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QUALITY CONTROL PROGRAM FOR SNAP 10A THERMOELECTRIC ELEMENTS

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CONTENTS

Page

Abstract v

I. Introduction 1
  A. The SNAP 10A System for Nuclear Auxiliary Power . 1
  B. Historical Background 1
  C. Fabrication Process Development 4

II. Quality Control Program 5
  A. Material 5
    1. Procurement 5
    2. Quality Verification 5
    3. Statistical Analyses 8
  B. In-Process Testing 10
    1. Process Control 10
    2. Reference 11
  C. Acceptance Testing 13

III. Conclusions 14

FIGURES

Page

1 Thermoelectric Converter Module 2

2 Typical Quality Verification Control Chart - Seeback Coefficient for "P" Type Pellets. 8

3 Scatter Diagram - Density of "P" Type Elements. . . 10

4 Typical Process Control Chart - Electrical Resistance of "P" Type Contacted Elements 14

5 Historical Control Chart - Resistance of Contacted Elements. 18

TABLES

I Correlation Between Manufacturers and As-Received Data. 11

II Acceptance Levels of In-Process Elements. 15

ABSTRACT

The development and implementation of a complete Quality Control Program for thermoelectric elements is described. The program was set up to aid in the fabrication process development of thermoelectric elements and modules for application to the SNAP (Systems for Nuclear Auxiliary power) 10A.

A brief description and historical sketch of the SNAP 10A is presented. The Quality Control Program of material procurement and quality verification, process control, in-process, and acceptance testing is described in detail. The results of the quality control effort are summarized.

I. INTRODUCTION

A. THE SNAP 10A SYSTEM FOR NUCLEAR AUXILIARY POWER

The goal of the SNAP program is to develop auxiliary power units capable of unattended operation for long periods of time. These systems have application to remote, isolated, or inaccessible places, such as arctic bases, undersea installations, and space vehicles. The specific goal of the SNAP 10A program is to develop an auxiliary power unit which will deliver 500 watts at 28 volts for a one-year period with a maximum weight limitation of 525 lbs.

The system utilizes a compact uranium-zirconium hydride thermal reactor as the heat source. The heat transfer medium, sodium-potassium eutectic alloy (NaK), is heated by the nuclear reactor and circulates to the thermoelectric converter where it releases heat. Useful electricity is produced by the direct conversion of nuclear heat by the thermoelectric elements which comprise the converter.

Many thermoelectric elements of N and P type are contained in modules through which the NaK flows. Each thermoelectric element is contained in a stack of components attached to the NaK tube. These components are an electrical insulator, an electrical conductor which connects adjacent thermoelectric elements, the thermoelectric element itself, and the individual radiator. Each stack is held under compression by a spring attached to a stud which is welded to the NaK tube. The end portion of a typical module is shown in Figure 1.

B. HISTORICAL BACKGROUND

The original SNAP 10 system provided for heat transfer by conductio only. The system required a higher temperature materials capability than actually proved available with existing thermoelectric materials at that time. Consequently, the system was redesigned to transfer heat by circulation.

The redesign, designated SNAP 10A, allowed for a greater radiator area and a consequent decrease in the operating temperature of the thermoelectric materials. At the time of the redesign, a development

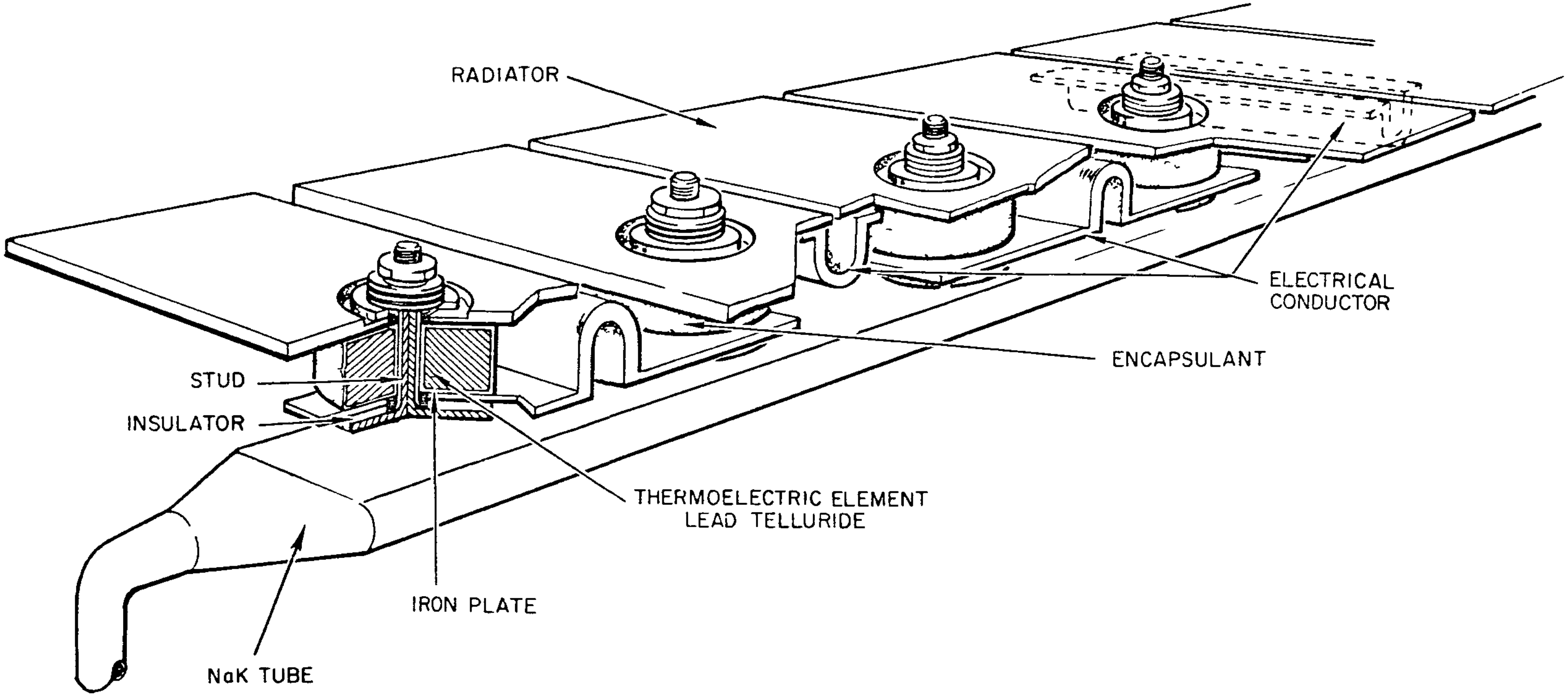

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Figure 1 - Thermoelectric Converter Module

program on thermoelectric materials was started to provide detailed knowledge of thermoelectricity and its related problems.

In the fall of 1960, a small pilot effort was begun. The effort consisted of the investigation of various means of contacting and encapsulating thermoelectric materials such as lead telluride. By the spring of 1961, the problem areas for lead telluride were defined. Due to the extreme tightness of the schedule for the SNAP 10A program, a task force was organized to work on these problems.

The task force was divided into four major areas as follows:

1. Design and Analysis

   This effort consisted of the review of the reference design of the thermoelectric generator and the evaluation of the results from performance and other tests. Alternate and backup designs were developed in case a satisfactory generator of the reference design was not producible.

2. Research and Development

   This effort included the investigation and development of backup and alternate means of fabricating, encapsulating, and assembling thermoelectric elements. A small scale investigation of the use of materials other than lead telluride was also made.

3. Fabrication Development

   This was the largest of the four efforts and was concerned primarily with the development of "reference" processes for contacting, machining, encapsulation, and assembly. This activity also produced the thermoelectric elements for the PSM-3 (Prototype System Mockup Number 3) SNAP 10A assembly.

4. Quality Control and Testing

   The prime responsibility of this effort was in assuring the acceptability of the final product; that is, completed thermoelectric elements and modules. It is with this activity that the major portion of this report is concerned. The effort consisted of the development of test instrumentation[1], the establishment of the test procedures[2], quality verification testing of incoming material, in-process testing, and acceptance testing of completed elements and modules. It also included the reporting, recording, and analysis[3] of test results. Because of the close relationship between the quality control and testing effort and the fabrication process development effort, a short review of the latter follows:

C. FABRICATION PROCESS DEVELOPMENT[4]

A program was set up to develop fabrication and assembly techniques for elements and modules. The major processing steps for the elements were contacting, machining, encapsulation, and module assembly. Other processing steps were used at different times in the program but were subsequently discarded because they were found to be unnecessary.

Because many materials adversely affect the thermoelectric properties of lead telluride, it was necessary to attach a diffusion barrier to the ends of the elements. Steel caps served the dual purpose of providing convenient electrical contacts and diffusion barriers. The contacts were pressed onto thermoelectric pellets by the application of high pressure and high temperature in a reducing atmosphere. Steel was used because it diffuses very slowly into lead telluride and in small amounts does not affect the thermoelectric properties.

After contacting, an axial hole was drilled through the elements. The purpose of the hole was to allow the stud brazed to the NaK tube to extend through the element. A spring fastened to the stud provided the compressive loading which was necessary because of the poor mechanical properties of lead telluride under tension. A design modification introduced during the program eliminated the need for the axial hole. The modification called for the elements to be supported in pairs. In this case, the stud was located between the elements.

The sublimation temperature of lead telluride is below the maximum design operating temperature of the material. In the encapsulation process, elements were coated with an impervious ceramic layer to inhibit this sublimation. This was the final element process prior to assembly into modules.

II. QUALITY CONTROL PROGRAM

A. MATERIAL

1. Procurement

During the pilot phase of the program, the number of pellets handled was quite small. Elements were treated as individuals and records were kept on an individual basis. No material specification existed because there were no specific requirements for the thermoelectric material. A purchasing and handling system was neither necessary nor desirable. As production development began, however, the increase in the number of elements "in-process" necessitated formalization of procedures.

In order to insure a steady supply of consistently high quality materials, the entire procurement system was revised. A material specification was established and all thermoelectric material was purchased to the requirements of the current issue of that specification. The consistency and quality of materials received improved measurably after the specification was adopted.

To facilitate uniformity, entire batches (300 to 400 pellets) of the manufacturers' material were purchased. This eliminated the variability inherent in the production of numerous small batches of material. It also allowed the suppliers to optimize their production methods.

2. Quality Verification

In the development of process techniques and the establishment of process variables, uniformity of input material is a necessity. Because of this need, detailed procedures were set up to verify the quality of incoming material.

Upon receival, manufacturers' containers were checked to see that they were properly identified to indicate the type of pellet, number of pellets in the container, manufacturer, and lot number.

Prior to identification and packaging, each pellet was examined visually for evidence of chips, cracks, excessive porosity or other unsatisfactory characteristics. Unsatisfactory pellets were withheld from processing for possible R and D use.

A receiving inspection system based upon statistical sampling and analysis of test data was established to verify the entire lot of material received in addition to those individual pellets actually tested. Using a table of random digits, the pellets were segregated into groups of twelve pellets each. The groups were referred to as "hot press groups" as this number coincided with the number of pellets that were processed together during contacting ("hot pressing"). Each hot press group of twelve pellets was packaged in a plastic box. They were protected against subsequent damage by nesting them in styrofoam. The entire lot was packaged in this manner except for fifteen pellets for a portion of the quality verification testing. Five each of the fifteen were tested for Seebeck voltage, chemical analysis, and density. Both Seebeck and chemical tests are destructive in nature but the density samples were kept for future reference as "standards" for the particular batch. In addition, three hot press groups or thirty-six pellets were selected for dimensional and electrical resistance quality verification testing.

In general, electrical, density, and dimensional checks were completed prior to processing but the schedule demands were often such that the pellets had to be released before the results of chemical analyses were available. To facilitate rapid analysis, the Shewhart Control Chart[5] technique was used to analyze the data. Charts were made and maintained for dimensions including diameter, parallelism (TIR), and length; density, electrical resistance, Seebeck coefficient, and chemistry (including major constituents and dopants). A typical chart is shown in Figure 2 for the Seebeck coefficient of "P" type pellets. The chart shows statistical control but with a rather high degree of variation. The variation

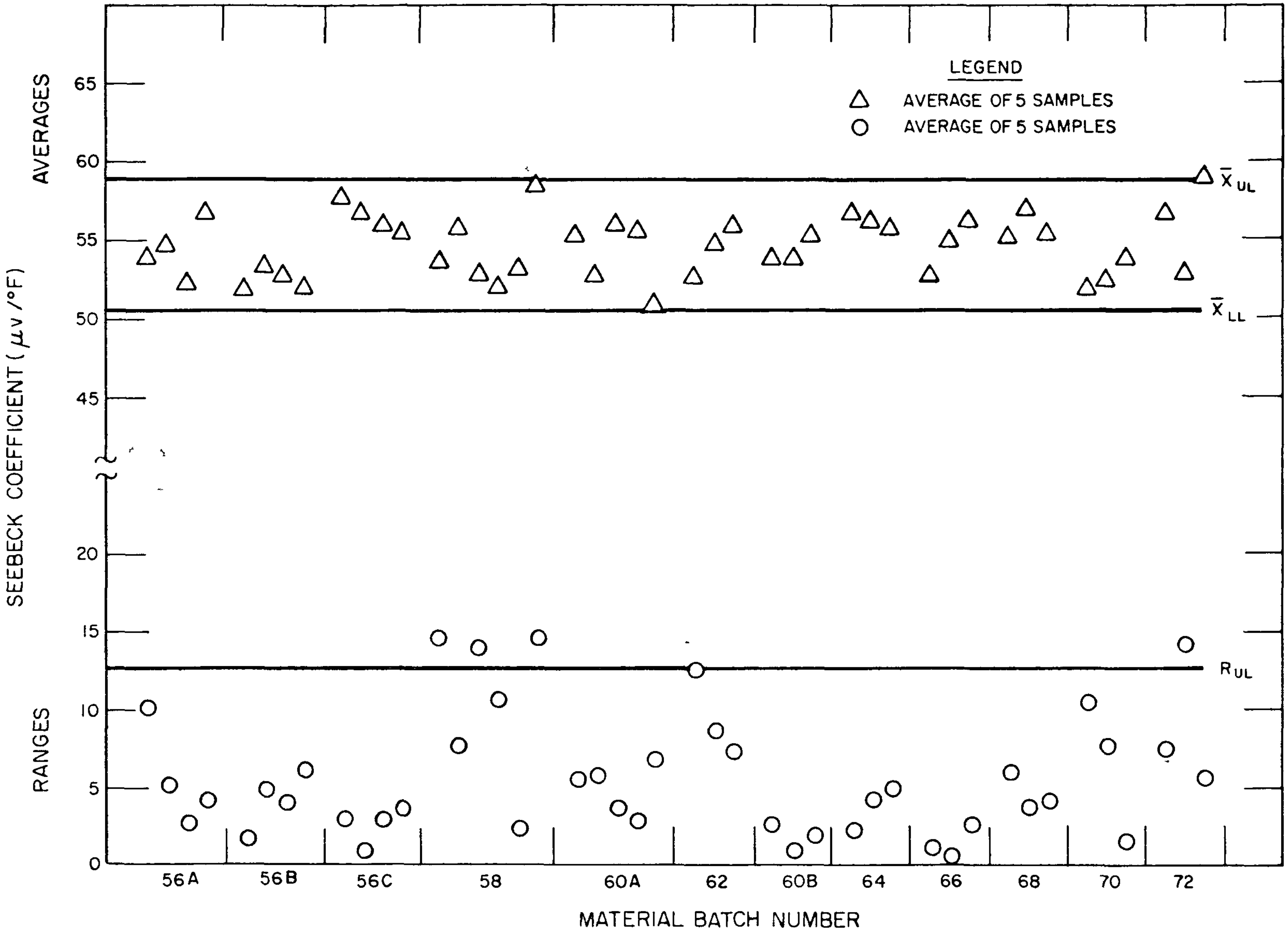

Figure 2 - Quality Verification Control Chart on Seebeck Coefficient
For "P" Type Pellets

may be inherent in the fabrication technique, the measurement technique, other causes, or some combination of causes. In such a situation, the material was accepted. In those cases where some measurement showed a particular lot to be statistically out of control, it was held for recheck or released to processing for R and D or other special purposes.

3. Statistical Analyses

In addition to control charts, other analytical work was performed on incoming data. Statistical analyses were conducted to determine the correlation between measurements made by the manufacturer prior to shipment and the measurements made on receipt of the material. The objective was to determine if shipping had any adverse effects on the pellets and to establish the validity of both sets of measurements.

In general, the samples chosen by AI and by the supplier to represent a given batch were composed of different individual pellets. Therefore, a strong correlation would not only validate the measurement technique but also the sampling technique. Lack of correlation however would show only that something was wrong without identifying the source of the trouble.

The data were analyzed using the standard statistical methods for determining the relationship between two variables. Scatter diagrams such as Figure 3 were plotted for the various parameters. The purpose of the scatter diagram is to discern visually whether there is a pronounced linear relationship between variables.

The degree of relationship is given by the correlation coefficient, r, which always lies between ± 1. Its magnitude indicates the strength of the relationship and the sign indicates whether one variable tends to increase or decrease with the other variable. It should be noted that the correlation coefficient does not indicate in any way the functional relationship between

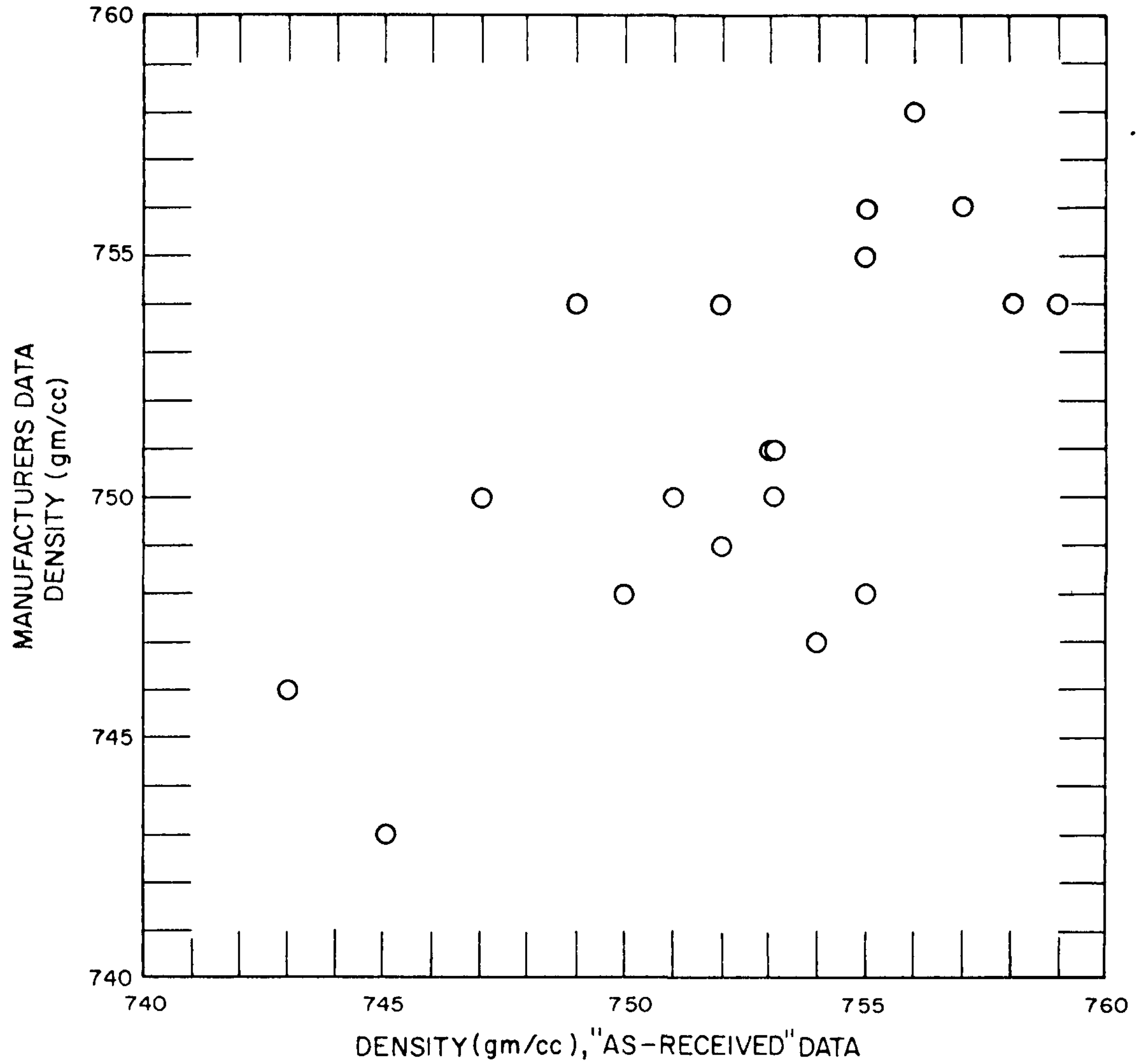

Figure 3 - Scatter Diagram for Density of "P" Type Pellets

variables but only the strength of their relationship. Correlation coefficients found to be statistically significant are given in Table I for the variables considered.

TABLE I

CORRELATION COEFFICIENTS FOR COMPARISON OF MANUFACTURERS AND AS-RECEIVED DATA

Property	Type Pellet	Correlation Coefficient
Diameter	P	0.90
	N	0.97
Length	P	0.69
	N	0.75
Density	P	0.67
	N	0.75
Electrical Resistance	P	0.24 (Not significant at 95% confidence level)
	N	0.63
Chemistry (for all constituents)	P & N	(Not significant at 95% confidence level)

The values in the table show good correlation for dimensional and density data validating the measurement and sampling techniques used. However, this is not so in the case of electrical resistance. The value for P type material indicates no significant correlation. While the value for N elements showed statistical significance, the functional relationship between the variables was not 1:1 as expected.

Correlation coefficients were not significant in the case of chemical data. However since the chemical composition of the thermoelectric elements is important only as it affects the electrical characteristics, no attempt was made to reconcile the differences. Electrical measurements made throughout processing were sufficient to provide control of the elements.

A further investigation was made into the distribution of pellet variables. Two entire batches were measured for electrical resistance, dimensions, and density to see if the distribution of these characteristics was approximately normal. As measured by plots on normal probability graph paper, the distributions approximated normality except for P element resistance.

The non-normal distribution is a possible contribution to the lack of correlation between manufacturers and in-house data for P element resistance. Another contributing factor is the actual method of measurement[1]. The instrumentation was set up primarily for the measurement of elements, i.e., pellets with electrical contacts on the ends. The method depends on a uniform electric current density through the element being measured. This criterion was met in capped (contacted) elements because the caps served to distribute the current. However, in uncapped pellets, such distribution was not guaranteed and the measurement reproducibility and accuracy were compromised. No further work was attempted to reconcile this problem since adequate control of the elements was maintained by one hundred percent testing of electrical resistance of elements after each processing step.

B. IN-PROCESS TESTING

1. Process Control

The purpose of the process control elements was twofold: 1) To determine if the particular batch would respond to fabrication in the same manner as previous batches and 2) to determine the consistency of the fabrication processes.

The process control elements were the first samples processed from each batch. All were tested one hundred percent for dimensions and electrical resistance. Batch acceptability and state of control of the processes were determined by comparison of the data with that obtained from previous batches by means of control charts of the measured quantities.

The nondestructively tested quality verification samples (36) from each batch were reissued as the "process control" samples. After contacting, the elements were identified by hot press run number and date, die cavity letter, and position in the die number. The identification remained with individual process control elements throughout processing. The continuous control of identity was effected by marking the plastic box with the hot press run number, batch number, and date of processing. Die cavity and position in the die were indicated by location of the element in the plastic box: Column for die cavity, row for position in the die. After each inspection or measurement, elements were replaced in the box in the same position from which they had been removed.

2. "Reference" Testing

The bulk of the elements, except those described previously, were referred to as "reference" elements because they were produced by the "reference" or established processes. All reference elements were tested for electrical resistance after each process step. Control charts such as shown in Figure 4 for P type contacted elements were used to ensure the continuing acceptability of both the process and the elements. Reference elements were subject to withholding or rejection if their electrical resistance was too high. Classification values are given in Table II.

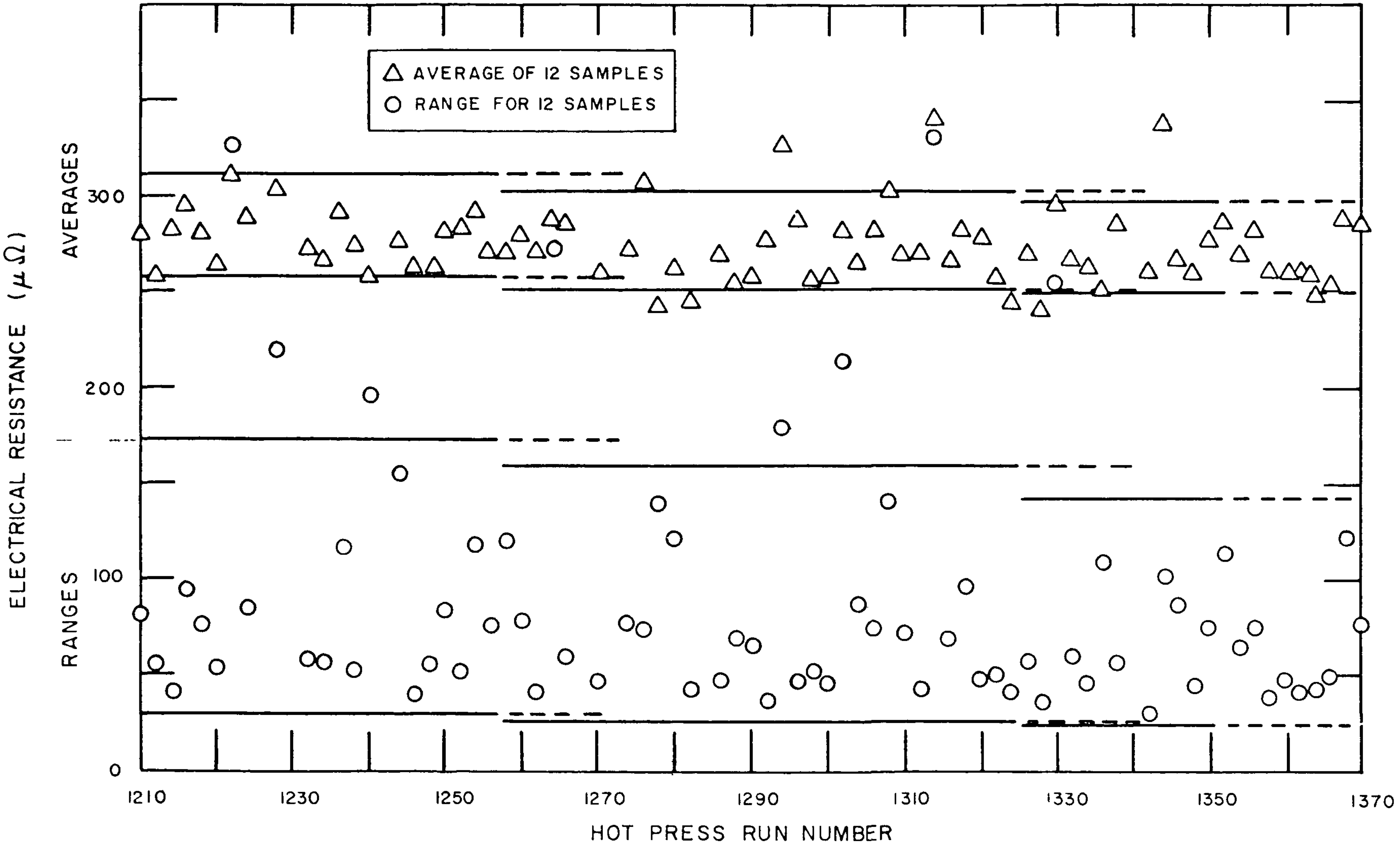

Figure 4 - Process Control Chart for Electrical Resistance of Contacted "P" Type Elements

TABLE II

ELEMENT RESISTANCE CLASSIFICATIONS

Condition of Element	Type of Element	Acceptance Level ($\mu\Omega$)	Withheld Range ($\mu\Omega$)	Rejection Level ($\mu\Omega$)
Contacted	N	150	>150-200	>200
	P	300	>300-400	>400
Machined	N	200	>200-300	>300
	P	400	>400-500	>600
Encapsulated	N	200	>200-300	>300
	P	400	>400-600	>600

After measurement, reference contacted elements were segregated into the three classifications. At this stage of processing, the elements were identified by resistance classification and batch number only. Acceptable elements were released for machining, withheld elements were kept for R and D or other special uses, and rejected elements were stored. A similar procedure was used for reference machined elements.

C. ACCEPTANCE TESTING

The final step in the processing of elements was encapsulation. This process involved the covering of the exposed thermoelectric material surfaces with ceramic coating impervious to PbTe sublimation. In addition to the electrical requirements mentioned previously, encapsulated elements had to meet dimensional requirements and were required to have a continuous encapsulation layer. The procedures for making these various measurements and inspections are given in Reference 2.

Reference and process control elements were subjected to identical tests and were withheld or rejected if they did not meet applicable requirements. Control charts were maintained for electrical resistance and, for documentation, summary sheets were prepared on the encapsulation inspection results for each encapsulation run.

Encapsulated elements were segregated into "accept," "withhold," and "reject" categories. In addition, to facilitate module fabrication, acceptable elements were further segregated into four acceptable classifications according to length. Elements were released for module processing only in groups having the same length classification. This procedure was necessary because of the methods used in module assembly operations.

III. CONCLUSIONS

A major purpose of the Quality Control Program was to systematize activities such as purchasing of material, routing of elements, recording of data, methods of measurement, and reduction and analysis of data. Semiproduction methods and techniques were substituted for R and D techniques in the areas of process control, routine measurement, and data reduction, analysis, and presentation. The effort assisted significantly in the completion of acceptable PSM-3 modules.

The net result of the Quality Control Program is well illustrated in Figure 5; a record of the electrical resistance of contacted elements as a function of time. The chart is a modified control chart of weekly averages with control limits based on monthly averages. The general decrease in resistance and dispersion with time is typical of data obtained at other stages of processing.

The objective of the program was to consistently produce low resistance elements. As evidenced by the steadily increasing yield of acceptable elements, this objective was, in effect, attained. At the close of the program, almost all "N" elements being fabricated were acceptable. The same was true of contacted and machined "P" elements although ~25% were still being withheld after encapsulation. Much of the reduction in resistance was due to the development of optimum processing parameters but a very sizable contribution was provided by the degree of control exercised over process variables, measurement techniques, and other phases of the over-all program.

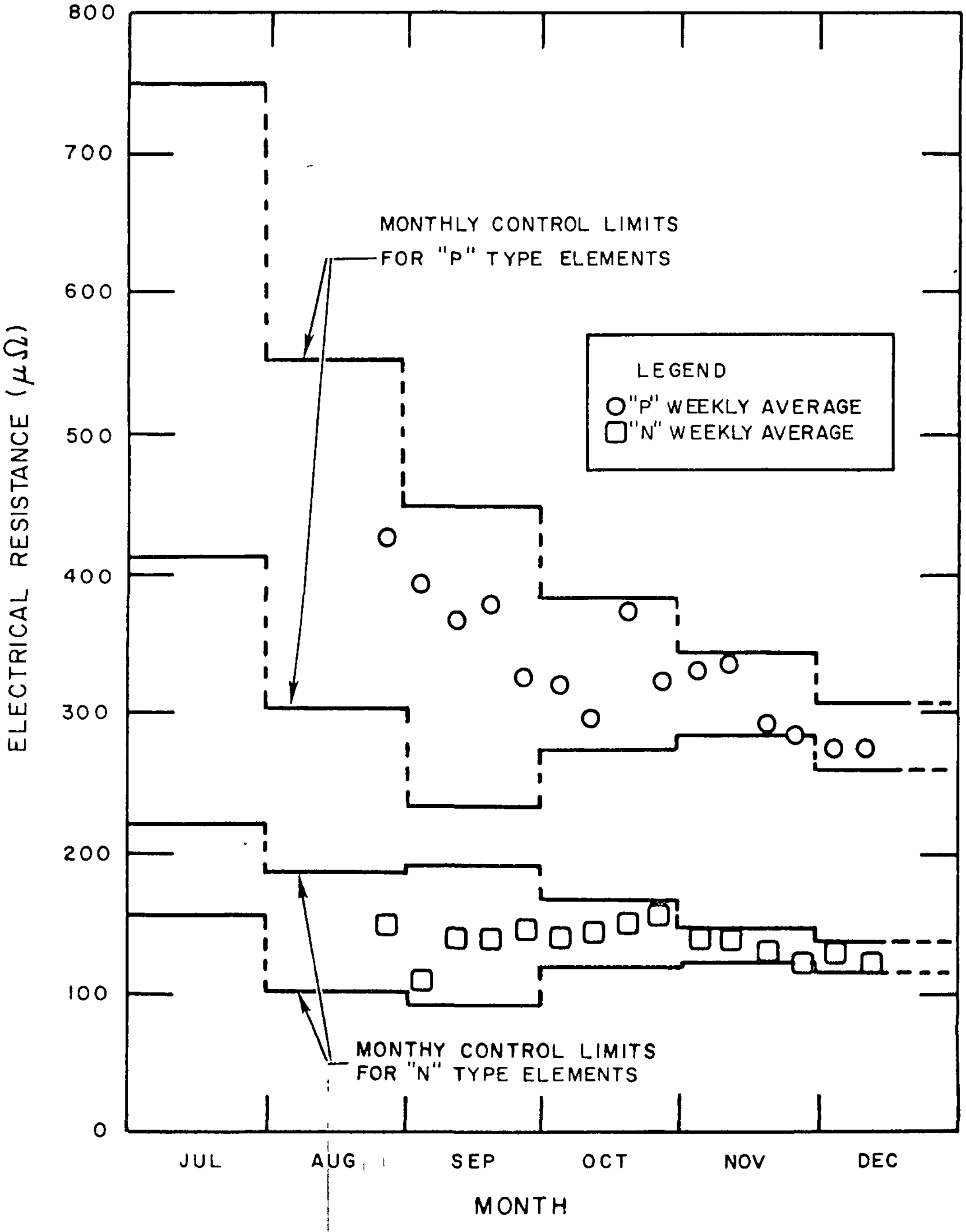

Figure 5 - Historical Control Chart for Electrical Resistance of Contacted Elements

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