RADIATION - INDUCED CONDUCTIVITY IN POLYETHYLENE

by

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NOMENCLATURE

A, A_γ	empirical constants
a	starting time of gamma-ray pulse
b	ending time of gamma-ray pulse
c	velocity of light in a vacuum, 2.998×10^{10} cm/sec
E_{kin}	electron kinetic energy, MeV
E_γ	gamma-ray energy, MeV
$\overline{E}_\gamma$	average gamma-ray energy, MeV
G_x	test specimen conductance, mhos
h	Planck's constant, 4.1352×10^{-15} eV-sec
I	current, Amperes
k_o	empirical constant
k_i	weighting factor
m	electron rest mass, 9.109×10^{-28} gm
n	integer number
R_x	test specimen resistance, Ohmn
T	temperature, °C
t	time
$U(t)$	unit step function
V_o, V	voltage, volts
Z	atomic number
$\dot{\gamma}$	gamma-ray dose rate, Rad(mat'l)/ unit time
δ	empirical constant
θ	angle of scatter, radians

Nomenclature (cont.)

μ	empirical constant
ν	gamma-ray frequency, sec^{-1}
σ, σ_o	conductivity, mho/cm
τ_o, τ_i	time constants, sec

1.0 INTRODUCTION

Much work[1-5] has been done concerning the volume resistivity of insulators subjected to ionizing radiation. Farmer[1] reported a decrease in resistivity of several orders of magnitude for polystyrene under X radiation. Yahagi and Shinohara[2] have measured the volume conductivity of polyethylene versus temperature and dose rate. Van Lint and Nichols[3] have discussed factors which cause nonlinearities to be present in the response of insulators subjected to ionizing radiation. A good description of current transients in insulators has been presented by Lindmayer[4], and the mechanism of radical decay in irradiated polyethylene has been researched by Geymer and Wegner[5].

The conductivity of insulating materials may be calculated by a direct application of Ohm's law. Basically, the measuring circuit consists of a series combination of a battery, the insulator whose conductivity is to be determined, and a current measuring device, such as an electrometer. The conductance of the insulator is first calculated from a knowledge of the measured current and applied voltage. The conductivity may then be easily determined by knowing the physical dimensions of the insulator.

It is the purpose of this paper to investigate the conductivity of polyethylene, both during and after irradiation by Co-60 gamma rays, and to compare the results with other data in the literature. Other authors have not stated the relative humidity at which their specimens were

conditioned and tested, but only the temperature and dose rate. All three environmental conditions are stated in this paper.

Section 2.0 contains a brief review of the predominant interactions of gamma rays with matter and describes the excitation of electrons in material according to band theory. The experimental procedure is described in Section 3.0, and the results and conclusions are presented in Sections 5.0 and 6.0, respectively.

2.0 THEORY

2.1 Excitation of Electrons by Gamma Rays[6]

Gamma rays interact with matter primarily through three mechanisms: the photoelectric effect, pair production, and Compton scattering.

The first mechanism, the photoelectric effect, is thought of as taking place between the incident gamma ray (or photon) and the entire atom since the participation of the atom as a whole is required. The interaction results in the ejection of an electron (usually from the K or L shell) with a kinetic energy given by

$$E_{kin} = E_{\gamma} - E_{bind} \tag{1}$$

where E_{γ} is the energy of the incident gamma ray and E_{bind} is the binding energy of the orbital electron. The interaction is strongest with the K-shell electrons and for photon energies just above the ionization potential for a given shell.

In pair production, the incident gamma ray disappears and a positron and an electron are produced. This process occurs only when the gamma ray energy exceeds the total restmass energy of the electron-positron pair, i.e., 1.02 MeV. The event must also occur within the Coulomb field of a nucleus. The kinetic energy of the pair is the difference between the gamma-ray energy and the rest energy of the pair, or

$$E_{kin} = E_{\gamma} - 1.02 \text{ MeV}. \tag{2}$$

The positron receives somewhat more than one-half of E_{kin} since it is repelled by the nucleus while the electron is attracted.

The nucleus must be present for pair production since it allows conservation of momentum when the photon transfers its entire energy to

the recoil particles. The probability for pair production to occur increases with the square of the nuclear charge.

In Compton scattering, the incident gamma ray may interact with any one of the orbital electrons which can be considered as free electrons for sufficiently high gamma-ray energies. The mechanism can be considered to be elastic collision between the incident gamma ray and the electron, resulting in a recoil electron and a scattered gamma ray. One can apply the laws of conservation of energy and momentum to the scattering process and solve for the kinetic energy of the recoil electron to obtain[6]

$$\left.\begin{aligned} E_{kin} &= h\nu - h\nu' \\ &= h\nu\left[\frac{(1-\cos\theta)h\nu/mc^2}{1 + (1-\cos\theta)h\nu/mc^2}\right] \end{aligned}\right\} . \qquad (3)$$

In Equation (3), $h\nu'$ is the energy of the scattered gamma ray and θ is the angle of scatter of the primary gamma ray. The probability that such an interaction will occur is proportional to the atomic number Z of the atom.

Figure 1[7] shows the relative importance of the three major types of gamma ray interactions. The photoelectric effect is dominant only for small $h\nu$ and large Z, pair production is of major importance only for large $h\nu$ and large Z, and Compton scattering predominates in the intermediate domain of $h\nu$, for all Z.

In the investigation of radiation - induced conductivity in polyethylene, $(CH_2)_n$, cobalt-60 gamma rays ($\overline{E}_\gamma$ = 1.25 MeV) were used. Since the highest atomic number of the atoms comprising polyethylene is 6, it is seen from Figure 1 that Compton scattering is the predominant interaction mechanism.

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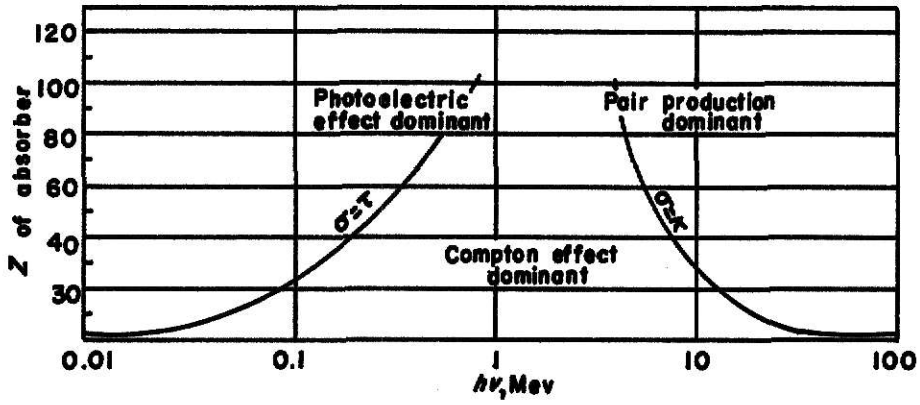

Fig. 1. Relative importance of the three major types of gamma-ray interaction.

It is important to realize, however, that the mechanism used to produce high energy electrons is immaterial as far as the electrons are concerned. The electrons so produced are usually referred to as primary electrons or delta rays and are so energetic that they produce many secondary ionizations as they traverse the material. Delta rays are almost the sole producers of secondary or conduction electrons since the ratio of conduction electrons produced by an incident particle to those produced by the resulting delta rays is about 1 to 10,000.

2.2 Electron Traps in Insulators

The concept of band theory to explain the increase in the conductivity of insulators under ionizing radiation was developed by Fowler[8] in 1955. A condensed review of his theory is presented in this section.

Consider a gamma ray with energy E_γ which is incident upon an insulating material. One of three interactions described in § 2.1 may occur, depending on E_γ and the Z of the material. If Compton scattering is the predominant reaction, the incident gamma ray creates primary

electrons or delta rays along its path. These delta rays have sufficient energy to cause secondary ionization.

If the energy transferred to an electron by a gamma ray (or by any other means) exceeds the binding energy of the electron, the electron goes into the conduction band. Once there, it is no longer bound to its parent atom and can wander freely throughout the material. Such electrons are called free electrons or conduction electrons. Figure 2 illustrates the elevation of an electron into the conduction band as a result of secondary ionization by delta rays.

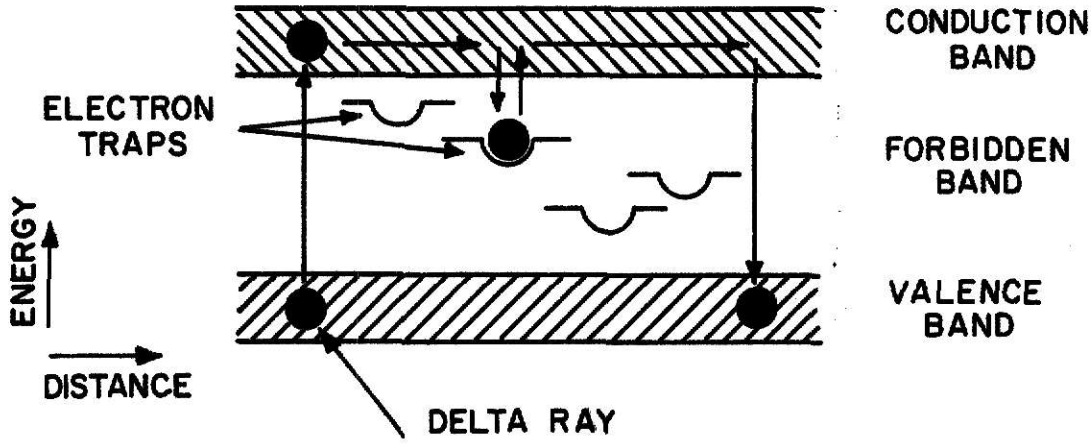

Fig. 2. Secondary ionization produced by the interaction of a delta ray with an electron. [Adapted from Fowler[8].]

Some insulators such as polyethylene (PE) and polytetrafluoroethylene (PTFE), also known as Teflon, contain lattice-like regions at room temperature, i.e., they contain regions of atoms arranged in a lattice-like structure which are interspersed between long carbon chains which have no regular lattice structure (amorphous regions). This causes electron traps

to be located at the edges of the lattice-like regions because of charge imbalances at these locations.

Trap sites are also present inside the pseudo-crystalline regions due to impurities, vacancies, or interstitial atoms. These sites act together with the electron-hole recombination process to reduce the number of conduction electrons in the material. Fowler[8] assumes the traps to be immobile and neglects conduction by holes. He also assumes that the traps have a continuous energy distribution in the forbidden band and that they are in thermal equilibrium with the conduction band so that trapped electrons are thermally released back into the conduction band. The latter assumption gives rise to time constants which may be very long.

As conduction electrons move about in the insulator, some of them collide with phonons (i.e., quanta of lattice vibrational energy). Since the electrons are assumed to be thermalized, their energy may be either increased or decreased by the collision. The electron-phonon collisions are elastic with the probability of gaining energy being the same as the probability of losing energy. Trapping occurs when a conduction electron approaches a recombination center close enough that the force of attraction between the electron and hole overcomes the kinetic energy of the electron. It is held in the trap until it acquires enough energy to leave the trap and return to the conduction band. Fowler[8] has assumed that the energy levels of the traps are continuously distributed within the forbidden band. The width of the forbidden band is at least 5 eV for most insulators.

2.3 Conductivity Response to a Rectangular Pulse of Gamma Radiation

S. E. Harrison et al.[9] have demonstrated that, for a rectangular function of steady-state gamma irradiation between 10^{-3} and 10^{4} Rad (H_2O)*/sec which is applied at time (a) and removed at time (b), the general form of a conductivity versus time curve has distinct features in three time intervals denoted as A, B, and C. Such a curve is shown in Figure 3. Interval A corresponds to the time in which the conductivity is responding to a step increase in radiation intensity but has not yet reached equilibrium. Hanks and Hammond[10] have found that this condition is closely approximated by

$$(\sigma - \sigma_o) = A(1 - e^{-t/\tau_o}) \qquad (4)$$

where σ_o = initial or "dark" conductivity,

σ = conductivity at time t,

A = empirical constant,

and $\tau_o = k_o \dot{\gamma}^{-\mu}$ = the time constant of the response, k_o and μ being empirical constants.

In time interval B, the conductivity has reached an equilibrium value and is characterized to a good approximation by[10]

$$(\sigma - \sigma_o) = A_\gamma \dot{\gamma}^{\delta} \qquad (5)$$

where A_γ and δ = empirical constants

and $\dot{\gamma}$ = gamma exposure rate in Rad (H_2O)/sec.

The equilibrium value for a specific material is determined by the gamma dose rate and the temperature of the material.

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*Rad(H_2O) = 100 ergs of energy absorbed per gram of H_2O = 6.24×10^{13} eV of energy absorbed per gram of H_2O.[9]

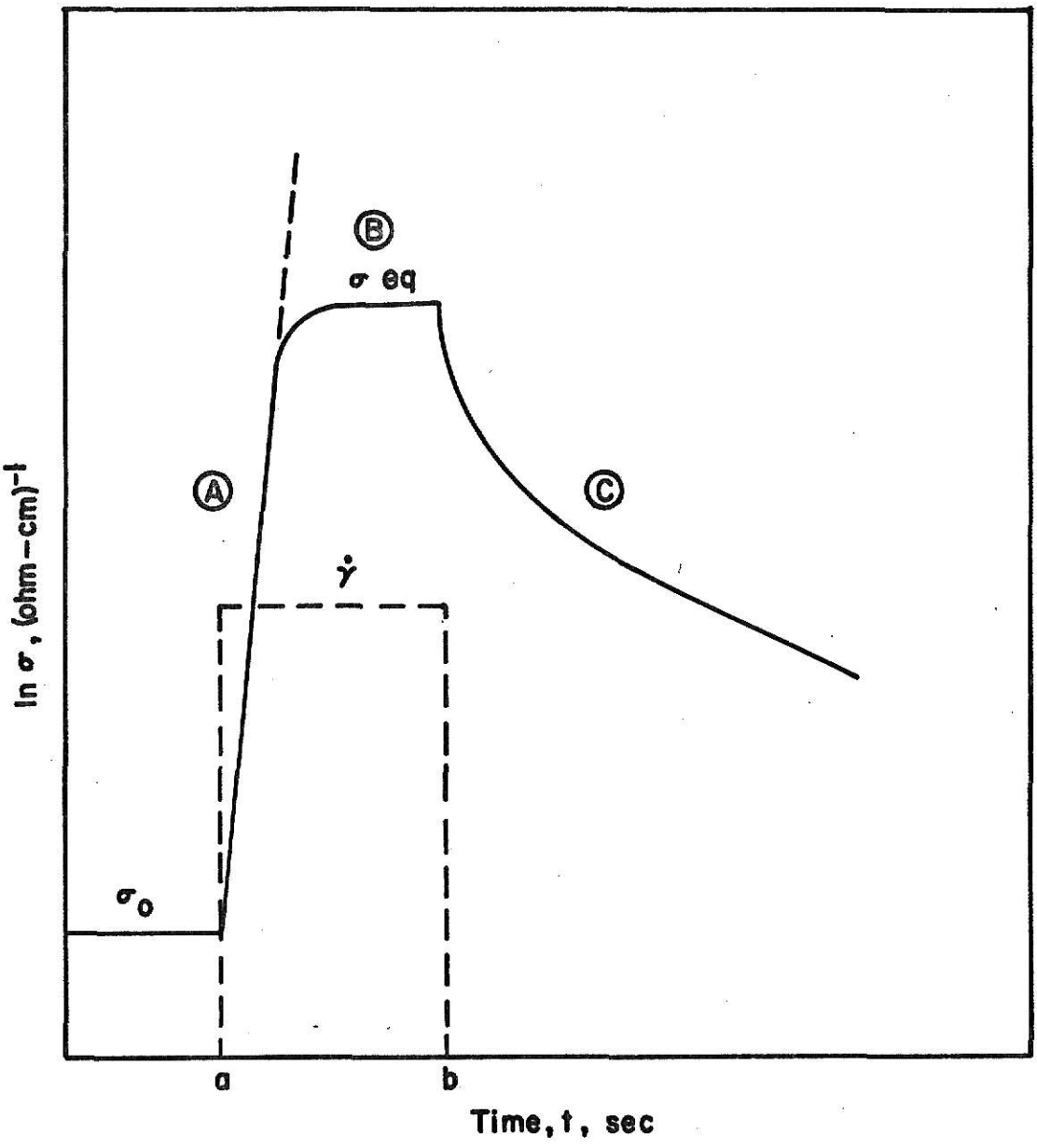

Fig. 3. Typical behavior of conductivity in response to a rectangular pulse of gamma rays. [After Harrison et al.[9]]

Typical values range from 4.0×10^{-18} mho/cm for polystyrene (T = 38°C, $\dot{\gamma}$ = 0.1 Rad (H_2) sec) to 1.2×10^{-13} mho/cm for Teflon (T=38°C, $\dot{\gamma}$ = 1000 Rad (H_2O)/sec).[9]

Interval C shows the recovery of the conductivity after the material is removed from the gamma ray field. This recovery is well approximated by[10]

$$(\sigma - \sigma_o) = A_\gamma \dot{\gamma}^{\delta} \sum_{i=1}^{n} k_i e^{-t/\tau_i} \qquad (6)$$

where n = the number of discrete decay time constants in the recovery process,

τ_i = the decay time constants of the recovery,

and k_i = a weighting factor associated with the ith τ_i.

Hanks and Hammond[10] have also combined the "unit step function," U(t), with Equations (4), (5), and (6) to yield the following generalized equation at a constant temperature and gamma dose rate:

$$\begin{aligned} \sigma(t,\dot{\gamma})\Big|_{\text{constant } T,\ \dot{\gamma}} &= U(t)\sigma_o + U(t-a)\{A_\gamma \dot{\gamma}^{\delta} \times [1 - e^{-(t-a)/\tau_o}]\} \\ &+ U(t-b) \times \{(\sigma_o + A_\gamma \dot{\gamma}^{\delta}) \sum_{i=1}^{n} k_i e^{-(t-b)/\tau_i} \\ &- [\sigma_o + A_\gamma \dot{\gamma}^{\delta}(1 - e^{-(t-a)/\tau_o})]\} . \end{aligned} \qquad (7)$$

Harrison et al.[9] have shown that Equation (7) holds for polyethylene, polystyrene, and other organic polymers.

3.0 EXPERIMENTAL PROCEDURE

Procedures outlined in the ASTM Standards Manual[11a-d] were followed in cleaning and conditioning the polyethylene samples. A testing procedure suggested by Winslow[12] was used to control transients in the measured current. Since the specimen preparation and testing procedures are rather lengthy, only a brief summary of them are presented in this section. Appendix I lists the procedures in detail.

Specimens 1-15/16 inch in diameter were cut from 0.060 inch polyethylene sheet stock. A short length of aluminum pipe which had one end machined to a sharp edge was used to cut the specimens. The cutting process resembled that of a cookie cutter. The specimens were then cleaned by washing them in distilled water and alcohol and drying them with a lint-free cloth. An oven was used to dry the specimens for four hours at 50°C after which they were conditioned for 40 hours at constant relative humidity in a glove box. The temperature and relative humidity within the glove box were periodically checked using wet and dry bulb thermometers. Next, the specimens were removed from the glove box and electrodes were painted on them by hand using silver conductive paint. The electrodes consisted of a high voltage electrode, a low voltage electrode, and a guard ring surrounding the low voltage electrode. The paint was allowed to dry at room temperature for 15 minutes.

Specimen holders were fabricated in accordance with Reference (11a). Two thicknesses of polyethylene were put together to form a test specimen. After insertion of the test specimen, the holder was completely coated with paraffin several times to prevent the loss of moisture from the specimen. Paraffin was used because it has been found to be a very

effective potting compound.[20] The specimen holder was put in an aluminum testing box which provided electrical shielding of the test specimen. This testing box was positioned inside the drawer of a Model 220 Gammacell and was connected to the measuring circuit by coaxial cables. Figure 4 shows the specimen holder and the testing box while Fig. 5 shows the positioning of the testing box in the drawer of the gammacell.

The measuring circuit consists simply of a power supply, the polyethylene specimen whose resistance is to be determined, a linear voltage amplifier, a RC low-pass filter to eliminate high frequency noise in the output of the amplifier, and a digital voltmeter. Figure 6 is a block diagram of the circuit. A schematic diagram of the testing circuit is shown in Fig. 7. The actual electronic equipment used appears in Fig. 8. The amplifier, filter, and voltmeter were used due to the unavailability of an electrometer. The electronic equipment was energized well in advance before the start of a test to allow it to become thermally stable. Also, the amplifier and voltmeter were zeroed before the start of each test.

The current versus voltage tests were run with the specimen in the radiation field of the gammacell following the procedure suggested by Winslow[12]. A voltage V_o was applied to the specimen for 5 minutes with no data being taken during this time. At t=5 min, the polarity of V_o was reversed and data were taken 5 minutes and 10 minutes after the reversal. At t=15 min, the polarity of V_o was again reversed and data were taken at 5 minutes and 10 minutes after the second inversion. The applied voltage was then increased and the above test was rerun. The range from 0V to 1000V was covered using 200V increments.

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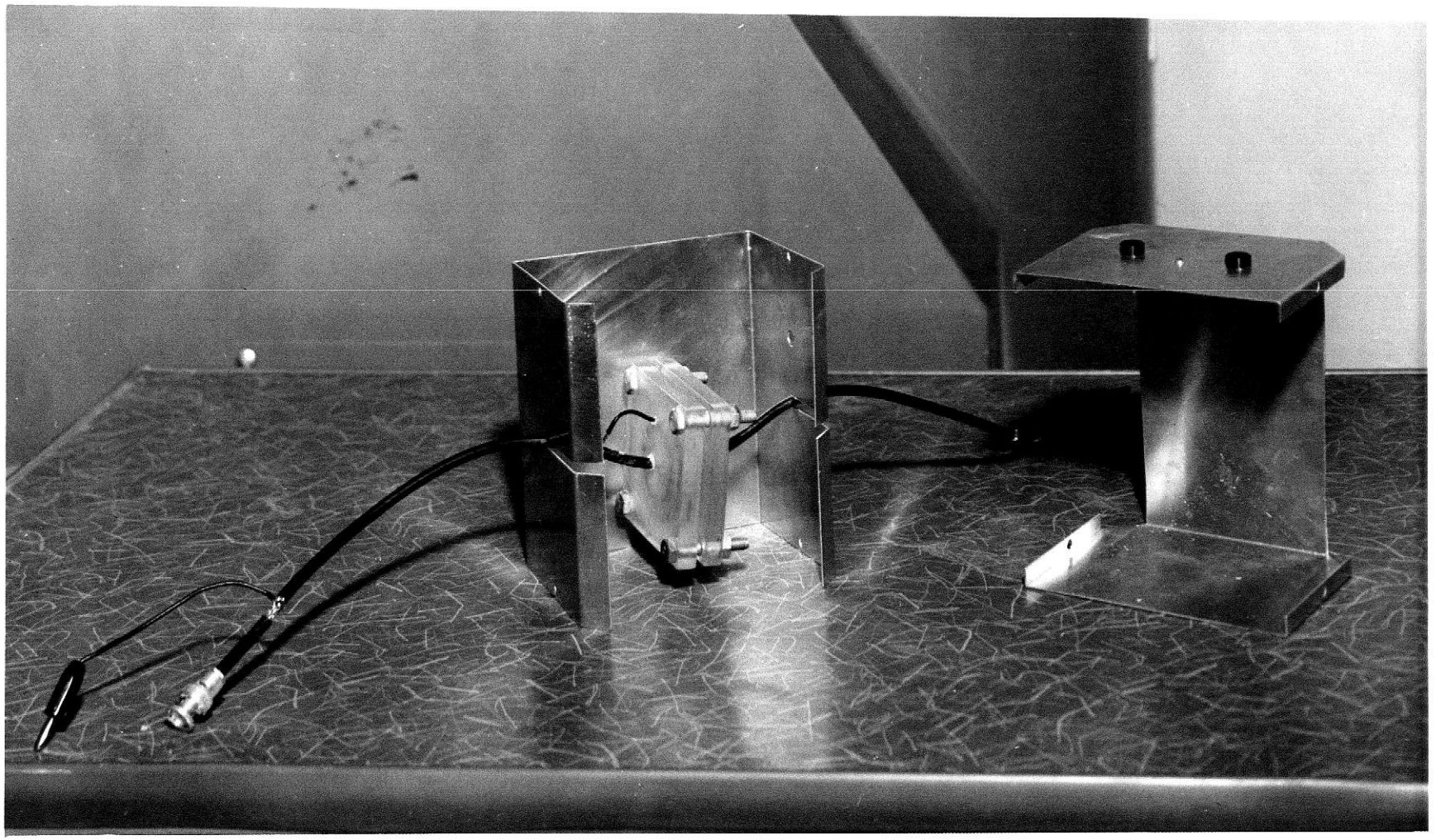

Fig. 4. Arrangement of specimen holder in testing box.

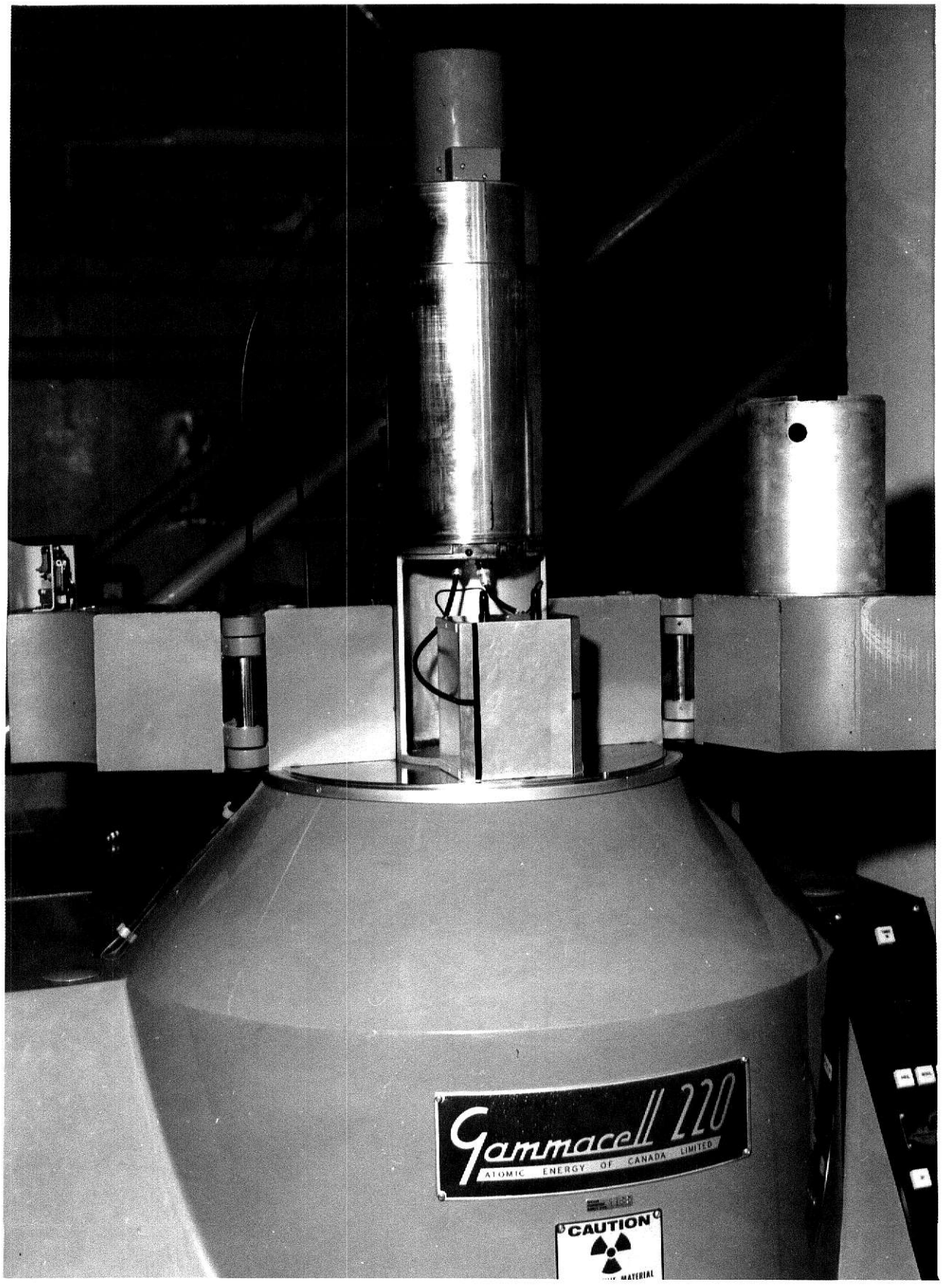

Fig. 5. Electrical hookup and positioning of testing box.

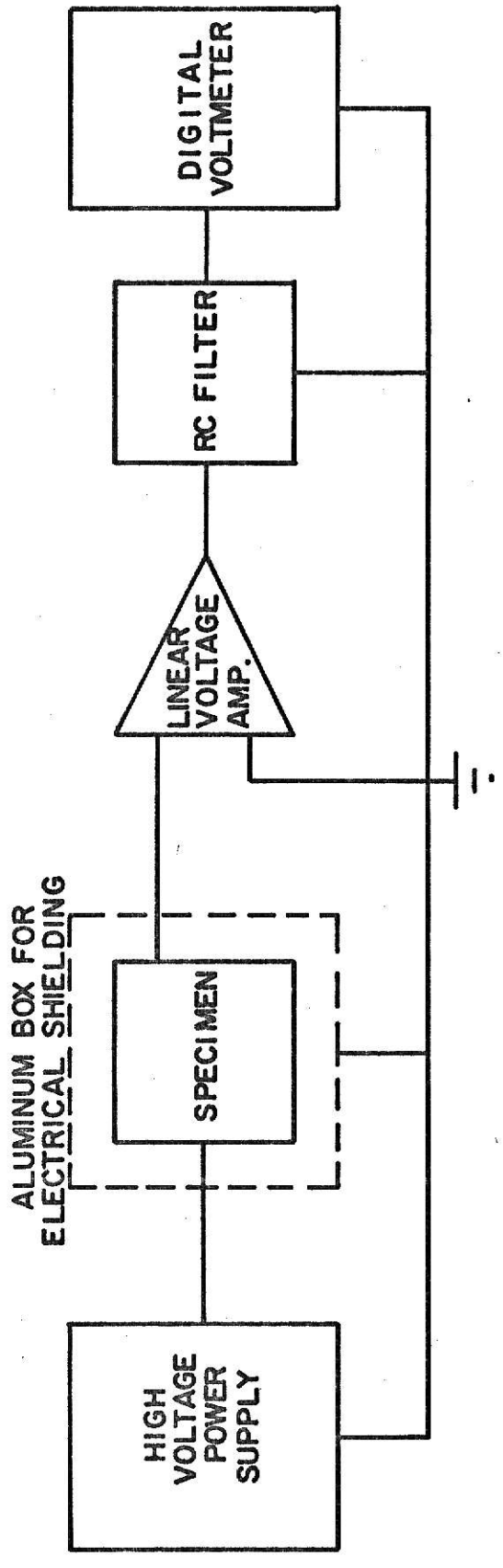

Fig. 6. Block diagram of testing circuit.

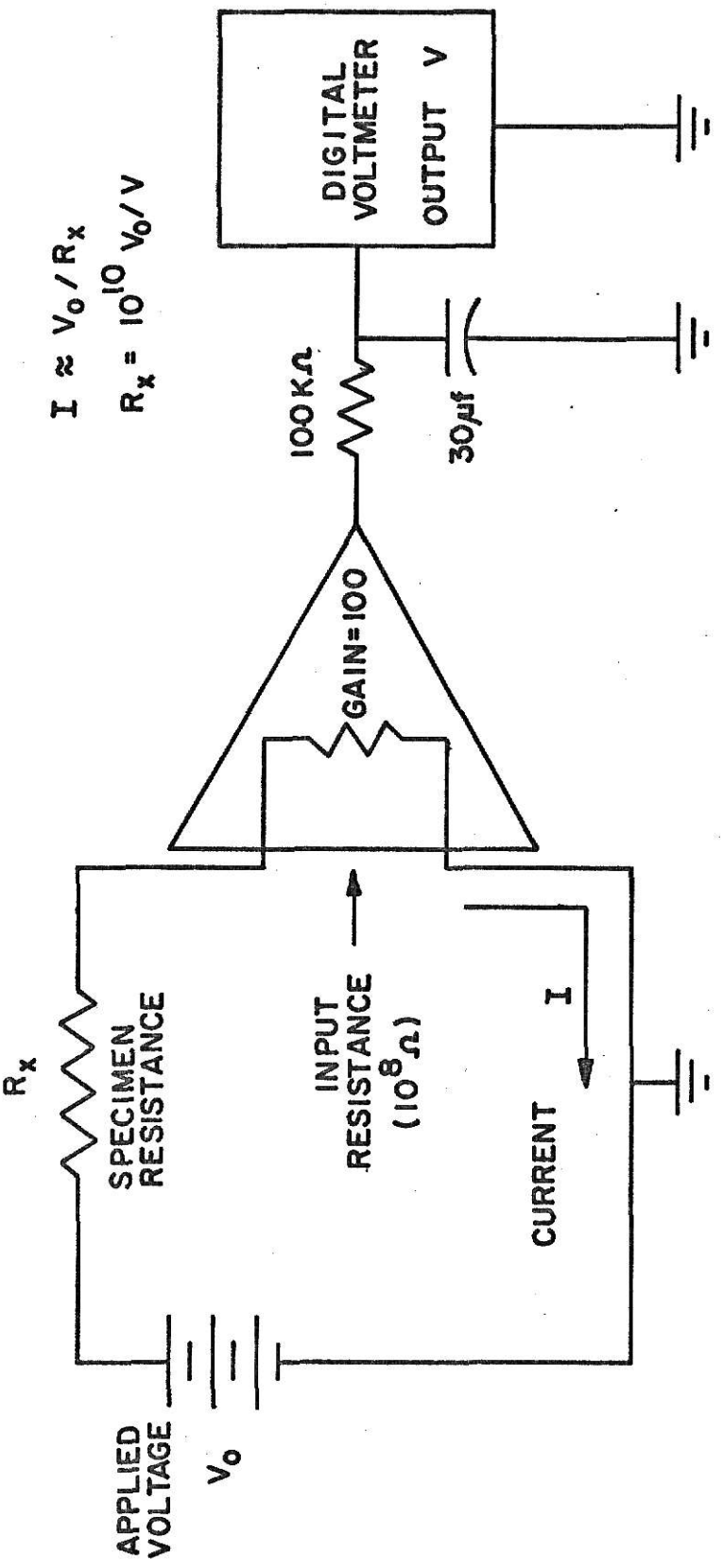

Fig. 7. Schematic diagram of testing circuit.

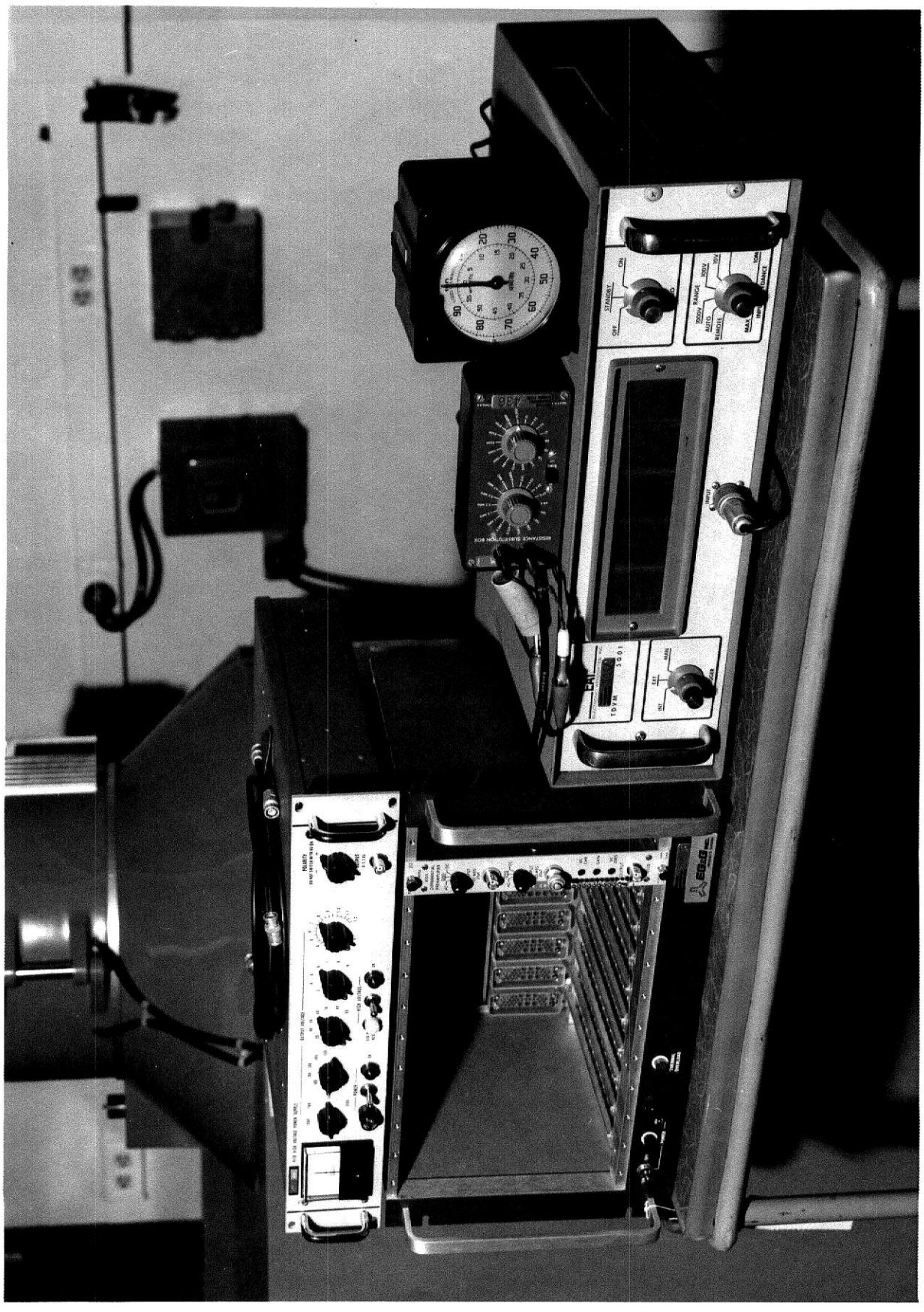

Fig. 8. Electronic equipment.

In the post-irradiation recovery test, the test specimen was removed from the gamma-ray field and the decay current was measured at intervals of one minute. The applied voltage used for this test was +1000V. The test was sufficiently long that a significant number of data points were obtained.

Finally, the dose rate at the surface of the test specimen was obtained by noting the position of the specimen in the irradiation chamber of the gammacell and from a knowledge of the isodose curves of the irradiation chamber. These are shown in Fig. 9[13].

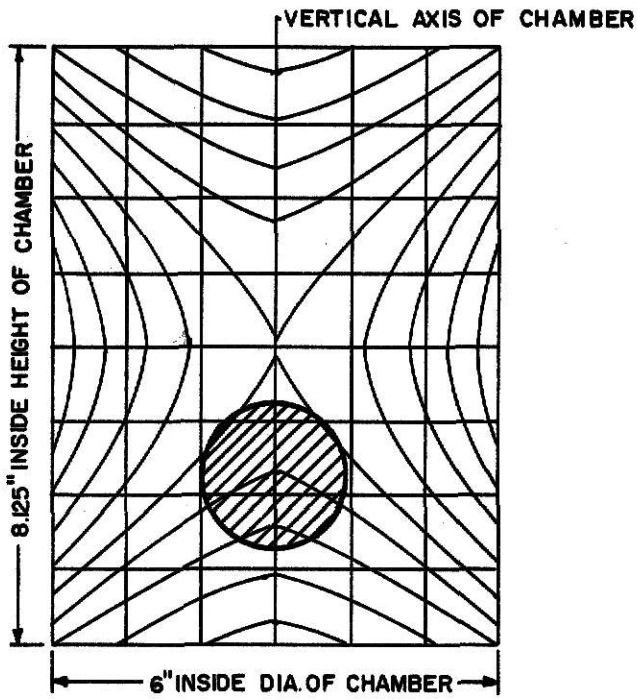

Fig. 9. Gammacell 220 isodose curves and position of test specimen in irradiation chamber.

4.0 DATA REDUCTION AND ANALYSIS

4.1 Determination of Dose Rate at Surface of Test Specimen

The approximate location of the test specimen in the irradiation chamber of the gammacell is shown in Fig. 9. The ratio of the dose rate at the center of the test specimen to the dose rate at the "100% dose location" at the chamber center was visually determined. The actual dose rate at the center of the specimen was then calculated by knowing the dose rate at the "100% dose location". (The "100% dose location" dose rate had been previously determined by the use of a Fricke dosimeter.) This calculated dose rate was assumed to be the average dose rate for the entire surface of the test specimen.

4.2 Calculation of A_γ and δ

The current versus voltage data for polyethylene samples irradiated at a particular dose rate were averaged and standard deviations were assigned. A weighted linear least squares computer program was then used to fit a straight line through the averaged data. The conductivity of polyethylene was calculated from the slope of the fitted line using Ohm's law in the form

$$G_x = I/V_o = 10^{-10}\ V/V_o \tag{8}$$

(see Fig. 7) and the relation

$$\sigma = G_x\left(\frac{\text{specimen thickness}}{\text{specimen area}}\right), \tag{9}$$

where the sample dimensions are given in Ref. (11a).

The empirical constants A_γ and δ were calculated from Equation (5), repeated here for convenience:

$$(\sigma - \sigma_o) = A_\gamma \dot{\gamma}^\delta \ . \tag{10}$$

Since the conductivity of an insulator increases by several orders of magnitude when the insulator is subjected to ionizing radiation, a good approximation for the left hand side of Eq. (10) is that

$$\sigma - \sigma_o \simeq \sigma \ . \tag{11}$$

With this approximation, Eq. (10) becomes

$$\sigma = A_\gamma \dot{\gamma}^\delta \ . \tag{12}$$

Logarithms of both sides of Eq. (12) may be taken to obtain

$$\log_{10}\sigma = \log_{10} A_\gamma + \delta \log_{10} \dot{\gamma} \ . \tag{13}$$

Here σ and $\dot{\gamma}$ are known. Eq. (13) is of the form

$$y = b + mx \ , \tag{14}$$

where $b = \log_{10} A_\gamma$

and $m = \delta$.

One gammacell located at Kansas State University and two gammacells located at the Nuclear Effects Laboratory at Edgewood Arsenal, Maryland provided three values of $\dot{\gamma}$ for use in Eq. (13). The resulting system of three equations and two unknowns was solved using the same linear least squares computer program mentioned previously. Statistics were propagated throughout the calculations.

4.3 Calculation of k_i and τ_i

The post-irradiation decay current versus time data for polyethylene samples irradiated at a particular dose rate were also averaged and standard deviations were assigned. An exponential curve-fitting computer program was used to fit a sum of exponentials to the averaged data. This

program gave the decay constants τ_i in the post-irradiation recovery equation

$$(\sigma - \sigma_o) = A_\gamma \dot{\gamma} \sum_{i=1}^{n} k_i e^{-t/\tau_i} .$$

Fits using 2, 3, and 4 exponentials were tried. The constants k_i were determined from the extrapolated zero-time intercepts supplied by the computer program and by the fact that $\sum_{i=1}^{n} k_i = 1$.

5.0 PRESENTATION AND DISCUSSION OF RESULTS

The density of polyethylene used in this experiment was found to be 0.983 gm/cm^3 by averaging measurements of specimen weight and volume. This value compares with a density of 0.953 gm/cm^3 calculated from data supplied by the manufacturer of the polyethylene sheet.[17]

From Fig. 9, the average dose rate at the surface of the test specimen was visually determined to be 96% of the dose rate at the "100% dose location" lines for the KSU gammacell. As mentioned previously, the dose rate at the "100% dose location" had been previously determined using a Fricke dosimeter and this was taken as a standard value in the computation of $\dot{\gamma}$. Standard dose rates for the two gammacells at Edgewood Arsenal were supplied by Sassé.[18] Table I lists the standard dose rates for the three gammacells which were used in this work.

Table I. Location and Standard Dose Rates of Gammacells

Location	Standard Dose Rate
Edgewood Arsenal, Md.	128 Rad(H_2O)/min
KSU, Manhattan, Ks.	2,500 Rad(H_2O)/min
Edgewood Arsenal, Md.	12,300 Rad(H_2O)/min

The measured background dose rate was less than 0.01 Rad(H_2O)/hr during all of the irradiation and post-irradiation tests and was not taken into account when computing the dose rates. The drift of the digital voltmeter was also neglected due to the long warmup time of the electronic equipment prior to the start of the tests. The amplifier drift was subtracted from the raw data, although this was really not

necessary since the drift never exceeded 6 mV and the amplifier was rezeroed after each of the separate voltage tests. The effect of shunt capacitance was determined to have little effect on the output voltage of the amplifier since in most cases the data obtained at times of 5 min and 10 min after inversion of the applied voltage polarity did not vary by more than 5% from each other. The data taken at the 5 minute marks were used in the calculation of A_γ and δ, as suggested by Winslow.[12] The conditioning and testing temperature was (25±2)°C and the conditioning relative humidity was (22±5)%.

The current versus applied voltage characteristic of polyethylene for various dose rates are shown in Figs. 10 - 12. The conductivity was obtained from the slope of the lines and by Eqs. (8) and (9). An effective test specimen diameter of 3.81 cm[11a] was used to calculate σ for the various dose rates. The logarithm of σ was then plotted against $\log_{10} \dot{\gamma}$ as shown in Fig. 13. The constants A_γ and δ were calculated from a straight line fitted through the points using a least squares technique. This method gave a value of A_γ of

$$(1.51 \pm 0.25) \times 10^{-16} [(\text{mho/cm})/(\text{Rad}(H_2O)/\text{min})^{\delta}]$$

and a δ of 0.66 ± 0.02 for a gamma dose rate from 123 Rad(H_2O)/min to 11,808 Rad(H_2O)/min. These compare with Harrison's[9] values of 5.2×10^{-16} [(mho/cm)/(Rad(H_2O)/min)$^{\delta}$] and 0.74 for A_γ and δ, respectively.

It is seen from Figs. 10 - 12 that the I-intercept is significantly different for each dose rate. The intercept appears to increase negatively with increasing dose rate in an unknown manner. No attempt was made to construct a function relating the I-intercept to the dose rate when the I versus V_o data were analyzed.

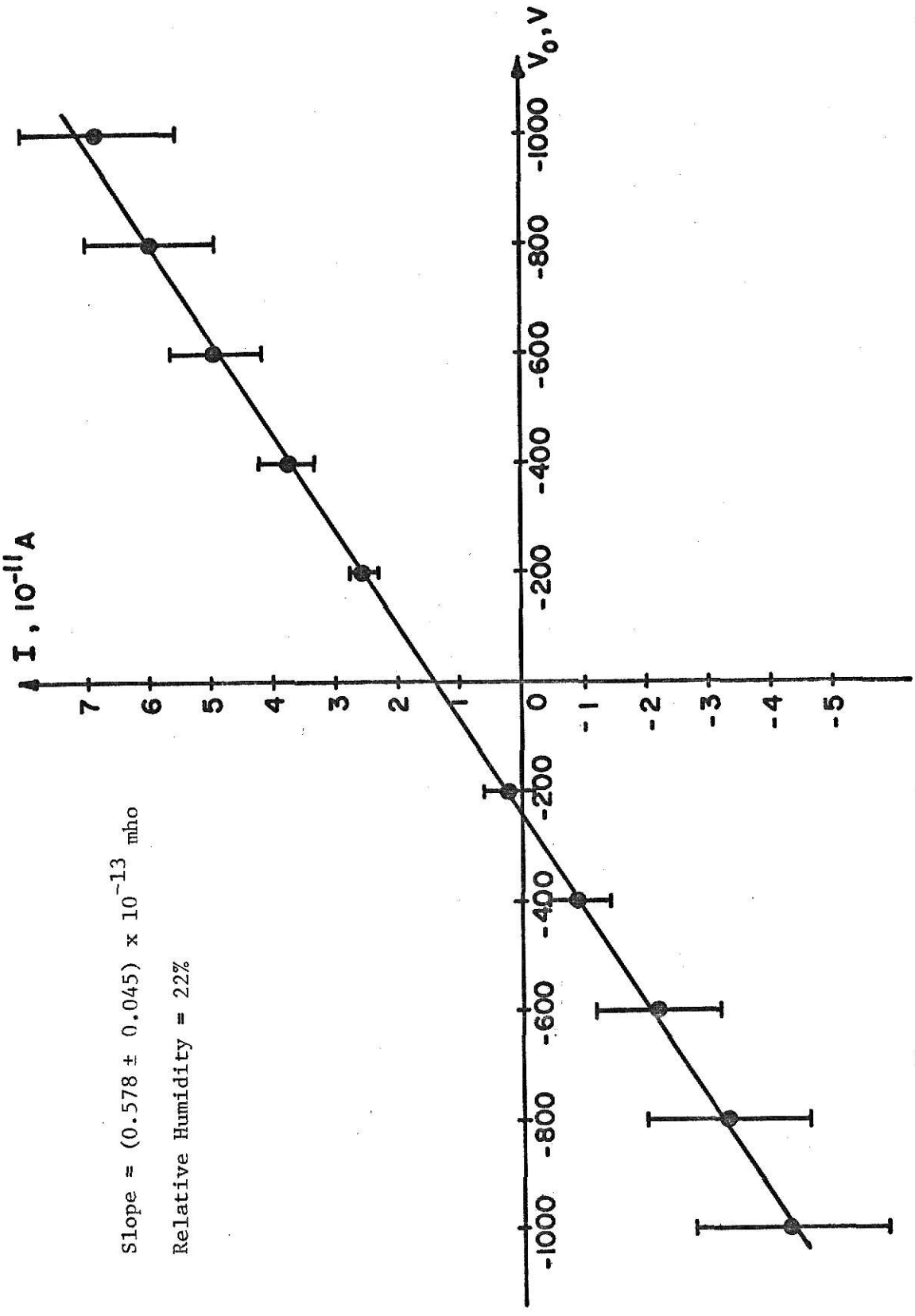

Fig. 10. Current versus applied voltage characteristic for polyethylene at a dose rate of 123 Rad(H_2O)/min at 25°C.

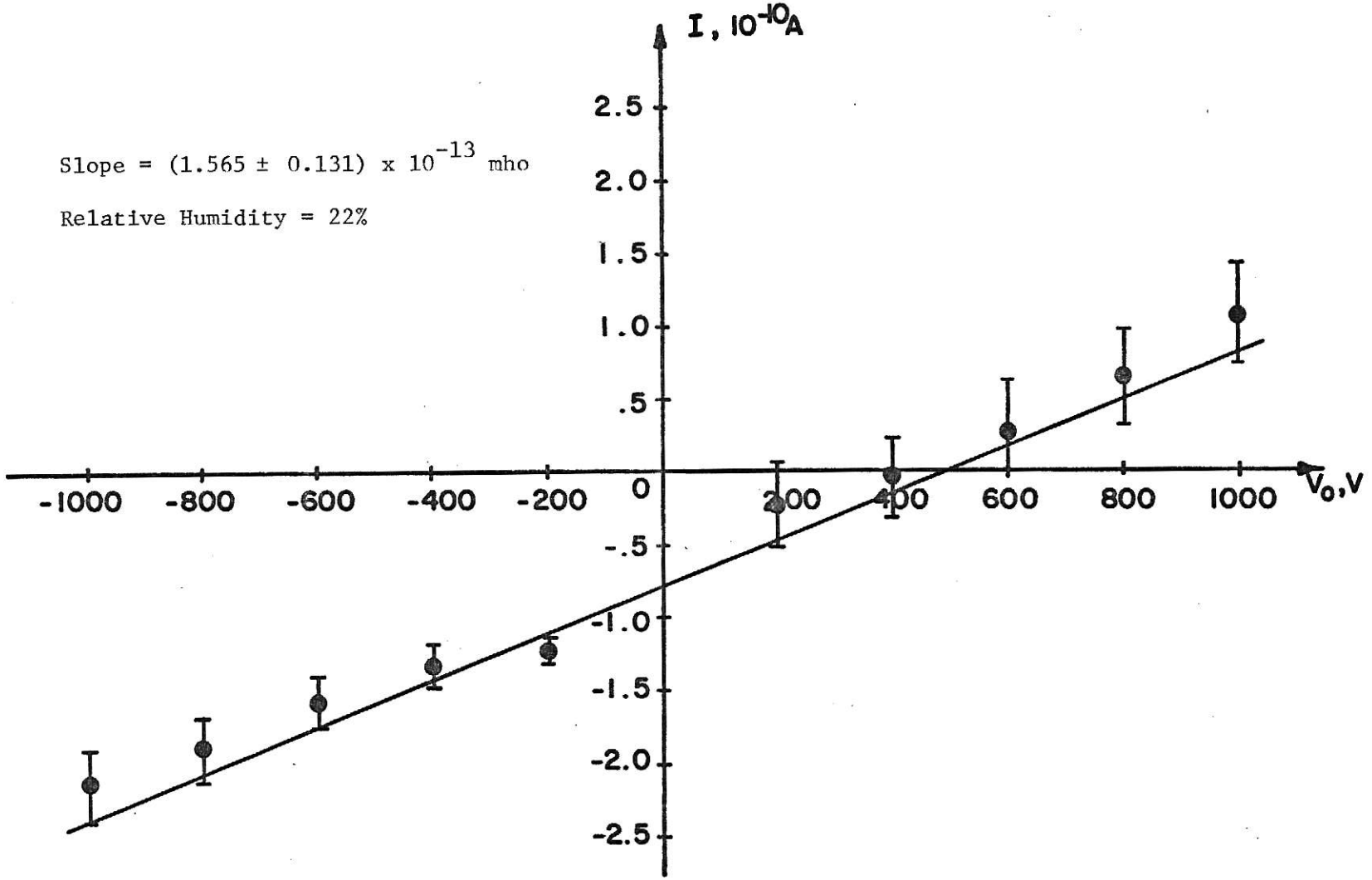

Fig. 11. Current versus applied voltage characteristic for polyethylene at a dose rate of 2400 Rad(H_2O)/min at 25°C.

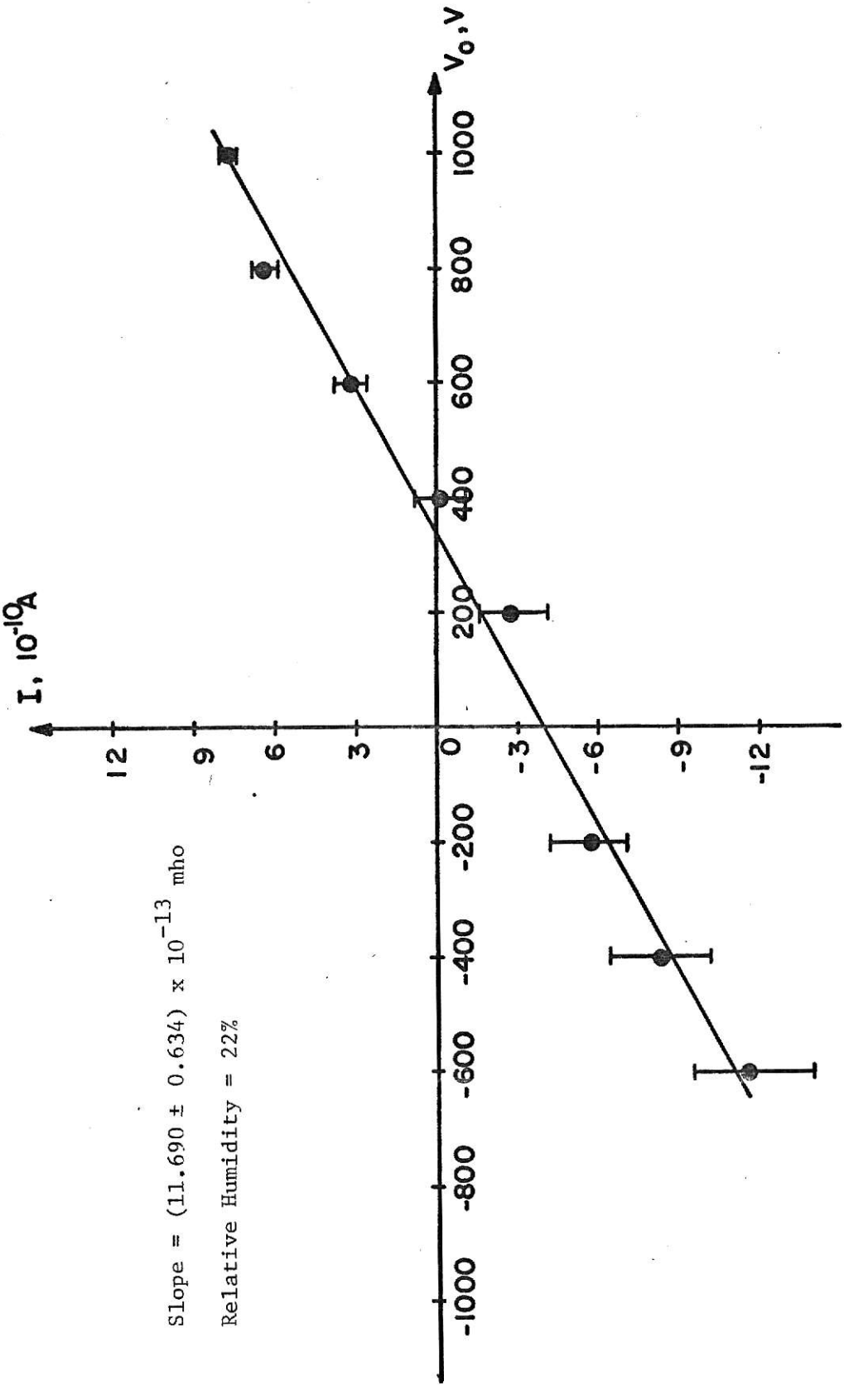

Fig. 12. Current versus applied voltage characteristic for polyethylene at a dose rate of 11,808 Rad(H_2O)/min at 25°C.

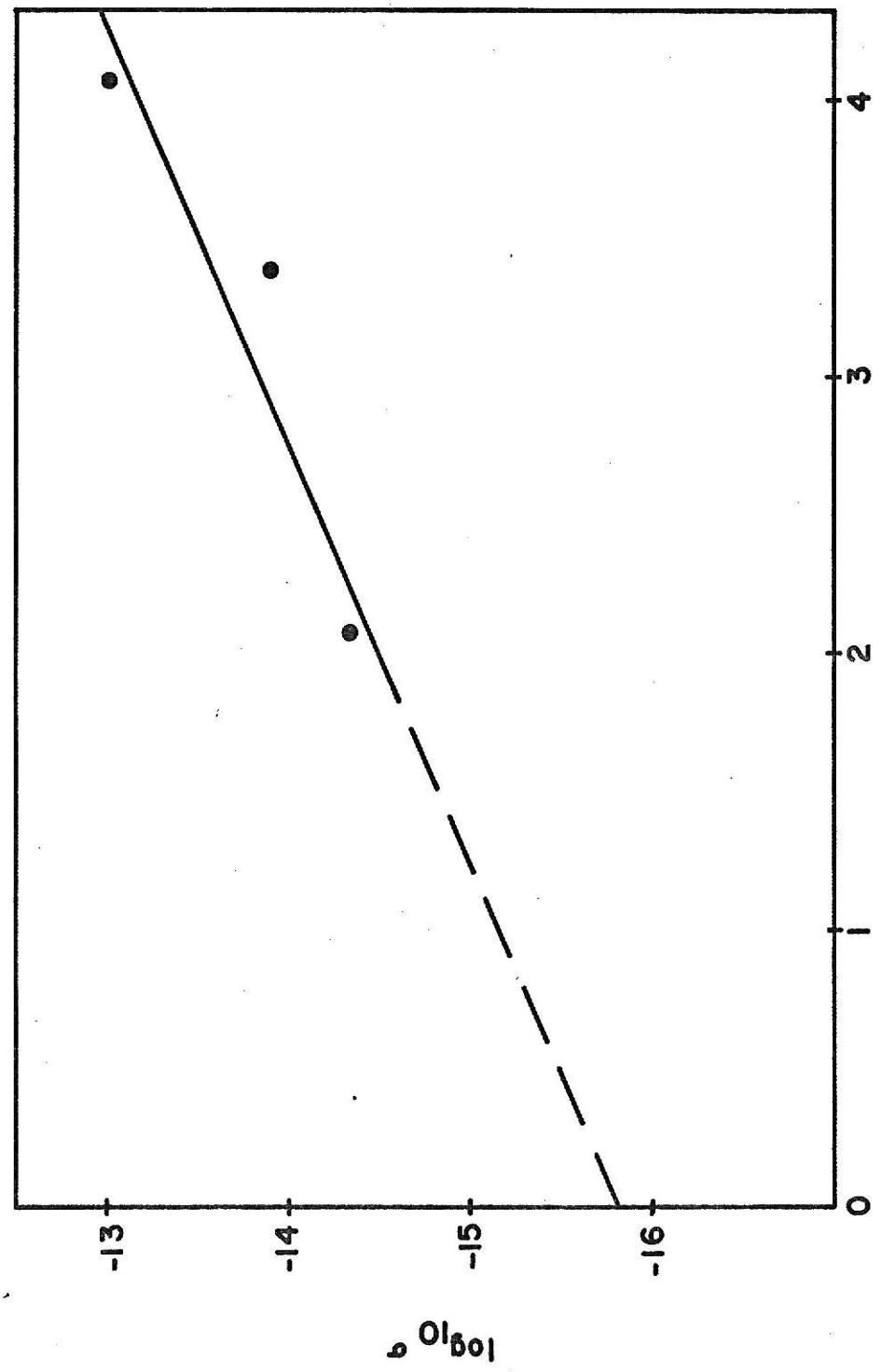

Fig. 13. Logarithm of conductivity versus logarithm of dose rate for polyethylene at 25°C.

Yahagi and Danno[19] have attempted to explain the reason for the negative I-intercepts. They state:

> "Concerning this reversed current, it seems probable that forward-scattered secondary electrons from the vacuum vessel wall, window and other metals were collected by the electrode. On the other hand, backward-scattered electrons from the sample and the parts underneath it did not affect the reverse currents appreciably, shown by the fact that the reverse current was not varied when the electrode toward the [gamma-ray] source was lifted from the specimen."

This author can offer no better explanation than the one given above to explain this phenomenon.

It was also found that the radiation-induced conductivity was about two orders of magnitude greater than the "dark" conductivity σ_o. However, σ_o could not be measured accurately because of the drift of the amplifier and the measuring range of the digital voltmeter. Had an electrometer been used, σ_o could have been measured more accurately.

Figures 14 - 16 show plots of decay current versus time for polyethylene after irradiation at various dose rates. The number of discrete time constants was found to be two for all three dose rates. The values of k_i and τ_i are listed for each dose rate in Table II along with an average value for each parameter.

Table II. Values of k_i and τ_i for n=2

Dose Rate Rad(H_2O)/min	k_1	k_2	τ_1, min	τ_2, min
123	0.495	0.505	2.741	141.153
2,400	0.630	0.370	2.050	91.559
11,808	0.610	0.390	3.169	51.701
	$\bar{k}_1 = 0.578$	$\bar{k}_2 = 0.422$		
	$\bar{\tau}_1 = 2.653$ min	$\bar{\tau}_2 = 94.805$ min		

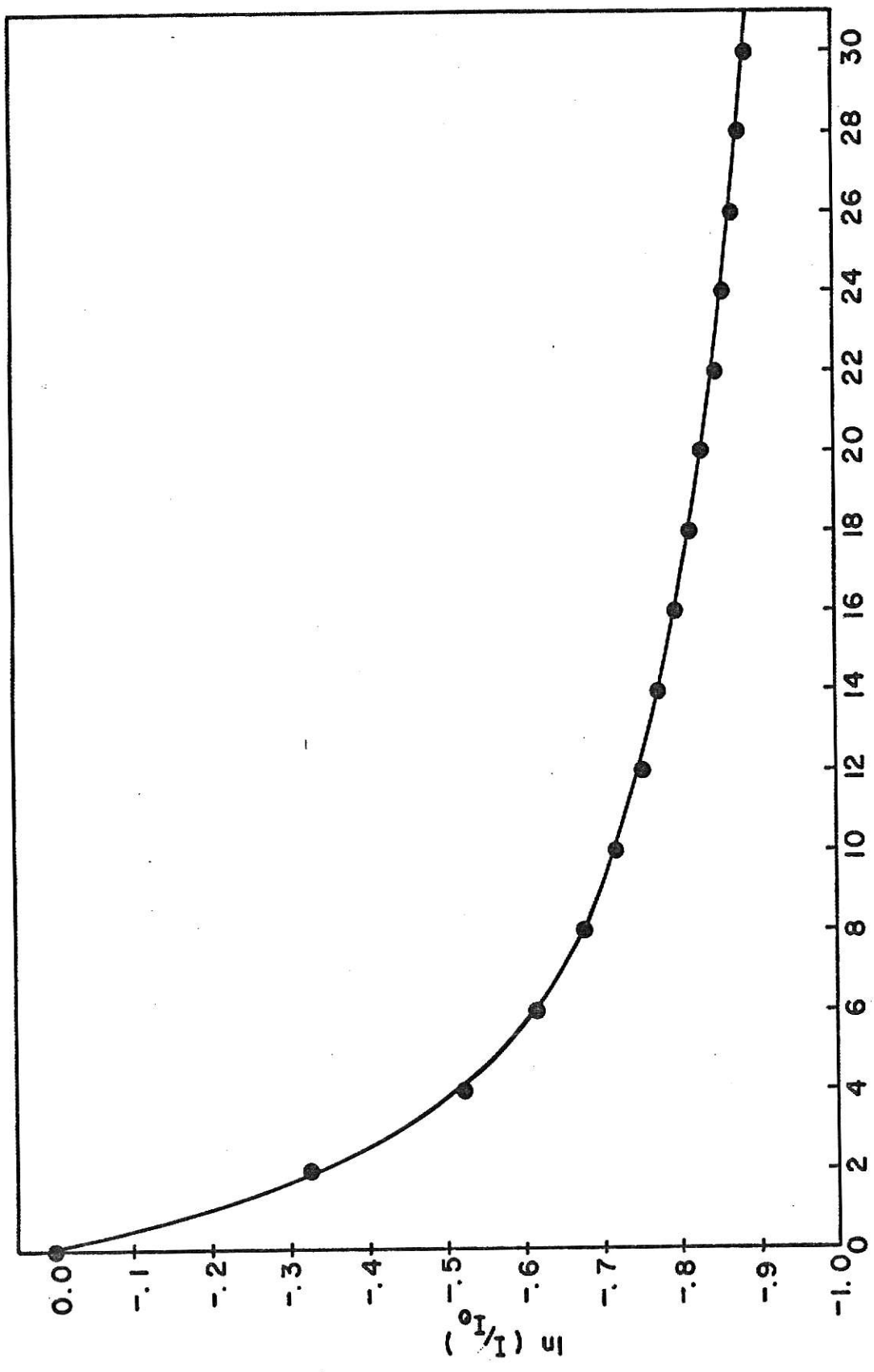

Fig. 14. Logarithm of decay current versus time for polyethylene after exposure to a dose rate of 123 Rad(H_2O)/min for 130 min at 25°C.

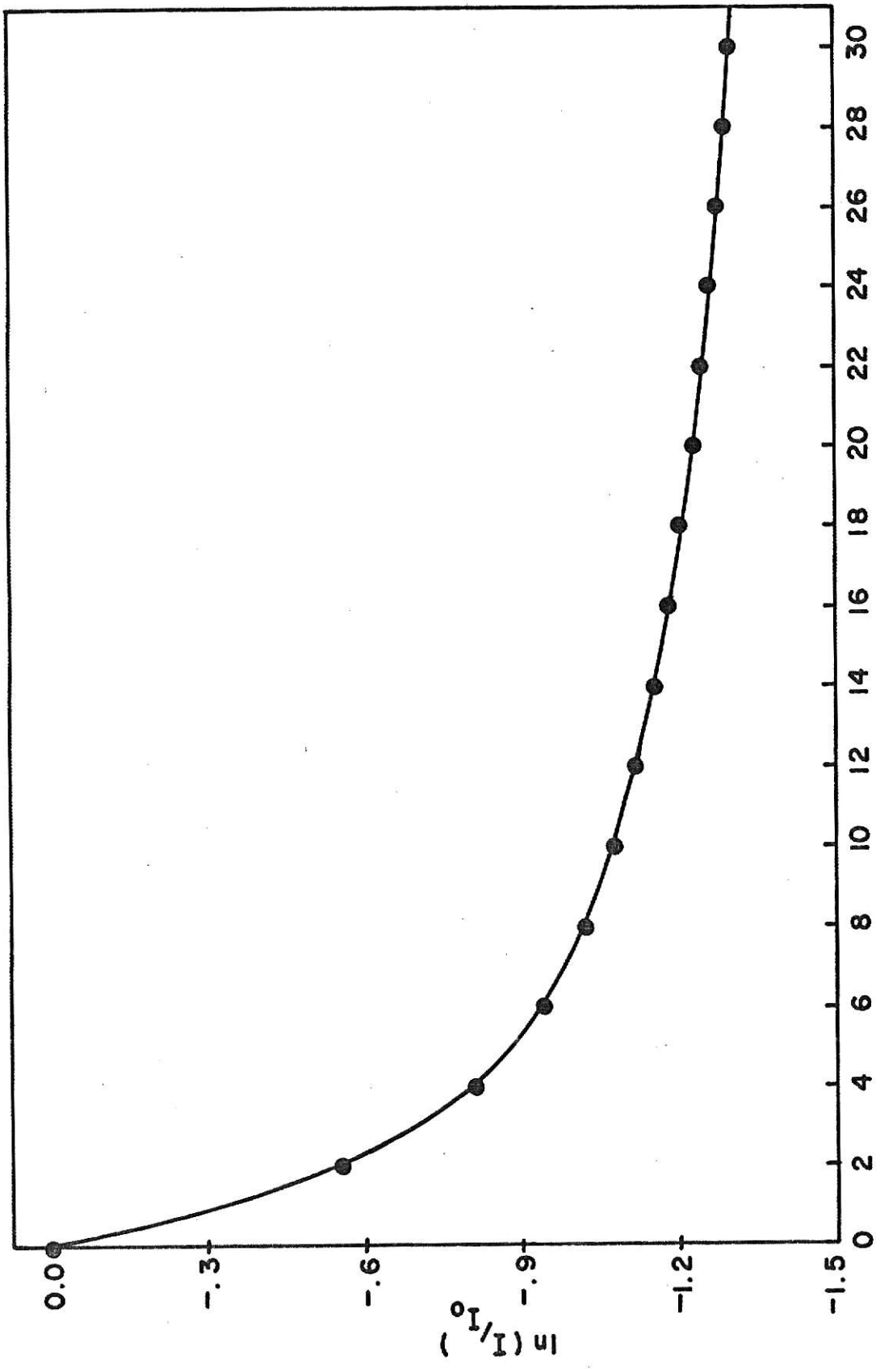

Fig. 15. Logarithm of decay current versus time for polyethylene after exposure to a dose rate of 2400 Rad(H_2O)/min for 130 min at 25°C.

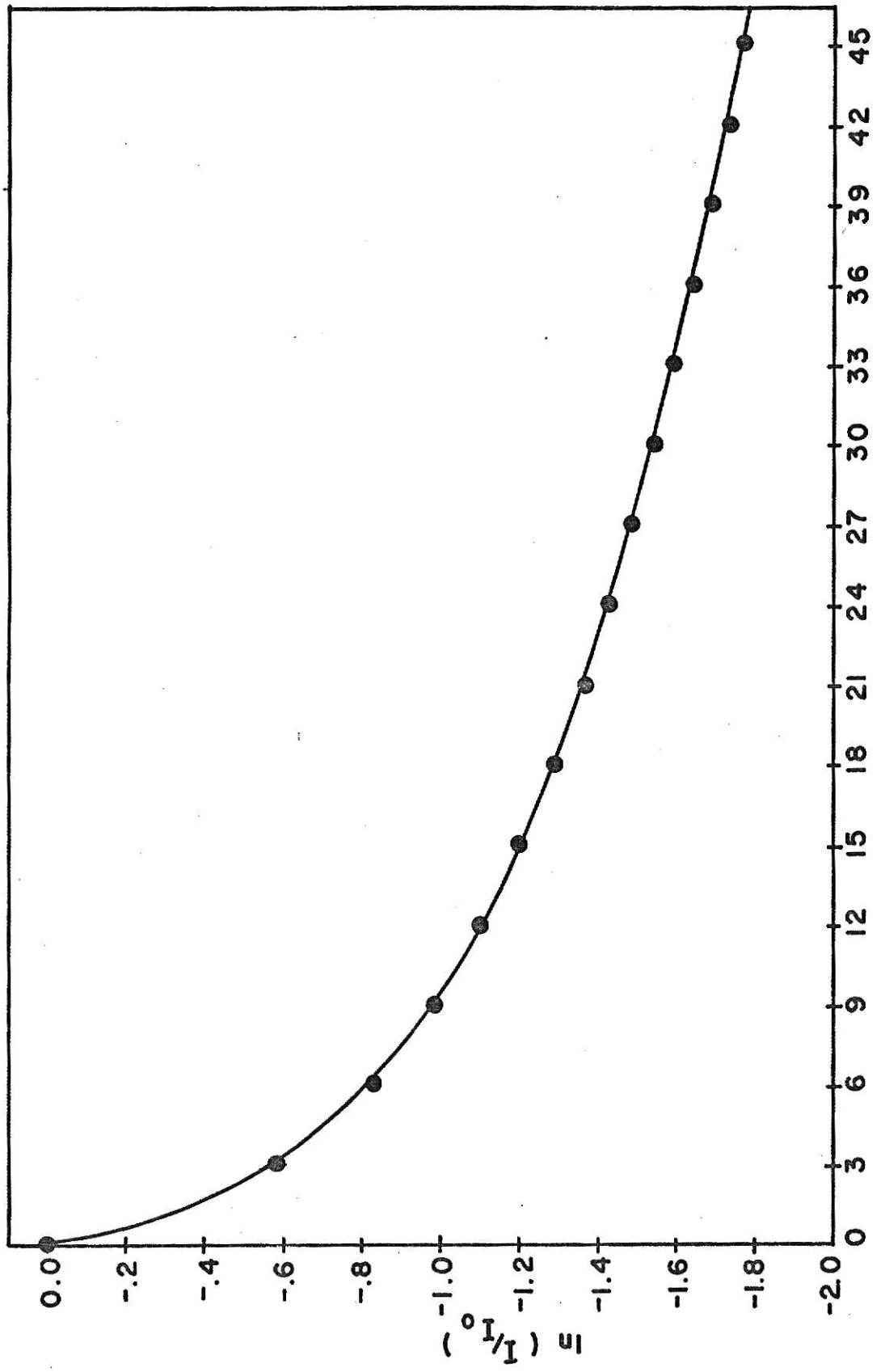

Fig. 16. Logarithm of decay current versus time for polyethylene after exposure to a dose rate of 11,808 Rad(H_2O)/min for 130 min at 25°C.

Although Harrison[9] does not report values of k and τ for polyethylene, he does suggest that time constants for polyethylene are very long when compared with those obtained for polystyrene. This is evidenced by Fig. 17. It is seen that the recovery of polyethylene is very slow which gives some credence to the values of k and τ obtained in this investigation, even though no accuracy can be assigned to them from Fig. 17.

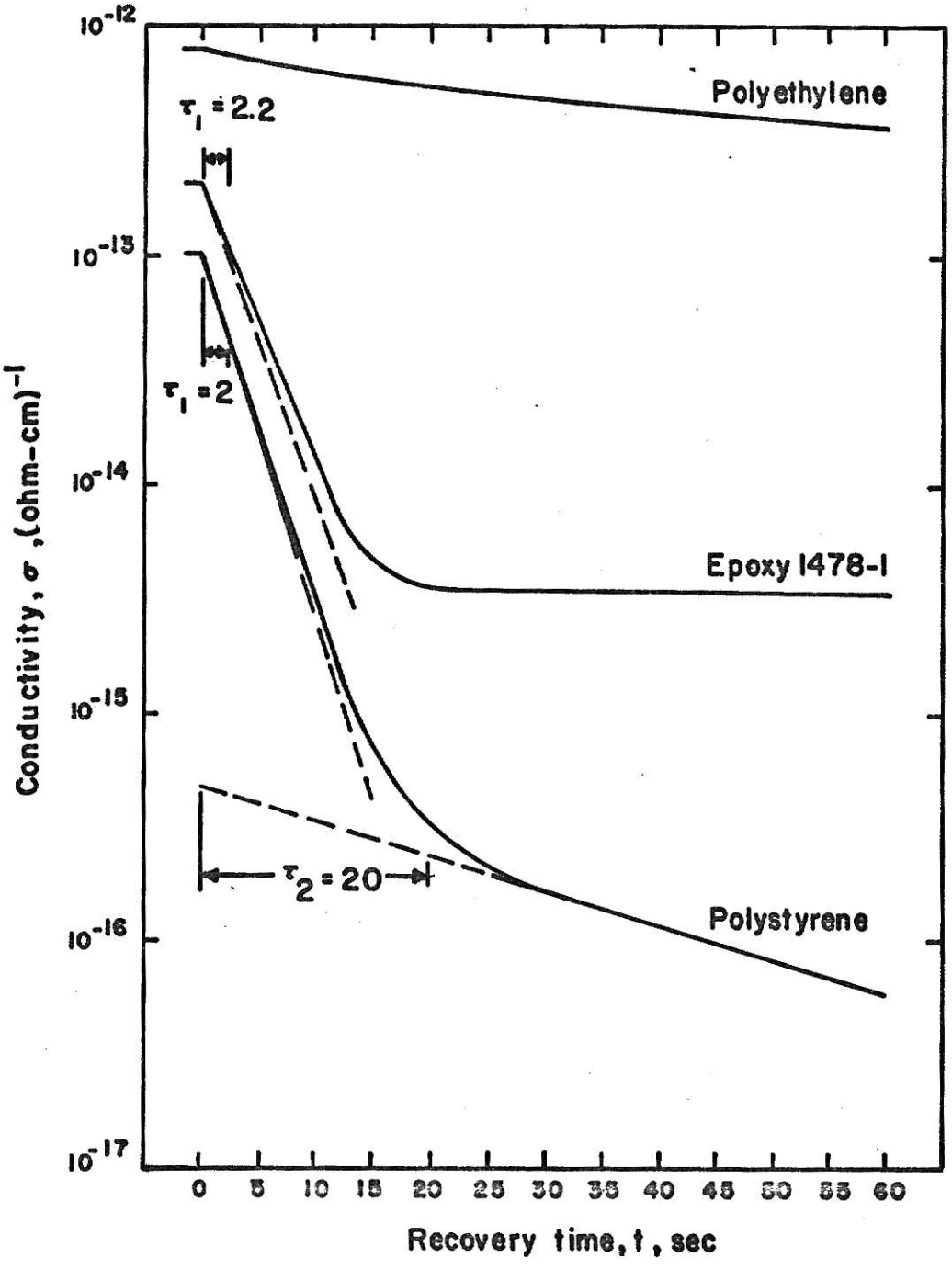

Fig. 17. Recovery curves at 54°C after exposure to 3.3×10^3 Rad(H_2O)/sec. [After Harrison[9].]

6.0 CONCLUSIONS

The results of this investigation compare favorably with other data in the literature.[9, 19] The data obtained for both the non-irradiation test and the irradiation test of polyethylene have shown that current measuring devices other than an electrometer can be used to collect accurate data for the irradiation test, but that an electrometer or other low current measuring device should be used during the non-irradiation test.

The values obtained for A_γ and δ agree well with data found in Ref. (9). Although no data existed with which to compare the experimental values of n, k, and τ obtained in this investigation, Harrison[9] has shown that the recovery time constants for polyethylene are very long in comparison with those for polystyrene and, from his comments, this author is inclined to believe that the values obtained here are plausible. It was found that there were two exponentials in the post-irradiation recovery of polyethylene and that the average weighting factors and time constants were

$$\bar{k}_1 = 0.578, \qquad \bar{k}_2 = 0.422;$$

$$\bar{\tau}_1 = 2.653 \text{ min}, \quad \bar{\tau}_2 = 94.805 \text{ min},$$

respectively. The equilibrium constants were found to be

$$A_\gamma = (1.51 \pm 0.25) \times 10^{-16} [(\text{mho/cm})/(\text{Rad}(H_2O)/\text{min})^{\delta}]$$

and

$$\delta = 0.66 \pm 0.02$$

for a gamma-ray dose rate from 123 $\text{Rad}(H_2O)$/min to 11,808 $\text{Rad}(H_2O)$/min.

It was also found that the I-intercepts of the current versus applied voltage curves became more negative as the gamma dose rate increased. Yahagi and Danno[19] have attempted to explain this phenomenon by suggesting

that the negative intercepts are due to forward-scattered secondary electrons from the specimen holder which were collected by the electrodes. This author can offer no better explanation.

7.0 SUGGESTIONS FOR FURTHER STUDY

A linear voltage amplifier and a digital voltmeter was used in this investigation due to the unavailability of an electrometer. It is suggested that an electrometer be used in subsequent work since it is especially suited to accurately measure extremely small currents.

It is also suggested that insulating materials be irradiated in a nuclear reactor at constant power to study the effects of neutrons on the materials. Pulsing or oscillating the reactor power might also be employed to study transient effects.

Finally, the same tests might be run at a different relative humidity to determine the effect of relative humidity on the conductivity of polyethylene.

8.0 ACKNOWLEDGMENTS

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10.0 APPENDICES

APPENDIX I: Detailed Procedure for Specimen Preparation and Testing

A. SPECIMEN PREPARATION

A1. Specimen Fabrication[11a]

1. Cut specimens 49 mm in diameter from polyethylene sheet 0.060" thick. A stamping process employing a circular "cookie cutter" cutting tool is recommended for ease and quickness in cutting the specimens.

2. After cutting, check the specimens for roundness and trim frays from the cut edges.

A2. Specimen Cleaning[11a]

1. Wash the specimens in distilled water and dry with a clean, lint-free cloth. Rubber gloves should be worn to avoid contaminating the specimens.

2. Rewash the specimens in isopropyl alcohol, then in distilled water, and finally dry with a clean, lint-free cloth.

A3. Specimen Drying

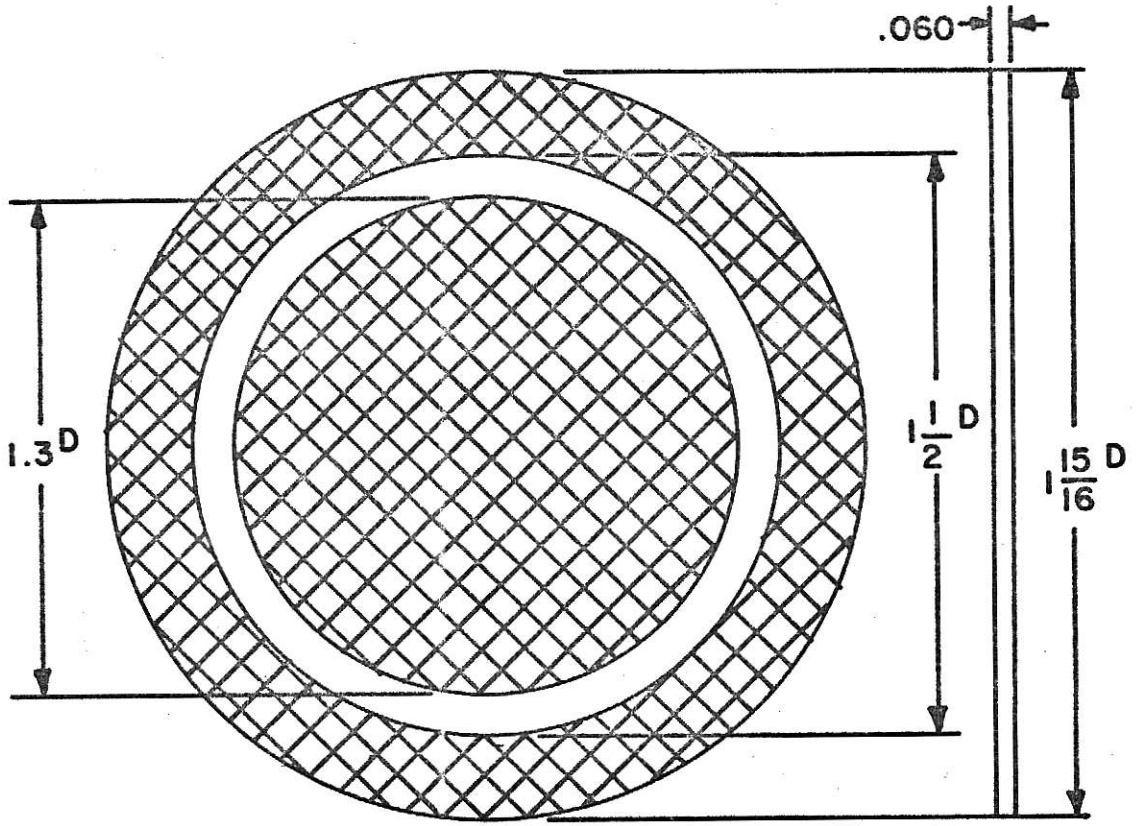
1. Dry the specimens by placing them on wire mesh inside a circulating air oven for 4 hours at a temperature of (50±2)°C.

A4. Specimen Conditioning[11b,c,d]

1. Place the specimens on wire mesh inside an environmentally-controlled region (e.g., a glove box) whose desired relative humidity has reached equilibrium at room temperature for a period of (40±¼) hours. (Room temperature is given by Reference (11c) as being (20 - 30)°C.) Method C of Reference (11d) was used to achieve a constant relative humidity at 20°C in this work.

A5. Electrode Application

1. Paint electrodes on the specimens using silver conductive paint (E-KOTE, type 3030) while the specimens are still inside the environmentally-controlled region. A small artist's brush and circular painting guides may be used. Since two thicknesses of material are put together to form the test specimen, three specimens should be painted in accordance with Figure I-1 and the remaining three specimens should have both sides completely painted. Allow the paint to dry for at least 15 min. It is suggested that the painting of the specimens be initiated at the 39¼-hour work mark.



NOTES

1. CROSS-HATCHED AREAS REPRESENT ELECTRODES. THE RING IS THE GUARD RING AND THE CENTER DISC IS THE LOW VOLTAGE ELECTRODE.
2. ELECTRODES ARE PAINTED ON SPECIMEN USING "E-KOTE" BRAND SILVER CONDUCTIVE PAINT AND ARE 0.001" THICK.
3. TWO PIECES ARE USED TO FORM A TEST SPECIMEN, GIVING A TOTAL THICKNESS OF 0.124".
4. EACH SPECIMEN THICKNESS WITH PAINTED ELECTRODES IS 0.062".
5. THE ENTIRE REVERSE SIDE OF THE SPECIMEN IS PAINTED AND IS THE HIGH VOLTAGE ELECTRODE.

Fig. I-1. Polyethylene specimen showing guard ring and low voltage electrode.

A6. Specimen Holder Fabrication[11a]

1. Fabricate four specimen holders as shown in Figure I-2. Solder the center lead of the coaxial cables to the high- and low-voltage electrodes; solder connect a small wire from the guard ring to the shield braid of the low-voltage coax cable. Also solder connect a short ground wire to the shield braid of each cable and terminate the wires with banana plugs as shown in Figure I-4.

A7. Specimen Arrangement in Holder

1. Polish the copper electrodes with steel wool to remove oxidation layer and reduce contact potential. Remove the specimens from the environmentally-controlled region. Sort the two types of specimens according to the painted electrode surfaces. Combine one specimen of each type so that the completely painted side (high voltage electrode) of one specimen is in contact with one of the completely painted sides (High voltage electrode) of the other specimen, as shown in Figure I-3.

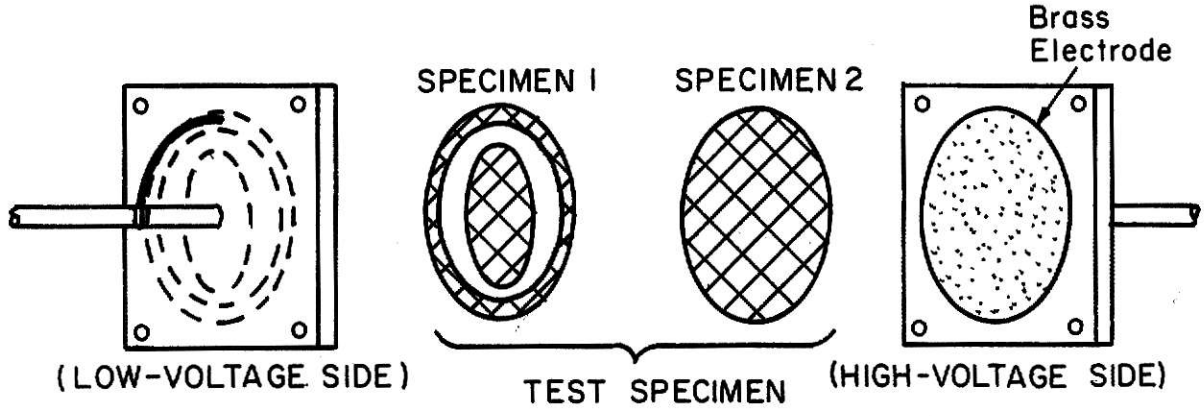


Fig. I-3. Arrangement of test specimen in specimen holder (exploded view).

As before, gloves should be worn to prevent contaminating the specimens.

2. Tighten the bolts of the specimen holder using an open-end wrench. Alternate the tightening process to insure a uniform torque on all of the bolts. Continue until the test specimen is snug in the holder. Be careful not to over-tighten the bolts as this compresses the test specimen; over-tightening is characterized by the bowing of the center of the specimen holder.

A8. Specimen Potting

1. Pot the specimen holder in paraffin by grasping the coax leads and dipping the holder into a container of melted paraffin. Since paraffin is flammable, it is recommended that an electric heater be used to melt the paraffin and that the temperature of the melted paraffin not be

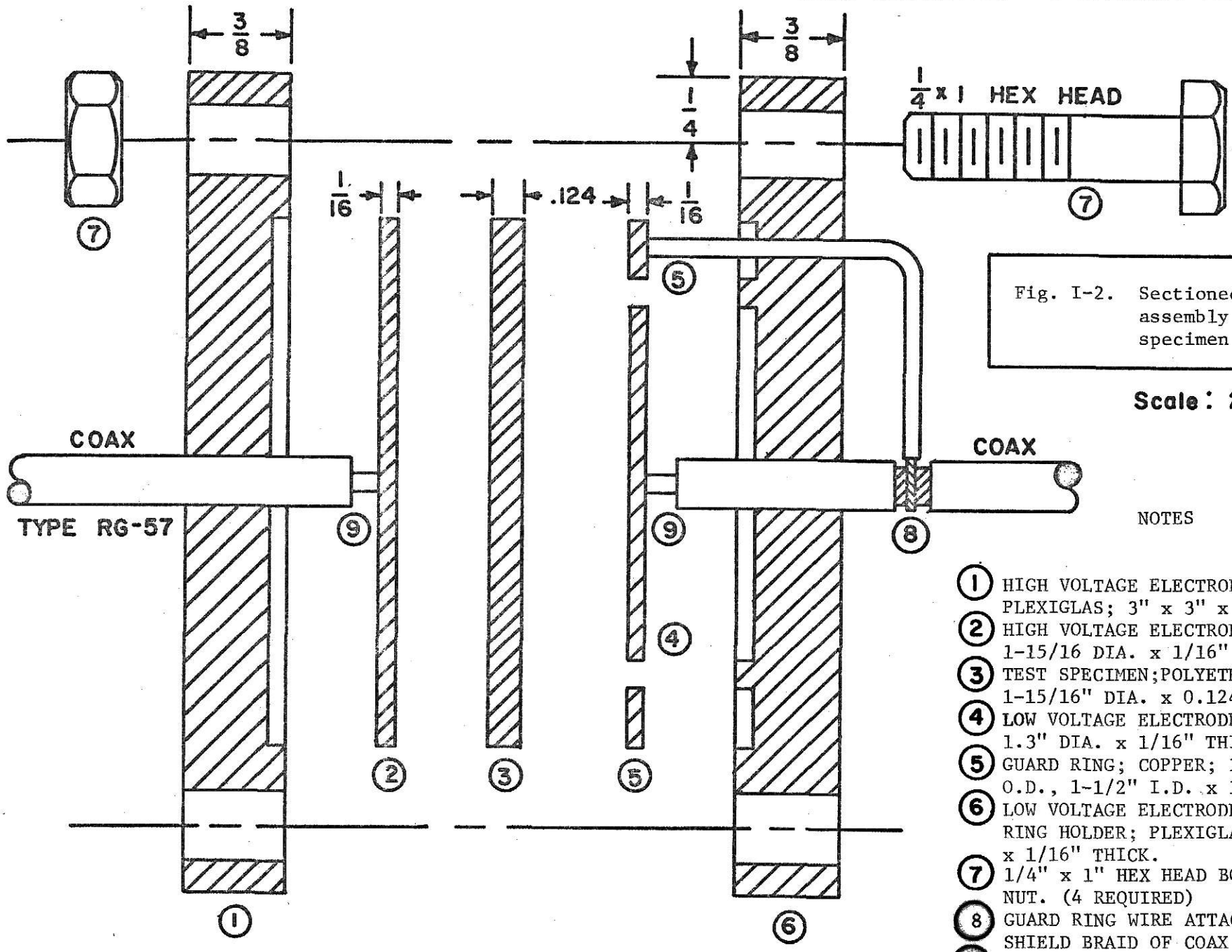


Fig. I-2. Sectioned and exploded assembly drawing of specimen holder.

Scale: 2" = 1"

NOTES

1. HIGH VOLTAGE ELECTRODE HOLDER; PLEXIGLAS; 3" x 3" x 3/8".
2. HIGH VOLTAGE ELECTRODE; COPPER; 1-15/16 DIA. x 1/16" THICK.
3. TEST SPECIMEN;POLYETHYLENE; 1-15/16" DIA. x 0.124" THICK.
4. LOW VOLTAGE ELECTRODE; COPPER; 1.3" DIA. x 1/16" THICK.
5. GUARD RING; COPPER; 1-15/16" O.D., 1-1/2" I.D. x 1/16".
6. LOW VOLTAGE ELECTRODE AND GUARD RING HOLDER; PLEXIGLAS; 3" x 3" x 1/16" THICK.
7. 1/4" x 1" HEX HEAD BOLT WITH NUT. (4 REQUIRED)
8. GUARD RING WIRE ATTACHES TO SHIELD BRAID OF COAX CABLE.
9. CENTER WIRE OF COAX CABLE ATTACHES TO ELECTRODES.

allowed to exceed 130°C. It is also recommended that a fire extinguisher be kept on hand. Be certain that the entire holder is coated with at least two dippings of paraffin. After the potting is completed, let the holder cool for 5 to 10 min.

B. SPECIMEN TESTING

B1. Fabrication of Testing Box

1. Use an aluminum "Combination or Screw Type Minibox" 6 in. long x 5 in. wide x 4 in. high mfg. by Bud Radio, Inc. (stock# CU-3007-A). Cut slots in the sides of the box for insertion of the coax leads of the specimen holder as shown in Figure I-4. Also mount two grounding jacks on the top of the box. Solder connect the jacks to each other and to the box. Bevel two edges of the testing box so that the box will fit inside the irradiation chamber of the gammacell with the cover on the chamber.

B2. Circuit Setup

1. Center the specimen holder in the testing box, assemble the box, and connect the ground wires on the holder leads to the grounding jacks on the box.

2. Run two type RG-57 coaxial cables (one for high voltage and one for low voltage) down the access well of the gammacell drawer by tipping back the green cap covering the well. Connect the high voltage cable to the high voltage lead of the specimen holder and the low voltage cable to the low voltage lead of the specimen holder (see Figure I-5).

3. Position the testing box inside the irradiation chamber of the gammacell so that the bevelled edges of the box face the experimenter (see Figure I-5). Replace the cover on the irradiation chamber. Close the swinging doors and reset the green cap on top of the drawer well to the vertical position.

4. Procure the following equipment:

   (a) John Fluke Model 415B High Voltage Power Supply (P/S).

   (b) Princeton Applied Research (PAR) Model 212 Differential Amplifier with power supply bin.

   (c) RC filter consisting of a 100 KΩ resistor and a 30 μf capacitor (τ_{RC} = 3 seconds).

   (d) Electronic Associates, Inc. (EAI) Model 5001 TDVM.

   (e) Standard Interval Timer (0 to 60 min).

   (f) Assorted hookup coax cables.

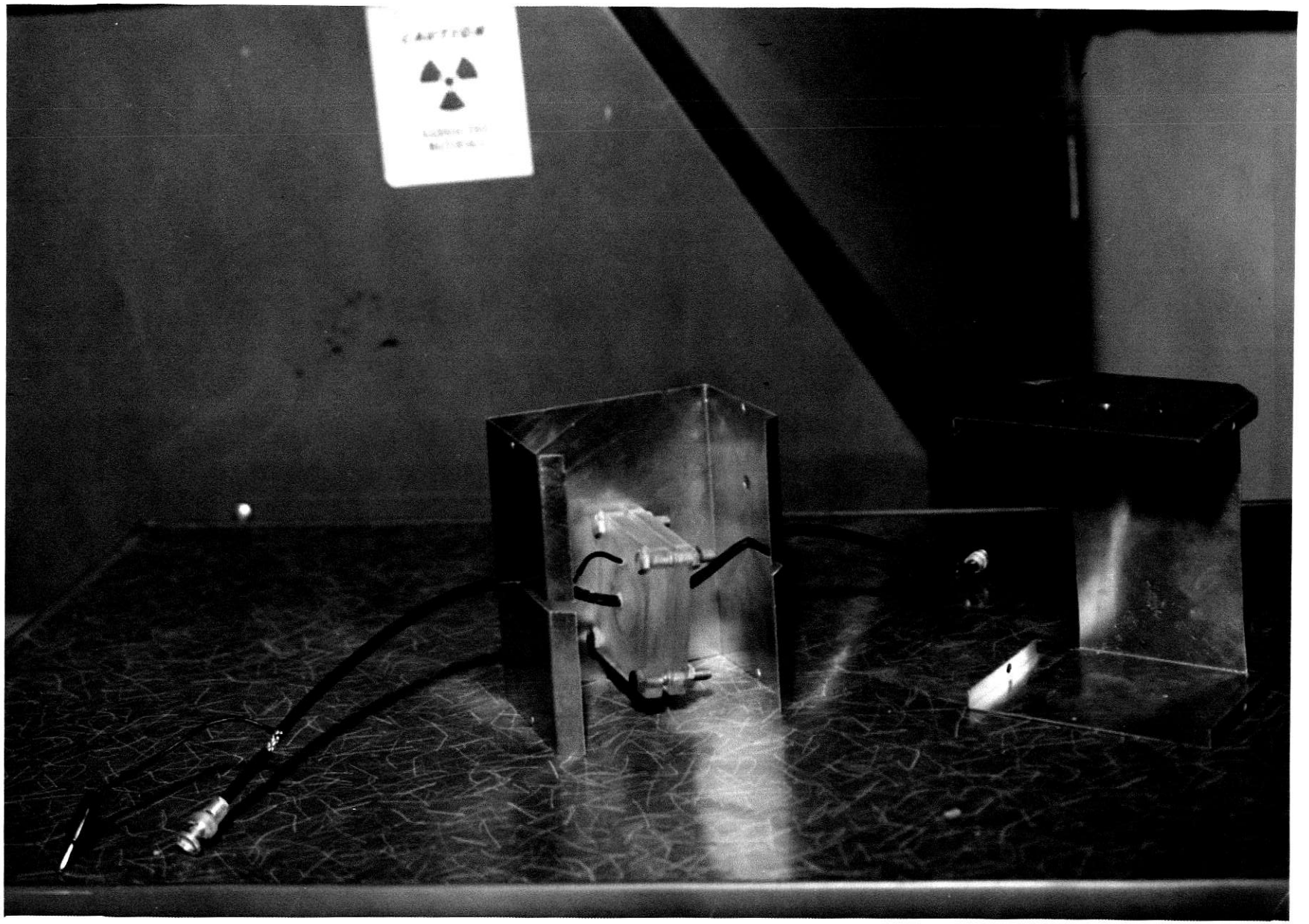


Fig. I-4. Arrangement of specimen holder in testing box.

Fig. I-5. Electrical hookup and positioning of testing box.

5. Connect the high voltage cable from the gammacell to the output of the Fluke P/S and the low voltage cable from the gammacell to input B of the PAR 212. The circuit setup is shown in Figure I-6.

6. Connect a cable from the output of the PAR 212 through the RC filter to the input of the EAI TDVM.

B3. Initial Checkout of Equipment[14-16]

1. Before testing any specimens, allow the Fluke P/S, PAR 212, and EAI TDVM to warm up for at least 2 hours prior to their use by setting their respective controls as follows:

   (a) Fluke P/S: all high voltage dials to zero; high voltage switch to "OFF"; power switch to "ON".

   (b) PAR 212: A input to "GND"; B input to "DC"; rear-panel slide switch to "10Ω".

   (c) EAI TDVM: range switch to "1V"; input impedance knob to "MAX"; trigger switch to "INT HI"; rear-panel REF switch to "INT"; power control selector switch to "STANDBY".

   (d) If a resistance substitution box is used as part of the RC filter, its top resistance dial should be set on "100 KΩ" with the range slide switch set to "HI".

2. Zero the equipment as follows:[15,16]

   (a) EAI TDVM: with the power control switch in "STANDBY", set the ZERO adjustment for +0.0001 volt with the range switch in the "1V" position.

   (b) PAR 212: connect a shorting cap to the B input with the A input switch set at "GND". Turn the power control switch of the EAI TDVM to "ON" with the range switch set to "10V". Adjust the DC ZERO control of the PAR 212 for a miminum reading on the 10V scale of the EAI TDVM. (Since this is a rather touchy adjustment, a "zero" setting of less than ±0.003V is acceptable.) Remove the shorting cap from the B input of the PAR 212 and reconnect the coaxial cable from the gammacell.

B4. Testing of Specimens

1. Neatly record the date, time, and temperature of the testing environment. Also record the radiation background level and the time constant of the RC filter.

2. Initially zero the EAI TDVM and the PAR 212 following the procedures outlined in §§B3(a) and (b), respectively. The 0 to 60 min. interval timer may be used as a time reference.

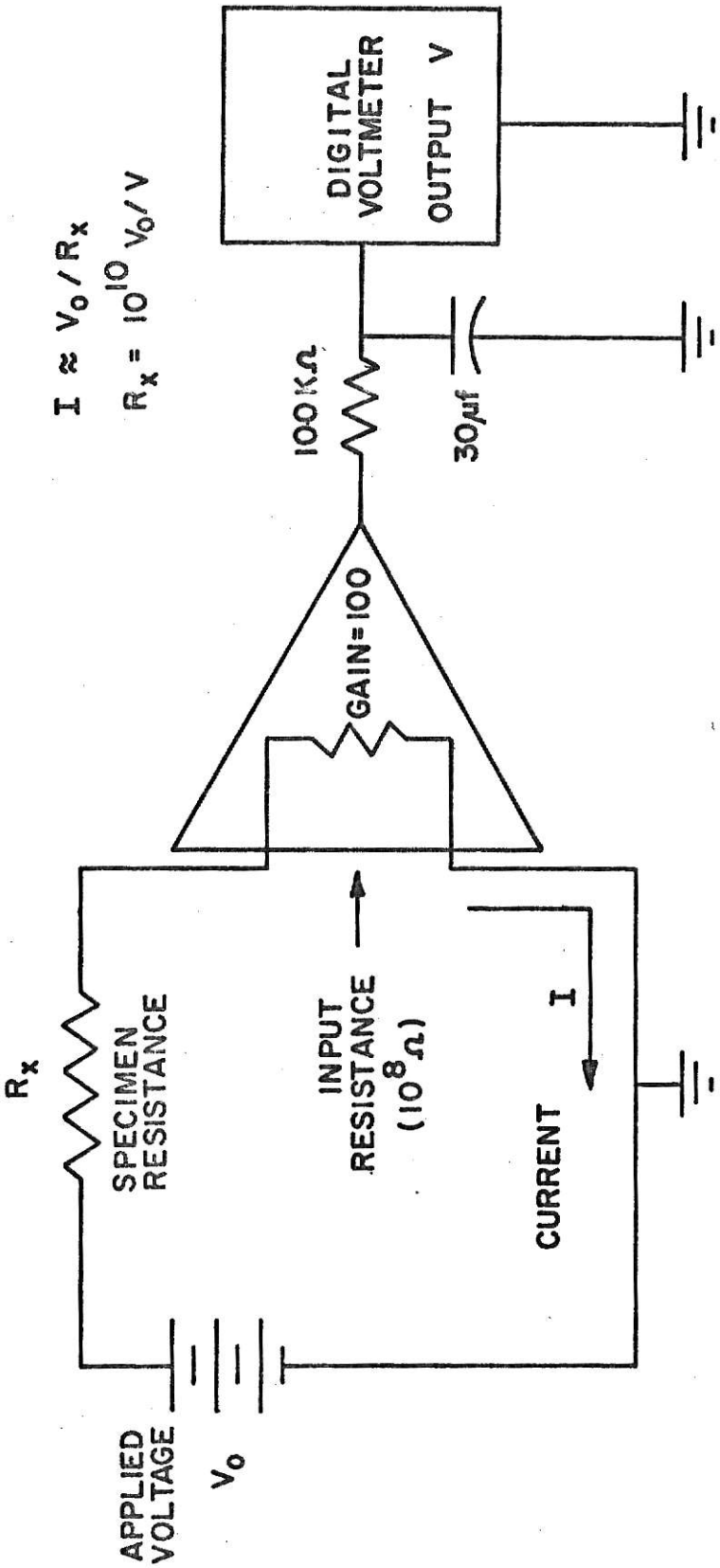


Fig. I-6. Schematic diagram of testing circuit.

B4(a). Non-Irradiation Test[12]

1. The following test is performed with the specimen outside of the radiation field of the gammacell. Set the EAI TDVM range switch to "10V" for this test.
   NOTE: For a better understanding of the test procedure, refer to Figure I-7 when performing steps 2 through 7.

2. At time zero (test start), set the Fluke P/S voltage selection and polarity for an output of +200 volts and move the high voltage toggle switch to "ON".

3. At t=5 min., reverse the polarity of the applied voltage by moving the Fluke P/S high voltage toggle switch to "STD BY-RESET", switching the polarity switch to "-", and then returning the high voltage toggle switch to "ON".

4. Observe and record the readout of the EAI TDVM at t=10 min. and t=15 min. (corresponding to times t'=5 min and t'=10 min., respectively, after the polarity change). Also record the drift of the PAR 212 after each observation by disconnecting the low voltage cable at input B of the 212 and connecting a shorting cap on input B. Wait from 20 to 30 sec after shorting before observing the amplifier drift due to the time constant of the RC filter. Reconnect the low voltage cable from the gammacell to the B input. Be certain to include an estimate of the uncertainty associated with each observation and any unusual behavior of the TDVM readout.

5. At t=15 min., again reverse the polarity of the applied voltage by moving the Fluke P/S high voltage toggle switch to "STD BY-RESET", switching the polarity switch to "+", and then returning the high voltage toggle switch to "ON".

6. Again, observe and record the readout of the EAI TDVM at t=20 min. and t=25 min. (corresponding to times t'=5 min. and t'=10 min., respectively, after the second polarity change). Also record the drift of the PAR 212 after each observation by following the procedure given in step 4 above. Be certain to include an estimate of the uncertainty associated with each observation and any unusual behavior of the TDVM readout.

7. After the reading at t=25 min. has been taken, the PAR 212 should be re-zeroed if its drift has exceeded ±6 mv. This tends to keep the drift to within a few tenths of a mv and provides more uniformity in the equipment zero.

8. Reset the timer to zero and increase the applied voltage to +400 volts. Repeat the test procedure as given in steps 3 through 7.

9. Repeat the above procedure for test voltages of 600V, 800V, and 1000V.

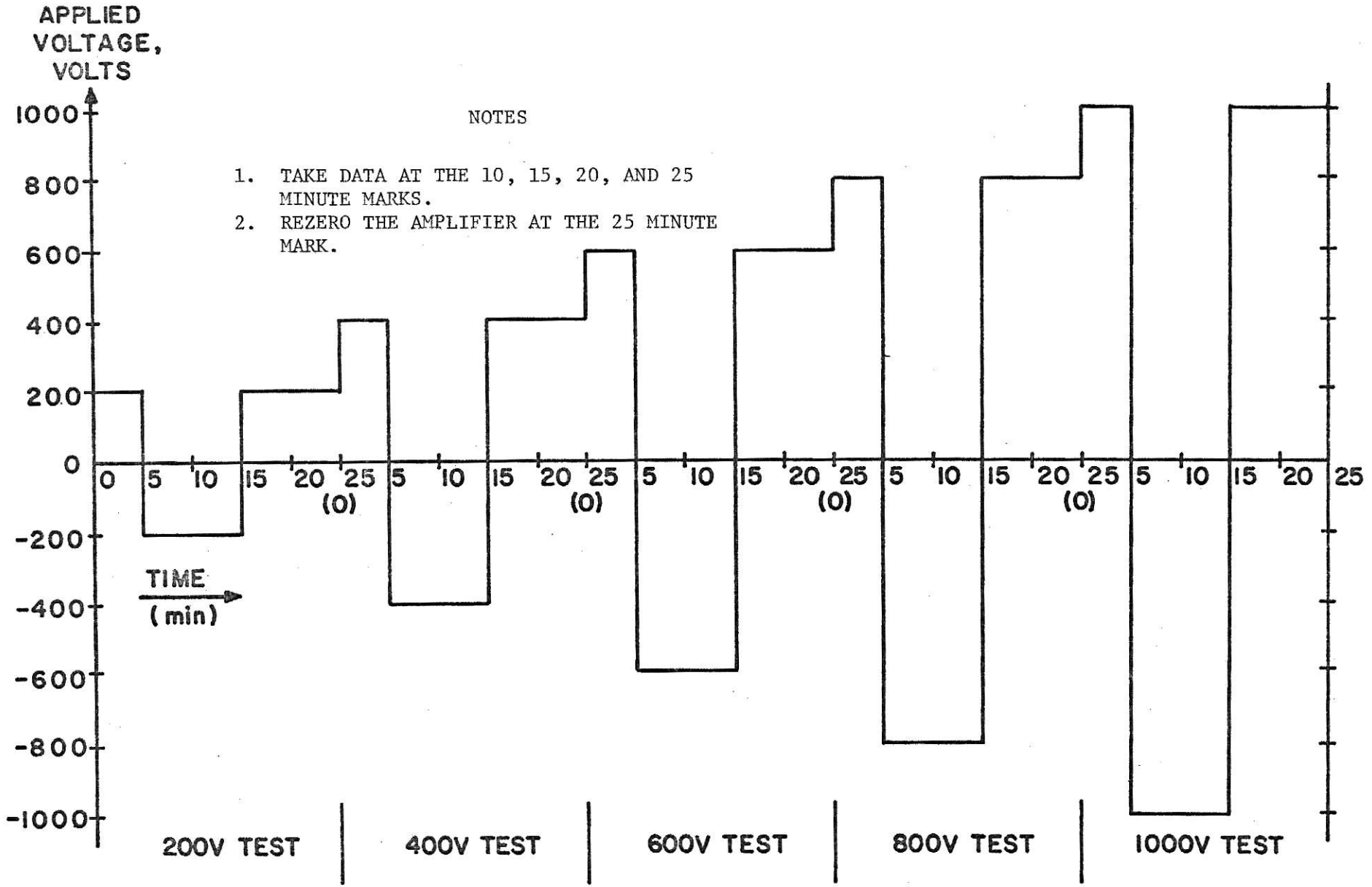


Fig. I-7. Applied voltage versus time curve.

B4(b). Irradiation Test[12]

1. The irradiation test should be started upon completion of the non-irradiation test. This test is performed with the specimen inside the radiation field of the gammacell. Set the EAI TDVM range switch to "10V" for this test. Since the radiation-induced conductivity of the specimen rapidly reaches its saturation value, no or very little error is present when the first reading is taken at t=10 min. after test start.

2. The irradiation test should cover the voltage range from 0 to 1000V and be run following the procedure given in steps 2 through 9 of the non-irradiation test.

B4(c). Conductivity Recovery and Radiation Damage Test

1. The conductivity recovery and radiation damage test should be started upon completion of the irradiation test. The range switch of the EAI TDVM should be set to "10V" for this test.

2. Record the test voltage used. It is recommended that +1000V be used so as not to introduce transients in the response at the beginning of the test.

3. At time zero (test start), remove the specimen from the field of the gammacell. Be certain that high voltage is applied to the specimen. Observe and record the readout of the EAI TDVM at one minute intervals.

   Include an estimate of the uncertainty associated with each observation, and note any unusual behavior of the data. Continue the test until a significant amount of data has been recorded. At the conclusion of the test, recheck the PAR 212 drift. The drift should be taken into account if it is significantly different from that recorded at the start of the test.

APPENDIX II: Listing of Electronic Equipment

1. John Fluke. Model 415 B High Voltage Power Supply (Serial No. 1275)

2. Princeton Applied Research (PAR). Model 212 Differential Preamplifier (Serial No. 207)

3. EG&G, Inc. Power Supply Bin (for use with the PAR 212 Differential Preamplifier)(KSU Dept. Nuc. Engg. Serial No. 1473)

4. Electronics Associates, Inc. (EAI). 5001 TDVM (KSU Dept. Nuc. Engg. Serial No. 1160)

5. Heath Corporation. Resistance Substitution Box, 15Ω to 100KΩ (KSU Dept. Nuc. Engg. Serial No. 436)

6. Standard Interval Timer, 0 to 60 min. (KSU Dept. Nuc. Engg. Serial No. 89)

7. Atomic Energy of Canada Limited. Model 220 Gammacell Irradiation Unit (KSU Dept. Nuc. Engg. Serial No. 1182)

APPENDIX III: Presentation of Raw Data

Table III-1. V versus V_o for $\dot{\gamma}$ = 123 Rad (H_2O)/min. (Data taken at t=5 min after polarity reversal.)

V_o, volts	V, volts	
	Specimen #1	Specimen #2
- 1000	-0.275	-0.586
- 800	-0.199	-0.458
- 600	-0.116	-0.315
- 400	-0.029	-0.163
- 200	0.062	-0.018
200	0.240	0.275
400	0.331	0.424
600	0.417	0.567
800	0.495	0.702
1000	0.559	0.808

Table III-2. V versus V_o for $\dot{\gamma}$ = 2400 Rad (H_2O)/min. (Data taken at t=5 min after polarity reversal.)

V_o, volts	V, volts				
	Specimen #1	Specimen #2	Specimen #3	Specimen #4	Specimen #5
-1000	-2.131	-2.345	-2.273	-1.772	-2.448
-800	-1.735	-1.945	-1.836	-1.679	-2.273
-600	-1.413	-1.615	-1.460	-1.467	-1.891
-400	-1.170	-1.370	-1.180	-1.313	-1.604
-200	-1.103	-1.233	-1.179	-1.255	-1.342
200	-0.134	-0.355	0.289	-0.511	-0.404
400	0.008	-0.138	0.445	-0.398	-0.132
600	0.332	0.173	0.804	-0.204	0.176
800	0.690	0.540	1.183	0.181	0.583
1000	1.078	0.961	1.589	0.419	1.235

Table III-3. V versus V_o for $\dot{\gamma}$ = 11,808 Rad (H_2O)/min. (Data taken at t=5 min after polarity reversal.)

V_o, volts	V, volts	
	Specimen #1	Specimen #2
-600	-9.057	-14.050
-400	-6.453	-10.154
-200	-4.201	-7.273
200	-1.599	-4.016
400	0.790	-1.132
600	3.743	2.583
800	6.807	5.937
1000	7.834	7.423

Table III-4. V versus t for $\dot{\gamma}$ = 123 Rad (H_2O)/min. (V_o = +1000V)

t, min	V, volts	
	Specimen #1	Specimen #2
1	.591	.500
2	.468	.408
3	.412	.367
4	.380	.341
5	.359	.325
6	.344	.313
7	.332	.303
8	.322	.296
9	.315	.290
10	.308	.284
11	.302	.280
12	.297	.276
13	.293	.273
14	.289	.270
15	.285	.267
16	.282	.265
17	.280	.262
18	.277	.260
19	.275	.258
20	.272	.256
21	.270	.254
22	.268	.253
23	.266	.251
24	.264	.250
25	.263	.248
26	.261	.247
27	.260	.246
28	.259	.245
29	.258	.244
30	.256	.243

Table III-5. V versus t for $\dot{\gamma}$ = 2400 Rad (H_2O)/min. (V_o = +1000V)

t, min	V, volts			
	Specimen #1	Specimen #2	Specimen #3	Specimen #4
1	.605	.792	.684	.875
2	.440	.582	.502	.669
3	.374	.495	.425	.572
4	.339	.446	.380	.528
5	.312	.414	.355	.493
6	.294	.391	.336	.467
7	.282	.373	.321	.448
8	.271	.360	.309	.432
9	.264	.348	.300	.419
10	.257	.339	.293	.407
11	.253	.332	.286	.398
12	.248	.325	.281	.389
13	.244	.319	.276	.382
14	.241	.313	.271	.375
15	.239	.308	.268	.369
16	.237	.304	.264	.364
17	.234	.300	.261	.359
18	.233	.297	.258	.354
19	.230	.294	.256	.349
20	.228	.290	.253	.345
21	.227	.287	.251	.341
22	.226	.285	.249	.338
23	.225	.282	.247	.334
24	.225	.280	.245	.331
25	.223	.278	.244	.328
26	.221	.275	.242	.325
27	.221	.273	.241	.322
28	.220	.271	.240	.319
29	.219	.269	.238	.317
30	.218	.268	.237	.314

Table III-6. V versus t for $\dot{\gamma}$ = 11,808 Rad (H_2O)/min. (V_o = +1000V)

t, min	V_o, volts	
	Specimen #1	Specimen #2
1	2.398	2.150
2	2.150	1.890
3	1.649	1.398
4	1.496	1.258
5	1.385	1.160
6	1.302	1.084
7	1.232	1.025
8	1.172	0.975
9	1.119	0.931
10	1.072	0.893
11	1.030	0.857
12	0.992	0.827
13	0.956	0.800
14	0.924	0.774
15	0.894	0.750
16	0.865	0.729
17	0.840	0.709
18	0.815	0.690
19	0.792	0.672
20	0.774	0.655
21	0.757	0.639
22	0.742	0.624
23	0.728	0.609
24	0.715	0.596
25	0.701	0.583
26	0.687	0.571
27	0.676	0.559
28	0.662	0.547
29	0.651	0.538
30	0.639	0.528

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(Table III-6, Continued)

t, min	V_o, volts	
	Specimen #1	Specimen #2
31	0.627	0.519
32	0.616	0.509
33	0.605	0.501
34	0.595	0.493
35	0.585	0.485
36	0.576	0.478
37	0.566	0.470
38	0.557	0.463
39	0.549	0.457
40	0.541	0.451
41	0.534	0.445
42	0.526	0.440
43	0.519	0.435
44	0.511	0.429
45	0.505	0.424

RADIATION-INDUCED CONDUCTIVITY IN POLYETHYLENE

by

STEVEN GREGORY FELLERS

B.S., Kansas State University, 1969

AN ABSTRACT OF A MASTER'S THESIS

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ABSTRACT

The conductivity of polyethylene during and after irradiation by Co-60 gamma rays was measured using a dc voltage source, a linear voltage amplifier, and a digital voltmeter. Three different dose rates were used which covered the range from 123 Rad (H_2O)/min to 11,808 Rad (H_2O)/min.

The constants A_γ and δ in the equation

$$(\sigma - \sigma_o) = A_\gamma \dot{\gamma}^{\delta}$$

which describes the equilibrium conductivity in polyethylene during irradiation were found to have the respective values $(1.51 \pm 0.25) \times 10^{-16}$ $[(\text{mho/cm})/(\text{Rad}(H_2O)/\text{min})^{\delta}]$ and 0.66 ± 0.02. Two distinct decay constants were found to be present in the post-irradiation recovery conductivity of polyethylene described by the equation

$$(\sigma - \sigma_o) = A_\gamma \dot{\gamma}^{\delta} \sum_{i=1}^{n} k_i e^{-t/\tau_i} .$$

The average weighting factors were $\bar{k}_1 = 0.578$ and $\bar{k}_2 = 0.422$. The recovery time constants were found to be $\bar{\tau}_1 = 2.653$ min and $\bar{\tau}_2 = 94.805$ min.