SPECIAL PROBLEMS IN NUCLEAR INSTRUMENTATION

PROGRESS REPORT

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ABSTRACT

Two new conducting plastic mixtures were successfully prepared. The one mixture consists of 30 percent carbon and 70 percent polypropylene while the other consists of 20 percent carbon and 80 percent polypropylene. Several improvements in the design and fabrication of small ionization chambers have been given a practical expression in the series of three 0.5 cc chambers reported earlier. Further experience and understanding of these chambers have been gained. Several examples of cooperation with other laboratories are given.

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INTRODUCTION

During the past year our principal activities were divided between; mixing special conducting plastics; the study and further development of small (0.5 cc) ionization chambers; and cooperation with other laboratories on sundry problems brought to us. The breakdown of the total effort over these three areas is approximately twenty-five, fifty and twenty-five percent respectively.

The work described in this report was performed by John J. Spokas as the sole principal investigator with the aid of supporting personnel consisting of one full-time scientific assistant, a technical specialist-machinist who contributed 75 percent of his time, a part-time (again approximately 75 percent) secretary, consultants and hourly help in the form of students working part-time. The principal investigator devoted 100 percent of his time to the research covered by this contract for the first three months of the reporting period; for the balance of the period, he devoted, on the average, upwards of 75 percent of his time. The remainder of the principal investigator's time was spent in other endeavors which were supported entirely by the college.

The accomplishments of the past twelve months in the area of mixing plastics are covered in the next section titled Mixing Conducting Plastics. Several batches totaling approximately 125 pounds were prepared with either 20 or 30 percent carbon added to polypropylene. The results of these efforts are in the process of evaluation. Thus far no problems attributable to poor mixing have been encountered.

As already indicated our major concentration during the past twelve months has been the series of thimble type 0.5 cc ionization chambers. These same chambers have been described somewhat in our two previous annual reports. The three chambers have identical geometry but are constructed of different materials. One chamber is made of tissue-equivalent plastic, another is made of magnesium in order to be insensitive to neutrons, and the third is made of an air-equivalent conducting plastic. Some refinements have been made in the fabrication of these chambers leading to significantly improved performance. At least two groups made extensive use of these chambers in the recently completed International Neutron Dosimetry Intercomparison at Brookhaven National Laboratory. Thus far experience has not revealed any defect in the design of these chambers. Appendix A gives the abstract of a paper which will be presented at the 5th International Congress of Radiation Research in Seattle in July, 1974. This paper will cover certain evaluations being completed on the T.E. chambers in collaboration with Frank S. Williamson and Gordon L. Holmblad of Argonne National Laboratory. The work of the past twelve months on these 0.5 cc chambers is reported below in the section titled Small Ionization Chambers.

As noted above a significant amount of time was devoted to cooperating with other laboratories. This type of activity has been a special feature of our particular program. Several examples of such cooperation are cited below in the section titled Cooperation With Other Laboratories. Such endeavors represent perhaps the most effective and meaningful way of sharing some of our peculiar resources with the wider scientific community.

Two items which are not otherwise covered in this report deserved to be mentioned. The first pertains to our involvement in the International Neutron Dosimetry Inter-comparison mentioned above. Our involvement consisted of accompanying one participant, Frank S. Williamson of Argonne with whom we have collaborated regularly. Officially our status was that of an observer. Practically we functioned as an assistant to Frank Williamson throughout the entire two week long exercise. We were deeply involved in every facet of the intercomparison measurements. This was a unique opportunity and on the whole a very valuable experience.

Some effort was devoted to various aspects of the ICRU chambers, a pair of spherical air-equivalent chambers having I.D. of 3/4 inches. An understanding of certain peculiar behaviors seen in particular units had been achieved. Several problems in the fabrication of these chambers have been solved. One notable problem concerns the manner of securing and attaching an electrical connection to the outer electrode. This is a particularly serious problem in the case of the thin-walled version of the ICRU chamber. Other improvements have been worked out for attaching a coaxial cable to the chamber. It is hoped that in the near future these ideas will be submitted to critical testing in the laboratory. The complete discussion of these improvements will be deferred until that time.

It is with much sadness and a deep sense of personal loss that the death of William P. Jesse on February 17, 1974 is noted. Dr. Jesse joined the Physical Sciences Laboratory upon his retirement from Argonne National Laboratory in the Spring of 1954. He carried on full time research from that time until his second retirement at the

end of august, 1972. Throughout this period Dr. Jesse was co-principal investigator under this contract. After his second retirement in 1972, Dr. Jesse consulted regularly for us spending one day per week on campus. He was a constant source of inspiration in addition to contributing much thoughtful and wise counsel to our scientific investigations. There is no question that he left a legacy which will have a lasting influence on our future research.

MIXING OF CONDUCTING PLASTICS

The effort spent on mixing was not as great as was planned for two reasons. Firstly, we discovered certain ambiguities in the composition of the standard tissue-equivalent (muscle), A-150, regarding the nylon component. These ambiguities could not be resolved in the short time available before mixing would have to begin if several hundred pounds were to be prepared. Secondly, our involvement in the intercomparison at Brookhaven occurred in the middle of July which is also the middle of the preferred mixing season. The mixing process involves much handling of extremely finely divided powders including carbon which are extremely difficult to contain. Therefore it has been customary to spare our laboratory and shop and only mix plastics when the weather is good so that the operation may be set up outdoors.

The mixture designated A-150 contains four distinct components, nylon 6/6, polyethylene, carbon and calcium fluoride. The atomic composition of the nylon is not entirely precise. At least two of the factors which contribute to this uncertainty are volatility of the plasticizer employed in the polymerization process and the strong water

absorption of nylon 6/6. It is possible to substitute another type of nylon such as nylon 6, nylon 6/10 and nylon 12 and to adjust the concentration of polyethylene and carbon to yield the very same atomic concentration as in A-150. Another type of nylon may inherently have a less variable composition and be more compatible with polyethylene the other plastic component in the mixture.

Another question concerns the possible advantages in substituting polypropylene for polyethylene. Both of these plastics have the same atomic composition though different atomic arrangement. The former polymer can withstand higher temperatures and could give improved physical properties to the tissue-equivalent mixture. Lacking the answers to these questions, having an ample supply of A-150 on hand, and being pressed for time suggested the delay in preparing a new batch of the tissue-equivalent mixture.

The Tri Service Radiac Working Group had sought our help in satisfying their need for a suitable conducting plastic for a tri service personnel dosimeter. This dosimeter project is being co-ordinated by the Defense Civil Preparedness Agency. Since we did possess the particular expertise they were seeking, we agreed to prepare sample batches of the material they desired. Our mixing efforts last Summer therefore, were confined to the preparation of two versions of conducting polypropylene, one having 20% carbon added the other, 30% carbon.

In preparing the two conducting polypropylene mixtures, an idea mentioned in a previous Progress Report (Progress Report No. COO-323-28) was tested. The idea was

to connect our extruder and mixer in a tandem arrangement for greater efficiency.

It has not been uncommon in the past in mixing plastics to first pass the extremely fine ingredients thru the extruder. Afterwards, the roughly mixed material, which is all that can be said for a mixture after a single pass thru an extruder, is first ground and then mixed in the patented intensive mixer designed and built by the late Dr. Shonka. What is achieved in the first step is an increase in particle size which then greatly facilitates the mixing operation (see page 9 of the Progress Report cited above) .

The tandem arrangement would bypass a grinding and a feeding operation since the material would be extruded directly into the mixer. The tandem hook-up did work though not particularly smoothly. The mixer operates in a cycle associated with the reversing rotations and consequent linear motions of the mixing screw. Control circuits were provided for the turning on of the extruder for a selectable interval each time the mixing screw reached the limit of its rearward travel. Actually, the best results were obtained with the extruder operating continuously. But even this was not satisfactory.

The overall rate with which material passed thru the extruder-mixer combination fluctuated considerably. In fact, at times the flow would completely stop despite the fact that the extruder screw was still rotating. Considerable effort using rams and such would be required to get the material moving thru the machinery again. It soon became apparent that time and effort would be saved by reverting to the former two-step approach, extrude first then mix afterwards. Of course, the extruded material must be ground before it can be fed into the mixer.

After the finely divided ingredients have gone thru the extruder once, thus increasing the effective particle size, the tandem arrangement operates beautifully. Since the necessary controls were available, the tandem arrangement was employed merely as a ready means to feed material into the mixer. Not only is the flow rate very constant, but the extruder only had to operate for only 1/4 second each cycle or about 5 percent of the time.

Approximately 130 lbs of conducting polypropylene were prepared. Both the 20 % and 30 % carbon mixtures show satisfactory mechanical properties and more than ample electrical conductivity for use as the basic material in the tri service dosimeter.

SMALL IONIZATION CHAMBERS

A considerable portion of our attention during the past year has been given to the series of three 0.5 cc chambers. The three chambers have identical geometry but each is constructed of particular materials in order to give either tissue, magnesium, or air response. The three versions are conveniently designated T.E. , Mg , and Air . The T.E. version is illustrated in Figure 1.

These chambers were described and discussed at some length in the Progress Report (Progress Report No. COO-323-30) submitted last year. This year the effort was directed to overcome certain weaknesses in the construction and to further testing and critical evaluation. A degree of perfection is sought in these chambers that will before long establish them as standards against which all other chambers of this type will be compared. Considerable attention was focused on the problem of

Figure 1 A sectional view of the 0.5 cc T.E. chamber.

the splitting of the shells and on a better means of fabrication of the base of the T.E. units. It should be stressed that the problem here only involve the T.E. chambers. Though the base fabrication is not the same in the Mg as in the Air versions as a consequence of the peculiar properties of the different materials involved, the results have proven to be without fault. An adaptation to a special low noise triaxial cable has been worked out specifically in the case of the Air chamber. This adaption is directly applicable to the T.E. and Mg units. This work is reported below.

The experience with the chambers has thus far shown them to leave little if anything further to be desired. It has been previously noted that both the participants in the International Neutron Dosimetry Intercomparison from the Armed Forces Radiobiology Research Institute and from Argonne National Laboratory used the T.E. and Mg chambers with apparently excellent results. These are but two examples of the favorable experience that has been found consistently in many separate experiments. Most of our recent effort directed toward the fuller evaluation and understanding of these chambers will be presented in the paper to be read at the 5th International Congress (see Appendix A) and therefore is not covered in this report.

An example of the energy dependence which is characteristic of the Air chamber is given in Table I. Here are presented the calibration factors found for one chamber over the range of energy of x-rays from 60 KVcP to 250 KVcP. The table shows that the energy dependence is constant to within approximately $\pm 1\%$ over this range. The slight dependence is of the form one ordinarily finds in ionization chambers. The calibrations were determined at the Regional Calibration Laboratory, Memorial Hospital

for Cancer and Allied Diseases, New York, New York.

TABLE I Calibration factors for a 0.5 cc Air chamber at several x-ray energies.

KVcP	Half-value layer		Exposure rate R/min	Calibration factor R/nC
	mm Al	mm Cu		
60	2.79	0.09	1.5	6.22
75	3.42	0.11	2.0	6.21
100	5.07	0.20	3.5	6.19
150	10.4	0.66	6.0	6.22
200	13.4	1.26	9.5	6.29
250	16.0	2.17	10.0	6.32

The matter of the shells splitting in the T.E. chambers was mentioned in the last report. This is a serious defect which must be eliminated if the chambers are to provide the ultimate accuracy of which they are otherwise capable. Several tests were completed during the past year to solve this problem. Firstly, it was important to establish whether the splitting was due to a shrinking of the shells over a period of time, an inherent mechanical weakness of the shells, or perhaps an entirely different cause.

The first possibility mentioned above may involve moisture. Plastics, notably nylons, absorb water to a considerable extent and the T.E. mixture, A-150, contains 35 % nylon. The following experiment was therefore performed. Eight shells were molded

under the same conditions and the I.D. of each was determined. Four shells were stored at 100 % r.h. while the other four were kept in the ambient atmosphere which averaged approximately 40 % r.h. over the period of the tests. The I.D. of each shell was taken as the mean of two perpendicular diameters read with a measuring microscope. After six months the first group showed an average I.D. of 0.3676 inches with an average deviation of .0010 inches. As can be seen, a slight effect appears perhaps on the edge of resolution but the magnitude seems hardly enough to account for the splitting.

The shells are being injection molded. However, to fill the mold cavity, even with the full injection pressure available, requires that the mold be pre-heated. The customary technique for pre-heating the mold is to place the mold on a hotplate heating first one side for a few minutes then flipping the mold over and heating the other side. Doubtlessly in this procedure the mold temperature varies considerably from one shot to the next. There is no question that the mold must be heated as the cavity does not otherwise fill. But even filling the cavity does not guarantee that the part will have been tightly compacted in the mold which might account for the lack of tensile strength in the molded shells. Higher mold temperatures would enable a greater fraction of the injection pressure to be transmitted throughout the plastic in the cavity, at least immediately following the filling of the cavity.

In order therefore to achieve higher temperatures prior to injecting and for more precise control of the temperature, a pair of heaters were fitted to the jaws of the vise used for clamping the mold in the injection press. With this setup several sets of ten

shells each molded at different temperatures. First 200 °F was used, then 225 °F, and finally 250 °F. At the highest temperatures, the material could not be contained in the mold as it would ooze out around the parting line of the mold. Though this was an impractical situation it did dramatically demonstrate that greater pressure is indeed transmitted to the plastic in the mold the higher the mold temperature. At the highest practical mold temperature of 225 °F, a second set of shells was molded using material that had been vacuum dried. The I.D. of each shell was determined as above. The results are given in Table II. The uncertainty indicated is merely the mean deviation of the readings in each set.

TABLE II The average I.D. of several sets of injection molded T.E. shells for different conditions of raw material and initial mold temperature.

Condition of material	Mold temperature - °F	Average I.D. - inches
Stored in ambient	200	$0.3709 \pm .0006$
Stored in ambient	225	$0.3721 \pm .0014$
Vacuum dried	225	$0.3709 \pm .0013$

It was planned to follow the history of each set of shells after they had been placed on chambers to determine whether particularly the vacuum dried material may show an appreciably lower frequency of splitting. However, before this information could be obtained it was decided to investigate the effect of annealing the shells on dimensional

changes. To this end three sets of eight shells each were molded under identical conditions. The shells were permitted to stabilize at room temperature for one day then the I.D. of each shell was determined as above. The three sets were placed in an annealing oven set at 150 °F and stored for various times. The shells were then removed and kept at room temperature for one day and then the I.D. of each was measured again. The first two sets were given subsequent annealing treatments to ascertain the time scale over which the observed changes occur. The results are given in Table III.

TABLE III Results of annealing three separate sets of A-150 shells at 150 °F for various times.

Set	Annealing Time - hrs.	Average I.D. - inches
I	0	.3711 $\pm$.0005
I	23	.36967 $\pm$.00036
I	144	.36833 $\pm$.00043
II	0	.37135 $\pm$.0005
II	72	.36866 $\pm$.00046
II	360	.3672 $\pm$.00035
III	0	.37097 $\pm$.00042
III	119	.36811 $\pm$.00034

The results presented in Table III show that all of the shells have closely the same initial dimensions and that there is a contraction due to annealing which is all but complete after 120 hours. A number of the annealed shells had been placed on chambers. Thus far the indication is that the frequency with which cracks develop is essentially zero. This may be partly due to the greater strength of the shells and partly due to more careful fitting of the shells to the base to the chambers. This annealing treatment, which significantly stabilizes the shell dimensions, ought to preserve the good fit indefinitely without splitting of the shells.

The integration of the shell-guard insulator and the guard, which combination constitutes what may properly be called the base of the chamber, is accomplished differently in each of the three versions of the 0.5 cc chambers. In the case of the Mg chambers the insulator is molded directly onto the magnesium guard electrode and the small stainless steel tubes providing means for gas inlet and exhaust (see Figure 1 of the last Progress Report). In the case of the Air chambers the insulator is bonded to the conducting plastic by a friction welding process. The result of this welding process is to make the two parts as though they are one except that certain regions are insulating while others are conducting.

When the first T.E. units were assembled three years ago, much effort was spent on attempting to friction weld the polyethylene insulator to the A-150 guard. At the very best one obtains a 30 % weld which was not completely repeatable. This was very surprising in view of the excellent results achieved in the Air units and the

fact that the two materials to be welded in the present case, namely polyethylene and A-150, have been welded together with excellent results in the case of the large environmental chambers. The extremely solid bond achieved in the large chambers has been noted in the last Progress Report (page 10-11). The weld geometry in the present instance is distinctly different than in the large chambers. In the former situation one desires to weld along a cylindrical surface whereas in the latter, the contacting surfaces are planar. This difference combined with the physical properties of the two materials involved evidently operate against obtaining a good bond by friction welding. It is not the geometry alone for, as has been noted, an excellent bond is obtained by friction welding in the case of the 0.5 cc Air chambers where the geometry is the same.

The bases of the first T.E. chambers were therefore fabricated by joining the insulator and conducting guard using a combination of heat and pressure in a compression mold. It was thought that a weld had been achieved by this process. However, close examination revealed that, where the surfaces were thought to be welded, there was merely an intimate contact but no bond as such.

A solid bond is needed to assure the long term stability of the chambers. Therefore a number of attempts were made to ultrasonically weld the parts together during the past year. Different configurations of the parts and holding fixtures as well as various welding parameters, such as ultrasonic intensity, dwell time and contact pressure, failed to yield a satisfactory result. At best only a partial weld could be produced and usually at the expense of distortion of the parts. Therefore, the additional T.E. chambers

made during the past year employed bases fabricated in the same manner as the earlier ones.

For some time it had been planned to adapt a low noise triaxial cable to the 0.5 cc chambers. The two main advantages to the use of triaxial cable are the improved shielding afforded the central conductor which normally carries the collector current and the neater arrangement achieved thru the elimination of the extra wire that would otherwise be needed for connection to the shell of the chamber. In using the triaxial cable, the collector is connected to the central conductor, the guard to the inner braid of the cable, and the shell to the outer braid.

Of the special low noise cables manufactured by Microdot Inc., one, cable No. 250-3822, was selected as being closest to the plain coaxial low noise type previously used. The diameter of the dielectric (polyethylene) is .040 inches nominally in the triaxial type while it is .075 inches nominally in the plain coaxial type. Therefore, it is necessary to employ a stainless steel tube, as the stem of the chamber and to which the cable is anchored (see Figure 1), which is smaller in I.D. than the tube used previously. Secondly, one will doubtlessly have to suffer an increase in the collector-guard leakage because of the thinner dielectric in the triaxial cable.

Setting a smaller tube in the base of the chamber only required minor tooling changes. Standard 17 ga hypodermic needle tubing, having an I.D. .042 inches, represents a perfect match to the new cable. For the purpose of determining the suitability of the triaxial cable, a number of 0.5 cc Air chambers were assembled using the new cable. The results of evaluating (11) eleven of these chambers were most encouraging. The

collector-guard leakage was actually about the same magnitude as before.

The collector-guard leakage is normally tested at a bias voltage of 6 Volts between the collector and guard. The procedure is to first neutralize the chamber and cable by holding the chamber for 24 hours or so with the collector lead shorted to the guard. The bias is then abruptly applied and the collector current monitored as a function of time. The collector current decays in a manner which is characteristic of polarization currents in polyethylene. Somewhat arbitrarily, the value of the collector current at 150 sec after the bias is first applied is taken as a measure of the leakage. Out of the eleven chambers tested, (3) three exhibited collector currents less than 6×10^{-15} Amp and only (2) two showed currents in excess of 12×10^{-15} Amp.

A further advantage, which was not anticipated, to the use of triaxial cables emerged from these tests. Namely, there appears to be a general reduction in noise, but particularly in microphonic noise. The collector current is simply not as sensitive to slight movements of the cable. The explanation of this result may be that the two additional layers, one dielectric layer the other a wire braid, give greater support to the central conductor. Consequently, a slight deflection of the triaxial cable as a whole does not produce the same relative motion of the central conductor and dielectric as in the plain coaxial cable. Whether or not this is the correct explanation, the results found in these tests demonstrate that triaxial cables ought to be used universally in three-terminal ionization chambers.

COOPERATION WITH OTHER LABORATORIES

Over the years a significant and particular expertise has been developed in the Physical Sciences Laboratory at Illinois Benedictine College. This expertise includes much experience in the preparation of conducting plastic mixtures and in the design and construction of various types of ionization chambers. Furthermore, a unique capability has been achieved in the way of special techniques for mixing and fabricating plastics. Particularly notable are several means for welding plastics. The only practical means available to share these peculiar resources with other scientists is thru cooperation. By "cooperation" is simply meant that certain help is extended to other scientists in specifically their own research. Several illustrations of this type of activity during the past year are given below.

In the last Progress Report, it was mentioned that a pair of polyethylene collars were friction welded to a spherical T.E. chamber having an O.D. of 8 inches for a group at Brookhaven National Laboratory. During the past year a second pair of polyethylene collars were welded to a second 8-inch chamber for the same group. The indications are that both chambers have held up exceptionally well thus far.

In connection with the participation of Frank S. Williamson in the International Neutron Dosimetry Intercomparison, certain parallel-plate chambers of his were rebuilt. New clamping and spacing rings were made incorporating thin teflon gaskets for high voltage insulation as well as to render the chambers reasonably air-tight. After modification these chambers were given preliminary testing to ascertain whether they could be counted on for the intercomparison. The preliminary testing

showed that one of the two graphite chambers had excessive collector-guard leakage and could not be used without certain corrective procedures.

Dr. Peter Fessenden of Stanford University sought our assistance in two areas. First he desired some of a special plastic mixture which Dr. Shonka had designed several years ago in collaboration with Dr. Raju then of the University of California. This particular mixture was intended to be muscle-equivalent with respect to pions. A small slug of this material was molded for Dr. Fessenden which he planned to use for a construction of small ionization chamber. Secondly, Dr. Fessenden desired a 0.5 cc T.E. chamber having a hollow collector. Such was achieved in the standard chamber (see Figure 1) by sectioning a collector at a point .015 inches above the base of the collector. The remainder of the collector was then bored out leaving a uniform wall .015 inches in thickness. The two parts were then re-joined by electrically welding them in a special fixture. The welding technique employed was one that had been used frequently in the past for joining small spherical shells. It consists of charging a capacitor to a certain voltage and discharging the capacitor thru the two parts held in contact with a definite force. Electrical continuity is achieved by electrodes attached to each of the two parts near the location of the weld.

APPENDIX A

"A Small Ionization Chamber for Measurement of Neutron and Photon Dose" *

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ABSTRACT

A small thimble-type three terminal chamber of ionizing volume equal to 0.5 cm^3 has been developed for accurately determining depth doses in human muscle. The chamber is homogeneous in that the entire sensitive portion of the chamber is constructed of Shonka tissue-equivalent (muscle) conducting plastic and has provision for circulating a matching tissue-equivalent gas continuously through it. Precise definition of the sensitive volume is achieved by means of a buffer region. Leakage currents are of the order of 10^{-16} Amperes and voltage soakage phenomena is virtually nonexistent. The angular dependence as well as the energy dependence will be discussed experimentally and theoretically. The measured calibration factor is 5 rad/nanocoulomb for Cesium-137.

* Work supported by the U.S. Atomic Energy Commission. Paper to be read at the 5th International Congress of Radiation Research, Seattle, Washington, July 14-20, 1974.