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One Dimensional Curved Wire Chamber for Powder X-Ray Crystallography

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ABSTRACT

A xenon filled single anode wire chamber with delay line readout has been constructed for use in powder X-ray crystallography using 8-20 keV X-rays. The entire chamber including the anode wire and the delay line which forms part of the cathode plane is a section of a circular arc whose center is the powder specimen. The anode wire--38 μm gold-plated tungsten--is suspended in a circular arc by the interaction of a current flowing through it and magnetic field provided by two permanent magnets, above and below the wire, extending along the active length of the chamber. When filled with xenon to 3 atmospheres the chamber has uniform sensitivity in excess of 80% at 8 keV and a spatial resolution better than 0.3 mm.

INTRODUCTION

In the past few years powder X-ray crystallography has become an important analytical tool for identifying samples and the impurities in them. For this purpose one desires a device which can detect quickly the characteristic spectra of the sample constituents. The usual method has been the use of X-ray film. Film has excellent resolution, but its main disadvantage is the very low sensitivity. Many of the

spectra of interest are very weak in intensity and the film may require many hours of exposure in order to blacken it sufficiently to be readable. Even for those cases where the exposure time is short, the film must be developed and then scanned manually with a microdensitometer.

Another method is to use a position sensitive X-ray detector which records the position of the detected X-ray electronically and stores the resultant spectra in a computer or multi-channel analyzer. A gas proportional chamber is very suitable for this purpose. When filled with xenon the efficiency can be between 90% and 70% for X-rays in the energy range 8 to 20 keV. Such chambers can be either linear with a single wire (1) or two dimensional multi-wire arrays (2).

DESCRIPTION OF CHAMBER

The detector consists of a single wire, xenon filled chamber whose sensitive volume has a cross sectional area $1.25 \times 1.25 \text{ cm}^2$ as shown in Fig. 1. A photograph of the detector is shown in Fig. 2. The readout is accomplished by a delay line (3) (delay 25 nsec/cm) with a bare copper helical winding which forms one surface of the cathode (4). In order to eliminate the position spread in detecting X-rays incident on the detector at angles other than 0° , the sensitive volume including the anode wire and delay line are curved to concentric arcs whose center is the position of the powder sample. Since it is desirable to have a uniform sensitivity along the anode wire without any dead regions which would be introduced by mechanical supports of the anode wire, it is held only at both ends by insulated supports. The wire itself is suspended at its appropriate radius of curvature by the magnetic force produced by a current through the wire interacting

with the magnetic field of the permanent magnets shown in Fig. 1.

The iron case of the detector serves as a flux return yoke.

The magnetic material is rubber bonded barium ferrite (5). After it is cut to the desired curvature it is magnetized. The field strength in the gap is about 800 Gauss. The magnets are covered with aluminum foil to prevent charge buildup on the rubber material.

The anode wire is 38 μm gold-plated tungsten. Wire such as stainless steel although often used in proportional chambers is not suitable in this case because of its high resistance; it melts at the necessary current. It is found that 15 mA is necessary to float the wire at the correct position.

A thin beryllium entrance window (1 mm) is used for minimum attenuation of the X-rays. Part of the cathode plane is formed by the electromagnetic delay line which is clamped in a lucite holder which bends the line to the desired shape. No degradation in the performance of the line is observed after it is curved.

The chamber was filled to 3 atmospheres with either a mixture of Ar(90%) CH₄(10%) or Xe(90%) CH₄(10%). The xenon mixture is used for high detection efficiency. The detection efficiency is estimated from the window attenuation, gas pressure, thickness of sensitive volume and is shown in Fig. 3 for xenon at pressures of 3 and 5 atmospheres. At 8 keV the efficiency is limited by the thickness of the beryllium window.

We readout each end of the delay line and also the anode wire. Fig. 4 shows the circuit which applies both the current and high voltage to the center wire and couples out the anode signal. A

filament transformer and rectifier provide the dc current to the wire. A current pulse in the anode wire due to an ionizing event induces a voltage drop across the inductors which is coupled through the dc blocking capacitor to the amplifier. The 60 Hz noise is filtered out using an inductor and capacitor filter.

The pulses from the anode and delay line are amplified and fed into zero-cross discriminators to determine the timing. A single channel analyzer is used to gate the anode wire signals in order to select only those pulses near the photopeak. Time to digital converters (TDC) are used to measure two time intervals: the time between the anode pulse and the delay line pulse from the left side and also the time between the anode pulse and the right side delay line pulse. These two intervals measure the distance between the event position and the left and right side of the delay line respectively. If we do an average of these two positions, we find that the result depends only on the timing of the delay line signals; the anode timing drops out, and the timing resolution improves by a factor of 2 (Ref. 6). This average is exactly equivalent to measuring the time difference between the arrival of the signal on the left side and its arrival at the right side of the delay line; it would be sufficient in most cases to readout only the ends of the delay line and use on TDC to measure the time difference. There are, however, some advantages to reading out the anode which may or may not outweigh the added complexity. The sum of the time differences between the anode signal and the respective signals from the ends of the delay line is simply the length of the delay line, plus some delay in the processing

electronics, a constant to within the timing accuracy. The width of this sum distribution is a measure of the spatial resolution of the detector since spatial accuracy is in large part determined by the timing resolution (6). This is often a more convenient method of monitoring the resolution than using a well collimated source; for example it can be done while taking data for other purposes. Another use of this time sum is for the identification of multiple hits in the detector; either from two particles striking at the same time or from a second particle arriving before the pulses from the first particle have cleared the delay line (7). These events for which the time sum is less than the correct value can then be rejected. This rejection capability is only necessary when the average rate is so high that the number of such two particle hits is non-negligible. If one is willing to allow a small fraction of the events to have position ambiguity due to these multiple hits, then with a 1 μ s long delay line the rate can be as high as 250,000 events/sec before the 1% level is reached.

In order to obtain high timing accuracy strict pulse shaping is required. This shaping was accomplished by decreasing the size of the amplifier output coupling capacitor to provide more differentiation of the pulse. For these chambers we found that a rise time of 20 ns and fall time of 25 ns gave the best timing results. The undifferentiated signals from the amplifier had rise and fall times of 70 and 250 ns for the anode and 45 and 100 ns for the delay line signals.

TESTING AND CALIBRATION

The chamber was tested using the 5.9 keV X-rays from ^{55}Fe . Both argon and xenon gas mixtures were used. Figure 5 shows the sum distribution obtained with an uncollimated source. The width is 3 ns which indicates a spatial resolution of 0.3 mm.

In order to test the uniformity of response and verify the spatial resolution, a slotted mask was placed in front of the detector with a ^{55}Fe point source located at the center of curvature (29 cm from the anode wire). The pattern was constructed from 0.13 mm Be-Cu with slots 0.2 mm wide at a spacing of 9.5 mm. Figure 6 shows the recorded position of each slot as a function of known location. The linearity is excellent. Also shown on the same figure is the total number of counts observed through each slot. The detector is quite uniform in efficiency. In Fig. 7 the observed spatial distribution is shown for events passing through one slot. The spatial resolution is found to be 0.3 mm, since the resolution of the 0.2 mm slit is included in the distribution.

CONCLUSION

In conclusion we have designed and constructed a curved one dimensional position sensitive proportional chamber for use in powder X-ray crystallography. The resolution is 0.3 mm and the uniformity of response is very good.

ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

- Fig. 1. A cross sectional view of the detector.
- Fig. 2. A photograph of the chamber showing the beryllium window.
- Fig. 3. The calculated detection efficiency of the mixture Xe(90%)
CH₄(10%) at 3 and 5 atmospheres in a chamber 1.25 cm thick
as a function of X-ray energy. Absorption by the beryllium
window is included.
- Fig. 4. A schematic of the circuit supplying dc current to the anode
wire and providing signal readout.
- Fig. 5. The time sum distribution for the ⁵⁵Fe source.
- Fig. 6. Test results with the slotted mask. The solid line and dots
show the time recorded by the TDC's for each slot as a function
of known distance. The small triangles give the number of
counts observed to come through each slot.
- Fig. 7. The spatial distribution for events passing through one slot.

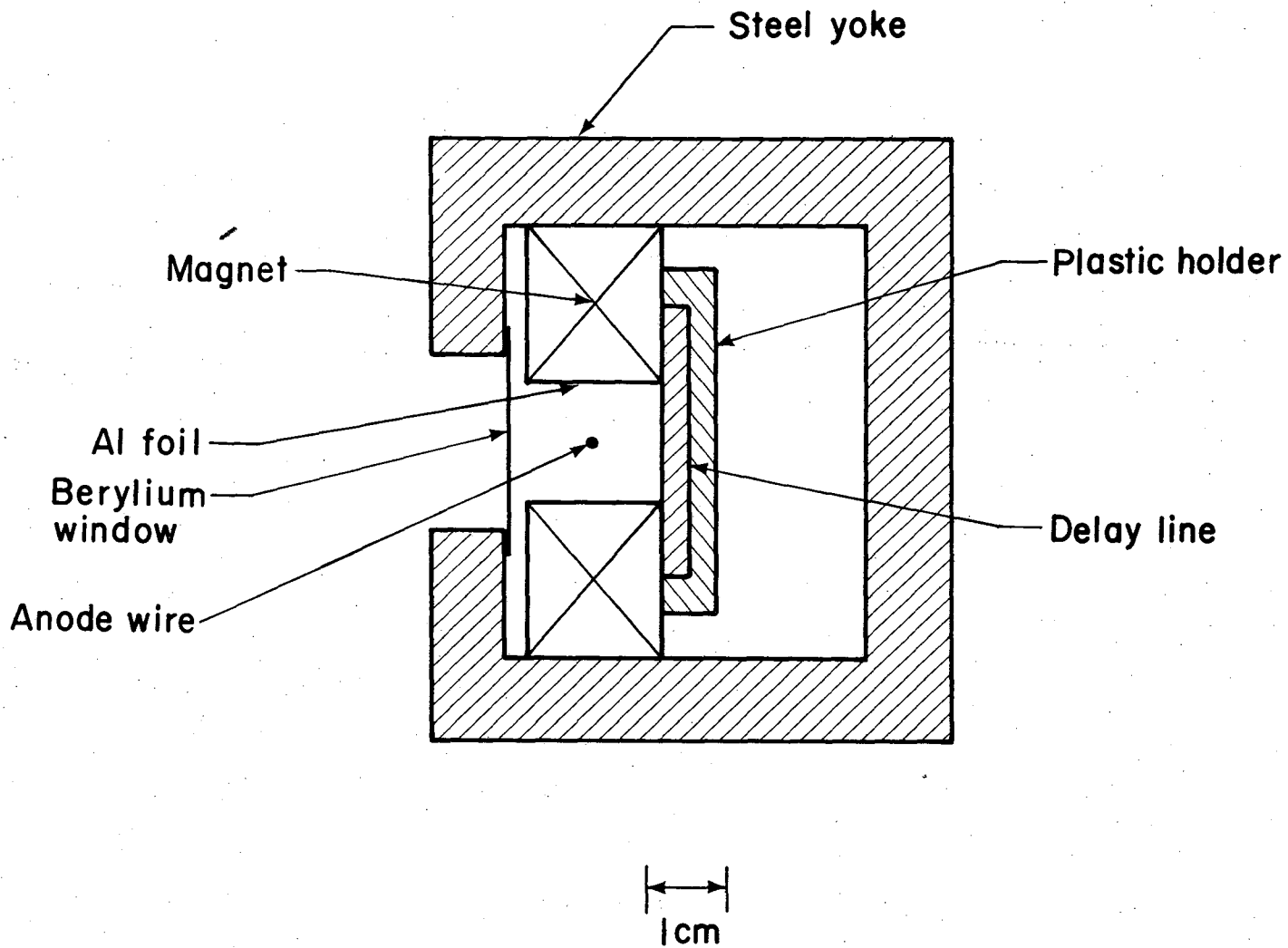


Figure 1.

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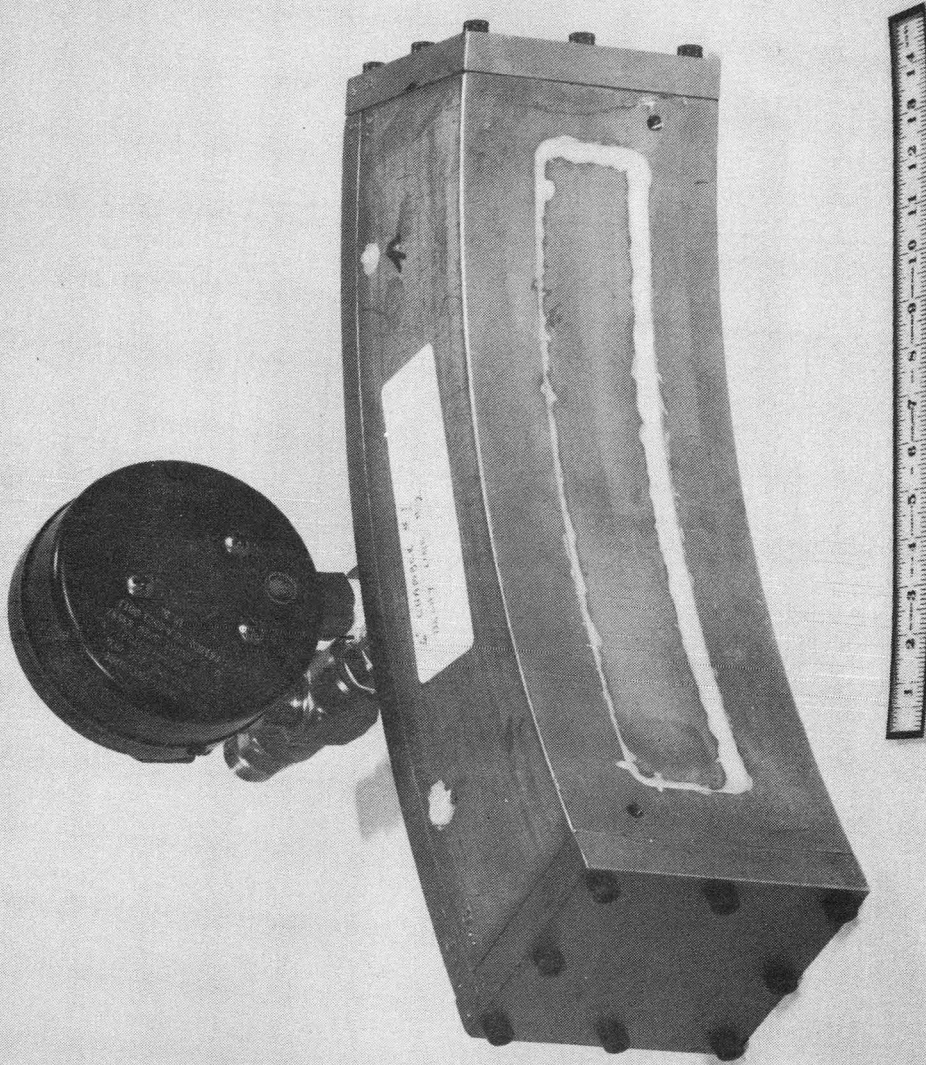


Figure 2.

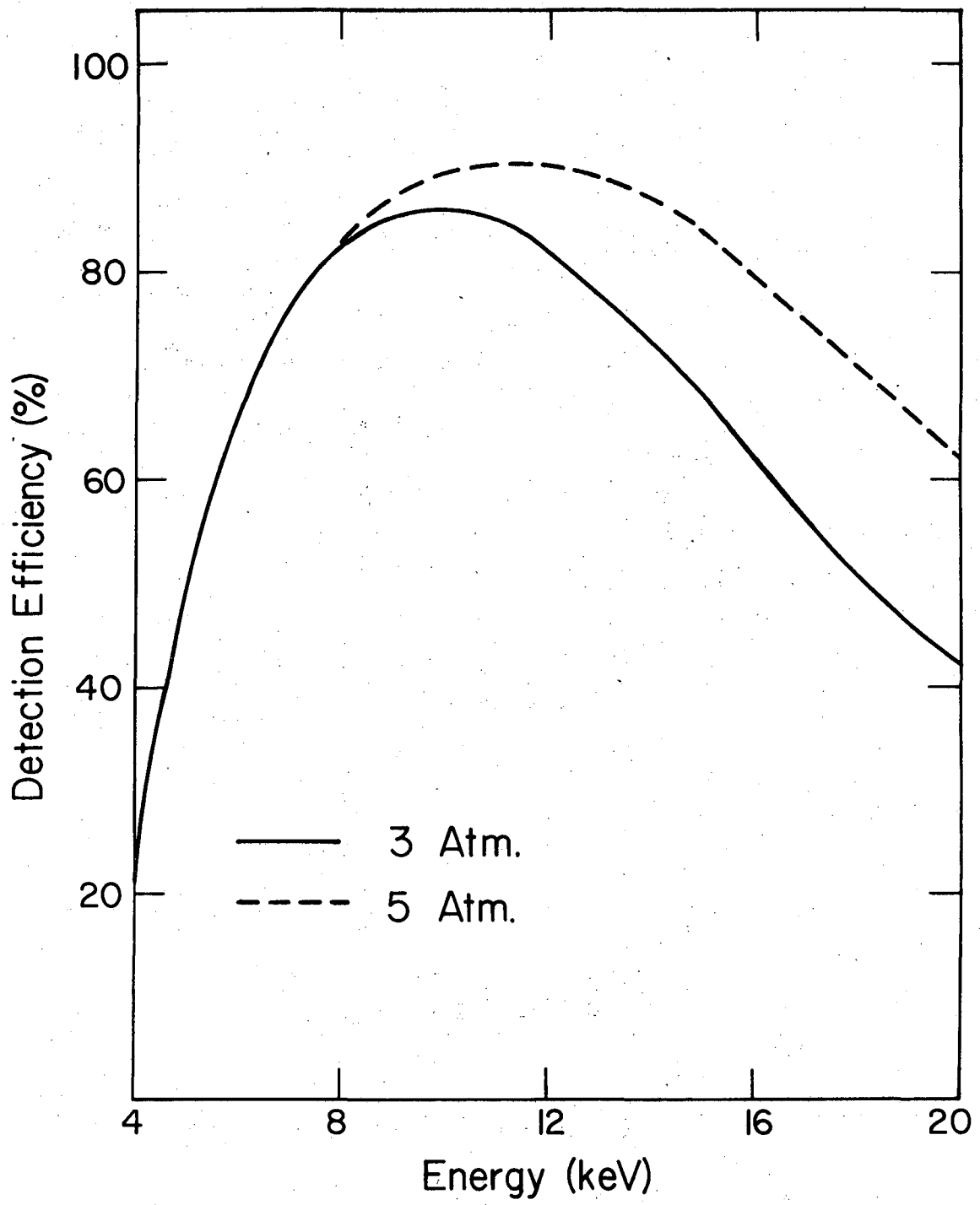


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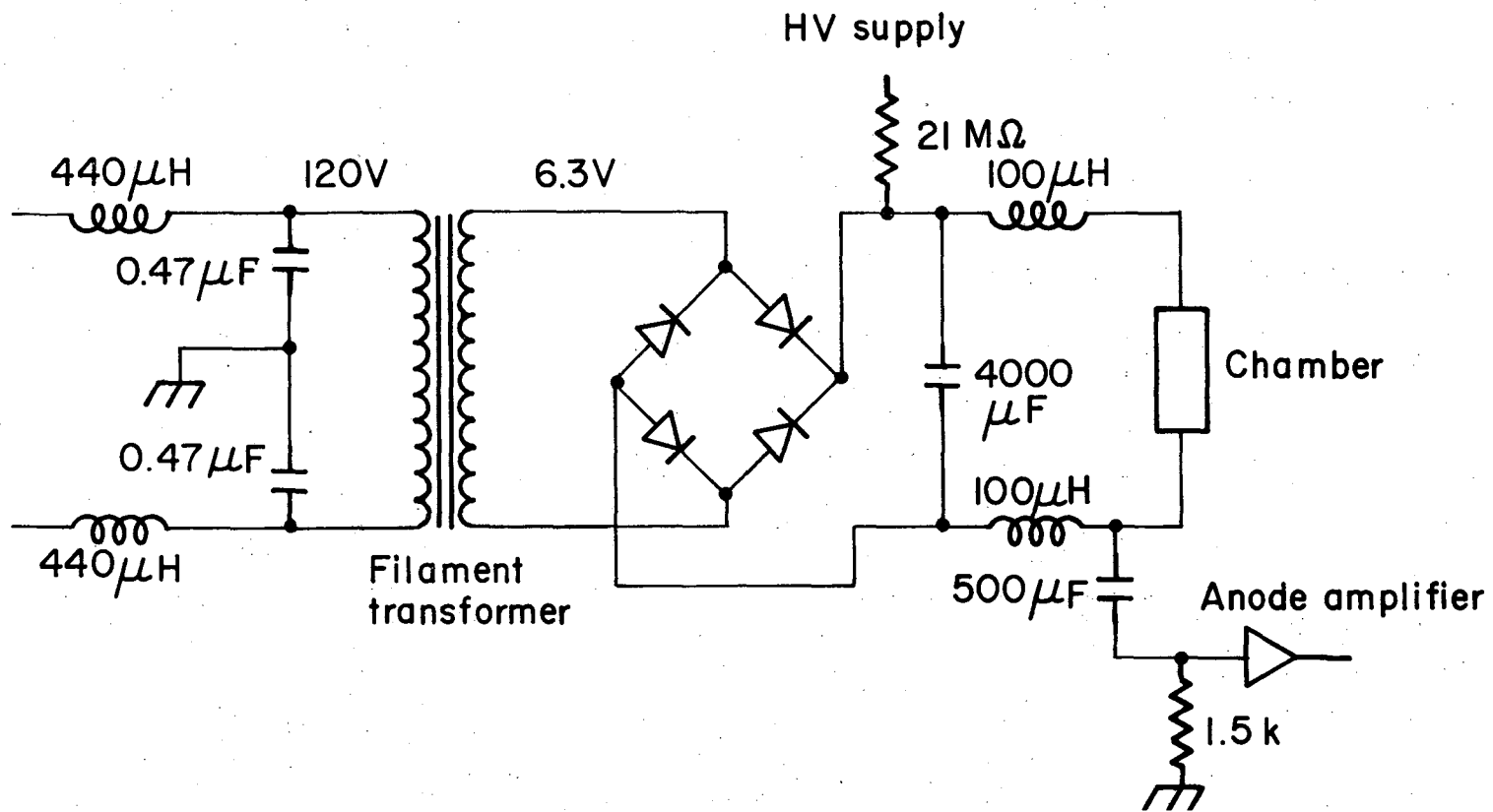


Figure 4.

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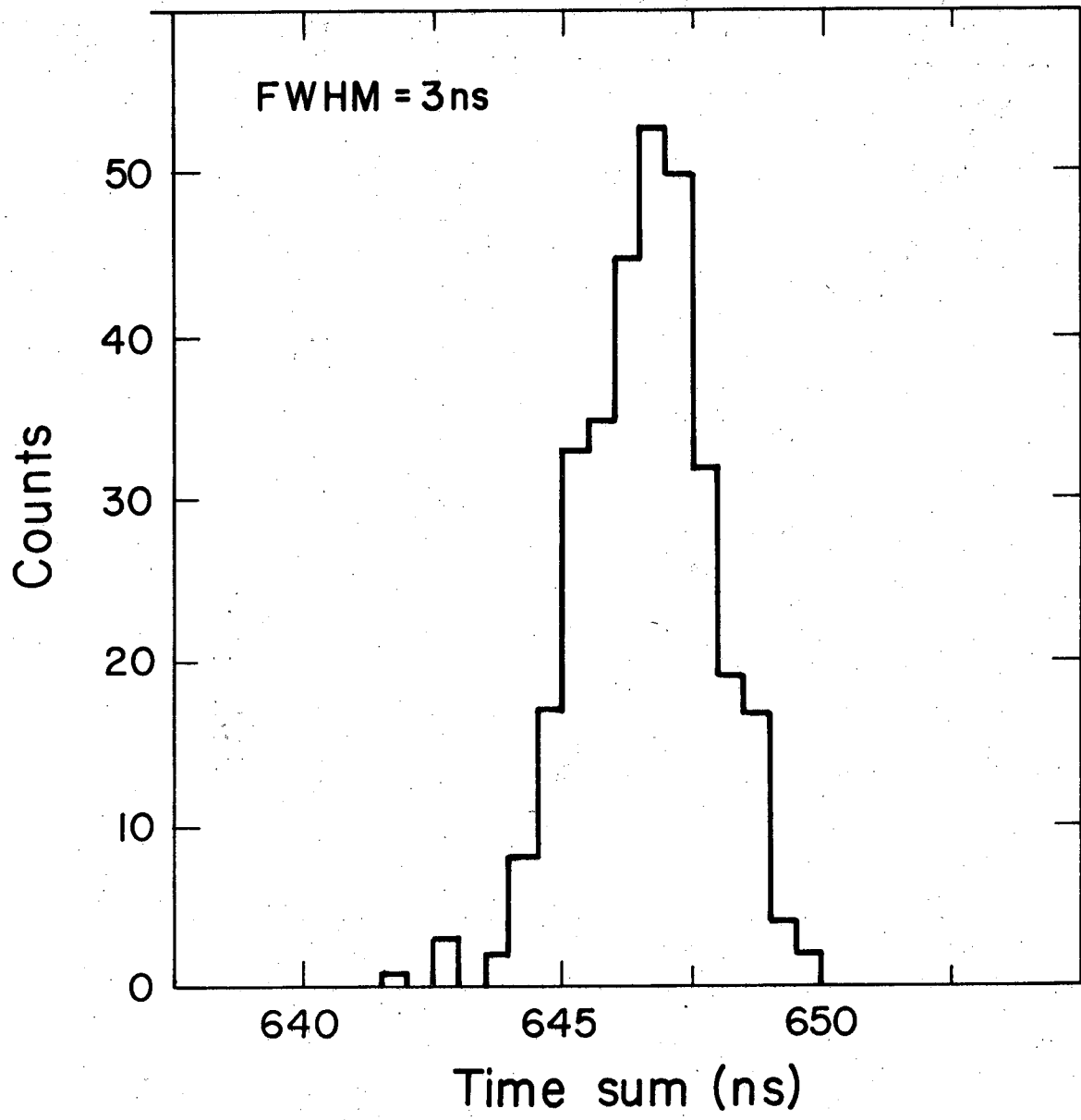


Figure 5.

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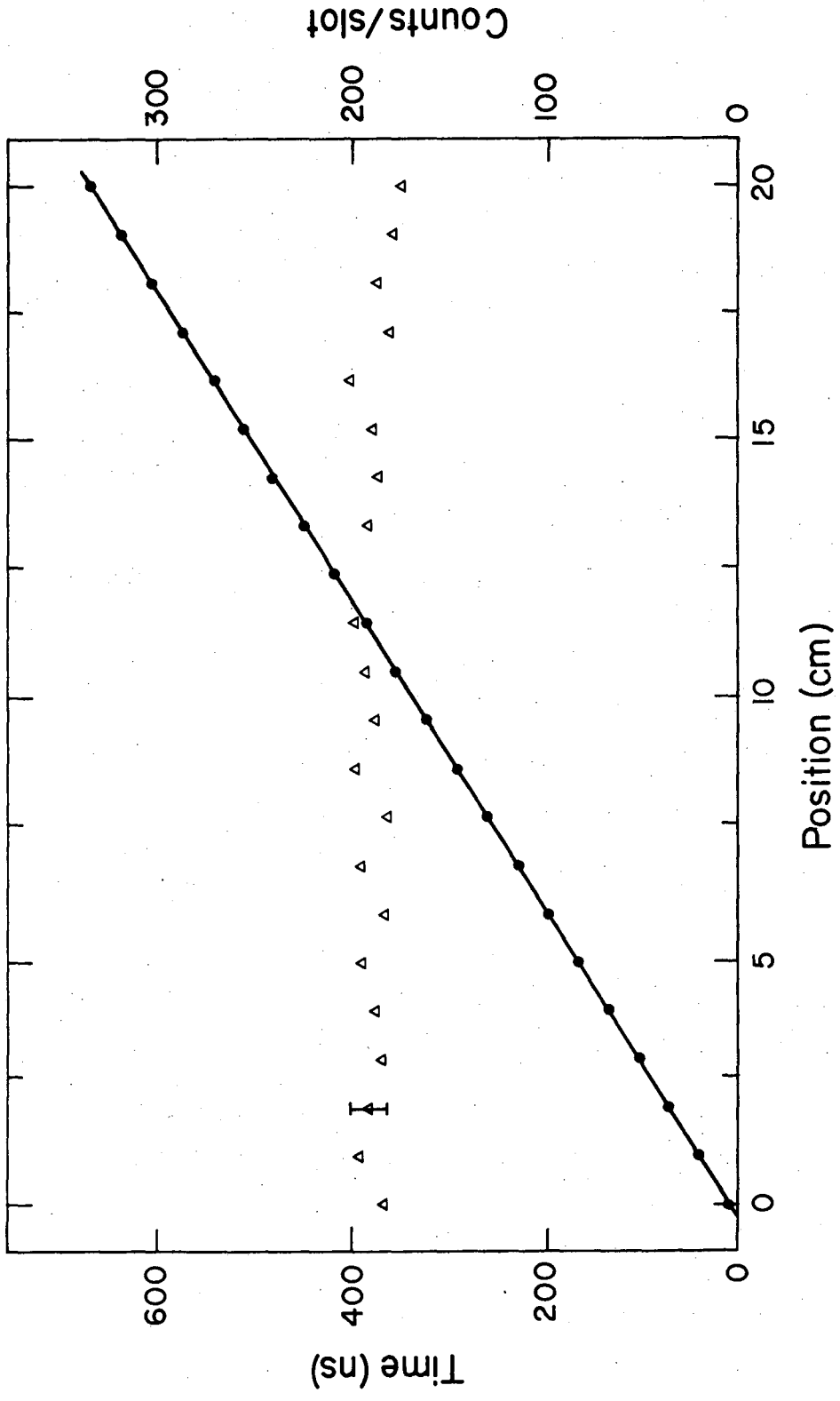


Figure 6.

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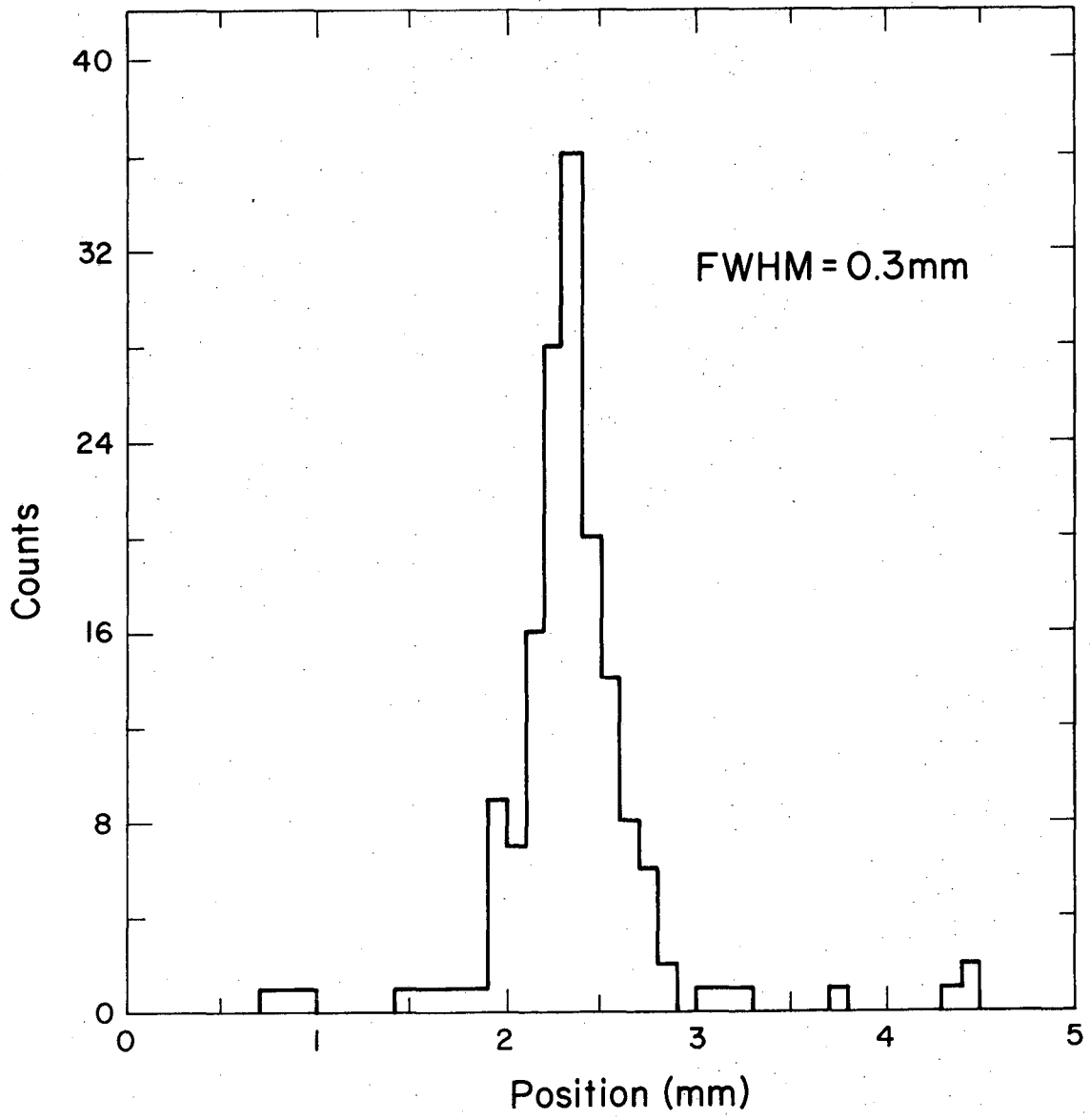


Figure 7.

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