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A HEAT TREATMENT WHICH EXTENDS THE USABLE RANGE
OF AgPO_3 GLASS DOSIMETERS

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LA-2575
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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA LOS ALAMOS NEW MEXICO

REPORT WRITTEN: June 15, 1961

REPORT DISTRIBUTED: September 13, 1961

**A HEAT TREATMENT WHICH EXTENDS THE USABLE RANGE
OF AgPO₃ GLASS DOSIMETERS**

by

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Contract W-7405-ENG. 36 with the U. S. Atomic Energy Commission



ABSTRACT

A method of extending the usable range of AgPO_3 glass rod dosimeters by a post irradiation heat treatment was investigated. It was found that heating rods at 325°C for one hour clarified most of the radiation-induced darkening but did not greatly erase the fluorescent centers. Removal of the darkening allowed more efficient fluorometer readings to be made on rods exposed to more than 10^5R . The net result was an extension of the useful range of the dosimeter by a factor of 25.

The above extension has a definite application in the glass dosimetry program. There had previously existed a region between the ranges of the AgPO_3 glass system and the cobalt glass system in which doses could not be adequately measured. The described clarification technique removes the uncertainty of dose measurements in this region.

ACKNOWLEDGMENTS

The authors wish to express their appreciation to Dr. Simon Shlaer, H-6, for his helpful discussions and to Dr. Payne Harris, H-8 Group Leader, for his encouragement and appraisal of this investigation.

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CHAPTER 1

INTRODUCTION

A method of measuring gamma radiation from 10⁰R to 10⁷R with special glass dosimeters has been proposed.¹ This proposal advocates the use of two glass dosimeter systems to cover the large range. The first system is based upon the radiophotoluminescence phenomenon of AgPO₃ glass, and the second system is based upon the radiation-induced optical density change in cobalt-activated borosilicate glass.

Silver metaphosphate glass in the form of rods 6mm long by 1mm in diameter is the more radiosensitive of the two glasses. Gamma radiation forms stable fluorescence centers in this glass which, when excited by near ultraviolet, produce light with a wave length of about 6400 Å⁰. The relative intensity of this orange light is a measure of the gamma dose. These glass rods are read in a fluorometer to determine their dose. A typical calibration curve of the glass rods obtained by the specially built Bausch & Lomb fluorometer (Fig. 1) is contained in Fig. 2. The curve indicates that doses from 10⁰R to 10⁴R can be measured by the system. At doses above 10⁴R the rods begin to

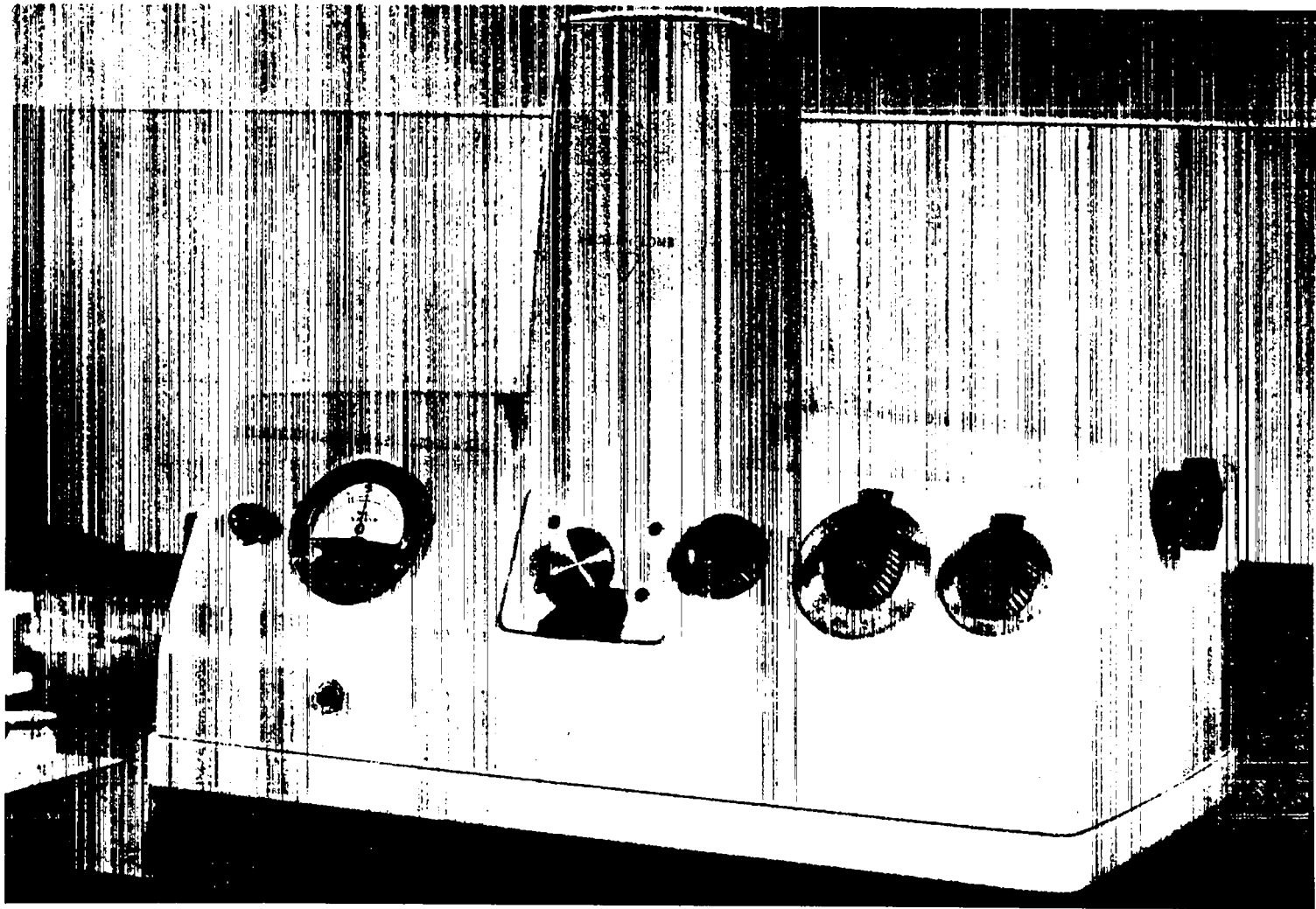


Fig. 1. Bausch & Lomb microdosimeter reader

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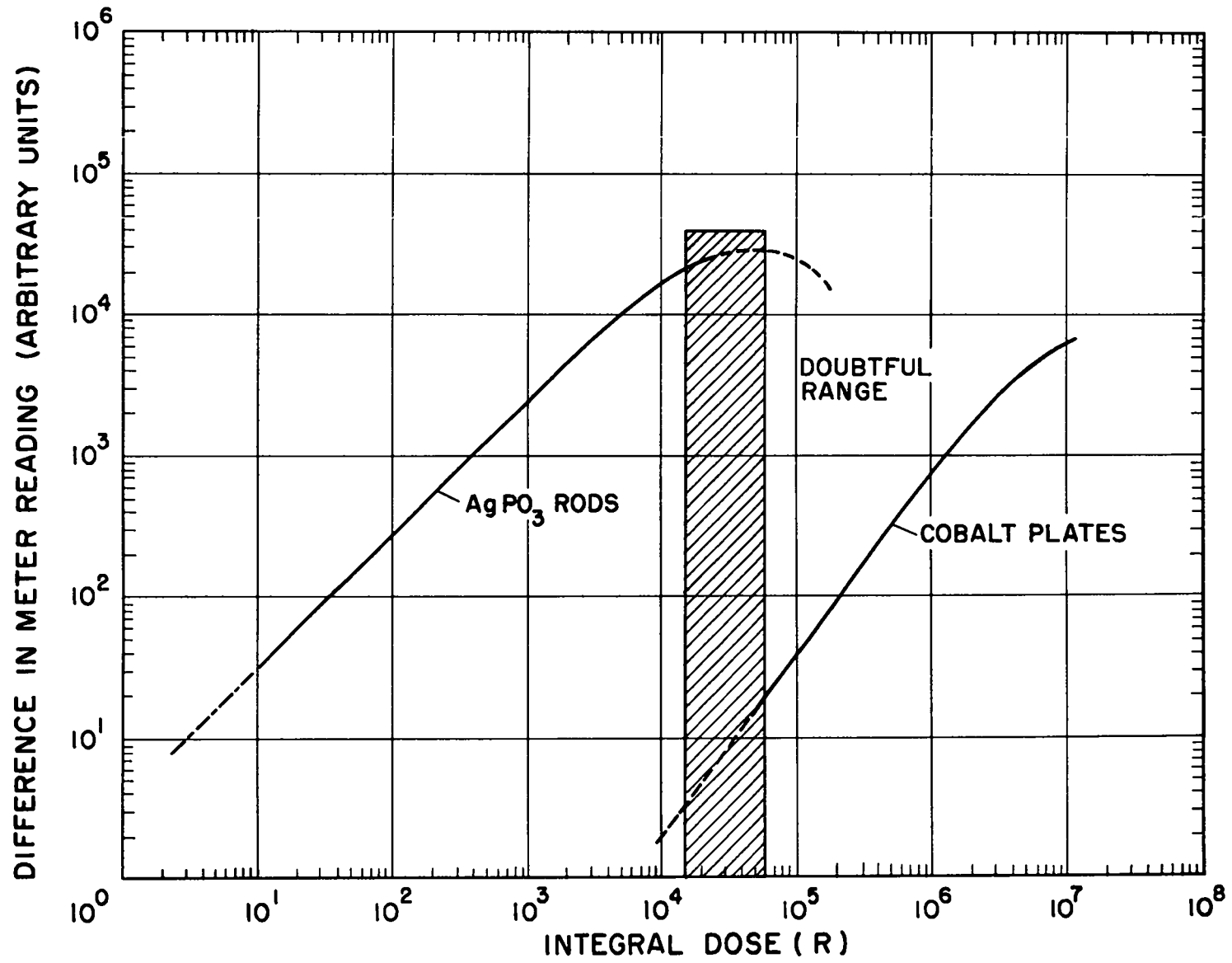


Fig. 2. Calibration curves of the AgPO₃ glass and cobalt glass dosimeters

undergo progressive radiation darkening, a characteristic of many types of glass. This darkening causes increased absorption of the incident-stimulating ultraviolet light as well as the orange fluorescence from the rods, thus producing a reversal of the calibration curve at doses in excess of 10^4 R.

Cobalt glass in the form of plates 15mm by 6mm and 1.5mm thick is used to measure gamma radiation doses from approximately 10^4 R to 10^7 R. The optical density of this glass for certain wave lengths increases with gamma radiation. Changes in the optical density are measured with a spectrophotometer. Figure 2 shows the typical response of irradiated plates as measured by the Bausch & Lomb Spectronic 20 (Fig. 3).

The calibration curves of the two glass systems as plotted on Fig. 2 indicate a region between 10^4 and 10^5 in which radiation doses are poorly defined. This poorly defined region is a result of an inadequate overlap of ranges afforded by the two systems. A method of extending the usable range of the AgPO_3 glass system and thereby allowing a measurement of doses in and well beyond the undefined region is introduced in this report.

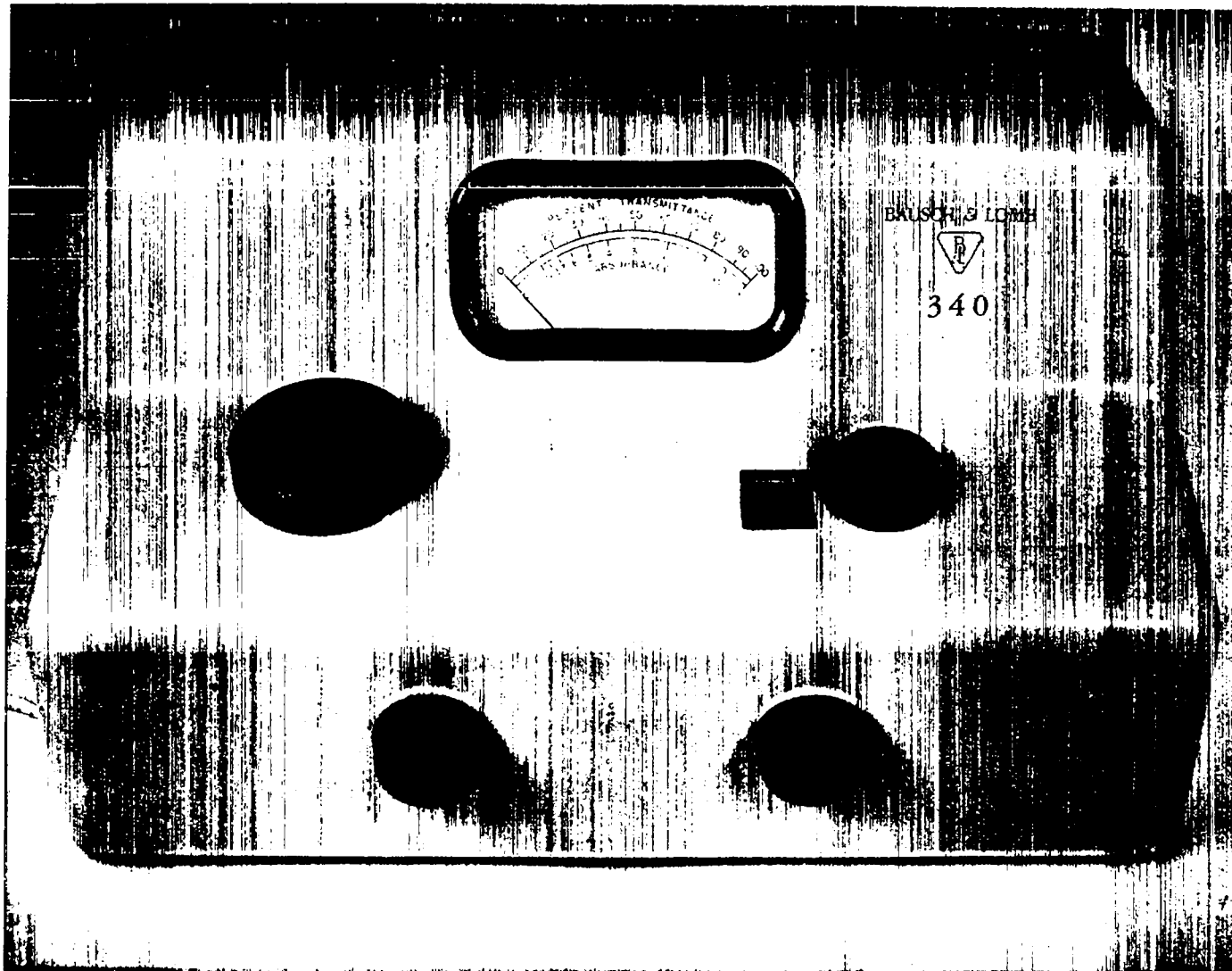


Fig. 3. Bausch & Lomb Spectronic 20

CHAPTER 2

METHODS AND RESULTS

It has been established that AgPO_3 glass is responsive to applied heat in several ways.² Perhaps the removal of the fluorescent centers of irradiated glass at temperatures in the neighborhood of 450°C is the most useful application. In an attempt to gain additional data about this erasure phenomenon, another effect of heating irradiated rods was found. When rods exposed to doses in excess of 10^4R were heated to temperatures below the erasing level of 450°C , the fluorometer readings were found to increase. These increased fluorometer readings were evidently the result of heat clarifying the rods by removing most of the radiation darkening without erasing an appreciable amount of the luminescent centers.

A study was initiated to investigate the feasibility of using a heat treatment for extending the usable range of the AgPO_3 glass system. Rods were exposed with a cobalt source to a series of doses ranging between 10^2R and 10^6R . These rods were then divided into groups with each group containing rods exposed to each of the doses.

Each group of exposed rods, with the exception of a control group, was then placed in a ceramic spot plate and heated at a particular temperature in a carefully controlled oven (Fig. 4). The oven temperatures used in the study were varied by intervals of 50° between 200° and 450°C . Half of the groups were heated for one hour and the other half for 16 hours. After being heated, the rods were read in a fluorometer. The readings of the heated rods were compared to those of the control or unheated rods.

This first appraisal indicated that, at a particular temperature, 16 hour heating times were more efficient than the one hour heating times for clarifying the rods. However, this slight advantage did not warrant the longer heating times, as the same gain could be attained by using a higher temperature with a shorter heating time. A suitable one hour heat exposure for maximizing the fluorometer reading of the high dosed rods was found to be in the 300° - 400° range.

A second study similar to the first was undertaken to test the reproducibility of the heat treatment and to define the temperature best suited for clarifying the radiation darkening with a minimum effect upon the fluorescent centers. For this study rods were exposed to 17 doses varying between 10^2 and $3.6 \times 10^7\text{R}$. These rods were then placed in groups as in the previous study. The various groups were heated at temperatures of 275° , 325° , 375° and 425°C . One hour heating times were used.



Fig. 4. Temperature-controlled oven and ceramic spot plate

The data obtained by reading the heated rods in the Bausch & Lomb microdosimeter reader are plotted on Fig. 5. Readings of unheated control rods are also plotted to serve as a means of comparison. The curves indicate that temperatures of 275°, 325° and 375° produce similar fluorometer readings. At temperatures much below 275° the coloration is not effectively removed. Heating the rods much above 375° appears to erase an appreciable number of fluorescent centers as can be deduced by comparing the 400° curve to the 325° curve (Fig. 5).

The temperature best suited for extending the range of the AgPO_3 rods would appear to be 325° when one hour heating times are used. At 325°, variations in oven temperatures do not introduce large differences in the fluorometer readings as shown by the agreement of curves in the 275°-375° range. These curves are linear up to about 10^5R and allow dosage determinations up to 10^6R . A comparison of the curves to the control curve shows that the usable range has been increased a factor of 25.

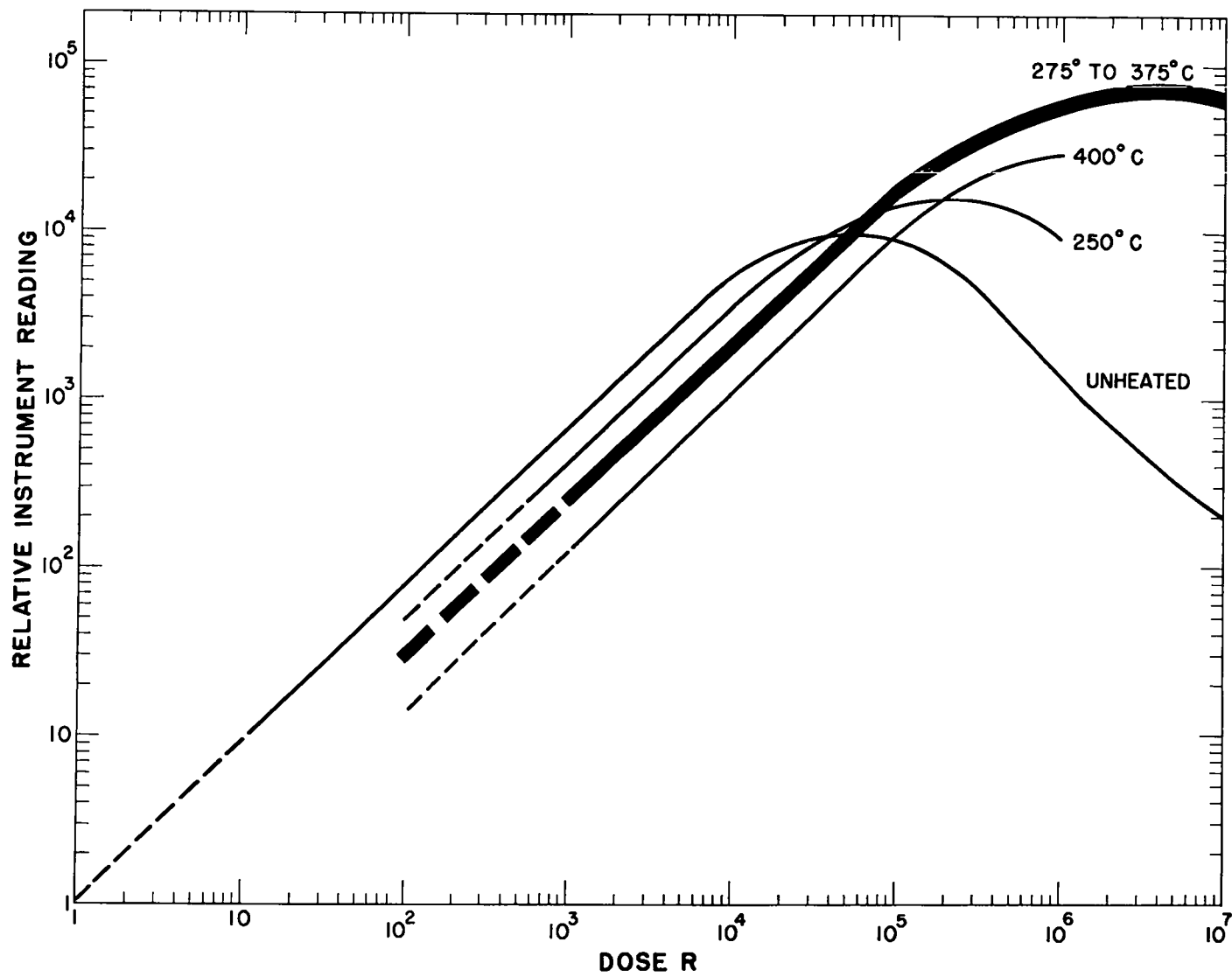


Fig. 5. Fluorometer readings after irradiated rods were heated for one hour at various temperatures

CHAPTER 3

DISCUSSION

Gamma radiation induces the development of both fluorescent centers and coloration in AgPO_3 glass. Basically both effects are a result of electrons being disturbed and raised to different energy levels by the high energy radiations.^{3,4} Doses of 10R or more produce enough fluorescent centers in the AgPO_3 glass rods to be measured by fluorometric techniques. Coloration is not noticeable until doses of approximately 10^4 R are reached. Above 10^4 R the coloration increases quite rapidly with dose. Fluorometer readings of the highly dosed rods are decreased because the coloration competes with the fluorescent centers in absorbing the ultraviolet light and because the coloration creates an opacity for the excited orange light.

Figure 6 shows the change in optical density of the AgPO_3 glass for the ultraviolet and orange light as a function of dose. These density measurements were made by measuring the density changes in AgPO_3 glass plates with a Bausch & Lomb Spectronic 20. The increasing

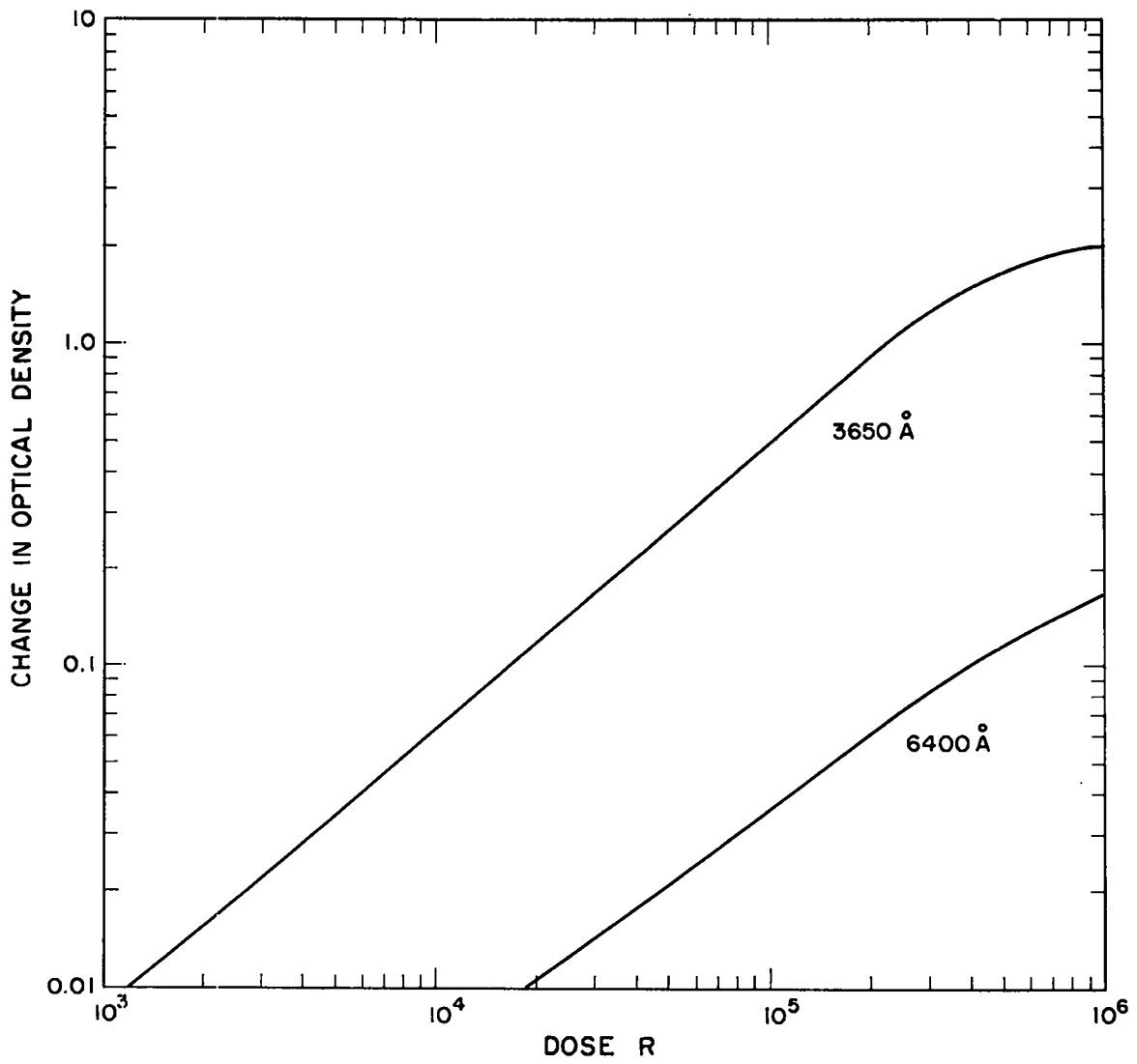


Fig. 6. Optical density changes in 1.5mm thick AgPO_3 plates

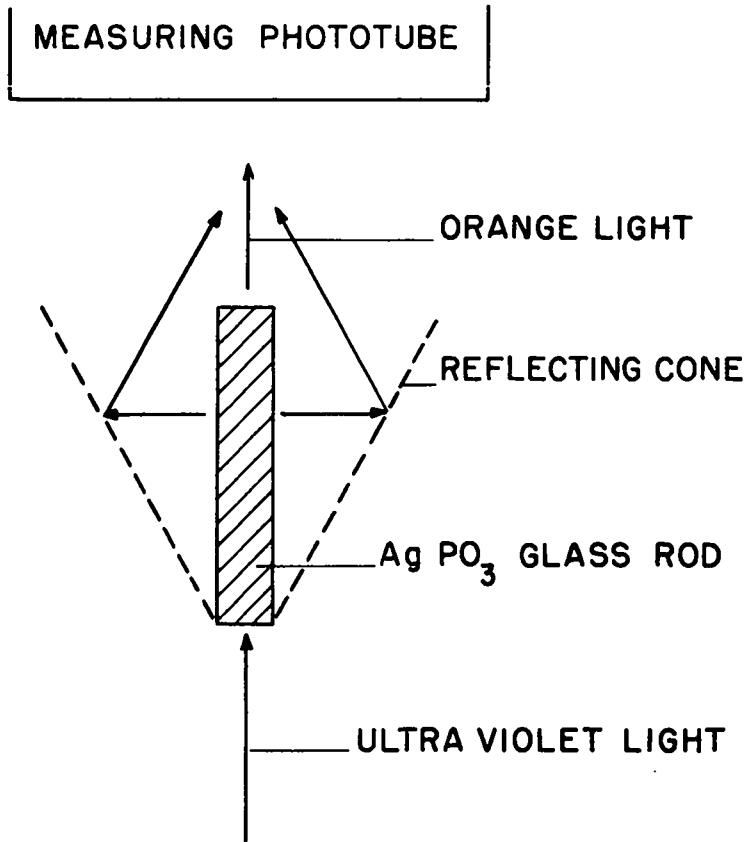
coloration of the glass is more efficient in absorbing the ultraviolet light than the orange light, as can be seen by comparing the two curves.

The adverse effects of the coloration upon fluorometer readings has been minimized in the design of some glass rod readers. Figure 7 shows sketches of the light paths in two different fluorometers as used to measure the fluorescent production of irradiated rods. Sketch A represents the early Bausch & Lomb microdosimeter reader and Sketch B represents the specially adapted Turner Model 110 (Fig. 8). In the Bausch & Lomb instrument the ultraviolet light must travel through the entire 6mm length of the rod. This distance is longer than the ultraviolet path length used by the Turner, which only needs to travel the 1mm diameter of the rod. Since the ultraviolet is highly absorbed by the coloration, the Turner should be a more efficient instrument for measuring the highly dosed rods.

A comparison of readings obtained by the two fluorometers is shown in Fig. 9. The Turner which is affected less by the coloration in the rods is able to read doses to about a factor of 5 higher than the Bausch & Lomb. The effects of coloration upon readings obtained with the Turner can be seen by a slope change and then a reversal of the calibration curve at doses above $10^5 R$.

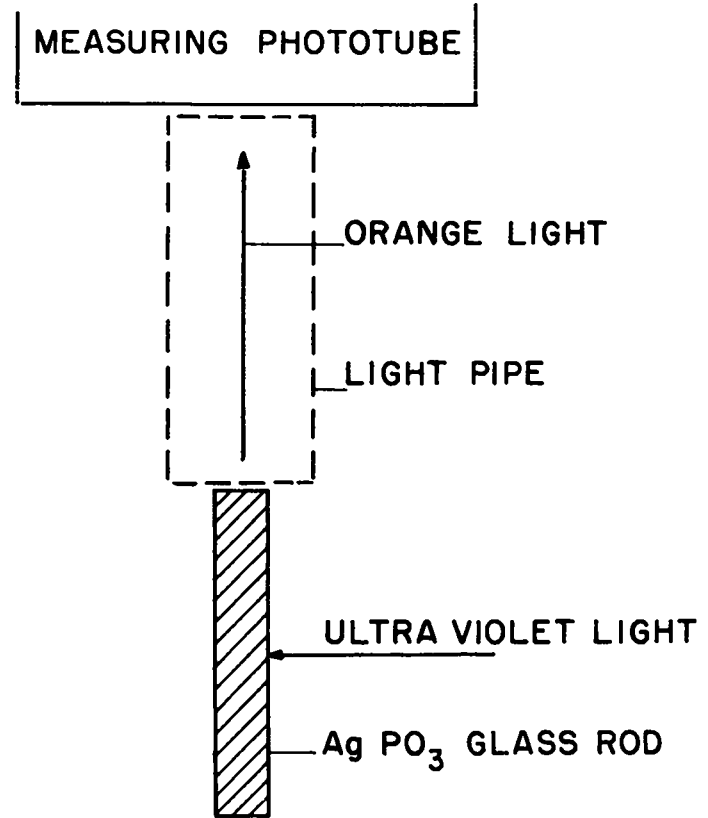
The coloration can be removed by heating temperatures which are lower than those needed to remove or erase an appreciable number of the fluorescent centers. This method is possible because the electrons producing the coloration are evidently more loosely bound than those

SKETCH A



BAUSCH AND LOMB
MICRODOSIMETER READER

SKETCH B



TURNER MODEL 110 FLUOROMETER
WITH GLASS ROD HOLDER ADAPTATION

Fig. 7. Optical schematic of two glass rod readers

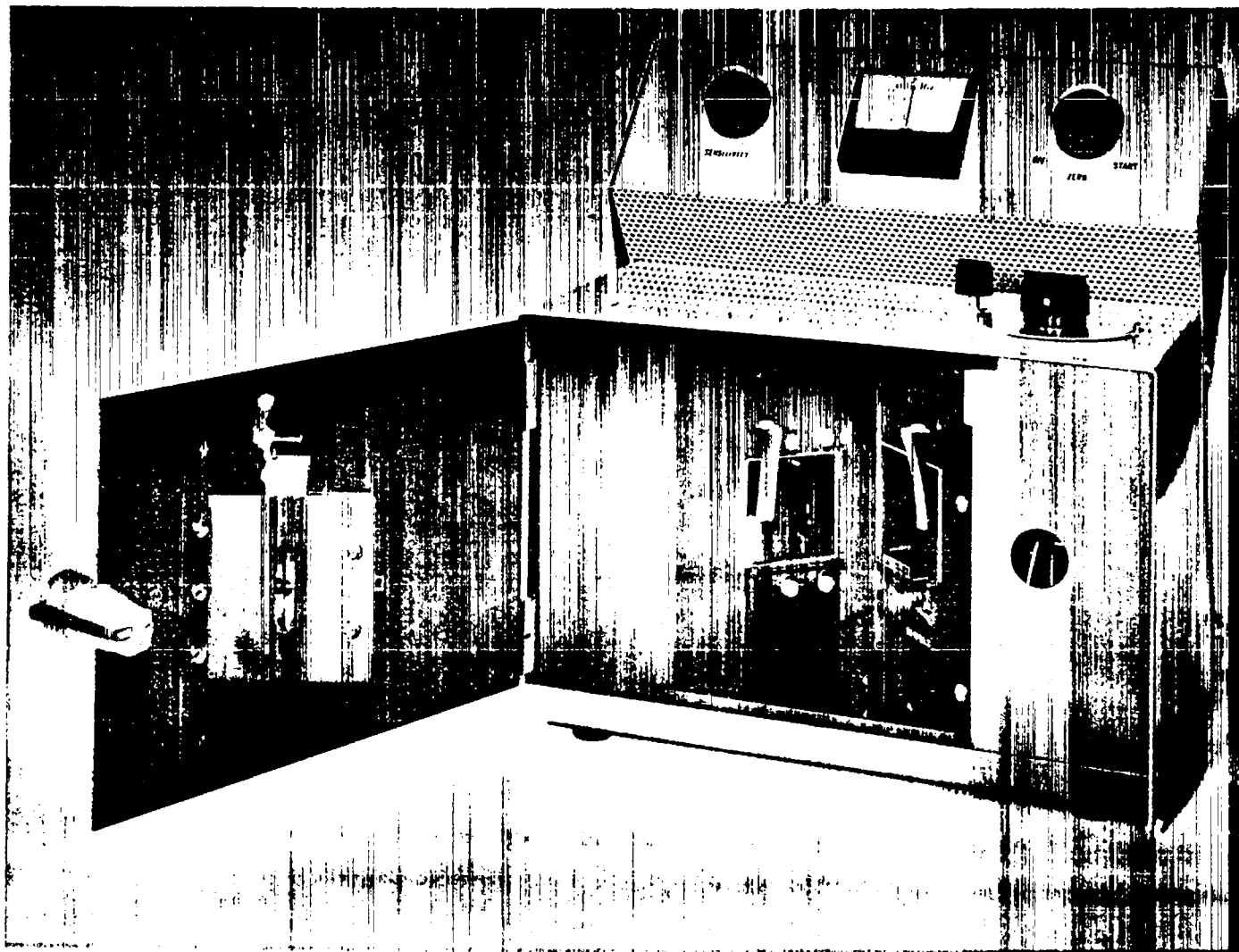


Fig. 8. Turner Model 110 fluorometer adapted to read glass rods

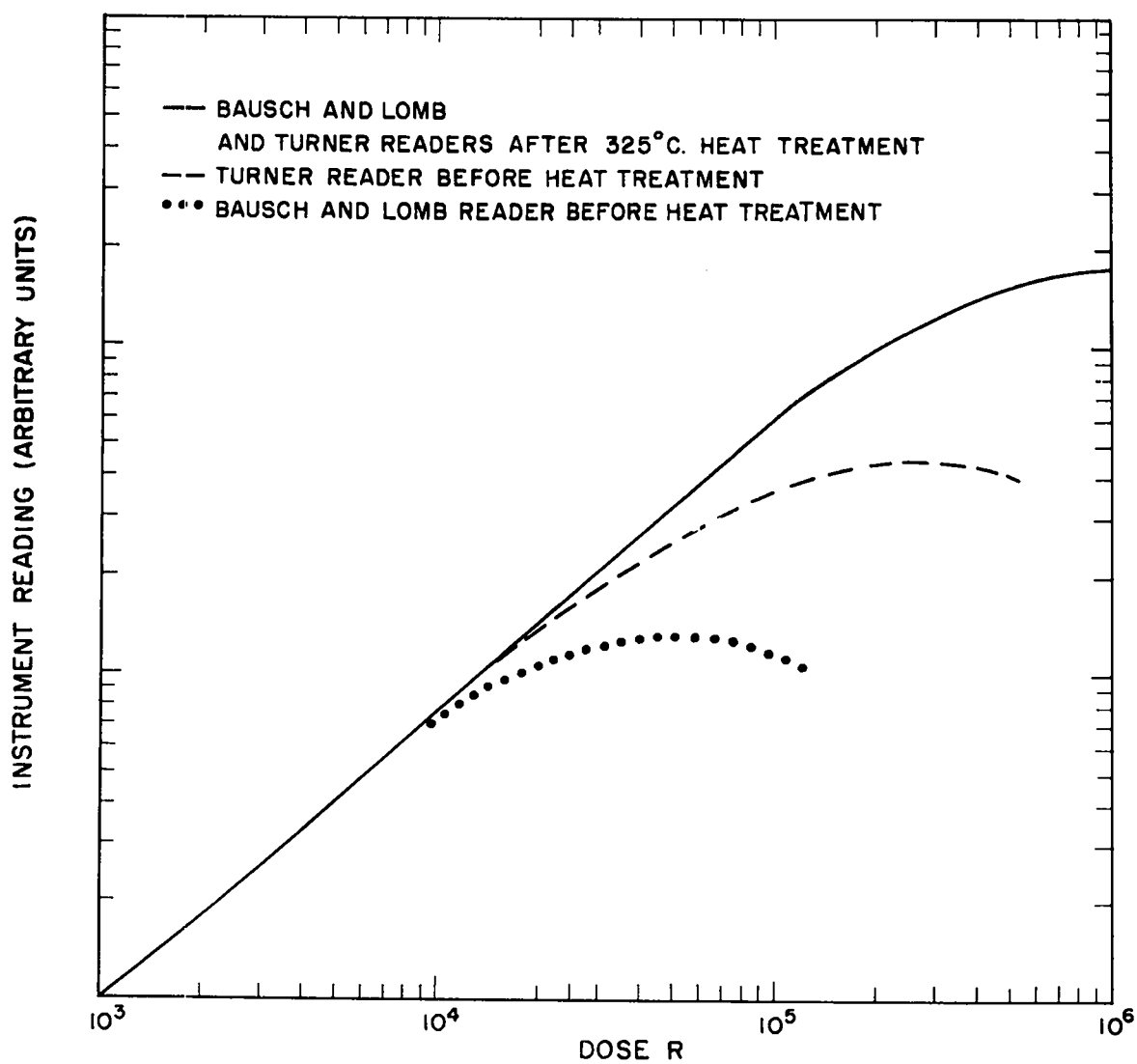


Fig. 9. Comparison of readings obtained by two fluorometers before and after irradiated rods were heated for one hour

producing the fluorescent centers. Therefore, less heat energy is required for their liberation. Temperatures in the vicinity of 325° appear to produce a maximum clarification of the rods and a minimal erasure of fluorescent centers. As a result of a clarification heat treatment, the fluorometer readings of both the Bausch & Lomb and Turner instruments are linear with dose up to 10^5 R (Fig. 9), and doses as high as 10^6 R can be measured.

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