
	2	–	+	–	–	+	–	–	+	–	–	+	–	–	+	–	+	+	+	+	+	V	b
	3	–	+	–	+	+	–	–	+	+	–	+	–	–	+	+	+	+	+	+	–	IV	b
	4	–	+	–	+	–	–	–	+	–	–	+	–	–	–	–	+	+	+	+	–	III	b
	5	–	+	–	–	–	–	–	+	–	+	+	–	–	+	+	+	+	+	+	–	II	b
	6	–	+	–	–	+	–	–	+	+	+	+	–	–	–	–	+	+	+	+	–	I	b
	7	–	+	–	+	+	–	–	+	–	+	+	–	–	+	–	+	+	+	–	–	VI	a
	8	–	+	–	–	–	–	–	+	–	+	+	–	–	+	+	+	+	+	+	–	V	a
	9	–	+	–	–	–	–	–	+	–	+	+	–	–	–	–	+	+	+	–	+	IV	a
	10	–	+	–	+	+	–	–	+	–	+	+	–	–	–	+	+	+	+	+	+	III	a
	11	–	+	–	+	–	–	–	+	+	+	+	–	–	–	+	+	+	+	–	–	II	a
	12	–	+	–	+	–	–	–	+	–	–	+	–	–	+	+	+	+	+	–	+	I	a

*Each entry represents 2 pellets

KEY

Symbol	Time	Temp.	Size	Density	Grain Size
–	Short	Low	¼"	90%	12–25 μ
+	Long	High	½"	96%	40–60 μ

units, one basic unit for each multicapsule assembly. The four major headings at the top of the table designate the assemblies, A, B, C, and D.

Within each major heading there are five columns titled Time, Temp., Size, Density, and Grain Size. The +'s and -'s under these headings designate the levels for these five variables according to the key given at the bottom of the table. Thus the basic unit in Position 1 of Capsule 1 of Multicapsule Assembly A will hold two 1/4" pellets of 40 - 60μ grain size in 90 per cent theoretical density. This unit will be maintained at the low temperature and will be irradiated for the shorter time.

At the right of Table I are two columns which identify the replicate, and block within the replicate, to which the four basic units in a row belong. Thus the very first row together with the very last row designate the eight basic units which belong to Block a of Replicate 1. Block b of Replicate 1 is composed of those eight basic units described in the seventh row from the top and in the seventh row from the bottom.

Table I shows, also, the actual positions of the pellets as they are stacked in the capsules and the positions of the capsules as they are stacked within the multicapsule assemblies.

V. STATISTICAL ANALYSIS

There are five different patterns which may be used to block a 2^5 factorial experiment into four blocks of eight basic units each. One of these patterns is undesirable, however, because it would result in sacrificing too much information on the two-factor interaction which measures neutron dose. This pattern was discarded.

All of the remaining four patterns were used. Two were used on two replicates of the design and two were used on one replicate.

A. Analysis by Replicates

The analysis for any experiment which is planned according to statistical principles may be sketched out in advance. A useful means for doing this is a skeleton of an Analysis of Variance Table. Such a table has been constructed and is included in this report as Table 2.

Replications I and VI use one blocking pattern to handle flux; Replications II and V use another blocking pattern; Replication III uses a third pattern and Replication IV a fourth. This may be seen in the headings of Table 2.

The term "degrees of freedom" may be thought of as pieces of information. Since there are 48 pellets in each replicate, there should be 48 pieces of information available in each replicate. These 48 pieces of information are broken up into independent groups by the analysis of variance.

The independent groups are, on the whole, different for each blocking pattern used. However, the patterns selected were those which would yield the same groups of degrees of freedom for all the main

Table 2. Analysis of Variance

Source of Variation	Degrees of Freedom			
	Replicates I and VI	Replicates II and V	Replicate III	Replicate IV
Total	48	48	48	48
Mean	1	1	1	1
Main Effects				
Time (H)	1	1	1	1
Temperature (T)	1	1	1	1
Size (S)	1	1	1	1
Density (D)	1	1	1	1
Grain Size (G)	1	1	1	1
Flux (F)	3	3	3	3
Two-Factor Interactions				
Dose (F × H)	2	2	2	2
F × T	1	2	2	2
F × S	2	1	2	2
F × D	2	2	1	2
F × G	2	2	2	1
H × T	-	1	1	-
H × S	1	-	-	1
H × D	-	1	-	1
H × G	1	-	1	-
T × S	-	-	1	1
T × D	-	-	-	1
T × G	-	1	-	-
S × D	1	-	-	-
S × G	-	-	1	-
D × G	1	1	-	-
Sacrificed	6	6	6	6
Three-Factor Interactions				
Dose × T	-	2	2	-
Dose × S	2	-	-	2
Dose × D	-	2	-	2
Dose × G	2	-	2	-
Experimental Error	16	16	16	16

effects and for the two-factor interaction which measures dose. The first nine lines of Table 2 reflect this.

The other two-factor interactions involving flux ($F \times T$, $F \times S$, $F \times D$, and $F \times G$) are measurable in all the replications but sometimes by one degree of freedom and sometimes by two degrees of freedom. This is shown in the next four lines of Table 2.

The remaining lines show which of the other interactions may be measured and in which replicate. A dash (–) in Table 2 means that there is no information in that replicate for measuring the effect.

B. Joint Analysis of Several Replicates

When a given test is included in more than one battery of examinations, the results will be analyzed by jointly analyzing the replicates which correspond to the batteries in which the test is included. Considerably more information will be obtained in this case.

If a test is to be included in exactly two batteries of tests the two batteries will not correspond to the replicates which employ the same blocking pattern, (for example, Replicates I and VI), for the following reason.

If the two replicates use the same blocking pattern the same pieces of information will be sacrificed twice. However, if the blocking patterns are different, some of the information lost in one replicate will be measurable in the other.

The joint analysis of several replicates is a little more complicated than that for a single replicate and, of course, it depends on which replicates are combined. There is no skeleton of such an analysis included in this report.

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