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PHASE II FINAL ENGINEERING REPORT FOR THERMOLUMINESCENT DOSIMETER COMFUTER-INDICATOR SYSTEM

## PHASE II

## FINAL ENGINEERING REPORT FOR THERMOLUMINESCENT DOSIMETER COMPUTER-INDICATOR SYSTEM

This Report covers the period: 15 June 1962 to 15 October 1962

EDGERTON, GERMESHAUSEN & GRIER, INC. Santa Barbara, California

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## NAVY DEPARTMENT BUREAU OF SHIPS

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## ABSTRACT

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During the preceding 5-month interval final design criteria have been established for the thermoluminescent dosimeter computer-indicator.

Techniques for the production of the CaF<sub>2</sub>: Mn phosphor, application of the phosphor to the dosimeter heater substrate, and electronic and mechanical design for the computer-indicator are described. Performance of the system, its reliability and accuracy are presented in as complete detail as is known.

#### 1. PURPOSE

1.1 The effort of this research and development program was directed toward the development of a system of personnel radiation monitoring based on the thermoluminescent properties of CaF<sub>2</sub>:Mn developed by the Naval Research Laboratories. To accomplish this effort it was necessary to investigate thoroughly the production, quality control, deposition, and response of the phosphor, and devise a practical dosimeter based on this study. It was necessary to construct a stable, reliable, and efficient device to measure the light output of the dosimeter and to provide automatic printout of the identification number and dose information. Included in the development program was the establishment of quality control methods for the production of the phosphor and the fabrication of a standard for the computer-indicator.

1.2 The Phase I portion of the thermoluminescent dosimetry research and development program was satisfactorily completed on September 3, 1961 and Final Phase I interim engineering reports have been issued.

1.3 The Phase II portion of the thermoluminescent dosimetry research and development program was satisfactorily completed on October 15, 1962 and the final Phase II Report and all interim engineering reports have been issued.

## 2. GENERAL FACTUAL DATA

#### 2.1 Laboratory and Equipment

2.1.1 To evaluate fully the production techniques and incorporate as many standard vacuum tube components in the final dosimeter as possible, laboratory space was acquired at the Boston facility of EG&G, Inc. A minor part of the development work was then assigned to this facility.

2.1.2 No additional Corporate or Navy funds were used since the last reporting period for equipment. Expendable supplies, i.e., chemicals, selected metals, were purchased as needed. Purchasing of parts to fabricate the required five computerindicators and 500 thermoluminescent dosimeters has begun.

#### 2.2 Technical People in Program

2.2.1 Dr. Ernest F. Blase is Project Scientist for this program and devotes full time to this program

2.2.2 Dr. Richard C. Palmer is responsible for all phases of the chemical research and devotes full time to the project. 2.2.3 Mr. David F. Rutland is responsible for the electronic development. Mr. Rutland is available full time for this program.

2.2.4 Mr. Roger E. Lagerquist is responsible for the mechanical design for the computer-indicator as well as the design of the dosimeter, case, and biological shield.

2.2.5 In addition, Mr. Vincent Poirier and Mr. Albert Malm are participating in the program.

2.2.6 Mr. Seymour Goldberg and Mr. William Soule are involved in the Boston research effort.

## 3. DETAIL FACTUAL DATA

#### 3.1 Phosphor Production

3.1.1 NRL Technique

3.1.1.1 The continuing research on techniques of phosphor production which extended through the entire research and development phase of our project were necessary in order to provide large-scale production methods. It had been supposed

the thermoluminescent dosimeter would eventually be made in lots of 1,000 to 100,000. It is unrealistic to consider mass production of a dosimeter if one must synthesize the phosphor in batches of 25 grams requiring 24 hours per batch. Satisfactory completion of a novel method of producing the phosphor will ultimately result in substantial savings to the Bureau of Ships at a later date.

3.1.1.2 Phosphor production has been satisfactorily accomplished by the technique developed at NRL. In this technique, an intimate mixture of 1.29 gm  $NH_4MnF_3$  and 25 gm of  $CaF_2$  is fired in a sealed platinum crucible with a secondary platinum cover and partially covered by graphite in a ceramic crucible for 16 hours at 1200°C. Maximum light output is at 280°C with a small peak at 125°C (~1% of total).

3.1.1.3 Although an active phosphor is produced when commercial grade  $OaF_2$  and  $MnF_2$  are mixed and fired, much greater sensitivity is achieved by firing specially prepared materials. The  $CaF_2$  is produced by treating  $CaCO_3$  with HF, while the  $NH_4MnF_3$  (source of  $MnF_2$  when heated) is produced by reacting  $MnCl_2$  with  $NH_4F$ . Details are presented in the previous engineering reports.

3.1.1.4 Raw materials for the production of  $CaF_2$  and  $NH_4MnF_3$  have been standardized to:

- 1. Special luminescent grade CaCO<sub>3</sub>;
- Special luminescent grade MnCO<sub>3</sub>;
- 3. Analytical reagent grade MnCl<sub>2</sub>. 4H<sub>2</sub>O;
- 4. Analytical reagent grade NH<sub>4</sub>F;
- 5. Transistor grade HF;
- 6. Distilled water.

3.1.1.5 While tests with other grade reagents have given phosphors with adequate sensitivity, the most consistent results with good sensitivity have been achieved with the above materials. All of these reagents are readily available with only a small difference in cost.

3.1.1.6 After the sample is fired and ground up, it is leached for 1 hour at near boiling point in concentrated HCL. Sensitivity of phosphor is increased in many cases. This is a technique developed by R. Ginther of NRL. The leaching technique is used for all phosphors, regardless of the preparative technique.

3.1.1.7 Experiments to increase the batch size during firing from 25 to 100 gm have been unsuccessful at SBL due to equipment limitations, however, Dr. R. Ginther in a letter of August 14, 1962, to Dr. E. Blasc states that he has been able to produce a 125-gm batch that "has essentially no low-temperature peak, and its sensitivity is equal to that of my usual standard." At SBL, poor sensitivity and a large percentage of the light output appearing at 125°C have resulted. Α large phosphor boule (75 gm) was sectioned and it was ascertained that the phosphor characteristics were varying throughout the boule. It is felt that this is because CaF<sub>2</sub>-is a good insulator and thus the large batches are not heated uniformly. (At the present time there are no plans to experiment further with large batches since SBL has been able to produce 25-gm batches of phosphor with good sensitivity from the coprecipitated materials with 30-minute firing times, see Section 3.1.3,).

3.1.1.8 Experiments to determine the effects of  $MnF_2$  content on the phosphor have been carried out for 1, 2, 3, 4, and 5 mole %  $MnF_2$ . The sensitivity increases gradually to a maximum

at 4 mole % and then falls off; the sensitivity being approximately 20% greater at this point than that at 3 and 5%. As no attempt was made to ensure the same particle size (see Section 3.3 for effect of particle size), it was concluded that the 3, 4, and 5 mole % were not sufficiently different to justify a change in the NRL technique from 3 mole %. 11

3.1.1.9 Experiments were run to determine the effect of varying the firing time on the quality of the phosphor. It was shown that the sensitivity of both the high-temperature peak (280°C) and the low-temperature peak (125°C) increases for the first 6 hours of firing at which time the low-temperature peak comprises some 30% of the total light output. The sensitivities of both peaks then decrease over the next 4 hours to a value which remains fairly constant up to 16 hours at which time the low-temperature peak comprises about 1% of the total light output. Thus the time of firing can be shortened to about 12 hours; however, due to the advantage of firing samples overnight (4 p.m. to 8 a.m.), the time of firing has been left at 16 hours.

3.1.1.10 Experiments have been performed to check the effect of temperature on the quality of the phosphor. In general, as the temperature is decreased below  $1200^{\circ}$ C, the sensitivity of the phosphor goes down and the time required to reduce the low-temperature peak to 1% of the total light output goes up. As the temperature is increased up to  $1400^{\circ}$ C (above the melting point) the sensitivity goes down to about 30% of the NRL best sample; but the time of firing is also subsequently reduced until at  $1400^{\circ}$ C, only 10 minutes is required for firing. Considerable loss of material is encountered above  $1200^{\circ}$ C.

3.1.1.11 To fire samples in controlled atmospheres a chamber was constructed for the Lepel Induction Heater, Figure 1. Attempts to fire samples of  $CaF_2$  and  $NH_4MnF_3$  have been unsuccessful. The crucible covers have all been blown off even though the temperature is raised in a gradual manner. If the covers are omitted the chamber becomes coated with a white deposit as 1200°C is approached. The resulting phosphor has very poor sensitivity.

#### 3.1.2 Coprecipitate Technique

3.1.2.1 Phosphor production has been satisfactorily accomplished by firing at  $1200^{\circ}$ C for 4 hours a coprecipitated mixture of CaF<sub>2</sub> and MnF<sub>2</sub>. The mixtures are fired in double platinum crucibles with double tops contained in a ceramic crucible containing graphite and a platinum cover.

3.1.2.2 Raw materials for the production of the coprecipitated mixture have been standardized to:

- 1. Special luminescent grade CaCO<sub>3</sub>;
- 2. Special luminescent grade MnCO<sub>3</sub>;
- 3. Transistor grade HF;
- 4. Distilled water.

3.1.2.3 The production of the unfired powder is accomplished by mixing the  $MnCO_3$  and the  $CaCO_3$  in a wet slurry and then adding HF. For a 3.45 mole %  $MnF_2$  sample, for example, 1.32 gm of  $MnCO_3$  and 32 gm of  $CaCO_3$  are mixed 50 ml of water, to which 25 ml of HF is added with much stirring. The precipitate is washed free of HF and then dried at 95°C for 48 hours. A pink powder which is weakly thermoluminescent before firing is obtained.



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FIGURE I. VACUUM CHAMBER CONTROL

3.1.2.4 Samples of coprecipitated material have been prepared with 2, 3, 3.45, 4, 5, 10, and 20 mole % MnF<sub>2</sub>. The best sensitivity is obtained with the 3.45 mole % although the 3 and 4 mole % are only down in sensitivity by 10 to 15 %. Again, particle size could account for the difference. The sensitivity of the 3.45 mole % samples averages from 80 to 100 % of that of the NRL Sample 9638-184-92961L as measured at SBL. No low-temperature peak is produced.

3.1.2.5 Experiments to check firing time and temperature have been performed with the best sensitivity achieved by firing for 4 hours at 1200°C In this case, however, it has been demonstrated that sensitivity is a nebulous quantity, for when the same particle sizes from samples fired for different times and temperatures are compared, one finds the same sensitivity. The smaller the particle size, the poorer is the sensitivity, a fact directly related to time and temperature of firing time (assuming no loss of  $MnF_2$  from the system) give larger crystallite sizes and hence, greater sensitivity).

## 3.1.3 <u>Coprecipitate Technique adapted to Controlled</u> <u>Atmosphere</u>

3.1.3.1 In the NRL technique of firing phosphor, one depends on the HF formed from decomposition of the  $NH_4MnF_3$  together with the graphite outside the crucible to furnish a slightly reducing atmosphere to protect the manganese from oxidation. In the Coprecipitate Technique, the reducing atmosphere and/or inert atmosphere is furnished solely by the reduction of the incoming oxygen to CO and  $CO_2$  (dependent on temperature). The controlled-atmosphere chamber for use with the induction heater was set up to

check the following atmospheres: (a) vacuum, (b) inert, (c) oxidizing, and (d) reducing. The atmosphere may be either static or flowing. 11

3.1.3.2 <u>Vacuum Firing</u>. Samples were inserted in a vacuum system and the temperature was raised slowly to  $1200^{\circ}$ C such that the pressure did not exceed 10 microns. Samples were fired at  $1200^{\circ}$ C for 30 minutes. Subsequent analysis revealed no low-temperature characteristic. Sensitivity was about 75% of that of NRL-9368-184-92961L. No change between a static vacuum or a pumping vacuum at  $1200^{\circ}$ C. At  $1360^{\circ}$ C (melting point of CaF<sub>2</sub>) extreme loss of materials was encountered.

3.1.3.3 <u>Inert Atmosphere</u>. Both dry argon and dry helium have been used as inert atmospheres. In both cases the results are identical. Samples are first vacuum dried at  $\sim 200^{\circ}$ C and then flushed with inert gas as the temperature is raised to 1200°C. Time of firing is 30 minutes after which time the samples are cooled, ground and analyzed. No low-temperature peak is produced. Over-all sensitivity is 85 to 95% of NRL-9368-184-92961L.

3.1.3.4 <u>Oxidizing Atmosphere</u>. The chamber is left open to the air as the sample is raised to 1200°C. The time of firing is 30 minutes at 1200°C. Dark-colored phosphor which does not leach white is obtained. Very poor sensitivity but high doses indicate the presence of no low-temperature peak.

3.1.3.5 <u>Reducing Atmosphere</u>. The reducing atmosphere used in the chamber was CO. In general, CO will reduce metal salts to corresponding metal or convert to metal carbide at temperatures between 300 to 1500°C dependent on the particular compound. The samples were first placed in the vacuum chamber and dried at 200°C. They were then flushed with dry

CO as the temperature was raised to  $1200^{\circ}$ C. The time of firing at  $1200^{\circ}$ C was 30 minutes. The sensitivity of samples was 85 to 95% of NRL-9368-184-92961L; however, in each sample there was a low-temperature peak which comprised 2 to 3% of the total light output. As all unfired samples were from the same batch, times of firing in various atmospheres were the same; temperatures of firing were identical as determined by optical pyrometer, and the only variable was atmosphere. The conclusion drawn was that a reducing atmosphere (in this case CO) introduced a low-temperature peak into the phosphor.

3.1.3.6 <u>Carbon Block</u>. To check the production of the lowtemperature peak further a carbon block was placed inside the platinum crucible on a platinum sheet above the coprecipitated material. The crucible was sealed and raised to 1200°C for 1 hour. The phosphor obtained had good sensitivity but contained 3% low-temperature characteristics. A sample fired without carbon block has lower sensitivity but no low-temperature peak.

3.1.3.7 <u>Graphite Powder</u>. Samples were fired as described in Section 3.1.2.1 but in this case the crucible was immersed in graphite powder. The phosphor that resulted contained 30% low-temperature characteristics but had over-all good sensitivity.

## 3.1.4 Summary

3.1.4.1 The NRL technique gives good sensitivity with < 1% low-temperature output and the phosphor produced thereby is adequate for use in the dosimeter. The firing time, of 16 hours, however, in a slightly reducing atmosphere at  $1200^{\circ}$ C with specially prepared materials introduces variables which at best are difficult to control and reproduce.

3.1.4.2 The SBL technique of firing coprecipitated samples with no controlled atmosphere in a double platinum and ceramic

crucible containing carbon for 4 hours at 1200°C gives a phosphor with good sensitivity with essentially no lowtemperature peak. The sensitivity is adequate for use in the dosimeter. The firing time of 4 hours at 1200°C from a single powder (coprecipitate) is an advantage over the NRL technique but like the NRL technique, the same variable atmosphere is hard to control and reproduce. 11

3.1.4.3 The SBL technique of firing coprecipitated samples in a controlled inert atmosphere in single platinum for 30 minutes at  $1200^{\circ}$ C gives a phosphor with good sensitivity and no low-temperature peak. The sensitivity is adequate for use in the dosimeter. From the experiments run so far by this technique it seems superior to both of the above techniques in that (a) the temperature of the phosphor can be more accurately determined and controlled, (b) the atmosphere is known and can be controlled, and (c) the time of firing at  $1200^{\circ}$ C can be accurately determined. (A phosphor contained in a series of crucibles in an oven does not reach equilibrium temperature in a "known" time.) The only inherent variable, one that is present in any technique, will be the starting material, namely its MnF<sub>2</sub> content and trace impurities.

## 3.2 Phosphor Evaluation

3.2.1 <u>Emission Spectrum</u>. The fluorescent emission spectrum of the phosphor which is the same as the thermoluminescent spectrum peaks at ~ 5000A with 95% of the light output between 4300 to 5900A. This is a close approximation to the quantum efficiency curve of an S-11 response tube, and hence such a tube is recommended for the thermoluminescent dosimeter reader.

3.2.2 <u>Manganese Content</u>. The sensitivity of the phosphor is affected by a change in MnF<sub>2</sub> content as measured by an

S-11 response phototube. A slight shifting of the emission spectrum is caused by an increase in  $MnF_2$  content which partially explains the apparent change in sensitivity. The difference between 3 to 5 mole %  $MnF_2$ , however, is negligible such that any phosphor with manganese content within this range may be used in the dosimeter.

3.2.3 <u>Particle Size</u>. Particle size as shown in Section 3.3 does affect the sensitivity of the phosphor. The actual size to be used will be determined by the dosimeter itself, that is to say, when it is deposited on a heating element it will be able to see 2 mr.

3.2.4 <u>Sensitivity</u>. Sensitivity of a phosphor is a nebulous term as mentioned before and many things will contribute to this quantity: (a) emission spectra, (and hence  $MnF_2$  content), (b) response of a given phototube, (c) particle size, and (d) amount of phosphor present in milligrams per square centimeter. Hence, one must specify all of these variables when standardizing the phosphor. The more nearly these quantities are controlled, the more lenient may be the requirements on the phototube itself.

#### 3.3 Phosphor Light Output Characteristics

3.3.1 Light emission during thermoluminescence is proportional to the dose received. Previous work at the Naval Research Laboratory indicates the light output is linear to at least  $10^5$ r. Comparative analysis between phosphors can be complicated by a variety of conditions. Figure 2 illustrates the effect of particle size on light output. For this experiment 1-gm samples of the phosphor were dosed to 95r. The samples were placed in a small platinum planchet and read in the laboratory photometer. It has been determined previously that the resulting layer of phosphor was "infinitely thick" with respect to its own light out. As can be seen from the h



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diagram, there is approximately three times the light out for equal phosphor weight and equal doses when one uses phosphor with a particle size of 150 mesh as compared to 400 mesh.

3.3.2 Similar studies were made on the effect of particle size versus thickness. Figure 3 shows the results of experiments with phosphors smaller than 400 mesh, between 150 and 400 mesh and larger than 150 mesh. The dashed lines indicated extrapolations based on the data in Figure 3.

#### 3.4 Bonding of Phosphor

3.4.1 In order to ensure the most economical production technique for bonding the phosphor to the heater substrate the probelm was approached at the Santa Barbara Laboratory and by the vacuum tube specialist at our Boston facility. Several techniques, i.e., use of DC-805, would have provided an emergency method for depositing the phosphor. If typical production techniques such as air spray deposition, dipping and electrophoretic deposition could not be used, the production of dosimeters would depend on development of special techniques or prohibitively costly hand manufacture. As a consequence of the extraordinary effort expended in depositing the sensitive phosphor revealed several applicable production techniques.

3.4.2 The preceding discussions of the phosphor apply to the pure phosphor. To bond the phosphor to the heater substrate a number of methods were experimentally evaluated. All of these tests were performed using the laboratory photometer. The phosphor and bonding agent were mixed in a 3/4 inch copper planchet and the glow curve determined in the identical fashion as the pure phosphor. A complete description of this system may be found in the Phase II, First and Second Interim Engineering Reports.



FIGURE 3. LIGHTOUT VS PHOSPHOR THICKNESS

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3.4.3 The original method (A) of using Dow-Corning 805\* silicon resin proved to be awkward for other then laboratory prototypes. Several disadvantages to the Dow-Corning resin are its long curing time (16 hours), volatility at the readout temperature of the dosimeter causing condensation of the organic material on the glass envelopes and lack of applicable production techniques. Nevertheless, a number of successful dosimeters were made to determine preliminary geometrical data, besides the experimental evaluation of the effect of DC-805 on the phosphor. The fact that the DC-805 introduced no low-temperature peak is shown in the glow curve in Figure 4.

3.4.4 A number of successful dosimeters were made by intimately mixing the CaF<sub>2</sub>:Mn phosphor with Amaco Metal Enamel\*\* and firing the mixture in an inert atmosphere until the glaze meits. The resulting fused mass is extremely hard and durable. There is no effect on the glow curve of the phosphor as is illustrated in Figure 5. As a preliminary evaluation of CaF,:Mn dosimeter, a number of these "buttons" were exposed to the SMALLBOY atomic event at the Nevada Test Site on July 14, 1962. The "buttons" were not shielded, and as a consequence, do not follow the chemical and glass dosimeter data exactly. Figure 6 is the plot of the three systems with arbitrary units for dose and distance. It is anticipated this particular dosimeter design will be continued as it has a variety of applications in other than personnel dosimetry. A particular advantage of the ceramic glaze is that the fused mass is transparent and allows all the emitted light to be

<sup>\*</sup> Can be obtained from Dow Corning Corporation, Midland, Michigan.

<sup>\*\*</sup> Can be obtained from American Art Clay Company, Indianapolis, Indiana.







FIGURE 5. GLOW CURVE, Ca F2: Mn + CERAMIC GLAZE

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used. A disadvantage is that the material is essentially heavy metal oxide and this complicates the calculation of effective shields.

3.4.5 Figure 7 is the glow curve for phosphor and Melbond Ceramic Adhesive Type CA-100.<sup>\*</sup> Though there is no apparent introduction of a low-temperature peak with the Melbond, the opaque adhesive eliminates all light from the phosphor except that exposed on the surface.

3.4.6 A similar loss of sensitivity was noted for the combination of phosphor and Sauereisen \*\* cement. Figure 8 illustrates the resulting glow curve for this mixture.

3.4.7 A method of air impacting the finely divided phosphor was investigated. To accomplish this, the pressure-limiting switch on an S.S. White "Airbrasive" unit was bypassed and the phosphor was blown by compressed nitrogen and impinged on a nickel or platinum foil. It was calculated the velocity of the particles was greater than 0.9 Mach. The particle size was 150 to 300 mesh and it was impacted on 5-mil platinum which was supported by a mandrel during the process. A photomicrograph, Figure 9, is the phosphor on platinum. The clear area is the plain platinum. Though quantitative data are lacking for this technique, it is estimated that less than  $1 \text{ mg/cm}^2$  is deposited on the phosphor. The glow curve, Figure 10, indicates no introduction of a low-temperature peak. However, the insufficient amount of matter caused the resulting dosimeter to be insensitive.

\* Available from Melpar, Inc., Falls Church, Virginia.

<sup>\*\*</sup> Available from Suaereisen Cements Co., Pittsburg, Pennsylvania.



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FIGURE 7. GLOW CURVE, CaF2: Mn AND MELBOND



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3.4.8 Planchet-shaped dosimeters were made by using nickel powder, 400 mesh, and the phosphor and pressing them at 25,000 psi. Serious consideration was given to developing this into a cylindrical, resistive-type element by adjusting nickel content and using this as the dosimeter element. After several experiments it was obvious that the required sensitivity could only be achieved by a considerable increase in surface area and calculable large increase in volume. It is anticipated that other applications will be found for this technique. 1

3.4.9 Vacuum-evaporated samples were completely impractical. An extensive research program would be necessary to determine experimentally the temperature of the  $MnF_2$ , the temperature of the  $CaF_2$ , substrate temperature, etc., in order to use this procedure.

3.4.10 A considerable effort was expended in the research and development phase to find satisfactory techniques to sinter the phosphor to the heater substrate. The technique of sintering metal oxide has been long used in the vacuum tube industry in the preparation of cathodes. The sintering of the  $CaF_2$  Mn phosphor proved to be a considerable more difficult task. First at the temperatures at which sintering occurred (over 900°C) the manganese content was considerably altered and, therefore, in some unpredictable fashion, so was the glow curve. A second problem was the failure of the sintered  $CaF_2$  to adhere to the heater substrate. The best compromise for sintering the phosphor was to use a standard phosphor ground to 300 to 400 mesh and spray the mixture on the heater substrate. The binder used was RCA Type 33-B-11.\*

<sup>\*</sup> Available from Radio Corporation of America, Camden, New Jersey.

After decomposing the binder, the phosphor on the substrate was heated by radiant heat in an induction furnace at 1000°C for periods of 0.25 to 20.0 minutes. During the heating cycle the device was in an argon atmosphere. A second method of accomplishing the heating was to resistance-heat the wire or metal substrate in a vacuum after depositing the phosphor. The problem of lack of adherence was solved by using nickel wire mesh. The phosphor sintered to itself around the wire mesh and formed a satisfactory bond. Figure 11 is a dosimeter in which the phosphor was sintered on a nickel mesh.

3.4.11 As was described previously (Section 3.1), the synthesis of the phosphor requires the activation to proceed at an elevated temperature. A number of experiments were conducted by spraying the unactivated material on the heater substrate and accomplishing the activation simultaneously with the sintering. Samples of  $CaF_2$  with 2, 3, 4, 5, and 10% MnF<sub>2</sub> were sprayed on nickel mesh heater substrates and fired for periods of 1 to 15 minutes. The most sensitive phosphors were made with 4% MnF<sub>2</sub> in the starting material. In general, dosimeters made by this technique were less sensitive than when pre-activated phosphor was used.

3.4.12 Figure 12 is the glow curve of a 50% Kasil<sup>\*</sup> and 50% phosphor mixture. Kasil, which can be applied at room temperature, dries to a translucent, hard, adhering mass. Mixtures of Kasil and phosphor were fired in the following proportions by weight: 10 parts phosphor to 1 part Kasil; 3 parts phosphor to 1 part Kasil; 1 part phosphor to 1 part Kasil. The best adherence was achieved with the latter. To determine the effect of prolonged heating on the phosphor-

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<sup>\*</sup> Can be obtained from Great Western Chemical Co., Los Angeles, California



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FIGURE II. SINTERED PHOSPHOR DOSIMETERS





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Kasil mixture, a planchet, such as described in Section 3.4 was made and the glow curve determined. This planchet was then heated at  $350^{\circ}$ C for 30 hours and the glow curve was reproduced. If there is any diffusion of the potassium ions into the CaF<sub>2</sub>:Mn phosphor it has no effect. A second test was performed by using a laboratory prototype dosimeter which was heat-cycled for 100 times and a 5-mr dose determined at the first, twenty-fifth, fiftieth, seventy-fifth, and one hundredth cycles. There was no change in the glow curve.

3.4.13 An attempt was made to deposit the phosphor by means of a "Plasmadyne" spray gun. The samples of phosphor were to be sprayed in an inert atmosphere box with argon as the carrier gas and shroud gas. Four samples were sprayed:

- 1. Simulated nickel substrates,
- 2. Corning glass resistors,
- 3. Platinum sheet, and
- 4. Nickel sheet.

The samples showed good adherence and retained the general shape of the glow curve but were badly discolored. The brownish appearance of the sprayed phosphor was due to oxidation of the MnF<sub>2</sub> in the phosphor.

3.4.14 Table 1 is a qualitative summary of the experimental approaches to the problem of bonding the phosphor to the heater substrate.

# TABLE 1. Qualitative Comparison of Bonding Techniques

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Bonding Method	<u>Sensitivity</u>	Effect on Phosphor	Production Applicability
DC-805	High	none	poor
Amaco Metal Enamel	Medium	none	fair
Melbond Ceramic Adhesive	Very Low	none	good
Sauereisen Cement	Very Low	none	good
Air Impaction	Low	none	good
Nickel Powder Sintering	Low	none	good
Vacuum Evaporation	Very Low	Introduced low- temperature pea in glow curve	poor ik
Sintering of Phosphor	Medium	Introduced low- temperature pea in some cases	fair k
Sinter and Activation of Phosphor	Medium	as above	fair
Kasil	High	none	good
Plasma-Spray	Medium	none	good

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## 3.5 Dosimeter Design

3.5.1 The original work of Schulman<sup>1</sup> described a dosimeter of about 2.5  $cm^2$  area with 40 mg/cm<sup>2</sup> of phosphor. With this design it was possible to read doses of 2 mr using an end on RCA 6199 photomultiplier tube and a General Radio 1230A electrometer. The device to be designed was to be non-directional for incident radiation except where electrical leads are positioned. Ideally, this would be a sphere but the production problems and design problems are very complicated. Other specifications called for the device to be as follows:

- 1. Phosphor bonding to be satisfactory to at least 400°C;
- 2. Glass envelope will not discolor at 20,000r;
- 3. No spurious pickup;
- 4. Re-useable to at least 100 times;
- 5. Operate over the temperature range -45 to +55°C;
- 6. Total volume of dosimeter not to exceed 0.5 inch<sup>3</sup>;

3.5.2 The experimental program dictated a design that was cylindrical with an end cap. Calculations, based on the preceding experimental evidence indicated a cylinder as illustrated in Figure 13 (Part A) would be adequate. The dimensions shown provide a minimum surface area of 2.88 cm<sup>2</sup>. The addition of 40 to 100 mg/cm<sup>2</sup> of phosphor of Kasil to the substrate will increase the diameter approximately 0.05 to 0.2 cm and, therefore, the area will be at least 3.0 cm<sup>2</sup>.

3.5.3 A number of materials were considered for the substrate heater. It was recognized that a desirable characteristic of the substrate heater would be high resistance so a lowcurrent power supply could be designed. The material selected was nichrome for use with bonded phosphor. Nichrome cannot

 Schulman, J. H., et al, Rev. Sci. Instr., <u>31</u>, 12, 1263 (December, 1960)


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be used with dosimeters made by sintering to phosphor because chromium diffusion at the sintering temperature changes the phosphor characteristics. Satisfactory dosimeters have been made by winding 15-mil nichrome wire on a 3/4-inch-long mandrel. A total of 13 turns are used. Figure 14 shows the sprial of wire before being coated with phosphor and also a complete dosimeter element. To determine the effect of the possible diffusion of the chromium from the nichrome into the phosphor at the maximum dosimeter temperature, a nichrome planchet coated with the phosphor-Kasil mixture was baked in the oven at 400°C for 30 hours. A glow curve was taken before and after and there was no detectable change.

3.5.4 A standard T-3 glass envelope was selected for the protective envelope. The dimensions of the envelope are 1.2 inches long and 0.359 to 0.389 o.d. The T-3 bulb is available in either lime glass or lead glass. Radiation exposures of 40,000r show a percent loss in transmission at 500 m# that is negligible. This is illustrated in Figure 15.

3.5.5 The prototype thermoluminescent dosimeter and the shielded and unshielded characteristics have been measured. An empirical response curve for the dosimeter was derived by utilizing the ratio of absorbed dose in the phosphor to the absorbed dose in air, backscattering corrections, and attenuation corrections at low energies. A comparison of the empirical data to an experimental determination of the energy response curve is shown in Figure 16.

3.5.6 Several shield designs have been evaluated experimentally in an effort to arrive at a configuration which will provide both an acceptable energy dependence and directional dependence. Figure 17 shows the response obtained with a 0.66-mm lead shield; this shield has holes drilled in it so that 10% of the shield area is open. The shield



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FIGURE 14. SPIRAL-WIRE DOSIMETER





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was not quite thick enough to attenuate the low-energy peak and the results of our calculations indicated that thicker lead would only produce an undesirable minimum response at 120 kev. Figure 18A shows a similar shield with 0.86 mm tin outside the lead shield. The shield seemed satisfactory from the standpoint of energy dependence but proved to be somewhat non-reproducible because a finite number of holes was used in a finite number of rows. A more satisfactory solution for providing the open area is shown in Figure 18B. The shield consists of cylinders separated with a spacer which establishes a cylindrical opening.

3.5.7 The directionality of the shield is expected to be superior to that which can be obtained with other methods and its reproducibility has been shown to be good. An open area has been provided near the end of the shield to enchance the directionality. The exact dimensions of this portion of the shield will depend somewhat upon the physical size of the finished dosimeter.

3.5.8 A series of tests were conducted to determine the minimum vacuum necessary in the dosimeter. The tests were run by mounting a dosimeter in the spherical reflector and attaching a vacuum line to the back end. A single dosimeter was then checked at  $\underline{2}$  different pressures of air and  $\underline{1}$  of argon. A summary of these tests is shown below.

Pressure	Dose	Relative Peak Height
1 x 10 <sup>-6</sup> mm Hg (air)	100mr	100
$1 \times 10^{-4}$ mm Hg (air)	100mr	91
760mm (Argon)	100mr	92

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On the basis of this and duplicate tests, the prototype dosimeters are being fabricated by performing all-glass sealing and tip-off operations in a flowing argon atmosphere and no vacuum system is required.

3.5.9 Dosed dosimeters were stored up to 20 days and read out to determine loss of dose during storage. The results indicate there is negligible loss of dose over the period of 20 days. This has been confirmed in a private communication with Dr. R. Ginther of the Naval Research Laboratory.

### 3.6 <u>Electronics Program</u>

3.6.1 Concurrent with the investigation of phosphor deposition and production, we proceeded with the design of a computerindicator capable of reading and printing seven decades of information from the dosimeter automatically. A variety of novel concepts were thoroughly investigated prior to building the experimental model. Actual performance of the model could not be evaluated until it was checked with an actual dosimeter, the availability of which was delayed by the unforeseeable effort required in the dosimeter development. As a result, certain modifications had to be made in the photomultiplier tube and high voltage circuitry in order to meet the sensitivity requirements of the specifications. We are pleased to report that the computer-indicator does conform to the sensitivity requirements over the required seven decade range. The mechanical portions of this device appear to represent the best technical approach to the exacting problem of measuring seven decades of light intensity. NRDL personnel have requested to use the approach in a primary standard apparatus they are developing.

3.6.2 The completed experimental computer-indicator (C-I) is shown in Figure 19. The unit has been developed using circuitry that was proved in the pre-prototype unit (Second



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# FIGURE 19. COMPLETED PROTOTYPE COMPUTER

Interim Report). The pre-prototype system has been simplified, repackaged on etched circuit boards, and a Nixie tube decimal number display added in place of the binary light systems. Altogether, ten custom made etched circuit boards and a plug-in chassis comprise the major portion of the electronics. The photomultiplier tube high-voltage supply, the dosimeter heater transformer and regulator circuit, and the power transformer are mounted separately. The electronics occupies over half of the C-I waterproof box which is 9 in. high by 14 in, wide by 13 in. deep. The frame holding the etched circuit boards is fastened to the front panel and is removed with the panel. An interconnecting plug allows the electronic frame to be quickly disconnected from the mechanical unit for service. All the plug-in units are furnished with test jacks at critical circuit points so that a faulty board may be easily located.

3.6.3 All controls and plugs are mounted on the front panel. The Nixie read-out lights are mounted behind a sheet of circularly polarized plastic. This plastic provides a dark background and accentuates the display as well as protects the lamps. The plugs for a-c power and the printer are in the lower right corner. Immediately above the read switch is the opening for inserting the dosimeter. The other controls and their function are:

- 1. Read Switch A momentary contact switch that starts the read cycle.
- Calibrate, coarse and fine Allows the unit to be calibrated after inserting the standard dosimeter and operating the read switch.
- 3. Zero Zeroes the instrument before reading the dosimeter.

NOTE: Controls 2 and 3 are provided with locks to prevent accidental error.

- 4. Reset Resets the digitizing circuit to zero. Used when calibrating the C-I.
- 5. Switch This switch allows the operator to keep the standard in the reader for longer than the 20-second normal reading cycle, and thus allows more time for the calibrate adjustment to be made.

The standard dosimeter, a-c power cord, and printer cable are housed in the cover of the C-I. The complete unit, with cover on, is watertight.

## 3.6.4 Computer-Indicator Operation

3.6.4.1 The computer-indicator is designed to read the peak light emitted from the dosimeter. The peak light is measured by means of a photomultiplier tube and a voltage digitizer. The digitizer is scaled so as to read directly in milliroentgens. The dose is displayed digitally on the front panel and may be printed on paper tape using an auxiliary printer. The dosimeter itself is mounted on a "handle" as shown in Figure 13. The handle carries a printed ID number along with a mechanical coding of the ID number. The ID codes are sensed by the C-I and printed along with the dose in milliroengtens. The C-I covers a range from 1 mr to 9,900r which corresponds to a range in peak light values of approximately 10<sup>'</sup>. Neutral density light attenuating filters in decade steps are used to reduce the light to the photomultiplier on higher ranges automatically. A Comming No. 4305 blue green filter is interposed between the dosimeter and photomultiplier tube to prevent the masking of low dose light levels by the infrared emission from the hot dosimeter. A rotating disk with three apertures rotating at 1800 rpm is also interposed between the dosimeter and photomultiplier tube to chop the light at a 90-cycle rate. The chopped light signal from the photomultiplier tube does not contain the d-c component of the

dark current and thus eliminates the need of frequent zero setting caused by changes in dark current with temperature. 3.6.4.2 The dosimeter, after being inserted into the C-I, is automatically driven into reading position located on the axis of the spherical mirror. The dimensions of the mirror have been chosen to couple the light to the photomultiplier tube efficiently. The superiority of the spherical mirror had been demonstrated in earlier tests. (Second Interim Report).

3.6.4.3 The block diagram of the C-I is shown in Figure 20 In the upper left, is shown the synchronous driving motor which drives the chopper disk, the neutral density filter disk advance mechanism, and the dosimeter drive mechanism. When the read push button is depressed the dosimeter drive mechanism drives the dosimeter past the identification number (ID) reading switches which sense the mechanical code on the dosimeter handle. During this operation, the drive commutator emits ID print control signals to the ID switches and thence via the transistor decoder and printer solenoid drivers to the keyboard of the printer, thereby printing the ID number. When the dosimeter is all the way in, and the active element positioned in the mirror, the drive commutator turns on the constant-voltage dosimeter heater supply and starts the dosimeter heating timer. The dosimeter element then begins to heat and emit light according to the dose. The chopped light is detected by the photomultiplier tube and amplified by the a-c amplifier.

3.6.4.4 The crests of the a-c amplifier output is converted to a two decimal place digital number by means of a decade counter. The counter controls, by diode switches, the amount of d-c current flowing into a summing junction. This current is proportional to the digital value represented by the counter





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FIGURE 20. COMPUTER-INDICATOR





FIGURE 20. COMPUTER-INDICATOR

and subtracts from the current peaks of the amplifier output. If the peaks exceed the d-c current by more than one decimal unit, the resulting error signal, amplified by a d-c amplifier, allows a pulse generator to supply pulses to the counter. As soon as enough pulses have been received by the counter to make the d-c current higher than the a-c crest current, the pulse generator is turned off, and the counter then indicates a number proportional to the value of the crest a-c current. The diode current switch and comparator has a resolution corresponsing to 0.1 of the least significant digit of the counter. This, plus a small distortion of the a-c amplifier, represents the error introduced by the digitizer. Two significant readout digits give an indicated precision less than 10% in keeping with the over-all system design accuracy.

3.6.4.5 The counting rate is sufficiently high for the counter to follow the rise in light intensity from the dosimeter. When the peak value is reached and the a-c amplifier output starts to decrease, the reading remains in the counter as it cannot count down and follow a decreasing voltage. Thus the peak light reading is retained for readout. The gain of the a-c amplifier is adjusted prior to reading by means of a standard dosimeter so that the decimal counter reads directly in milliroentgens. This corresponds to range 1 shown in the table (Figure 20). If the glow of the dosimeter becomes more intense, and reaches full scale of the counter (99 mr), the scale is automatically changed, first by reducing the gain of the a-c amplifier (which also reduces photocell noise on the higher ranges), and then by introducing neutral density filters of densities of 1, 2, 3, and 4. The instrument is therefore capable of indicating 1 mr to 9,900r.

3.6.4.6 After the dosimeter heater has been on for a length of time determined by the timer, and the glow peak has been reached, the counter indicates the first two significant digits of the dose, and the position of the filter disk, as indicated by the filter commutator in conjunction with the control logic, indicates the required number of readout zeroes. The dosimeter drive mechanism is the actuated to drive the dosimeter out. The drive commutator then scans the decimal counter and filter commutator so as to print the dose on the printer. When the dosimeter is completely ejected, the ID number and dose appear on the printed record and the dose appears on the Nixie display.

### 3.6.5 Special Features of the Electronic Circuit

3.6.5.1 The coding of the ID number on the dosimeter handle is accomplished in a 1, 2, 4, 2 code for each decimal digit (Figure 13). The decimal counter also operates in a 1, 2, 4, 2 code for each decade. The Nixie lamp display and printer operate from a 10-wire decimal code. A single decoder is shared between the display and printing functions where normally three decoders would be required. When the C-I is idle, the decoder drives the two significant figures of the Nixie light display. In this mode, the decoder input is switched at a 60-cycle rate between the two decades of the counter by means of the digit switch. The Nixie tubes are also supplied by a 60-cycle source so that they in effect are switched to the output of the decoder on alternate half cycles. Although each Nixie tube is only on for half the time, the display brilliance is still quite adequate. When the printer is being operated, the Nixie display is turned off and the decoder then receives its input either from the ID reading switches or from the counter.

3.6.5.2 The a-c amplifier that amplifies the signal from the photomultiplier tube has a narrow band pass to reduce the fluctuation noise from the photomultiplier. A good signal-to-noise ratio for very low light levels requires a very narrow bandwidth. However, the narrower the band, the longer time it takes for the amplifier to respond to input signals. Thus the bandwidth must be wide enough to allow the amplifier to reproduce faithfully the increase in light from the dosimeter over the 15-second heating cycle. An amplifier with an equivalent 1/4 second time constant (bandwidth of about 2/3 cycle) has been found to be adequate. However, when a larger dose than 99 mr is read, the gain of the amplifier must be reduced by one tenth. Due to the long time constant of the narrow band amplifier, it would require a few seconds for the change to take place and the output of the amplifier to decay. This would not be sufficiently fast to catch the peak level of light which only lasts for about 1 second. This problem is solved by increasing the bandwidth at the same time that the gain is reduced. The wider bandwidth allows the amplifier to recover quickly and the scale changes are made in approximately 0.5 second. The signal-to-noise ratio is not impaired by the wider bandwidth as the photomultiplier tube output is well above the noise level on the higher ranges.

3.6.5.3 A block diagram of the amplifier is shown in Figure 21. The photomultiplier tube current develops a voltage across the standardized controls which adjust for variations in sensitivity of the photomultiplier tube. This voltage is applied to a high input impedance stage which drives a high gain feedback amplifier. Normally, the transistor switch in the feedback loop is closed, shorting the



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FIGURE 21. BLOCK DIAGRAM A.C. AMPLIFIER COMPUTER - INDICATOR

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feedback resistors to ground. The feedback is then entirely through the parallel "T" null network, which produces a very sharp amplifier response at the chopper frequency. When it is desired to make the first scale change (as indicated by 99 on the counter) the transistor switch is automatically opened and introduces sufficient extra feedback to lower the gain by a factor of 10. The feedback resistors also shunt the null network, rendering it less effective and increasing the bandwidth. The  $\frac{2}{3}$  10 adjustment allows the gain reduction to be accurately set to a tenth. The use of feedback in all the amplifiers reduces gain variations due to temperature and ensures a stable system.

3.6.5.4 In order to increase reliability, no relays are used throughout the computer-indicator. The drive control solenoids and printer solenoids are driven from power transistors. The digital counters and control logic use resistance-coupled logic which is economic of component parts. The breakdown by function of transistors and diodes is as follows:

	<u>Transistors</u>	Rectifiers and Diodes
AC Amplifier	7	2
Counter and Digitizer	28	36
Control Logic	33	6
Decoder and Digit Switch	26	
Nixie Lamp Drivers	20	6
Printer Solenoid Drivers	48	1
Power Supplies	4	
TOTAL	166	68

All voltages are regulated against line voltage variations to ensure against calibration errors during the reading cycle. 3.7 Thermoluminescent Dosimeter Reader - Mechanical Program

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3.7.1 Four major functions are performed by the mechanical portion of the thermoluminescent dosimeter reader. They are dosimeter insertion and positioning, identification number readout, light chopping, and filter indexing. A single synchronous motor drives all the mechanisms which perform these operations.

3.7.2 Dosimeter Insertion Drive. General Description: When a dosimeter is to be read, it is pushed through the port in the front panel of the reader, and latches into position in the dosimeter drive carriage. See Figure 22 for positions of the mechanical portion. The "read" button on the front panel is depressed and the carriage pulls the dosimeter through the identification number decoder and positions it in the reflector as is shown in Figure 23. Electrical contacts on the carriage pass heating current to the dosimeter, which causes it to emit light in proportion to the radiation dose to which it has been exposed. When the reading cycle is completed, the carriage drives the dosimeter back out of the reader.

3.7.3 Detailed Operation (Figure 24). When the dosimeter drive solenoid is energized momentarily it trips a springtype clutch. The clutch couples the drive mechanism to a shaft which is continuously rotating at 164 rpm. The spring clutch makes one revolution and is stopped by the solenoid plunger. The single revolution of the spring clutch is transmitted to a crankshaft through 2:1 gearing and results in 1/2 revolution of the crankshaft. The down stroke of the crankshaft is coupled to the dosimeter carriage through a connecting rod and pulls the dosimeter carriage downward to the limit of its travel and positions the dosimeter in the reflector. At the end of the readout cycle, an electrical pulse to the drive solenoid causes 1/2 revolution of



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# FIGURE 22. COMPLETE READER MOUNTED IN CASE, FRONT PANEL REMOVED



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FIGURE 23. DECODER - REFLECTOR MODULE



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FIGURE 24. DOSIMETER INSERTION DRIVE

of the crankshaft and the up stroke of the crankshaft moves the dosimeter upward to the limit of its travel. Depressing a button on the front panel unlatches the dosimeter so it can be removed from the reader.

3.7.4 On the upstroke, the crank stops a few degrees short of top dead center and an overrunning clutch prevents rotation of the crankshaft in the reverse direction. This allows a dosimeter which is latched in the carriage to be pushed up and down with moderate force without damaging the mechanism.

3.7.5 Identification Number Readout. As a dosimeter is drawn into the reader by the insertion drive, the dosimeter handle passes through the ID number decoder. As each digit button passes beneath the four decoding switches, indentations in the digit button close the switches which correspond to the binary code of the digit represented by the button. After the four decoding switches are "made up" into the binary code of a particular digit, a fifth switch (the synchronizer switch) is closed by the digit button and the digit is entered into the printer.

3.7.6 Light Chopper. The light chopper consits of a rotating shutter which is driven directly by the synchronous motor. A motor speed of 1800 rpm and a shutter with three equally spaced openings are used to produce a chopping frequency of 90 cycles/sec. A motor speed of 3600 rpm and a two-aperture chopper were used formerly. (120 cycles/sec chopping frequency.) The changes in motor speed and chopping frequency have resulted in smoother mechanical action and less susceptability to 60 cycles/sec electrical interference. The thickness of the chopper disk has been increased in order to recain enough inertia in the system to prevent fluctuations in chopper speed when the filter indexing solenoid is actuated.

3.7.8 Filter Indexing Mechanism. General Description: Five neutral density filters are carried in a disk located between the dosimeter and the photomultiplier tube. During the readout cycle, electrical pulses are applied to the indexing mechanism whenever it is necessary to position a different filter in front of the photomultiplier tube. The filter disk quickly makes 1/5 revolution to position the proper filter. When the readout cycle is completed, a continuous electrical pulse causes the filter disk to rotate around to the starting position if it has not remained there during the readout cycle.

3.7.9 Detailed Operations. When the filter indexing solenoid is energized momentarily it trips a spring-type clutch (see Figure 25). The clutch couples the filter indexing geneva mechanism to a shaft which is driven continuously at 102 rpm. One revolution of the geneva input shaft advances the filter disk one position and locks it in place as shown in Figures 26A and 26B. After one revolution of the geneva input shaft, the spring clutch is stopped by the solenoid plunger and the geneva mechanism remains locked until the next electrical pulse is received. When the solenoid is energized continuously during the filter homing operation, the spring clutch remains engaged and the geneva mechanism keeps advancing the filters until the home position is reached.

3.7.10 Materials Used. The filter disk is made of magnesium (subject to Bureau of Ships approval) in order to keep its moment of inertia to a minimum. This allows rapid indexing of filters without producing undue stress in the indexing mechanism. The geneva drive components are made of stainless steel and permanently lubricated with Electrofilm dry film lubricant.

3.7.11 Dosimeter Construction. The major components of the thermoluminescent dosimeter are the dosimeter tube, tube base, identification number assembly and the biological shield. Figure 13 is the layout of various dosimeter parts. The thermoluminescent dosimeter tube contains the phosphor and an electrical heating element, vacuum encapsulated in a glass envelope. Construction of the tube is described in detail in Section 3.5.

3.7.12 The tube base is made of plastic with metal contacts for carrying the heating current to the dosimeter tube. A groove in the tube base allows the dosimeter to be latched into the reader carriage during the reading cycle. A key on the tube base holds the dosimeter in the proper orientation with respect to the electrical contacts and the ID number decoder switches.

3.7.13 The dosimeter handle accomodates eight identification number buttons. Each button contains indentations corresponding to the binary code of the numeral which it represents. This numeral is also stamped on the button so that when all the buttons are assembled on the dosimeter handle, the ID number can be read visually as well as being automatically recorded by the printer when the dosimeter is read. The number buttons are so constructed that they cannot be assembled backward or in the wrong orientation on the dosimeter handle. The number buttons are designed for easy adaptation to automatic assembly.

3.7.14 The lead and tin inserts of the biological shield are mounted in a plastic housing which also serves as a protective cover for the dosimeter. An O-ring seals the dosimeter from moisture and the shield is locked in place with a catch which requires a special tool for disassembly, minimizing the possibility of personnel tampering with the dosimeter. With the proper tool, removal of the biological shield is quite simple.



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26. FILTER INDEXING GENEVA MECHANISM FIGURE

B. LOCKED POSITION

A. BEGINNG OF FILTER CHANGE CYCLE

# 3.8 Standards Program

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3.8.1 At a meeting with representatives of NRDL and NRL, the Santa Barbara Laboratory was assigned the responsibility of providing a method of calibration of the photometric device as additional work to be accomplished during Phase II. It was considered essential that a unique method be found for standardization of the photometric device due to the complex relationship of the emission spectra of the thermoluminescence and the variable response of the individual photomultiplier tubes. Very close rapport was required with NRDL for the accomplishment of this task as they were to provide a primary standard for comparison of dosimeters and photometric devices from different manufacturers. Our solution to this problem, based on extensive research in phosphor charactersitics, resulted in a simple, nonchanging. inexpensive, "standard" dosimeter that can quickly calibrate and check major portions of the photometer for accuracy and reproducibility. The idea has been included in the more fundamental apparatus being designed by NRDL and assures the Navy that dosimeters and readers made by other manufacturers will be compatible with existing systems.

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3.8.2 Two distinct types of standards are necessary for the satisfactory completion of the thermoluminescent dosimeter development program. The first of these is a replica dosimeter to be used with the computer-indicator as a calibration device. The requirements for this device are as follows:

- Identical emission spectra to thermoluminescent dosimeter,
- 2. Identical geometry to thermoluminescent dosimeter;
- 3. Constant light emission;

4. Safe to personnel;

5. Economical.

Previously it was reported<sup>2</sup> that the fluorescence emission spectra of the  $CaF_2$  Mn phosphor is the same as the thermoluminescence emission. An ideal dosimeter standard can be made by intimately mixing the phosphor with a radioactive material. Three standards have been prepared in this manner. Figure 27 shows the two standards that were built in planchet shape. The original standard was made by dissolving C<sup>14</sup> labeled "Lucite" and mixing the phosphor with it. The second standard was built by U.S. Radium Co. and used the same techniques. In this standard 3 mC of C<sup>14</sup> activity was used. The fluorescence output from the planchet placed under the photomultiplier tube is equal to a dose of 9r from a real dosimeter.

3.8.3 The U.S. Naval Radiological Defense Laboratory fabricated a more practical dosimeter by mixing  $BaCO_3 C^{14}$ labeled with the phosphor and putting the mixture in a small glass tube. The mixture was sealed in the tube with a small plastic plug. On the prototype dosimeter reader this was equivalent to 2.5-4r. It is anticipated the standard to be supplied with the computer-indicator will be fabricated in an identical manner. The light emitting portion will have the same size and shape as the active dosimeter element. The standard will deliberately be made to calibrate the high ranges of the computer-indicator and will be fitted with several metal thimble-shaped covers which have a calculated number of holes in order for the emitted light to be equivalent to smaller doses.

Phase II Second Interim Engineering Report, Edgerton Germeshausen and Grier, Inc., Report S-200-R.

<sup>2.</sup> 



# FIGURE 27. PLANCHET - SHAPED DOSIMETER STANDARDS

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3.8.4 A more complicated problem involves the standardization of dosimeters and computer-indicators when made by several manufacturers. To standardize dosimeters the USNRDL has designed and built a dosimeter comparator. Figure 28 is a preliminary print received from NRDL and shows the plan and elevation of the device. As can be seen from the figure it will be possible to compare an EG&G dosimeter with a fluorescing standard and an NBS standard lamp. The comparator uses the EG&G spherical reflector and light chopping assemblies as integral parts

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3.8.5 In order for manufacturers of the computer-indicator to be sure their devices are of the required sensitivity and accuracy it is planned to supply them with calibrated fluorescing dosimeters, identical to those described in 3.8.3.

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FIGURE 28. DOSIMETER STANDARD COMPARATOR ASSEMBLY

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# PART II

# 1. PROGRAM FOR NEXT INTERVAL

1.1 During the next 4-month interval, production techniques will be evaluated while constructing the 500 dosimeters.

1.2 At the Santa Barbara Laboratory five computer-indicators will be completed and delivered to the Bureau of Ships.

1.3 Reports, Instruction Sheets, and Technical Manuals shall be provided as described in the contract.

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### PHASE II

### Legend

# Project Performance

- 1. Order necessary equipment and M & S.
- 2. Produce first phosphors.
- 3. Modify phosphor synthesis to larger batches and simplify chemical techniques.
- 4. Investigate effect of phosphor particle size, thickness, substrate and bonding agent on light output.
- 5. Establish quality control and phosphor standards.
- 6. Select geometry of dosimeter and assemble prototype.
- 7. Design case and shield.
- 8. Evaluate shield.
- 9. Evaluate prototype dosimeter and modify to finalize all end points of geometry, shielding and holder.
- Study computer-indicator (C-I) problem and construct first laboratory device.
- Investigate design Preprototype Model of heater-timer,
  C-I logic circuits and readout.
- Construct design Preprototype Model of heater-timer, C-I logic circuits and readout.
- 13. Test same under environmental conditions.
- 14. Design preprototype C-I.
- 15. Construct prototype C-I.
- 16. Final testing of dosimeter and C-I system.
- 17. Interim engineering reports.
- 18. Discuss concepts and results with BuShips and NRL representatives.
- 19. Phase II Final Report.

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20. Work with NRDL on standards program.

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# TABLE I PROJECT PERFORMANCE AND SCHEDULE CHART

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