Improvements in the Making of Special Photographic Emulsions for Nuclear Physics

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In 1945 (1) the author prepared concentrated, very fine-grained emulsions such as emulsion formula 2 (2), in which it has proved possible to record, with good detail and discrimination, the tracks of fission fragments (3), alpha rays, protons, and even of slow electrons (1, 2). Experience has shown us that the properties of these emulsions are not easily reproduced (4). The critical factors have been found to be: the mechanical and stoichiometric conditions during the mixing operation; the temperature of the wash water, which should be preferably below 5° C; and the gelatine used.



FIG. 1. Schematic view from above of emulsion-making machine. Sliding parts are shown in heavy black. Part 1 represents a stirrer; 2, a beaker containing solution C; 3, pouring tips; 4, tanks of solutions A and B; 5, three-way stopcocks; 6, 100-ml syringes; 7, pushing rods; 8, sliding carriage (bench carrying 8 and motor moving it are not shown); 9, right angle racks sliding inside 8 and linked through pinion (not shown); 10, sheet with slot guiding the movement of racks 9; 11, potentiometer regulating speed of motor 12; 13, flexible shaft from motor 12 to stirrer 1.

To control mechanical and stoichiometric factors, a machine has been designed and built that is illustrated schematically in Fig 1. To the stirred C solution, solutions A (600 g AgNO₃/1000 ml solution) and B (420 g KBr/1000 ml of solution) are added simultaneously, through the action of syringes, pushing rods, and carriage, moved by a motor sliding along a bench. Volumes of solutions A and B are kept constantly equivalent or nearly so, B being in slight excess. The double rack mechanism with its guiding slot introduces an adjustable difference between the movements of the pistons of syringes containing A and B, and thus allows one to cope with a difference of diameter between pistons, and with inaccuracies of concentrations of A and B. Also, through the peculiar shape of the slot chosen, it is possible to vary the excess (B-A) at will during mixing. The system also fills up syringes when they are empty. Here are numerical data for solution C: 15 g gelatine, 167 ml water, and 83 ml ethyl alcohol; use at 40-50° C. First add 1.3 ml of B. Then deliver at equivalent rates 100 ml of A and B, taking 2-3 min. Increase the stirring rate from 240 to 550 rpm. Chill and set at $0-2^{\circ}$ C (1 hr). Break the jelly in small pieces and wash at $0-2^{\circ}$ C (1 hr). Collect, melt, coat, dry, and sensitize 1 min in 6% triethanolamine before use.

In this method, the mixing phase is highly critical but there is no high temperature treatment. In preparing ordinary emulsions, mixing may be less critical but the heat treatment is a delicate phase.

This machine has been used with uniformly good results. A prototype of it, using 30% syringe sizes and quantities in mixing, has been in operation for nearly a year with consistently good results. Under its standardized conditions, 13 gelatine samples have been tried, and all gave good tracks of alpha rays and protons, and at least a peculiar granular appearance with radium D electrons. The best electron tracks (40-60 kv) resulted from gelatines labeled "fast negative," that is, from gelatines recommended for obtaining the greatest light sensitivity.

This gives us further reason to believe that similar active centers are essential both for light and for ionizing particles. Still unexplained is the lack of connection noted between light and particle sensitivity for various emulsions, which probably depends on the disposition of active centers on the AgBr grains.

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Sensitivity of Gamma-Ray Counters¹

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Radioisotopes can be investigated in many cases only by detection of their gamma radiation. Measurements of the distribution and the amount of an isotope used for tracer studies or for therapeutic purposes are often based on counting the gamma rays emitted in a certain direction (3). The sizes of source and gamma-ray counter, and the distance between them, determine the fraction of the total gamma radiation which is incident on the detector. The actual number of registered counts is again only a fraction of the incident gamma rays, depending on the sensitivity of the counter. It is known that even counters made from high atomic material detect only 0.7-2% of the gamma rays with energies between 0.5-3.0 Mev when the gamma radiation penetrates the counter walls perpendicularly (\mathcal{Z}) . This low counter sensitivity is a serious limitation for many investigations. Different methods which result in a higher counting sensitivity will be outlined here.

Somewhat higher sensitivities can be obtained by using

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Geiger-Müller counters end-on. The probability for the secondary electrons, produced by the gamma rays in the counter walls, to enter the sensitive volume of the counter increases when the gamma rays strike the walls at a small incident angle. With calibrated I¹⁵¹, Co⁶⁰, and radium sources, sensitivities of about 2% for I¹³¹, and 4% for Co⁶⁰ and radium were observed with commercial end-window tubes, such as the North American Philips No. 62019. The same results were obtained with a multicellular type of counter.¹

FIG. 1.

A large number of organic and inorganic crystals have come into use recently as scintillation counters (1, 4). The gamma rays absorbed in the crystal produce light flashes, which are detected and amplified by a photomultiplier tube. Due to their high density and high atomic weight components, calcium tungstate crystals show a very high gamma-ray sensitivity. Under conditions described herein, sensitivities of 24%, 38%, and 36% for Co⁶⁰, radium, and I¹⁸¹ gamma rays, respectively, were observed, with highly polished, clear crystals² of area $\frac{1}{2} \times \frac{1}{2}$ in. and thickness $\frac{1}{5}$ in.

A 1P21 photomultiplier tube operating at about 90 v per stage and the CaWO₄ crystal were kept at room temperature. A model 500 Los Alamos preamplifier and discriminator, together with a scaling circuit, counted photomultiplier pulses greater than 12 mv. Fig. 1 shows the counting rate versus integral pulse height for Co⁵⁰, Ra, and I¹³¹ sources. The tube background, also given in Fig. 1, has been subtracted from the source measurements, which were adjusted to the same extrapolated counting rate. The three curves indicate the differences in gammaray spectra of the three isotopes.

² The Linde Air Products Company, Tonawanda, N. Y.

Taking into account only the primary gamma rays entering the crystal through its front surface $(\frac{1}{3} \times \frac{1}{2}$ in.), the percent crystal sensitivities mentioned were obtained. For the $\frac{1}{3}$ in. thick CaWO₄ crystal, the sensitivity for I³³¹ corresponds to the expected gamma-ray absorption in the crystal. However, the Co⁶⁰ and Ra sensitivities are at least twice that expected from the absorption of the incident primary gamma rays alone. Since these results were found to be independent of the resolving time of the counting equipment, which was varied from 5 to 300 µsec, the CaWO₄ crystal used under these conditions caused no double pulsing.

All three pulse height distributions of Fig. 1 were obtained with a Lucite rod, 1 in. in diam and 4 in. long, placed between the crystal and the photomultiplier tube. The CaWO₄ crystal was covered with an Al foil as reflector, except for the surface touching the Lucite rod. Fig. 2 shows how the radium pulse height distribution is affected by varying this arrangement.

Curve (a) of Fig. 2 repeats the results with the radium source given in Fig. 1. Curve (b) was obtained when the Lucite rod was removed and the crystal was mounted directly on the photomultiplier tube but without being covered with Al foil. The decrease in large pulse heights shows that the light absorption in the Lucite is smaller than the amount of light gained by the Al foil. Curve (c) shows a further decrease in light intensity when the Lucite rod was again placed between crystal and photomultiplier but without using the Al foil reflector.

All three curves intersect at about the same extrapolated counting rate. This indicates that the results were not affected by a change of the crystal arrangement when the extrapolated counting rates were used. The application of the Lucite rod provides a better geometry

¹U. S. Patent #2,397,661, The Texas Company.

for most investigations. Its replacement by a quartz rod would improve the light conduction.

Finally, curves (d) and (e) of Fig. 2 show the results for the crystal placed with the $\frac{1}{2}$ in. $\times \frac{1}{2}$ in. surface facing the Lucite rod, with and without Al foil. In this position the surface area and therefore the number of incident primary gamma rays was only $\frac{1}{4}$ as compared with the conditions for curves (a), (b), and (c). The extrapolated counting rates, however, were nearly the same as before. The crystal sensitivity, therefore, seems to increase in proportion to the crystal thickness. These results are in agreement with the approximately 100% sensitivity for Ra gamma rays reported by Moon (4), for a CaWO₄ crystal about $\frac{3}{2}$ in. thick.

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Adapter For Visual Monitoring with Portable Geiger-Müller Equipment

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The application of a neon-filled strobotron (SN4) tube as a visual means of continuous monitoring of areas in which radioactive materials are used has proved itself very convenient.

Fig. 1 is the schematic diagram of a circuit developed for using an SN4 (manufactured by Sylvania Electric Company) as a visual indication of instantaneous changes in counts, that may be due to contamination or changes in background.

The circuit is enclosed in a metal box which has a large bull's-eye lens directly in front of the SN4. This circuit was developed to operate directly from the earphone jack of the Herback and Rademan Geiger-Müller Radiation Measuring Set, Model GLR-200.

The signal is superimposed on a d-c voltage which in effect produces a pulsating d-c voltage having a positive sign. The d-c voltage is adjusted to a value just below the firing point of the OA4 by sensitivity control R7. Thus, the incoming signal increases the potential of the d-c value to a point that will cause this tube to fire.

Resistor R6 has to be of a value that will keep the potential on the starter anode at the same value as the cathode, and yet of high enough value to prevent excessive loading of the output stage of the Geiger-Müller circuit.

When the OA4 fires, condenser C2 is momentarily shorted to ground through resistor R4, at the same time the plate voltage is reduced to a value which will not support operation. With the OA4 in an extinguished state, condenser C2 recharges through R5, thus impressing a positive voltage of sufficient value on the starter anode of the SN4 to cause it to fire. The SN4 will become extinguished when the potential of C1 falls below the critical operational potential. Following these events C1 becomes recharged through R1, thus conditioning the circuit for another firing sequence.

The operation of the circuit is dependent upon the height of the voltage pulse from the output stage of the Geiger-Müller circuit. This pulse must have a value of not less than 5 v.

The sensitivity of the instrument is directly dependent upon the Geiger-Müller tube used and the preliminary cir-

