Effect of High Temperature
on
A Typical NRD Ni-63 Gas Chromatography Source
Report NC-400-1PT

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Introduction

Beta radiation sources having the form of metal foils plated with metallic Nickel 63 are widely used in the gas chromatographic technique of chemical analysis. Normal and correct application of the GC method and equipment typically involves high temperature exposure for long time periods. The Nickel 63 type of radioactive ionization source is considered to have exceptionally good resistance to change of characteristics when subjected to elevated temperature, but it is of importance to demonstrate this through specific testing.

An earlier series of tests performed by one of the authors of the present study had demonstrated that Nickel 63 sources produced by him at that time did exhibit anticipated temperature resistance. The present series substantially extends the earlier work, while also demonstrating reproducibility of the results.

Purpose of Current Tests

Using a standard NRD Model N1001 Ni-63 chromatography source, to examine the effect of prolonged high temperature exposure on the release rate of Ni-63, and on the available ionization current.

To determine reproducibility of results outlined in our study dealing with Ni-63 sources of earlier manufacture in another laboratory.

Description of Source Tested

NRD Source Model	- N1001
Source Serial Number	- A599
Source Construction	- Nickel metal plated on one surface of gold foil .007" thick
Source Dimensions	- 0.5" x 1.5"
Nickel 63 Content	- 11 millicuries
Pretreatment	- Source fired at 430°C for 15 minutes in argon atmosphere

Test Procedures

1) Initial Room Temperature Tests - Source in form of a flat foil was measured for ion current by means of standard NRD ion chamber. (3" parallel plate chamber with 1" air gap.) It was then formed into cylindrical shape (approximately 1/2" diameter and 1/2" height), and inserted into a concentric design detector cell of the high temperature electron capture type. This cell was then assembled into the equipment layout shown in Figure 1. With dry nitrogen gas flowing at a rate of 100ml/minute, the initial ion current was checked at room temperature. (Ion current data is shown in Graph No. 1.)

2) Exposure Tests at 400° to 450°C.

a. Detector cell was heated and maintained at 400° to 450°C for 500 hours. Cell was purged continuously with dry nitrogen gas at rate of 100 ml/minute throughout this period. Ion current measurements were taken at approximately 100 hour intervals at the operating temperature. (Ion current data is shown in Graph No. 2, showing a typical periodic measurement. A plot of ion current measurements taken at a representative voltage and temperature is shown in Graph No. 3.)

b. Nickel 63 volatilization and radioactive leakage tests were performed at approximately 100 hour intervals during the course of the cell operation described in (a) above. Dry filter paper wipes were taken from inner surfaces of dry trap #1 of Figure 1; aliquots of 1 ml each were taken from wet traps #2 and #3, evaporated to dryness, and measured for activity. (Measurement data is given in Table #1.)

c. After 500 hours of operation, the cell was cooled to room temperature. Ion current measurements were taken as temperature dropped. (Measurement data show in Graph No. 4.) Cell was then disassembled, and its inner and outer surfaces were wipe tested for presence of activity. Nickel 63 source was removed from cell, flattened to its original form, and examined for possible discoloration, flaking, cracking, etc. (Data re wipe tests and surface characteristics shown in Table #2.) Measurements of the flat foil source were made in the standard ionization chamber with 1" air gap as in section (1) of test procedure.

3) Exposure Tests at 500° to 530°C

The Ni-63 source tested above was again formed into cylindrical shape and inserted into the detector cell. With nitrogen gas flowing as before, the ion current was determined at temperature 50°C. (Data given in Graph No. 5.) Cell was then heated and maintained at 500° to 530°C for 92 hours with continual nitrogen purge. At completion of this run, heating was terminated and cell was disassembled. Final leak tests were performed through wiping inner and outer surfaces of cell, as well as inner surfaces of dry and wet traps. (Measurement data given in Table #3.) Final measurements of the flattened foil source were made in the standard ionization chamber with 1" air gap as in section (1) and section (2c) of test procedure. (See Graph No. 6 for comprehensive comparison of results of ionization chamber measurements before and after each high temperature exposure series.)

Discussion

The tests and data obtained provide clear assurance that high temperature exposure of this type of Ni-63 plated source does not result in a detectable release and volatilization of Ni-63 into the environment. It is considered that the detectable but

insignificant beta count rates found on inner cell surfaces after completion of testing are the result of mechanical transfer of removable activity from the source surface during assembly and disassembly operations. In this connection one should recall that the Ni-63 plated foil is considered to be a "bound" source, not a "sealed" source.

Although not of prime interest in the present test series, the appearance of the family of ionization curves in Graph No. 6 deserves comment. The apparent decrease in ionization current after each high temperature series, even though moderate, must be considered as an indication of slight changes occurring in the available activity of the source. Since no active material has migrated from the foil, the pro ability of diffusion of Ni-63 into the substrate remains for investigation. Even though the temperature ranges of the present tests (400°C to 530°C) are considerably higher than in field usage of the Ni-63 type GC cell, this simply suggests an accelerated rate of change as contrasted with the effects which may be experienced in the field during many thousands of hours of use.

Sketch of setup used for high temperature test

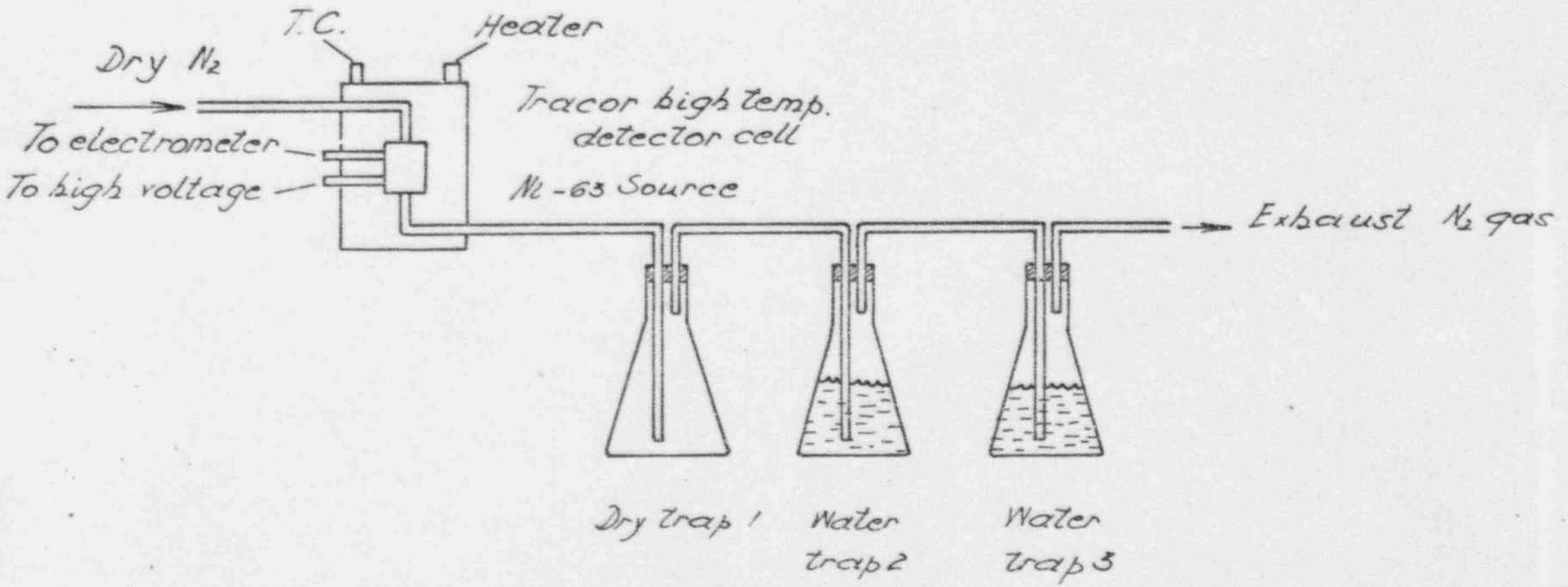

Figure 1.

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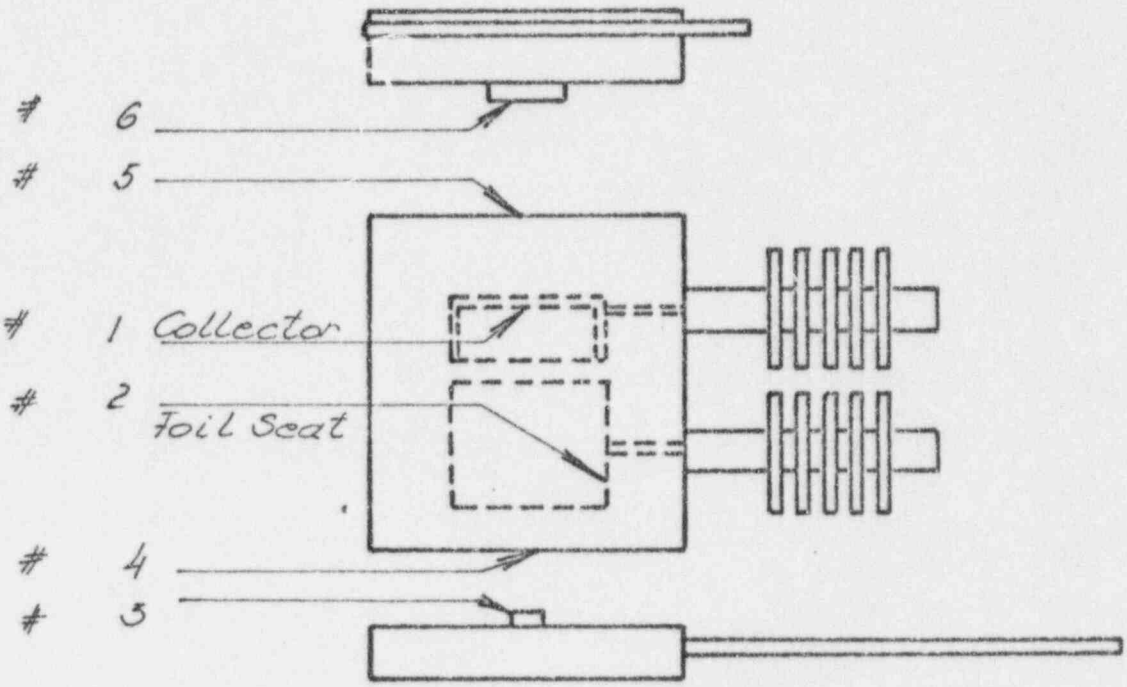


Wipe Take Scheme

Hrs Elapsed	Dry Flask	Flask #2 Aliq.	Flask #3 Aliq.
96	BkG	BkG	BkG
288	-1-	-1-	-1-
432	-1-	-1-	-1-
480	-1-	-1-	-1-
500	-1-	-1-	-1-

Table I

Detector Cell Wipe	Characteristic of Foil cell	Results
No activity on outer cell surfaces has been detected	Discoloration	No
	Cracks	No
	Flaking	No
	Bending	No

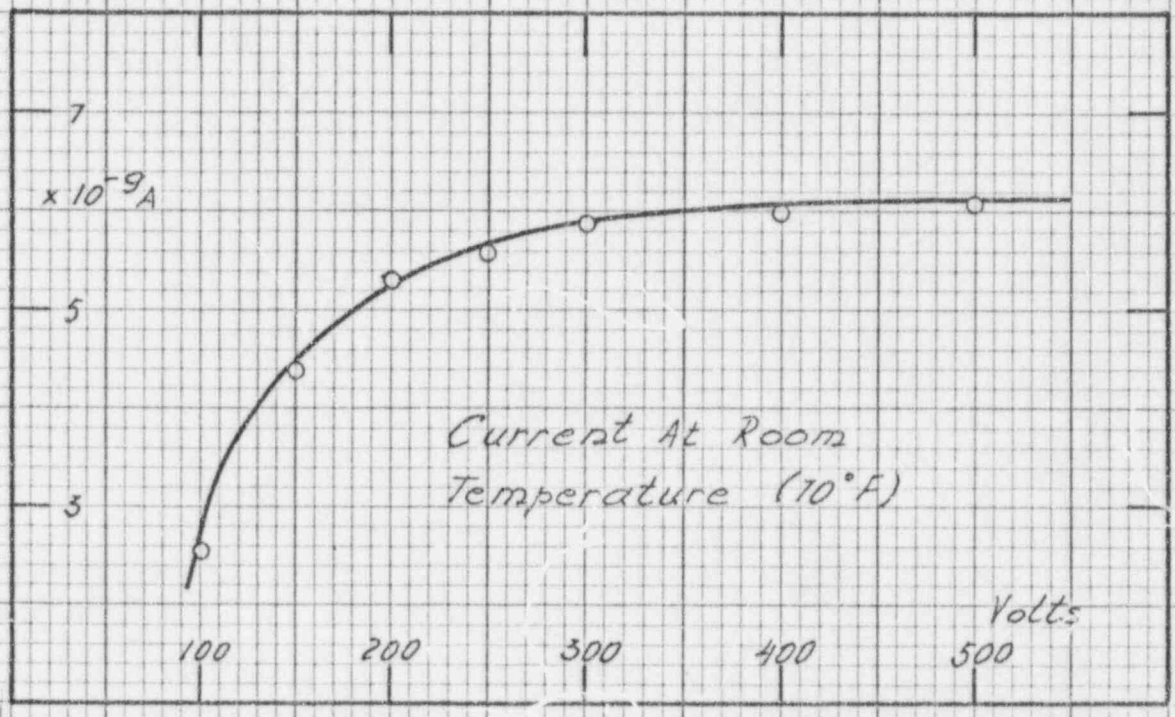
Table II

Evaluation of Detector cell surfaces

Inner Surfaces		Outer Surface	Dry Flask Wipe	Flask #2 Aliq.	Flask #3 Aliq.
Wipe #	Cpm				
1					
2	11.300				
3	21	3kG	BkG	BkG	BkG
4	31				
5	54				
6	5				

Table III

Wipe test evaluation of aparatus after 500-530°C series



Graph N°1

Graph N°2

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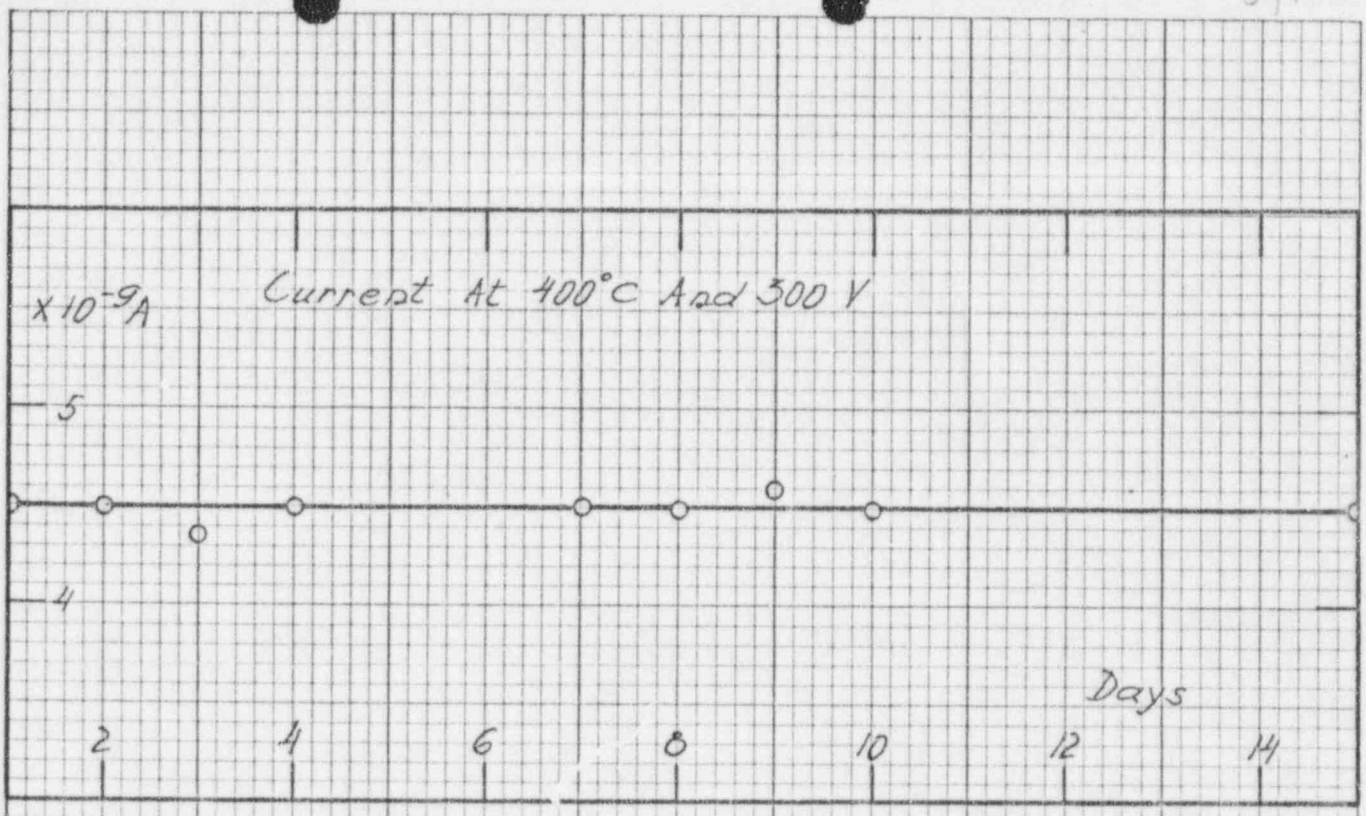
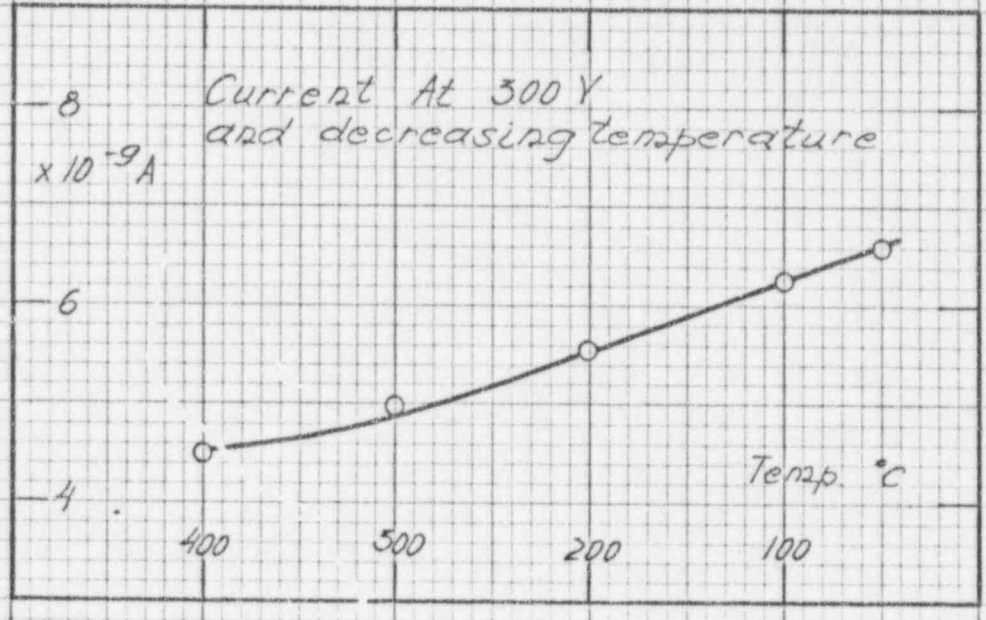


Graph № 3



Graph № 4

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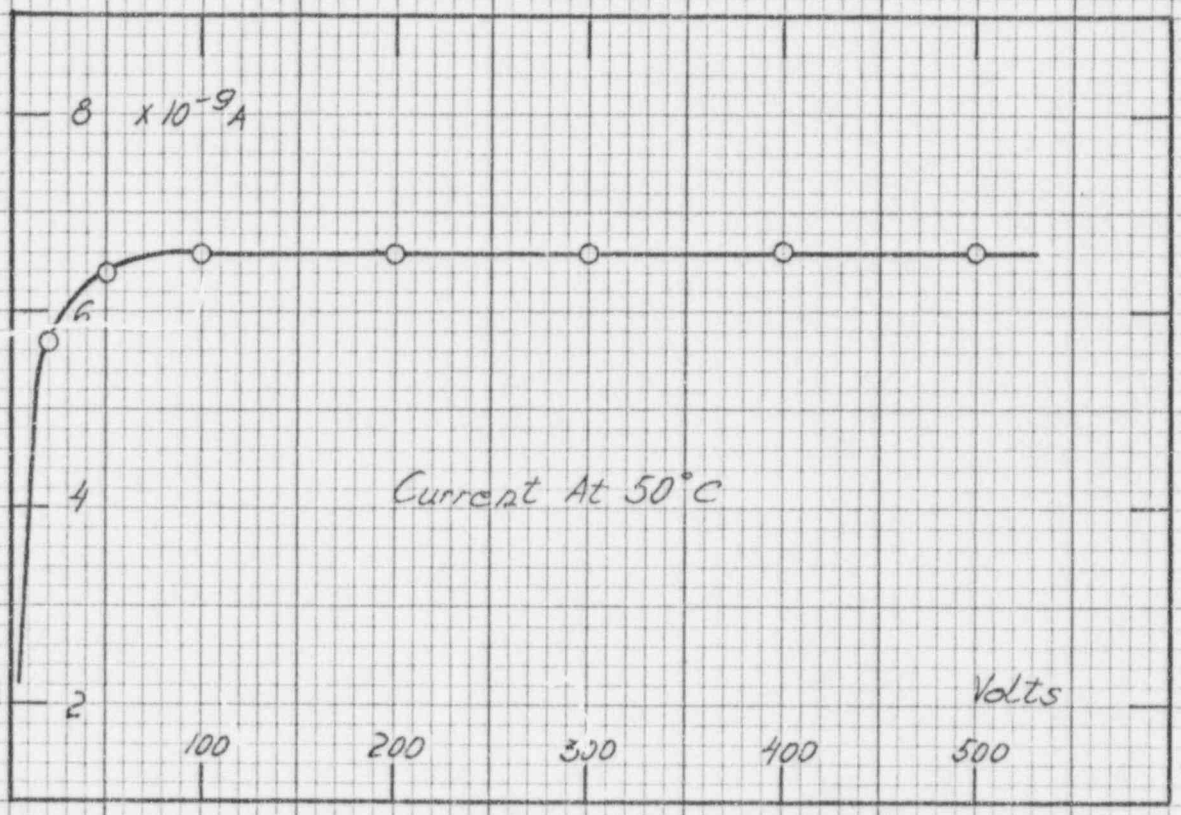
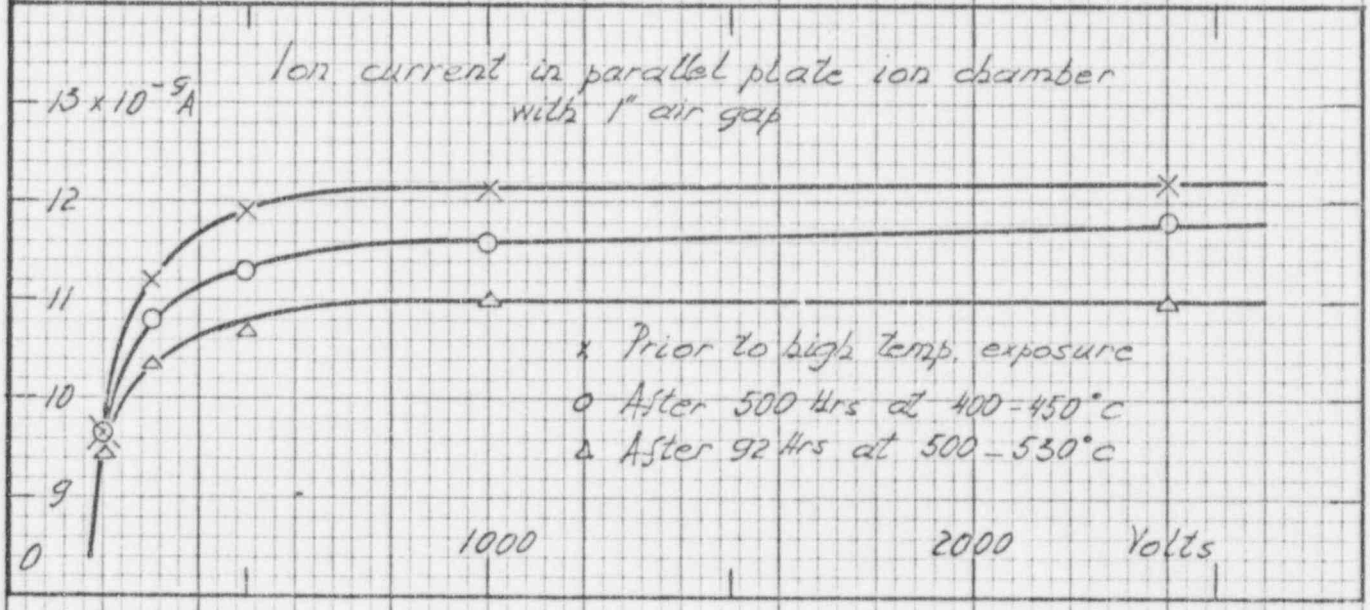


Graph N° 5



Graph N° 6

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NRD

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April 28, 1970

Mr. Matthew Whittier
Hewlett Packard
Avondale Division
Route 41 and Starr Road
Avondale, Penna.

Dear Mr. Whittier:

As promised during our telephone conversation earlier today I am enclosing two copies of the Ni-63 test report. If you should require additional copies please do not hesitate to let me know.

Please accept our sincere apologies for the length of time that it has taken this report to reach you.

Very truly yours,

Robert H. Bull

Robert H. Bull
Manager, Technical Sales

RHB:Me
Encl.