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CLEVELAND, OHIO

Semi-Annual Status Report

for the period March 1, 1966 to August 31, 1966

NASA Grant NsG-655

Principal Investigator: Karl J. Casper

Summary

This status report covers research on silicon and germanium detectors:

(a) Large volume lithium drifted germanium detectors,

(b) tests of large volume silicon detectors,

(c) temperature dependence of the energy resolution of silicon detectors.

In addition, preprints of two papers now in press ~~are appended~~. Removed This research is being published under the auspices of this grant and concerns applications of radiation detectors to nuclear spectroscopy.

Finally, research on the internal Compton effect which was finished during the period covered by this report has been discussed in a separate report issued August 10, 1966.

Lithium Drifted Germanium Detectors

An 8 cm^3 lithium drifted germanium detector has been in operation for more than six months. This detector is a five-sided coaxial type and has a resolution of 4 keV with the Co^{60} lines at 1.17 MeV and 1.33 MeV using an uncooled FET preamplifier.

The limited supply of driftable germanium has proved to be the primary delay in this area. Orders to Sylvania have taken over five months to fill and little or no material was obtained from them until August.

In an effort to ease this situation, Semimetals, Inc. was contacted as a possible supplier. Their material has previously been of poor quality for lithium drifting. However, several suggestions were made to them by us to improve the quality of the germanium. They cooperated primarily by rebuilding their furnace to make it vacuum tight and by expending considerable effort in eliminating lineage from their germanium. In September, a sample slice was supplied to us. This piece exhibited normal drift characteristics. The total drifted depth was 6 mm when drifted at 100 volts and 47°C for fifteen days. The rest of the ingot has been delivered and will be tested.

A preliminary version of the preparation of lithium drifted germanium detectors is given below.

Preparation of the crystal for diffusion.

The crystal is sanded lightly, cleaned in trichloroethylene and alcohol, and placed in a saturated solution of copper sulfate in hydrofluoric acid. The copper will plate onto the crystal and is then diffused into the crystal in air on a hot plate. The crystal is sanded using a sanding wheel and all corners are slightly rounded. After cleaning and scrubbing with trichloroethylene and alcohol, the surfaces which are not to be diffused are painted with Alkadag #154 available from Acheson Colloids, Inc.

Diffusion.

Lithium is evaporated in a vacuum of 10^{-5} mm or better onto all faces of the crystal which are to be diffused. Without removing the crystal from the vacuum, the lithium is then diffused into the crystal at about 400°C for twenty minutes.

Several methods of heating the crystal have been tried. The most successful has been to place the crystal on a carbon block which has two Vulcan 550 watt Thunderbolt heaters mounted inside. The temperature is monitored with a platinum wound resistor. The block is heated to the correct temperature and the system is then filled with helium gas to a pressure of 1 mm. This permits overall heating of the crystal.

After diffusion, the helium gas is pumped out of the system and the crystal is allowed to cool to room temperature in the vacuum. It is relatively important not to allow air to reach the crystal since lithium precipitates very readily in the presence of oxygen at elevated temperatures.

Preparation for drift.

The crystal is removed from the vacuum system, washed in distilled water and the surfaces are lightly sanded. The depth of the diffusion can be determined by a copper plating procedure. The crystal is submerged in a 20 g/liter $CuSO_4$-H_2O solution and a nine volt reverse bias applied to the crystal. Copper plates heavily at the junction on the p side of the junction but not on the n-side. The diffusion is acceptable if it is greater than 0.3 mm.

The copper is then removed by immersing the crystal in HNO_3. Those regions of the crystal corresponding to the exposed p-n junction are sanded on the lapping wheel with #320, #600 sanding discs and then a pellon disc which is kept wet with a cerium oxide slurry. The crystal is etched in 6:2:1 HNO_3, HF, fuming HNO_3. The etch is usually performed twice, the first etch lasting until significant etching action has ceased or the solution has become quite hot; the second etch serves as a cleanup etch and is usually quenched after one or two minutes.

If the crystal is a planar device, both the top and bottom surfaces are covered with gallium-indium eutectic. For a coaxial device, the n-type lithium diffused surface is covered with eutectic and a small drop of eutectic is placed in the center of the p-type region.

Drift.

Drifting is carried out in Freon-113 which has a boiling point of 47°C. A power supply capable of providing 0-300 volts at 3.5 amps is used and the diode is placed in series with light bulbs to limit the current. For the first three hours, the voltage applied is only 100 volts in order to build up the junction. After that, a voltage slightly less than the breakdown voltage is applied.

Two types of drift have been observed. In general planar devices continue drifting for nearly one centimeter without requiring further diffusion. On the other hand, it has been found necessary to rediffuse coaxial devices. If there is no degradation of the diode characteristics with time, i.e., the current does not steadily increase, it has been often found that the lithium is drifting very slowly. Usually, the current gradually increases with time, which is characteristic of lithium precipitation, and it is occasionally necessary to rediffuse the crystal.

After drifting, the diode is rediffused to renew the lithium contact. At this point two cleanup drifts are performed. The first is carried out a few degrees below room temperature dissipating 30 watts in the diode. This drift lasted for two days. The crystal is then mounted in its dewar system and cooled to liquid nitrogen temperature. Both the current voltage curve and capacitance as a function of voltage are measured. The capacitance curve should exhibit nearly a flat characteristic above approximately 10 volts/mm if the material is well compensated. If it is not flat, this means that the compensated region is not intrinsic and the depletion depth is a function of the bias voltage. This variation in depletion depth is reflected by a variation in capacitance.

The temperature of the crystal is now adjusted so that the second cleanup drift at 200 volts has a current of 0.3 ma/cm^2 of surface area. This is most easily accomplished by slowly feeding liquid nitrogen into the dewar and monitoring the current personally. After four or five hours of drift, the crystal is cooled to liquid nitrogen temperature and the current and capacitance characteristics are again measured. This process is repeated until the capacitance characteristic becomes flat.

This cleanup process is very important from the standpoint of optimizing the resolution and producing symmetric gamma ray photopeaks. These are dependent on exact compensation of the material. Since the initial drift is usually carried out at high currents, the compensation is not exact since there will be some distortion of the electric field from this current. In general, the lithium ions which see this current in addition to the field of the impurity atoms will be slightly displaced from a position where they will compensate the material, and a drift at much lower temperature is necessary to move the lithium ions into the correct interstitial position. The last cleanup drift provides the last small necessary displacement. Some further adjustment will occur at liquid nitrogen temperature and a slight improvement in resolution as a function of time has been noted.

Large Volume Silicon Detectors

The detection of high energy particles such as protons above 100 MeV requires silicon detectors of unusually large depletion depth. The path length of the particles increases rapidly with energy and for protons whose energy is 160 MeV the range is about 9 cm. It is not feasible to lithium-ion drift this distance. However, a detector has been fabricated in the following way.

As shown in Fig. 1, the silicon ingot has been squared off to form a rectangular bar. In this particular case, the dimensions of the bar were 1.5 cm x 1.5 cm x 11 cm. Lithium was diffused into one of the 1.5 cm x 11 cm faces and drifted to a depth of 1 cm. The detector thus formed can be used to detect high energy protons if the particles are incident normally on one of the 1 cm x 1.5 cm faces.

This detector was completed and has been tested at the Harvard University cyclotron where 160 MeV protons are available. The leakage current of the detector at room temperature was 100 μa at 100 volts. Since very high voltages are necessary for this detector in order to obtain good timing and complete charge collection, it was cooled to liquid nitrogen temperature before using. The detector was mounted in the system shown in Fig. 2. The entrance window to the detector was a 10 mil thick beryllium plate which, by calculation, should spread the resolution by less than 100 keV. At liquid nitrogen temperatures the current at 2000 volts was less than 10^{-9} amps which was sufficiently low for the electronics used.

In the initial tests at the cyclotron, the detector was placed directly in the beam using only the residual ions to produce the beam. A single lead collimator was used with a thickness of 2" and an entrance

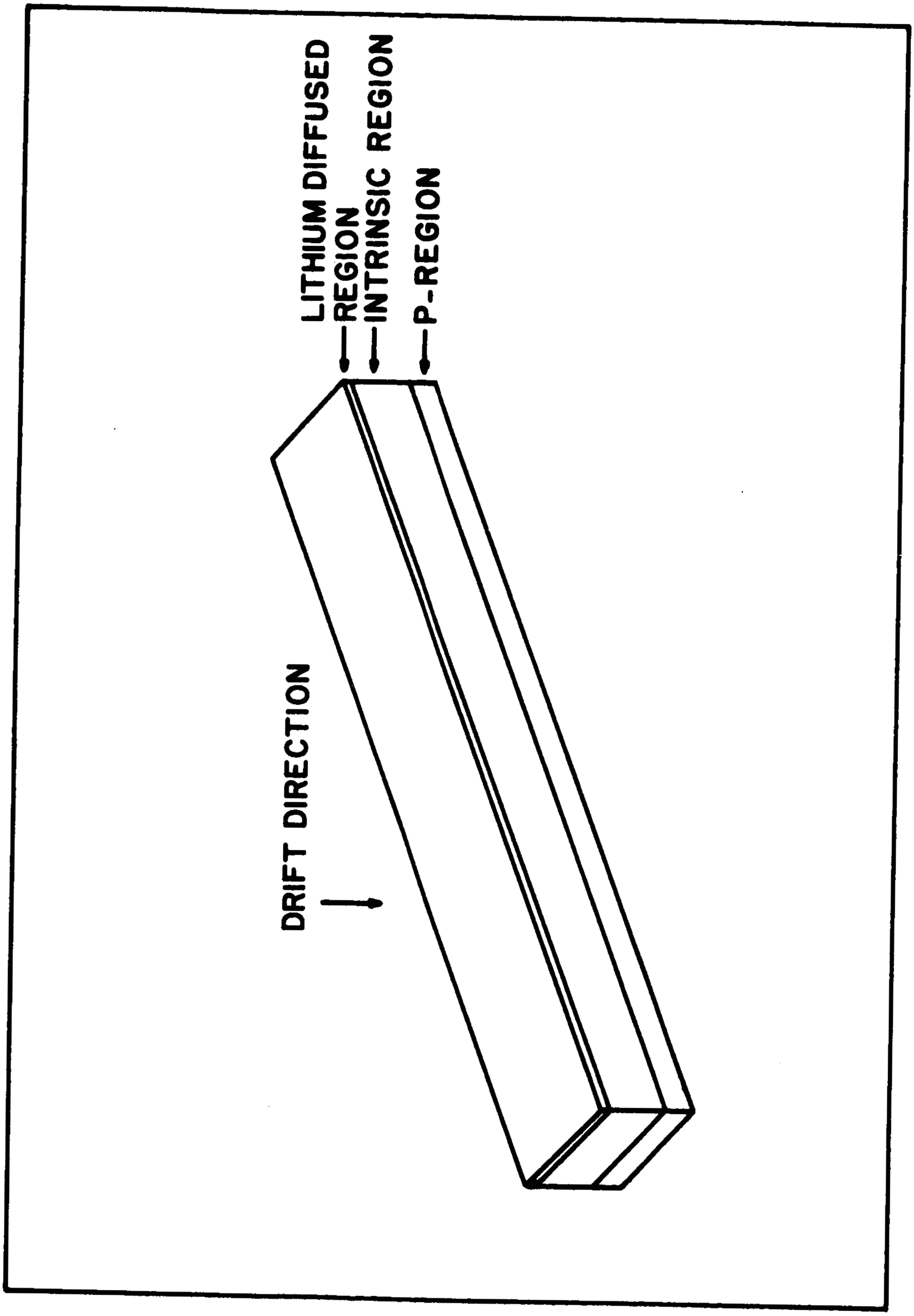
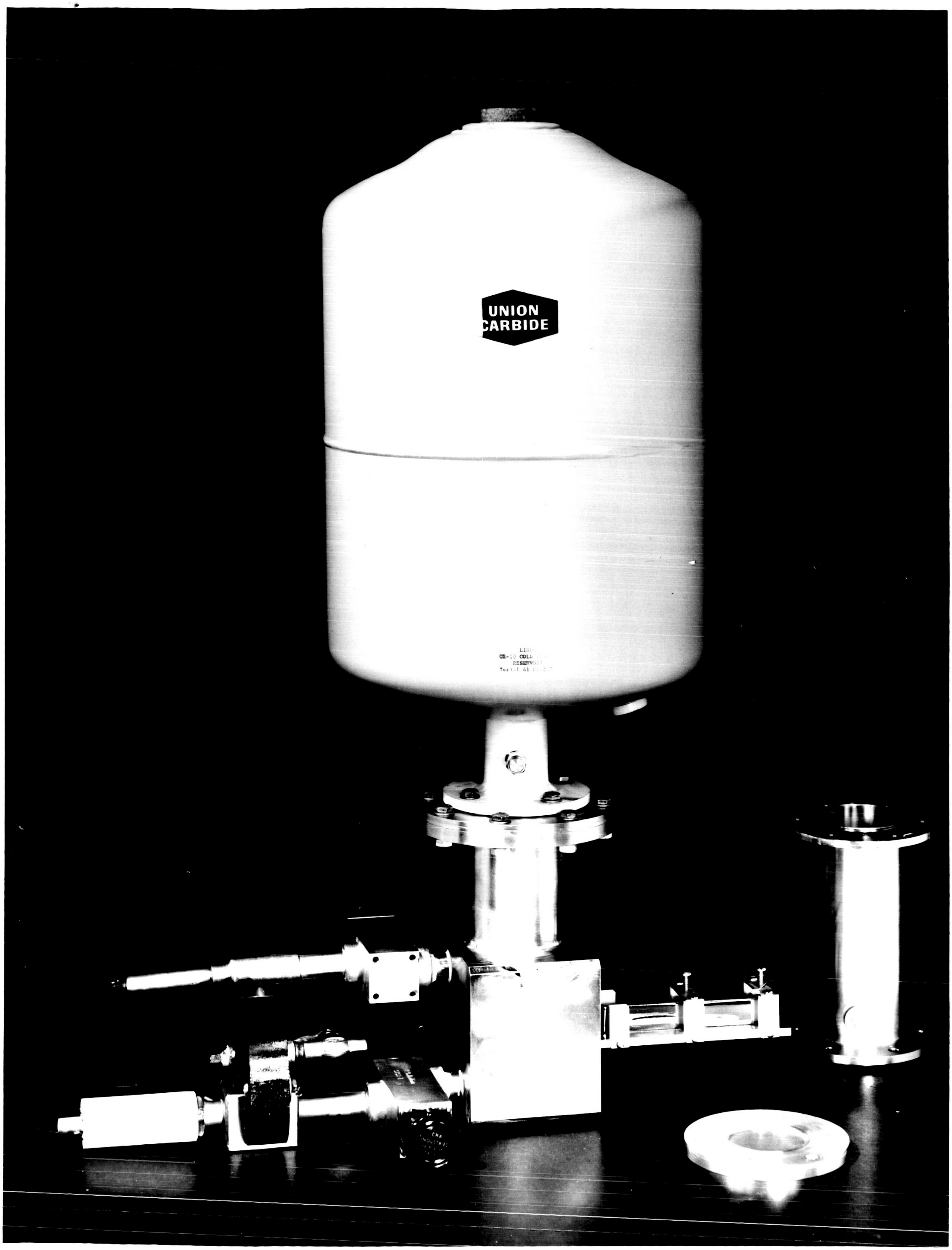

LITHIUM DIFFUSED REGION
INTRINSIC REGION
P-REGION
DRIFT DIRECTION

Fig. 1

Fig. 2. Large volume silicon detector with associated cryostat.


UNION
CARBIDE

hole 1/8" in diameter. A typical spectrum obtained with the detector is shown in Fig. 3. Considerable effort was made to reduce the excessively large number of counts in the tail of the spectrum. The horizontal alignment and the vertical height were altered, and double lead collimators were used. To see if all of the charge was being collected, the bias voltage was varied between 500 and 2500 volts. No change in pulse height was seen above 1500 volts, and the spectra taken above this voltage were identical. None of these methods eliminated the tail of the spectrum.

At this point, the beam energy was degraded to provide 100 MeV protons. A spectrum of these particles is shown in Fig. 4. The resolution of the peak has been reduced, but it is quite obvious that the contribution of the tail is considerably smaller. It was concluded that the primary problem was multiple scattering of the protons within the detector. Multiple scattering involves no large single scatterings, but merely the collaboration of a large number of scatterings which would individually be so small that no deflection of the incident particle would be detected. These scatterings occur in the production of the ion pairs and may add in such a way as to give a rather large overall deflection of the incident particle. This process is susceptible to theoretical treatment on a statistical basis since so many scatterings occur. Such a treatment has been done, although not published, by W. M. Preston of Harvard. For this experiment the beam would be expected to spread out radially with a Gaussian distribution. The locus of the half maximum points of this distribution forms a circle with a diameter of 1.5 cm. This is greater than the vertical thickness and equal to the width of the detector. As a result it was felt that this effect was the one responsible for the tail in the spectrum and the initial experiment was concluded.

Fig. 3. Spectrum of 160 MeV protons measured with large volume silicon detector.

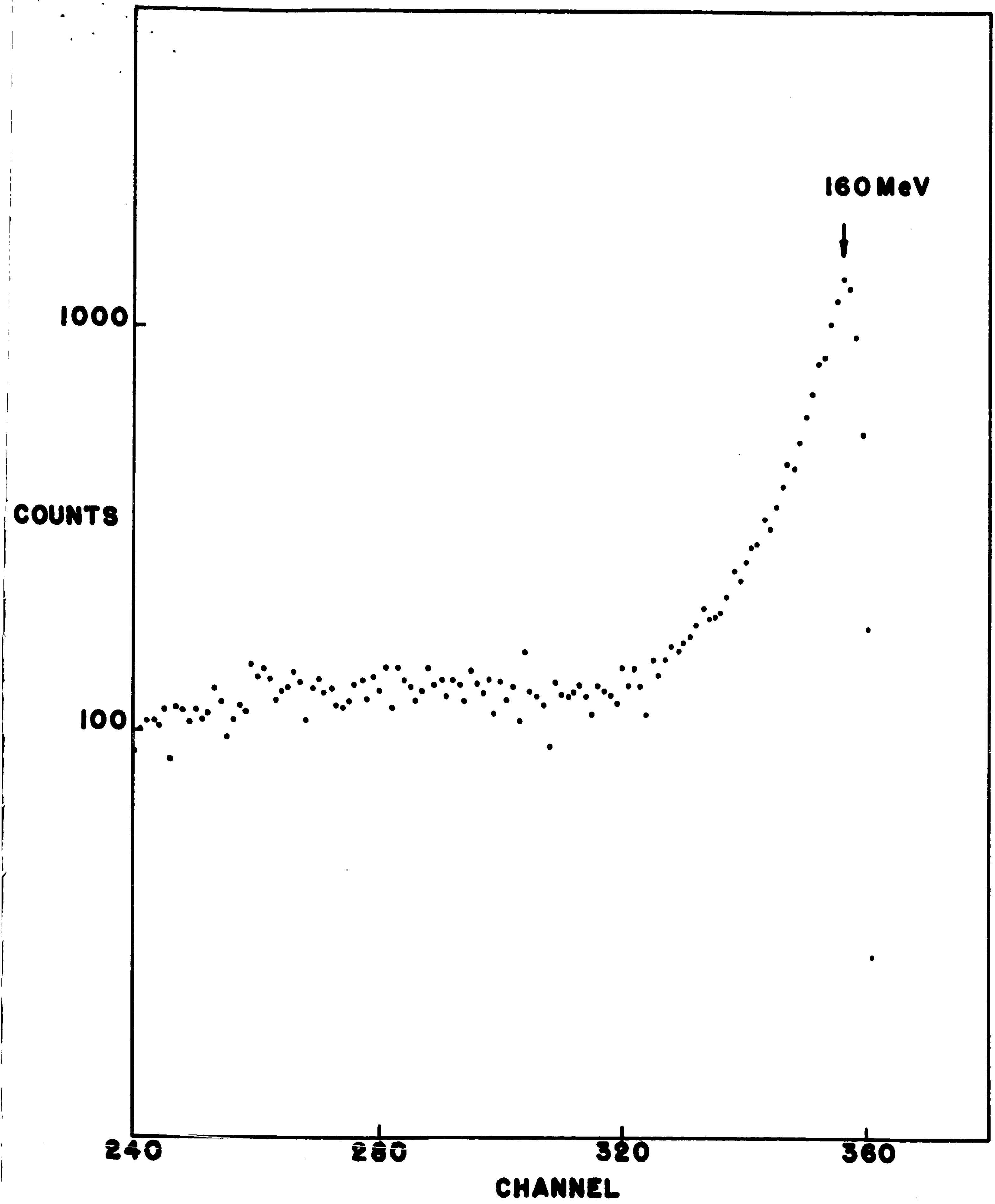


Fig. 3

Fig. 4. Spectrum of 100 MeV protons obtained by degrading 160 MeV protons with absorbers.

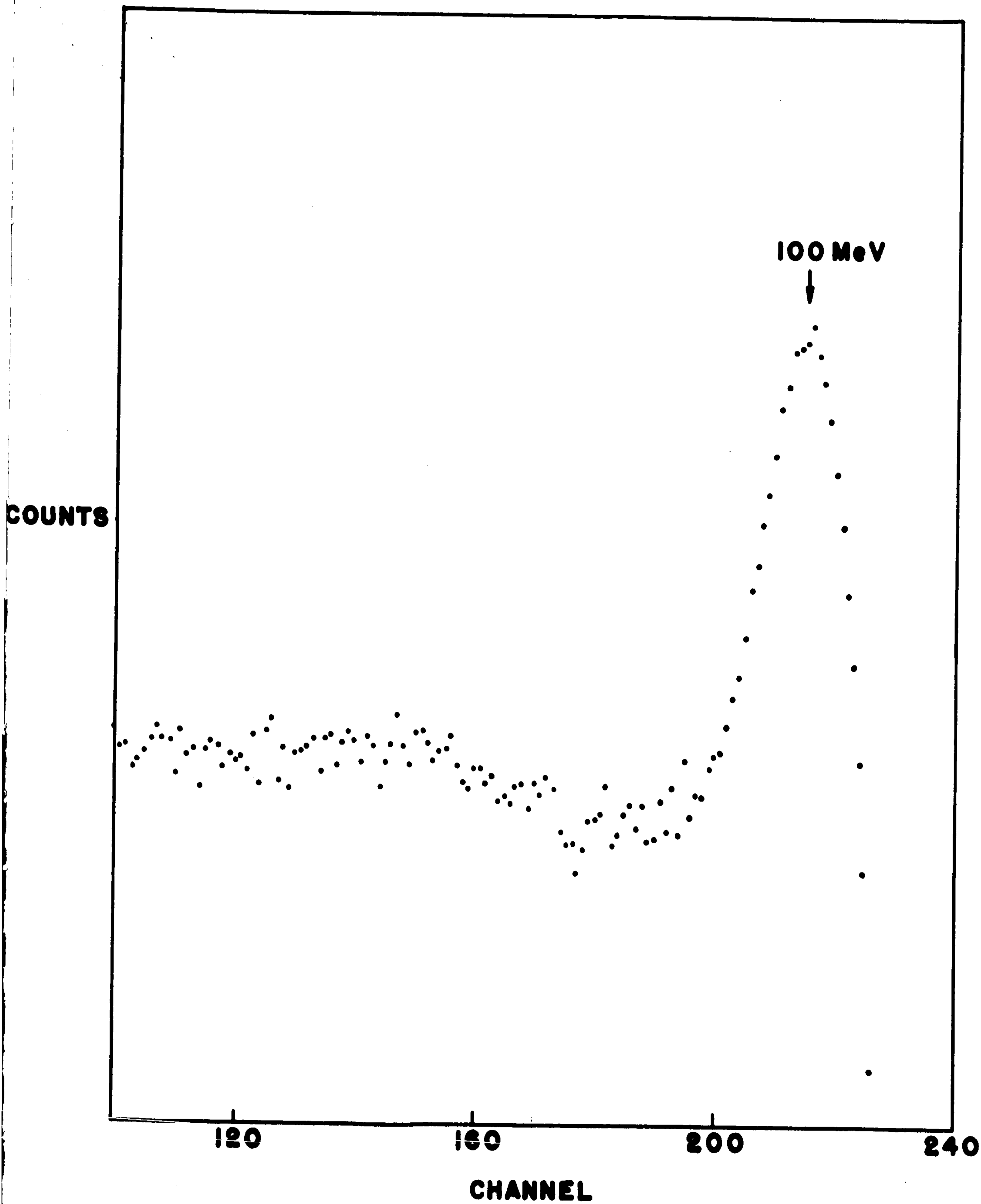


Fig. 4

However, further experimental work to check this possibility has been performed by A. M. Koehler with the cyclotron. A series of aluminum plates of increasing thickness was placed in the beam of protons with a photographic plate at the back of these plates. The divergence of the beam was measured as a function of plate thickness and the circle produced at the end of the range had a diameter no greater than 0.5 cm which is not in agreement with the theoretical prediction and is well within the detector dimensions.

Several possibilities therefore remain to be explored. A method for adjusting the vertical alignment of the detector will be tested. The alignment is most critical since a misalignment of only 7° is sufficient to reduce the effective depletion depth seen by the proton to less than a maximum range of 9 cm.

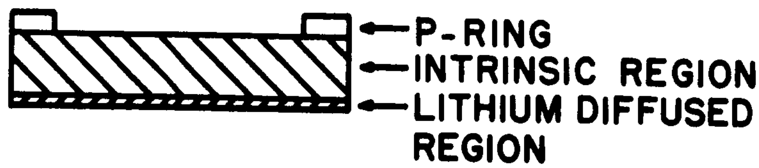
In addition, a much larger detector is now being drifted. An ingot 12 cm long having a diameter of 3.6 cm was obtained from Dow-Corning. This was sanded into the form of a rectangular bar 2.5 cm x 2.5 cm x 12 cm. It is intended to drift this detector to a depth of 2 cm thus increasing the cross-sectional area by more than a factor of three. At the present time, the drifted depth of this detector is 1.1 cm. Both this and the first detector will be checked again at the Harvard Cyclotron in the middle of December. The vertical alignment will be varied; the second detector which will not be completely drifted until late spring will have a vertical thickness equal to that of the first detector but will have nearly twice the width. A comparison of the spectra recorded by these two detectors will also help settle the multiple scattering question.

Temperature dependence of the energy resolution of silicon detectors.

One of the aims of this research was to measure possible fluctuations in the energy resolution as a function of temperature of lithium drifted silicon detectors. Some improvement in resolution is to be expected as the temperature is reduced to liquid nitrogen temperatures since the reverse leakage current is decreasing and the carrier mobility is increasing. However, it has been found that the resolution of germanium detectors is best at temperatures slightly above liquid nitrogen. By analogy, one might also predict a similar effect with silicon detectors.

As part of this experiment a lithium drifted silicon detector with the p-ring configuration shown in Fig. 5 was used initially. This p-ring structure permits direct contact of the ring with the metal holder, and the detector is easily cooled to a particular temperature. It should be noted, however, that this type of detector has not given us optimum resolution at room temperature. Direct comparison of slices from the same ingot shows that the configuration generally used by us and previously reported yields a better resolution by a factor of two. One of the main reasons for this is that it is nearly impossible to lap the front surface and be certain that the intrinsic region has been reached. However, the ability to cool this detector was the primary motivation for use of this structure.

The system used in these measurements was pumped only to forepump vacuum. The copper block on which the detector was mounted was insulated with mica. In order to achieve a set of stable detector temperatures between room temperature and liquid nitrogen temperature, simultaneous cooling by a liquid nitrogen reservoir and heating by a cartridge heater



CROSS SECTION OF P-RING DETECTOR

Fig. 5

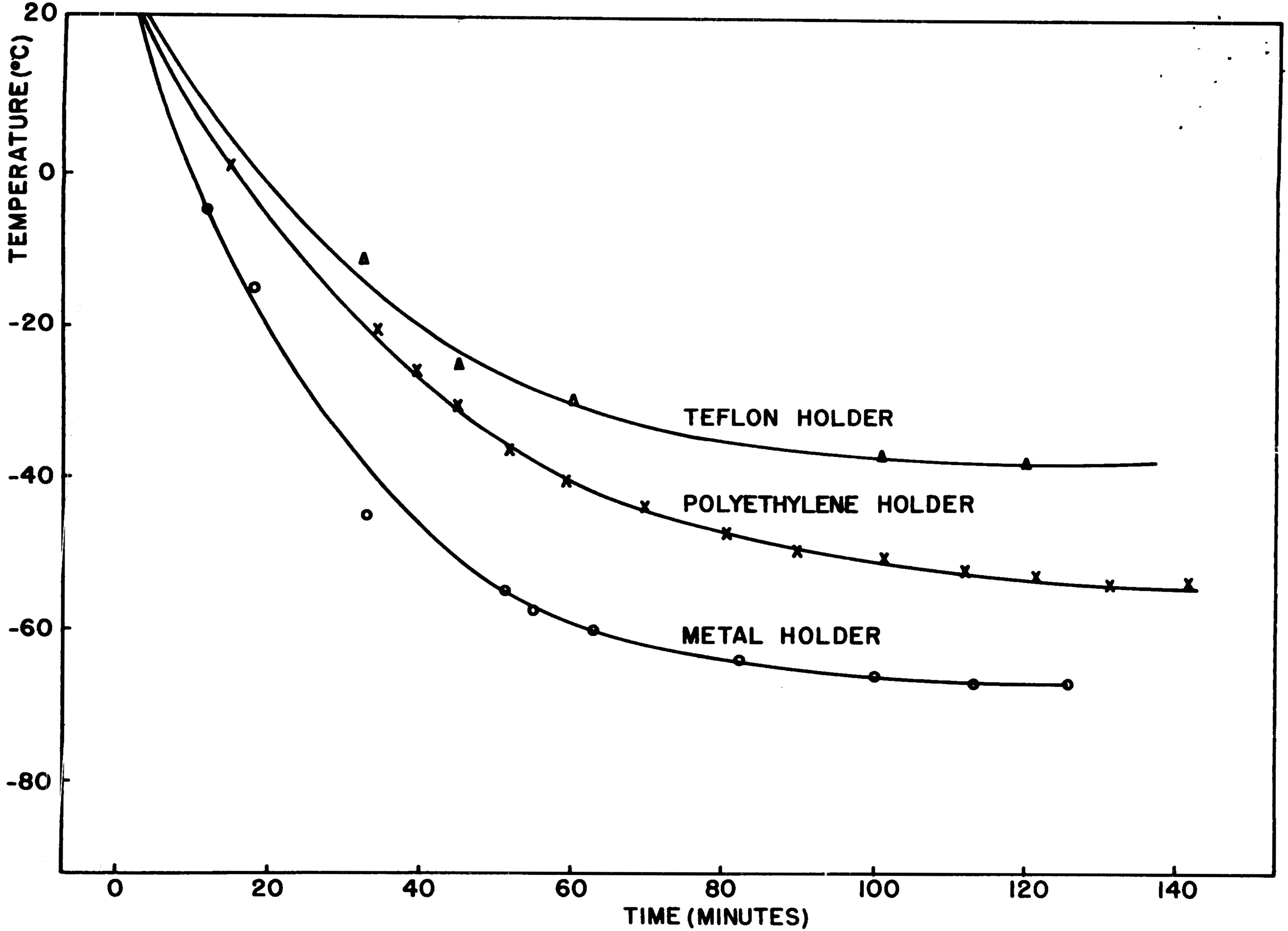
were employed. The block on which the detector was mounted was insulated with mica from the liquid nitrogen reservoir while the cartridge heater was mounted in the block itself. Dynamic equilibrium was possible although half an hour was usually necessary to be certain that equilibrium had been established.

A Bi^{207} source which has a set of conversion lines from the 569 keV and 1063 keV transitions was used for the resolution measurements. Both resolution and leakage current were measured. The leakage current continued to decrease as the temperature decreased. However, the resolution as shown in Fig. 5 actually displayed a minimum at about $-100^{o}C$. Since most of our detectors are fabricated without a p-ring structure, it was desirable to determine the characteristics of these other detectors. Three types of holders are used: Teflon, polyethylene, metal with a thin polyethylene insulator. Each of these detectors was mounted in the vacuum system, and the leakage current and resolution were measured as a function of temperature. In order to monitor the temperature, a platinum resistor was attached to the clip holding the detector and the system was allowed an hour to reach equilibrium. It was assumed that the detector temperature and the temperature of the mount were the same at that time. Surprisingly, the resolution showed no minimum as before. Moreover, the leakage currents rarely fell below 20 nanoamps. These results led the suspicion that the temperature of the detector was not being accurately measured.

This point was checked in the following way. A brass tube containing a platinum resistor for temperature measurement was epoxied with silver bearing epoxy to a drifted silicon detector. Then the mounting block temperature and the detector temperature were both monitored as the system was cooled to liquid nitrogen temperature. The mounting block temperature fell quickly to liquid nitrogen temperature, but the temperature of the detector did not drop nearly so much as can be seen from Fig. 6. The insulation of the detector was simply too good to permit low-temperature cooling.

Thus, the temperatures achieved in these holders never reached the point at which the minimum was observed in the p-ring device. A change in the insulation of the metal holder from polyethylene to sapphire is now being made. Sapphire is particularly advantageous since it has good electrical insulation properties, but has a thermal conductivity nearly two orders of magnitude better than Teflon or polyethylene. The results of these measurements will be reported later.

Fig. 6. Minimum temperature achieved in detector using different holders clamped to liquid nitrogen reservoir. Metal holder has a thin polyethylene insulator between it and the detector.


TEMPERATURE (°C)
20
0
-20
-40
-60
-80
TEFLON HOLDER
POLYETHYLENE HOLDER
METAL HOLDER
0
20
40
60
80
100
120
140
TIME (MINUTES)

Abstract

A test of time reversal invariance in electromagnetic transitions is proposed in which all possible two gamma-ray angular correlations in a triple cascade are measured. The results can be combined to yield a term proportional to $\cos \eta$.