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Final Report

Contract No. NAS8-27336

Remote Analysis of Planetary Soils:
X-Ray Diffractometer Development

by John C. Gregory

**School of
Graduate Studies
And Research**

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**The University
Of Alabama
In Huntsville**

Submitted to:

National Aeronautics and Space Administration
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X-RAY DIFFRACTOMETER DEVELOPMENT

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Huntsville, Alabama 35807

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ABSTRACT

A system is described suitable for remote low power mineralogical analysis of lunar, planetary, or asteroid soils. It includes an x-ray diffractometer, fluorescence spectrometer and sample preparation system. The development described in this report concerns the x-ray diffractometer which is the principal component of the system. This work has been published elsewhere (Gregory and Parnell, 1972). A one Curie ^{55}Fe source provides a monochromatic x-ray beam of 5.9 keV. Seeman-Bohlin or focusing geometry is employed in the camera, allowing peak detection to proceed simultaneously at all angles and obviating the need for moving parts. The detector system is an array of 500-600 proportional counters with a wire-spacing of 1 mm. An electronics unit comprising preamplifier, postamplifier, window discriminators and storage flipflops requiring only 3.5 milliwatts has been designed and tested. Total instrument power is less than 5 watts. Powder diffraction patterns using a flat breadboard multiwire counter have been recorded.

INTRODUCTION

Results of mineralogical analysis of samples of moon rock and soil returned during the Apollo program have greatly enriched knowledge of the geochemistry and geological history of the moon. Some differences between moon and earth rocks of compositional variety have been reported by earth-based laboratories. Some lunar minerals contain relatively large amounts of titanium for example. Apart from American and Russian lunar soft landing missions, experimental data on the composition of the solar system was derived from three principal sources: analysis of the earth's crust, the study of meteorites, and spectral analysis of solar radiation.

While the amount of information obtained from an earth-based laboratory examination is much greater than that from remote controlled unmanned instruments, the added cost of systems capable of returning a sample from Mars or the asteroids is tremendous, and it is expected that the next few decades will see a number of unmanned scientific probes making soft landings on various bodies in the solar system.

It is generally agreed that x-ray diffraction is the single most powerful tool for determining the composition of mineral mixtures in rocks and soils. Each mineral component in a rock produces a characteristic diffraction pattern which is not altered by the presence of other minerals, and computer programs are commonly used to unscramble superimposed patterns from crystal mixtures. Relative abundances of minerals in a rock may be obtained from comparison of certain peak intensities of the different minerals present.

Accordingly, in the conceptual design of a lunar and planetary soil analyzer,

(Gregory, Guenther and Parnell, 1969), an x-ray diffractometer was the principal method employed in the instrument, which also used high resolution x-ray fluorescence spectroscopy for rapid elemental analysis. The soil analyzer was designed for the un-manned lunar roving vehicle (since cancelled) which was to make a 1000 km traverse across the surface of the moon. Information to be obtained was:

- Rapid in-situ determination of chemical composition over the unprepared lunar surface or rock sample for elements of atomic number 9 to 92.
- Quantitative analysis of the chemical composition by both empirical comparison with rock standards and analytical methods.
- Identification of the minerals forming the rocks and estimation of their relative abundances.
- Statistical plotting of the mineral groups encountered to aid in the preparation of a precise geological map of the moon's surface along the traverse of the lunar rover.

The traverse of a rover would have permitted the collection of some hundreds of soil and rock samples. An on-board x-ray spectrometer-diffractometer with telemetry to earth permitted selection of materials of more interest or more varying nature for eventual return to earth, while still retaining the information about the distribution of a possibly more common regolith composition. In addition, if the vehicle were lost or otherwise unrecoverable, important information will still have been obtained.

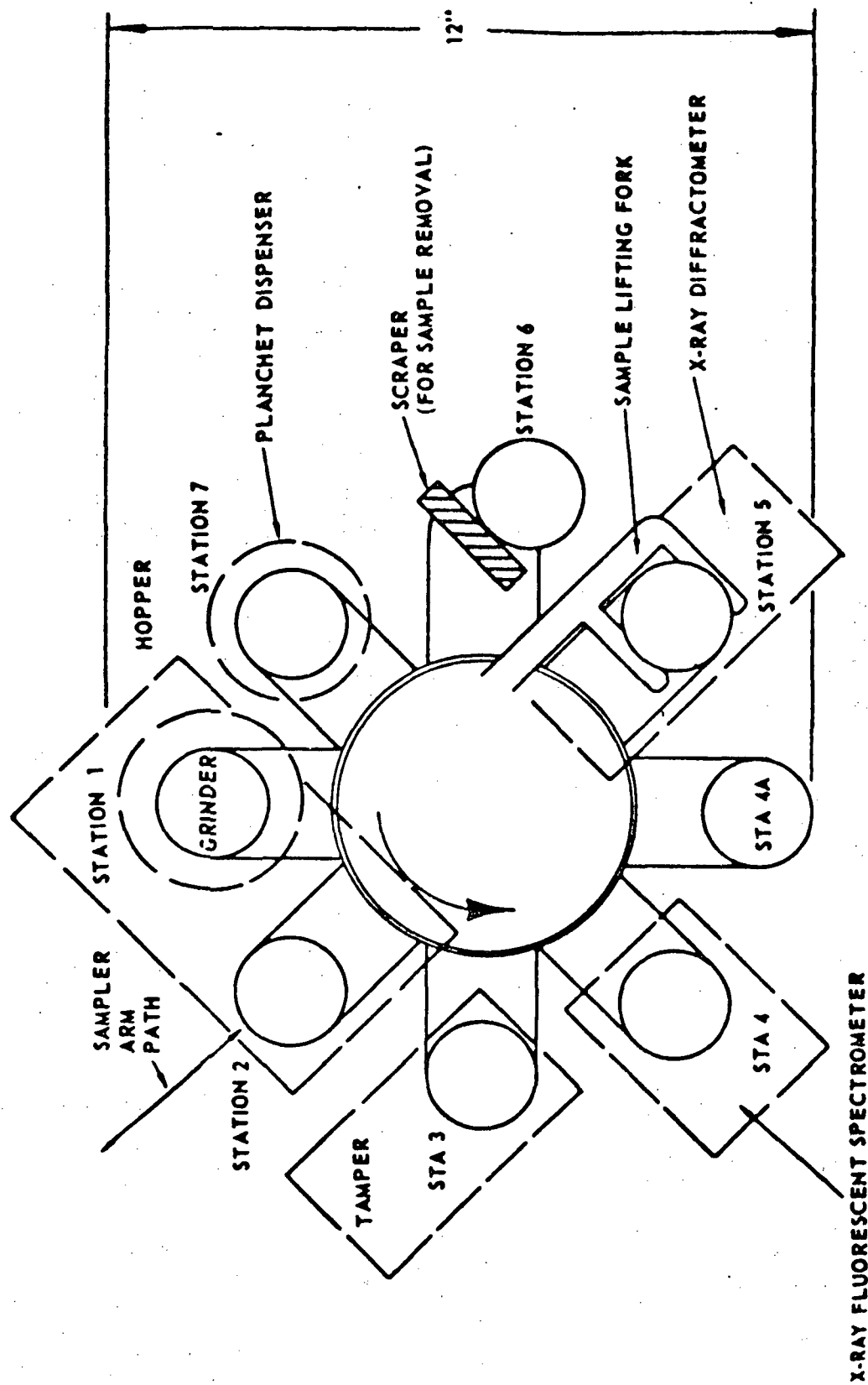
The instrument is equally suited for a soft landing on Mars, one of the asteroids, or one of Saturn's or Jupiter's moons, with or without a mobile platform. The advantages

ages of a roving vehicle are great, however, though it is not of course practicable in the case of asteroids. With a diffractometer and spectrometer so mounted, an investigator may perform a mineralogical survey of large portions of the body's surface giving insight into the fundamental questions of its genesis and subsequent formative and modificatory processes.

Sample acquisition systems have been developed by a number of workers and some are summarized in Philco-Ford Report VG-4410. Details of such systems are not relevant here since they will depend on the type of mission. The integrated sample delivery system and analysis devices are shown in Figure 1 (Gregory, Guenther and Parnell, 1969). The positions or stages through which a sample passes are shown in more detail in Figures 2 and 3.

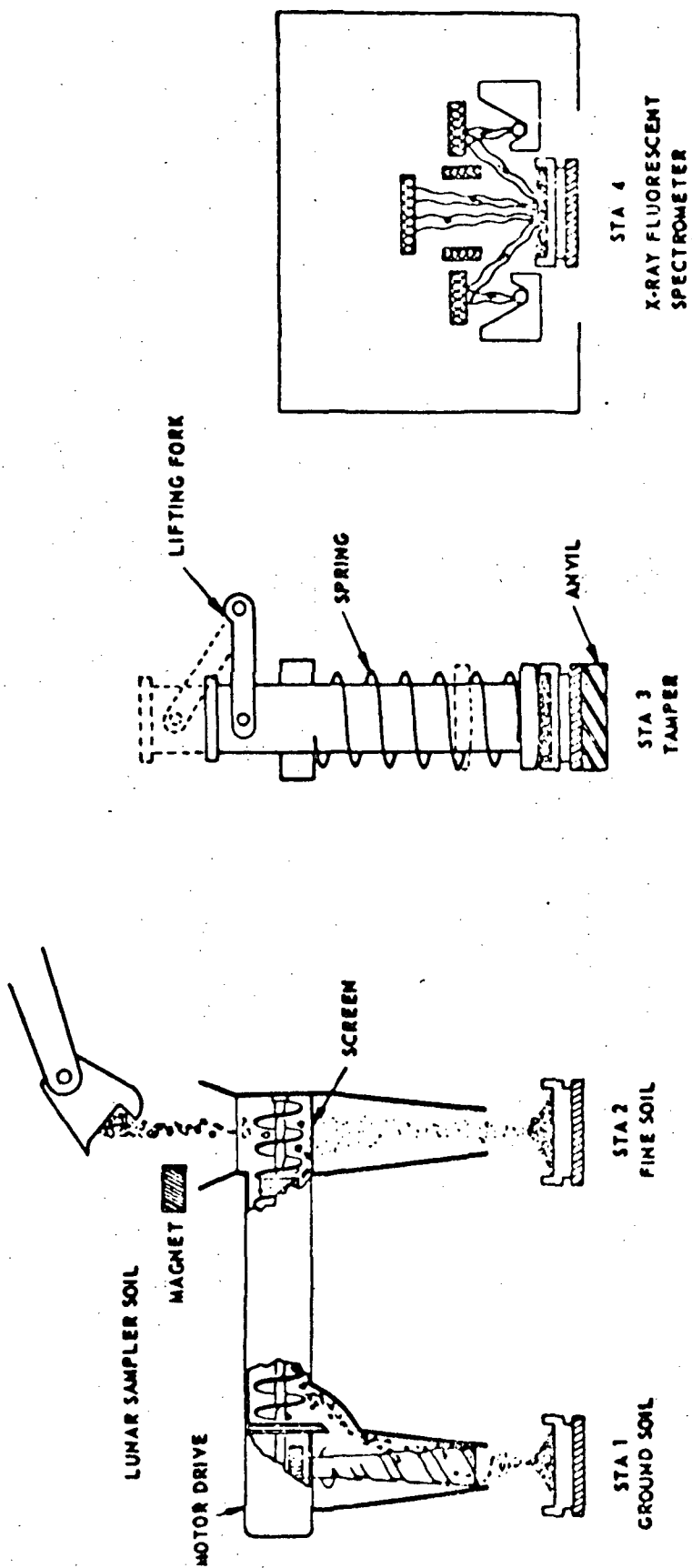
The sampling system proposed will accept a sample from the scoop and separate it using a mechanically activated sieve into two fractions with a particle size limit of 140 microns for the fines. The larger pieces will be ground in a rotary crusher to the same maximum size and deposited in a separate planchet. After tamping and shaping the samples will pass in succession to the fluorescence spectrometer. Identical or similar fluorescence results for both will indicate that the time-consuming diffractometry need not be performed on both samples when there are other power demands.

Rotation of the circular sample array will bring the first sample under the diffractometer. On command the sample will be lifted clear of the rotary sample delivery system and retained in the focusing circle of the diffractometer. All planchets will be pushed off their holders after the diffractometer position by the rotation of the holder arms under an obstruction. A new planchet will be placed into position on each



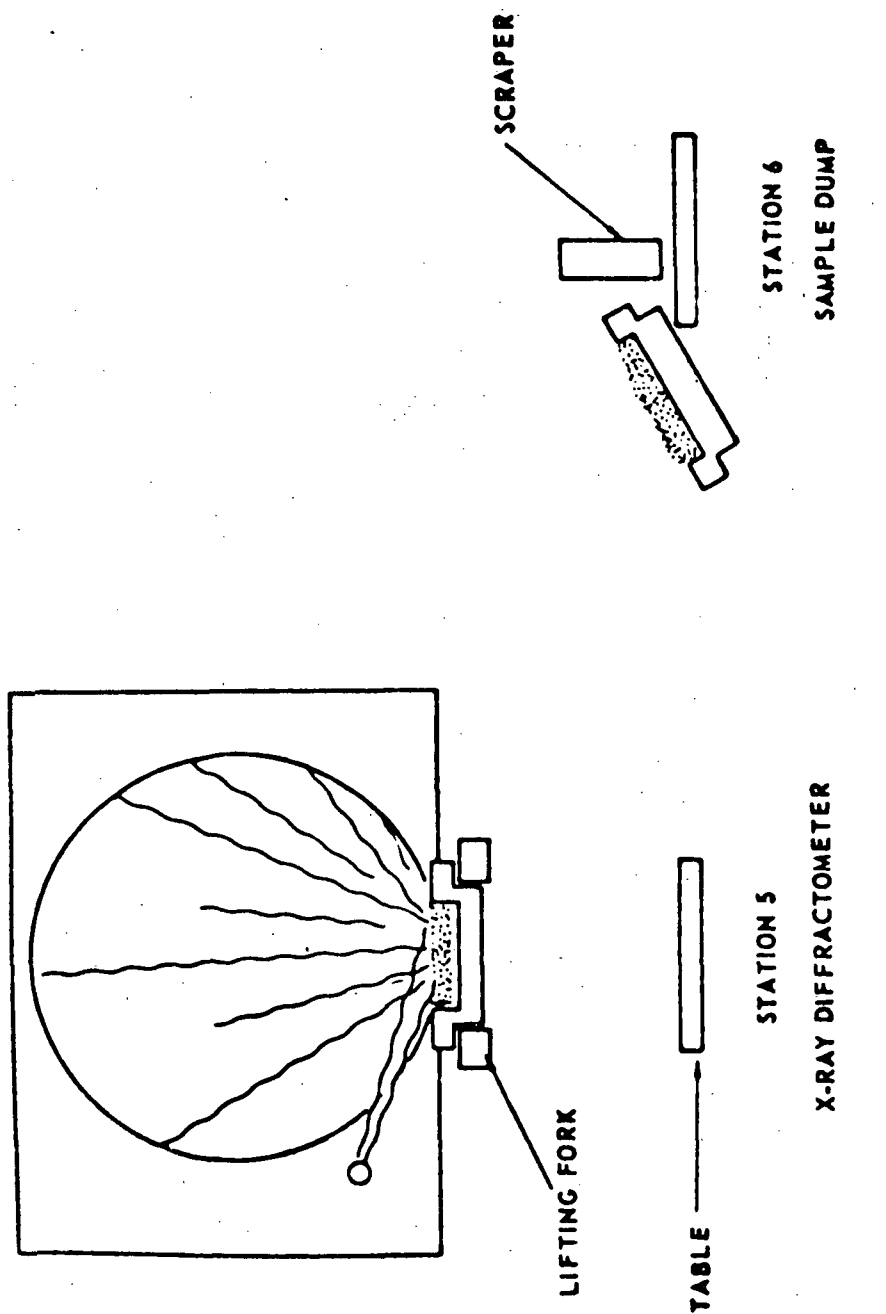
PLAN VIEW OF SAMPLE PREPARATION & TEST ARRAY

FIGURE 1



SAMPLE PREPARATION & TEST

FIGURE 2



SAMPLE PREPARATION 3 TEST

FIGURE 3

arm ready for filling at the hopper.

The work performed under this contract included the design and construction of a laboratory feasibility model of the low power x-ray diffractometer, the demonstration of its practical application to acquire x-ray diffraction patterns of minerals, and the preliminary design of a flight model to satisfy the usual constraints of space flight instrumentation while still providing good crystallographic data. The bulk of this report is concerned with diffraction camera research. The fluorescence spectrometer sub-system design has not changed substantially since it was described (Gregory, Guenther and Parnell) and it will not be further discussed here. Some improvement in energy resolution of the Si (Li) detector has been achieved in the past two years, however.

X-RAY DIFFRACTOMETER INSTRUMENT DESCRIPTION

The diffractometer described here is designed to fit the following constraints of space application among others:

- minimum power requirements (less than 5 watts)
- minimum weight
- maximum instrument life (includes consideration of high voltage problems, number of moving parts, source reliability, detector reliability)

The diffractometer must also conform to the following requirements:

- high sensitivity
- good angular resolution
- short analysis time

(1) X-ray Source

The chief difficulty with techniques employed in commercially available x-ray units in connection with space applications is their large power consumption. Most x-ray diffraction systems may be conveniently divided into four constituent parts: the source, the mechanical section (camera, goniometers, collimators, etc.), the detector, and the electronics. The power requirement of the x-ray tube normally exceeds that of the other three component systems by at least an order of magnitude. In addition, x-ray tubes are subject to degradation of performance which makes standardization of quantitative assays more difficult. The operating voltage of 25 kV could also pose problems for space applications. The radioactive source proposed for the diffractometer requires no power or high voltage and is, of course, 100 percent reliable. It is essential

therefore that an x-ray diffraction system required to operate on less than 5 watts should employ a radioactive source of x-rays. Such sources are technically feasible, and have been used (Preuss, Toothacker and Bugenis, 1966 and 1967) to demonstrate diffraction from crystals. Considerations of source availability, monochromaticity, wave length and counter efficiency indicate that an ^{55}Fe source is most suitable. The ^{55}Fe source does not show a bremsstrahlung background, being superior in this respect to an x-ray tube.

The source will be a ^{55}Fe rectangle of total activity 1 Curie and specific activity approximately $700 \text{ Curies gm}^{-1}$, 1 centimeter in length and of width 1 millimeter, electroplated onto a platinum substrate. ^{55}Fe decays by electron capture emitting $\text{MnK}\alpha$ rays of energy 5.9 keV. Even such a strong source produces a photon flux much less than that of an x-ray tube; however, special geometry and multiple detectors can compensate for this.

(2) Mechanical Arrangement

The camera used in this design is based on the self-focusing principle of Seeman and Bohlin, in which the narrow line source, the powdered sample, and the detectors all lie in the same focal circle, as shown in Figure 4. The optical analogy of this arrangement is the Rowland circle. For similar camera dimensions the Seeman-Bohlin system gives considerably higher intensity and resolution than the Bragg system which is employed in conventional diffractometers. An added advantage is that the sample can be mounted rather simply, and curved solid samples can also be analyzed. As the total integrated x-ray flux from a 1 Curie ^{55}Fe source is $10^{10} \text{ photons sec}^{-1}$ as compared with 10^{12} to $10^{15} \text{ photons sec}^{-1}$ for the x-ray tube, we use simultaneous counting by

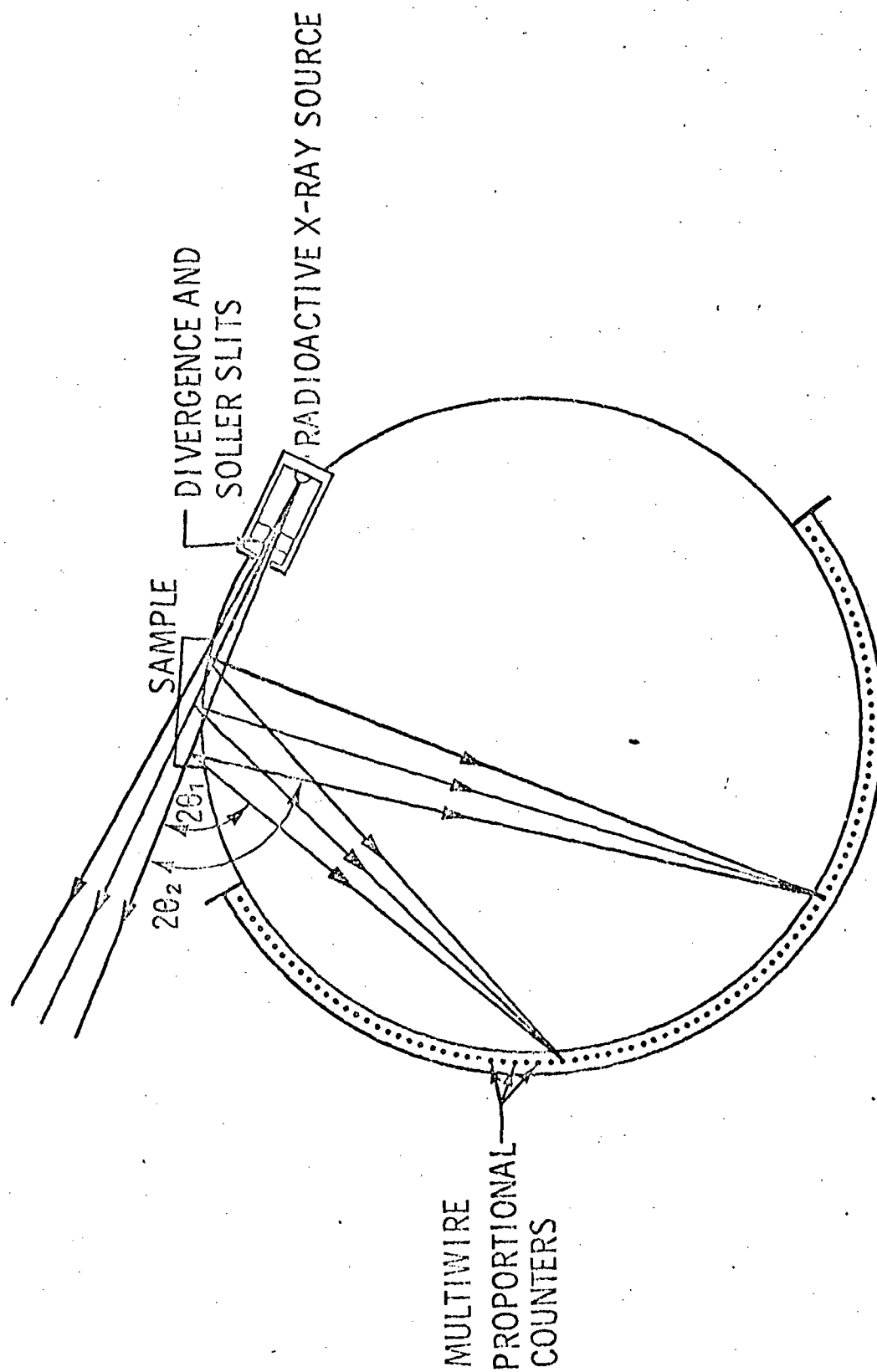


FIGURE 4: X-RAY POWER DIFFRACTOMETER USING SEEMAN-BOHLIN GEOMETRY

multiple array of x-ray detectors. The normal time for recording a powder diffraction pattern with a movable counter is of the order of 10^3 to 10^4 seconds.

Calculations of reflection efficiency of x-rays in an intense Bragg reflection from a crystalline powder (e.g., 200 plane of LiF) yield a count rate of nearly 100 counts per second for a 1 Curie ^{55}Fe source. This result has been experimentally verified at lower activities by Preuss and Toothacker (1969). It is calculated that minerals present in quantities greater than 10 percent of a powder mixture could be identified by the diffractometer described after a counting time of one to two hours.

Some factors affecting line width in powder patterns are:

1. Inhomogeneous strains
2. Composition variations
3. Mean dimension of crystallites in the direction normal to the reflecting plane (particle-size broadening)
4. Thickness of diffracting layer of sample
5. Range of wavelength in incident radiation
6. Width and height of slit defining incident beam.
7. Width and height of exit beam

Factors 1 to 4 are sample parameters: the first two are beyond control and the third is affected by the sampling techniques used such as drilling, grinding, and sieving. Some particle size distribution curves for returned lunar soils are shown in Figure 5. These factors will not be discussed here, but it is perhaps unlikely that the sample handling and conditioning equipment (drill, grinder, sieve, former) included in an automated Mars or asteroid surface landing probe will be sophisticated enough to allow overall

EARTH RETURN LUNAR SOIL SAMPLES GRAIN SIZE DISTRIBUTION CURVES

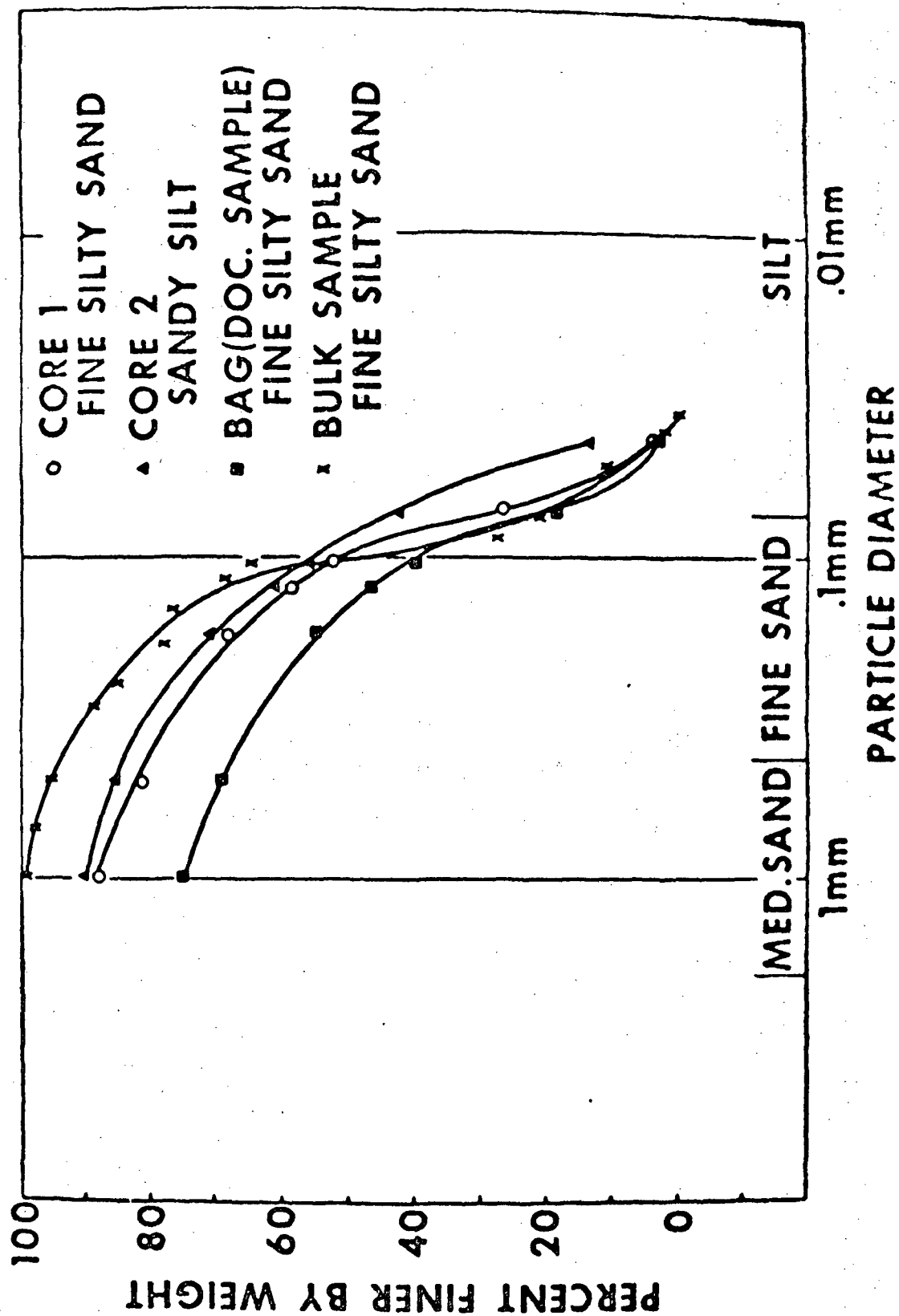


FIGURE 5

angular resolution to equal the best angular resolutions routinely obtained in the laboratory.

A disadvantage of the Seeman-Bohlin arrangement is that the sample to detector distance varies rather widely with θ . With the multiwire detectors, however, this intensity variation can be compensated for by increasing the active length of the detector at values of θ near 90° where the projection of the diffraction cones on the detector surface is nearly linear. For example the active length of a detector wire at low θ might be 0.5 cm and might increase to several cms at $\theta = 90^\circ$. This is easily achieved by a variable shaped aperture on the outside of the beryllium counter window.

The geometry of the camera will allow measurement of crystal lattice d-spacings up to about 5 Å for this wavelength of primary photon beam. Angular resolution of the camera (diameter 10 inches) is 0.25 degrees 2θ at optimum values of Bragg angle θ . This will allow deduction of the presence of most minerals constituting a significant proportion of a rock. Under conditions of some complexity ambiguities can arise in the interpretation of diffraction patterns. Two powder patterns obtained with different wavelength primary beams would provide a different set of d-spacings to help to eliminate ambiguities. At this stage it does not seem that another intense source of low-energy x-rays is available. X-ray fluorescence spectra from the same sample would, however, provide additional data which would extend the usefulness of the analysis.

In summary; the use of self-focusing geometry eliminates mechanical rotary drive of the sample and allows a large sample area with concomitant sensitivity, and the use of a multiple array of detectors eliminates rotation of the detector goniometer. The advantage of simultaneous counting by all detectors outweighs the disadvantage of the

lower photon flux of the radioactive sources vis-a-vis a conventional x-ray tube, and there are no instability, failure, or high voltage problems.

The flight version of the diffractometer for which engineering concepts are shown in Figures 6 and 7 is designed to be made almost entirely of non-volatile materials such as metal and ceramics within the detector region. Many of these materials problems are common to the large area proportional counter hodoscopes being designed at MSFC for the High Energy Astronomy Observatory. These counters have a required operating lifetime in orbit of one to two years.

(3) Detectors

In the flight counter design a gas-filled multiwire proportional counter of approximately cylindrical geometry forms the fixed detector array. Six hundred counter wires are arranged in an arc as shown in Figures 4 and 6. Proportional counters were selected instead of other particle detectors because of their particular advantages, which are:

1. Good energy resolution with charge pulse proportional to photon energy gives good background rejection from cosmic radiation, natural and artificial radio isotopes in the vehicle, and specimen fluorescence.
2. High efficiency for low energy x-rays.
3. Low noise.

The use of six hundred proportional counter wires each with its own amplifier and single channel analyzer might be regarded as a somewhat unsophisticated approach compared with the resistive wire technique so elegantly employed by Borkowski and Kopp (1968, 1970). These workers used pyrolytic-carbon-coated quartz fibres of very

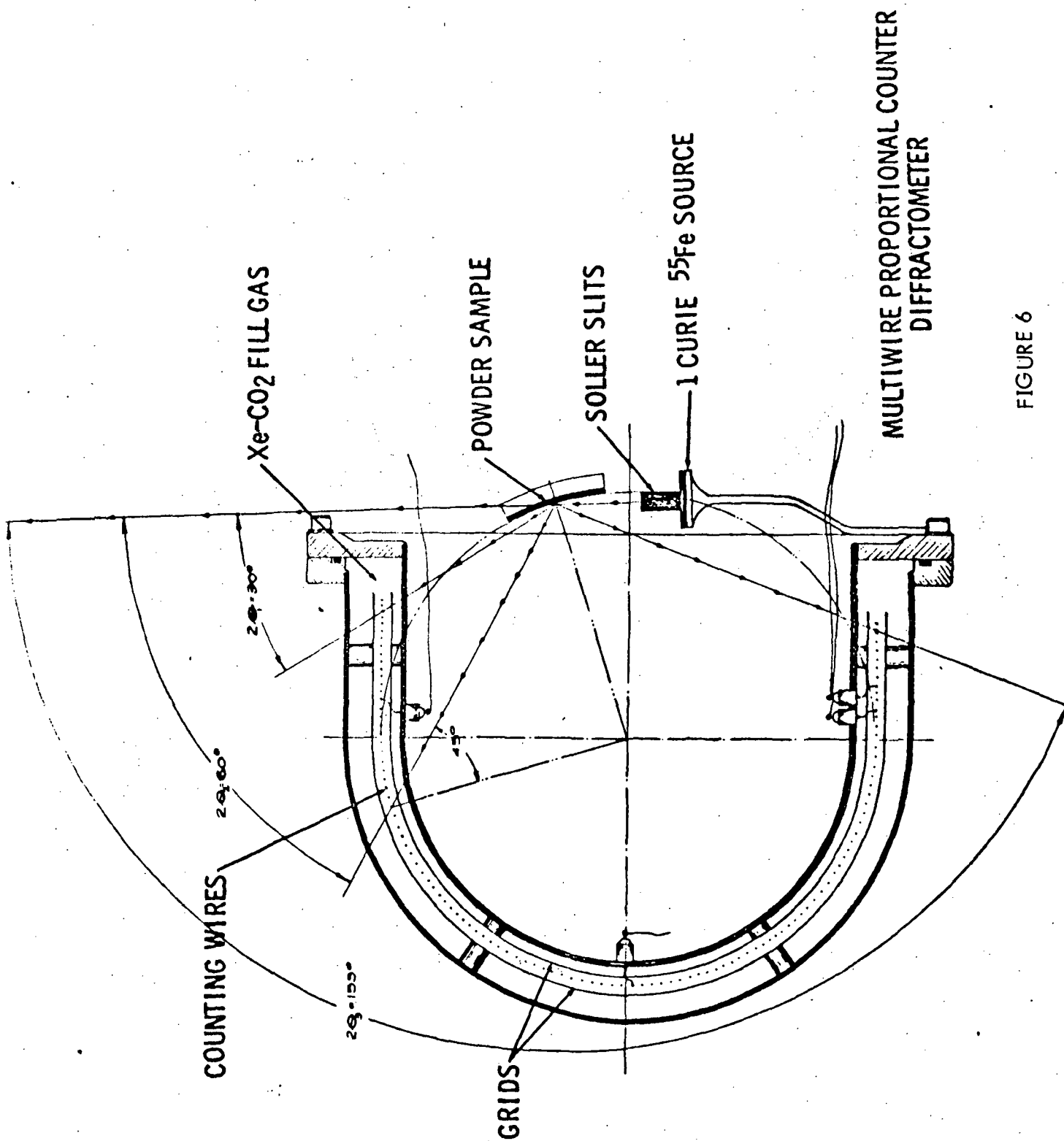


FIGURE 6

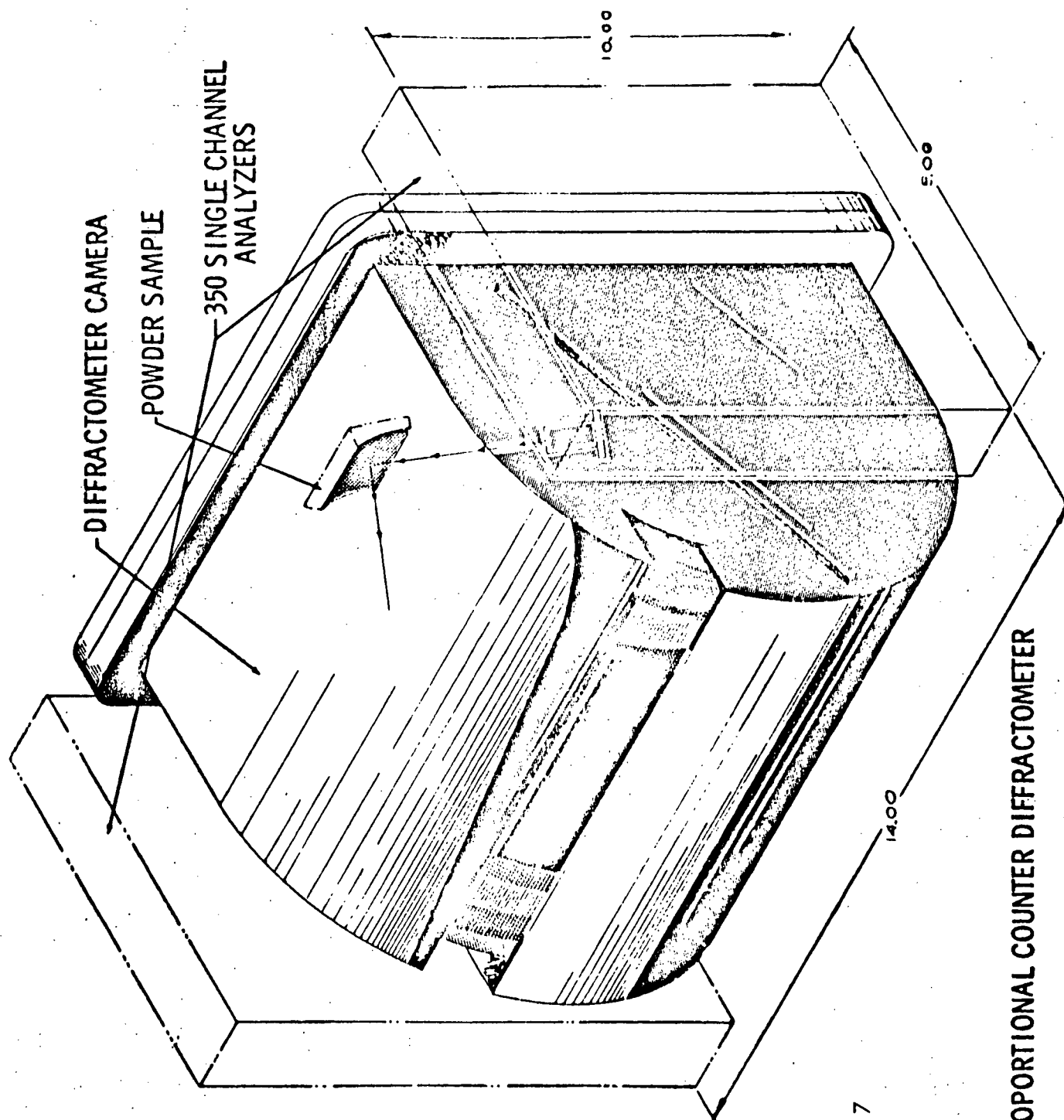


FIGURE 7

MULTIWIRE PROPORTIONAL COUNTER DIFFRACTOMETER

high uniformity, 25×10^{-6} m. (.001 inch) diameter, and approximately 10^4 Ohms mm^{-1} as anode wires in proportional chambers. They report position resolutions along the wire of 0.15 mm for 5.9 keV x-rays. They also report a diffraction pattern obtained with such a counter. The arrangement is, however, linear and thus rather large and restricted in angular range ($\theta = 6^\circ$ to 35°). The difficulty with a single curved resistive wire is the incompatibility of the requirements for mechanical rigidity with those of good electrical properties. Wire supports grossly perturb the electric field.

Multiwire proportional counters have received a considerable amount of attention in the last few years by a number of research groups in nuclear and cosmic ray physics in this country, the U. K. and at CERN. At wire spacings below a few millimeters the electrical and mechanical constraints on construction and properties of the counters require special consideration. The construction and properties of multiwire proportional counters have been described in several publications, notably those of Charpak and coworkers at CERN, (1968, 1970).

In an application such as this the counter parameters of major importance to the performance of the diffractometer are wire-spacing, gap-width or detector thickness, and the length of the counter being actively used for detecting x-rays. In this design the active length of the proportional counter wires is varied as a function of θ to allow for greater line curvature at low θ , and lesser intensity at high θ . At $\theta = 40^\circ - 50^\circ$ the active length is 4 cm. This decreases to 0.5 cm at low θ . Resolution is poor below $\theta = 20^\circ$ ($1^\circ \approx 2 \theta$ approximately) and no useful information may be obtained below $\theta = 15^\circ$. The angular range of the system is $\theta = 15^\circ - 80^\circ$ which corresponds to a d-spacing range of $4.5 - 1.1 \text{ \AA}$.

At θ values around 50° diffracted photons strike the detector 'plane' almost normally. At lower θ however, diffracted photons impinge at increasing angles to normal. At $\theta = 30^\circ$ for example, a photon passes through the counter at 45° to the normal to the wire plane at that point. For a proportional counter gap width of 3mm the photon count may appear on any of ten wires centered at that angle, though not with a symmetrical distribution. This means that the angular resolution is degraded to a proportional amount since each wire spacing round the focusing circle subtends the same angle at the sample at all values of θ for the camera geometry described. Also efficiency will be lost due to photon absorption in the longer path through the beryllium window and cathode and the dead space in between.

Difficulties caused at low θ values by photons passing into the detectors at angles far from the normal can be relieved somewhat by modifying the Seeman-Bohlin geometry. The detector array is displaced out of the focusing circle at low angle θ as shown in Figure 6. Though this reduces the number of wires over which a particular Bragg reflection is counted, it also removes the detectors from the focal point of that reflection. For a very thin detector the resolution would thus be degraded by an amount proportional to the displacement out of the focusing circle and proportional to the divergence angle of the reflected beam. This angle is the same as the divergence angle of the rays from the source itself. The spread due to the beam divergence is much less than that caused by the beam striking the detectors at an oblique angle, and distortion out of the focusing circle improves performance as well as facilitating fabrication. For a given sample length (2 to 3 cms along the circumference) we cannot reduce the divergence angle by moving the source nearer to

the sample. This movement does however produce another beneficial effect.

A smaller angle between the x-ray beam and the tangent to the sample surface permits reflections at smaller Bragg angles to be recorded. The disadvantage to moving the source nearer is that sample alignment and surface condition become much more critical, even though the sample subtends the same angle at the source no matter where the two are placed round the focusing circle. Low angle scattering also increases. Reduction in the circumferential length of the sample while reducing the x-ray beam divergence angle also reduces the sensitivity. A compromise is struck between these conflicting factors with a source to sample angle of about 30° . At this angle the beam strikes the center of the sample at 15° to the tangent at that point.

At beam incidences other than normal to the detector the angular resolution is clearly a function of the thickness of the detector as well as of the angle of incidence. The thinner the counter, the better the angular resolution will be. Counter efficiency must also be considered at this point, as well as the effect on the electrical properties of the counter of reducing the gap-width. Xenon is chosen as the counter gas because of its density and consequent efficiency. At 1 atmosphere of 93% Xe - 7% CO_2 , and a gap-width of 3 mm. (counter thickness 6 mm.), the efficiency is calculated to be 45%.

(4) Electronics

A block diagram of the portable diffractometer read-out scheme is shown in Figure 8. Each of the 600 wires is connected by a short lead to a counter and storage unit (CSU) consisting of a charge-sensitive preamplifier, a postamplifier, a single channel analyzer

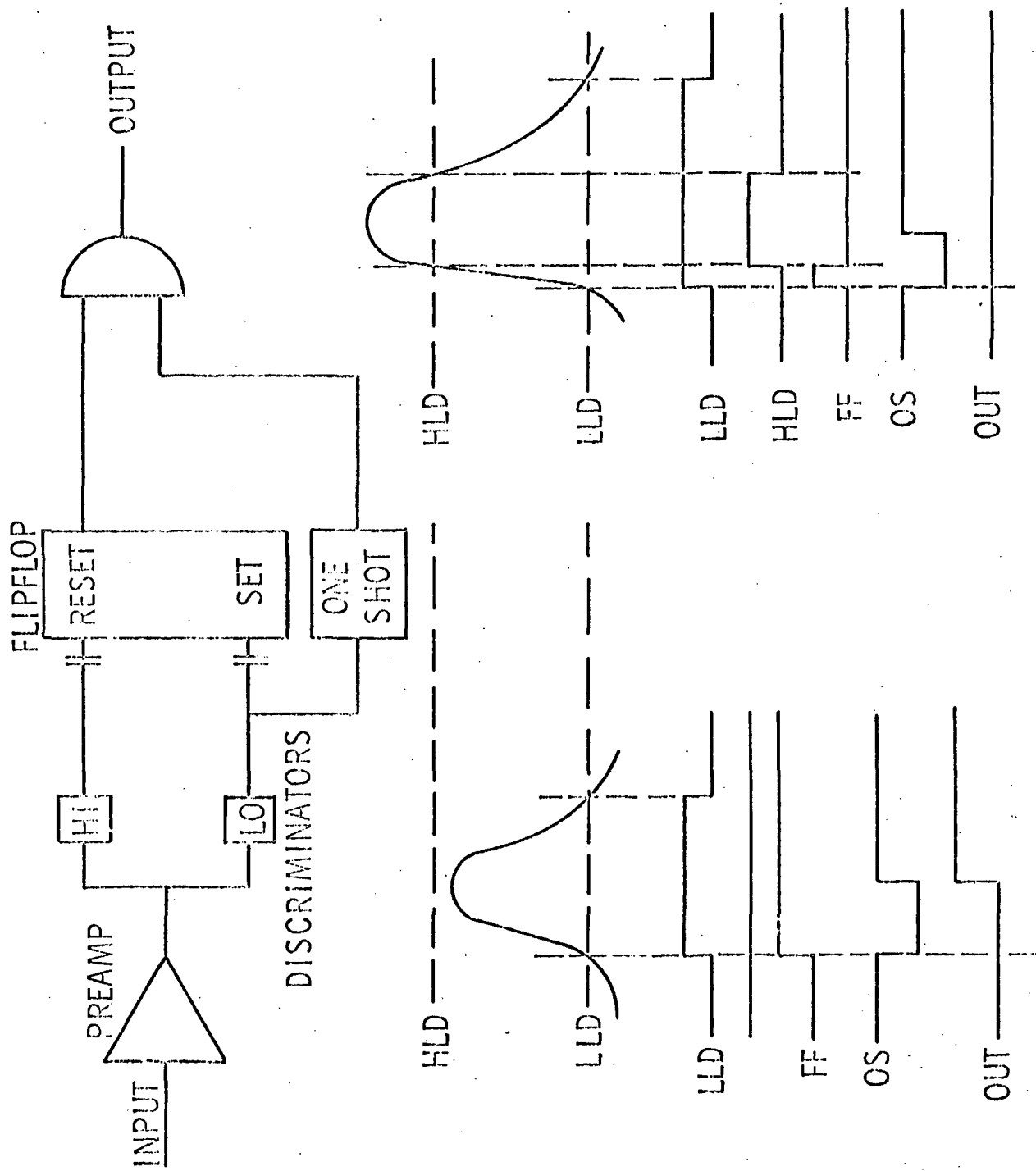
with a window from 2 to 7 keV and two storage flipflops. The scanner inspects this storage for counts, taking the CSUs sequentially in groups of four. The scan rate will be ample to prevent overflow of the CSUs for the anticipated count rate.

Readout proceeds continuously into a memory from where it may be telemetered on command. Counting occurs simultaneously on all other groups than that undergoing inspection at any moment. If, in any group, counts are registered, the clock is stopped and the counts transferred to the memory with the appropriate address. After completion of this, the data buffer is cleared and the clock allowed to continue. Upon reaching 151 the counter resets to group 1 and continues.

The use of proportional counters as detectors requires individual preamplifier/single channel analyzers for each of the 600 wires. We desire to operate the counters at a conservative gas-gain of about 10^3 for stability and long life. This produces a charge pulse of 3×10^{-14} coulombs for ^{55}Fe x-rays.

A charge sensitive preamplifier was chosen for low noise and reproducibility of gain. This is followed by the single channel analyzer (SCA) shown in Figure 9. Reproducibility of the discriminator settings is $\pm 10\%$. The discriminator levels may be trimmed over a range of 1/2 to 5 from the nominal values of 2 and 7 keV. The SCA is followed by a two bit scaler which is used to de-randomize partially the count rate for each wire, and as temporary storage for the scanning circuits. Counting rates of approximately 100 sec^{-1} in the peak channels allow an average scan rate as low as 100 kHz before significant data is lost.

The requirement for 600 preamplifiers and SCA units is at first sight a rather significant problem. The laboratory counter has been developed with standard PC



TIMING DIAGRAM FOR SINGLE CHANNEL ANALYSER

FIGURE 9

techniques using commercial discrete and IC units. This unit has a parts cost of approximately \$15 per wire and consumes 40 milliwatts of power per channel. Requirement for a similar unit has existed for a large area proportional counter hodoscope being studied for the HEAO spacecraft (Ormes, et al., 1970). A unit has been developed for that application containing a charge-sensitive pre-amplifier, dual discriminators, dual gates and dual flip flops. This unit,* which is shown in Figure 10, is packaged in hybrid thick film form, weighs 9 grams and consumes less than 4 milliwatts of power. The preamplifier meets the specifications for the diffractometer and the parts count is the equivalent of the pre-amplifier SCA scaler. Thus, these units for the 600 wires will require a total of less than 2.4 watts of power.

* Space & Tactical Systems Corporation, Bedford, Mass.

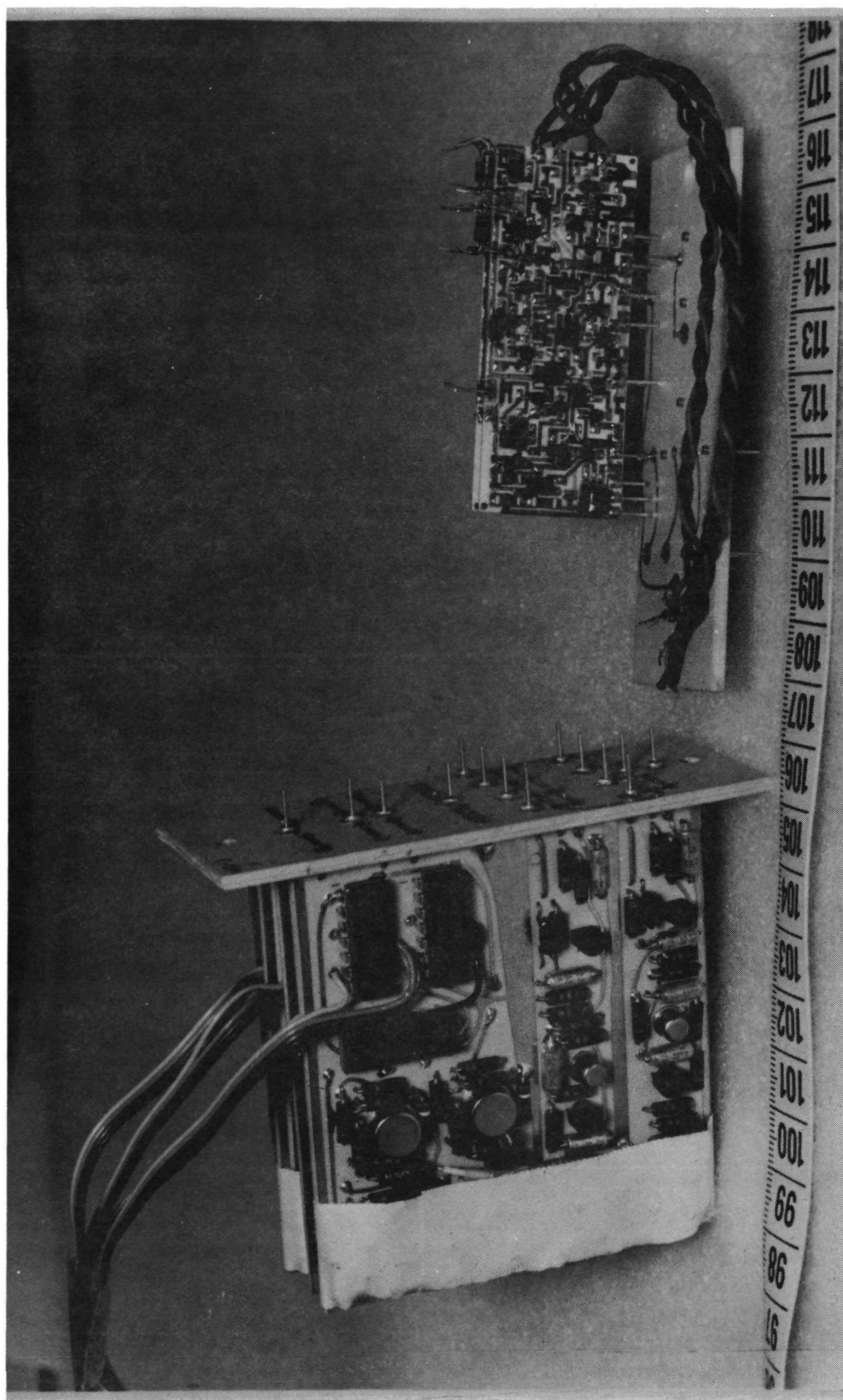


FIGURE 10

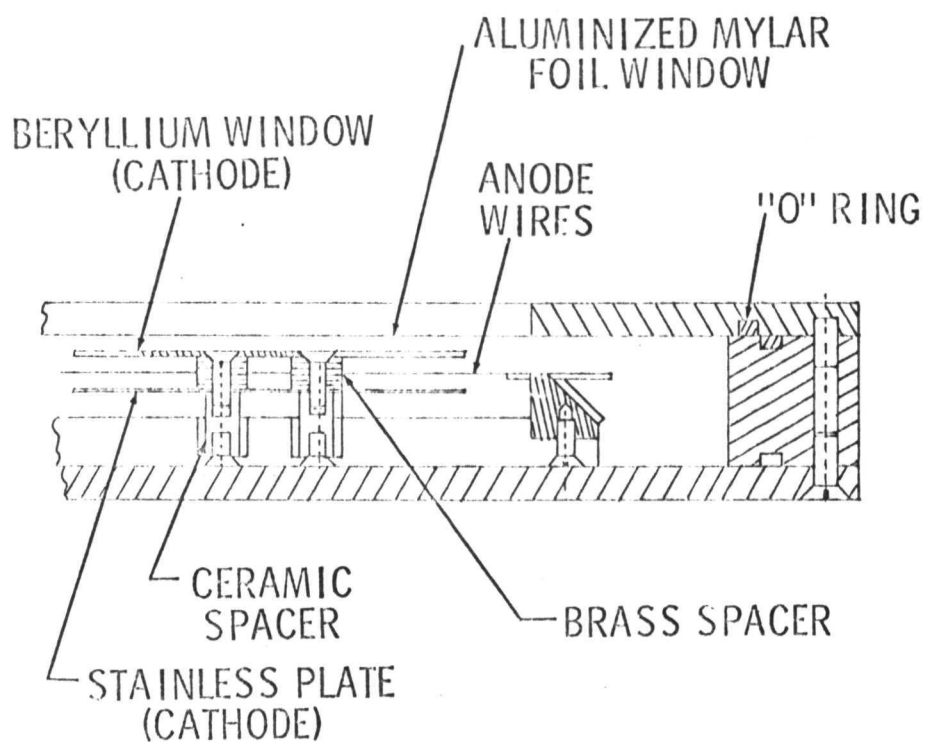


FIGURE 11 PLANAR 30-WIRE TEST COUNTER:
SECTION SHOWING CONSTRUCTION

EXPERIMENTAL RESULTS

The small flat proportional counter shown in Figure 11 was designed so that the wire spacing and gap-width could readily be altered. This system was used to study the multiwire proportional counter parameters to give a detection system of maximum position and energy resolution consistent with good electrical properties and high efficiency. It was operated with the anodes at ground potential and directly connected to the charges-sensitive preamplifiers. The lateral dimension of the negative high voltage planes parallel to the anode wires was smaller than the length of the anodes, and the cathodes did not touch the same insulator as that which supported the anodes. This arrangement conferred two advantages:

1. The high field region was confined to the relatively perfect center portion of the anode wires.
2. Any leakage or noise caused by the high voltage insulator was not seen by the preamplifier.

In the version used to obtain the diffraction patterns shown in this paper, the counter anodes were gold-plated tungsten 18×10^{-6} m. (.0007 inch) diameter, spaced 1×10^{-3} m. (.040 inch) apart, with gap-width or anode to cathode spacing of 3×10^{-3} m. Anode wires were pretensioned and soft soldered to copper strips. The wires passed through notches cut in Kel-F blocks at the ends of the array which served to position the wires parallel and coplanar to within 13×10^{-6} m. (.0005 inch). In a flight version these plastic blocks would be replaced by those of ceramic.

This counter had 30 such wires and operated well at voltages from -2300 to -2800 volts. Breakdown did not occur until much higher voltages. Various gases have been used in the counter and studies are currently being made to define counter lifetime as a function of gas and construction material characteristics.

Although we can readily produce multiwire counters with energy resolution approaching the theoretical value (approximately 16% FWHM at 5.9 keV) with wire-spacings and gap-widths of 5mm, the energy resolution is degraded in the 1 mm. wire-spacing counters. Using Ar-25% isobutane, a resolution of 22% FWHM is obtained at 1 mm. spacing as shown in Figure 12, with lower resolutions given by Ar-CO₂ mixtures. Energy resolution is not a critical factor in this application, and 30 - 40% FWHM would be adequate to perform background rejection.

Many preliminary measurements were also made with the counters shown in Figure 13. This had a wire-spacing and gap-width of 5 mm. and operated with the anodes at high voltage. This counter proved very useful as it was much more easily handled than the 30 wire, 1 mm spacing counter. Resolutions of 16% FWHM at 5.9 keV were commonly obtained.

The flat 30 wire counter was used to demonstrate the position sensitivity of the technique for low energy x-ray and to obtain diffraction patterns. The counter was mounted on the goniometer wheel of a standard Phillips x-ray diffractometer, after removal of the conventional proportional counter and exit slit collimators. This arrangement is shown in Figure 14 and 15. The plane of the wires was perpendicular to the diffracted beams, and the wire and the axis of rotation of the goniometer wheel were parallel. The radius of the arc

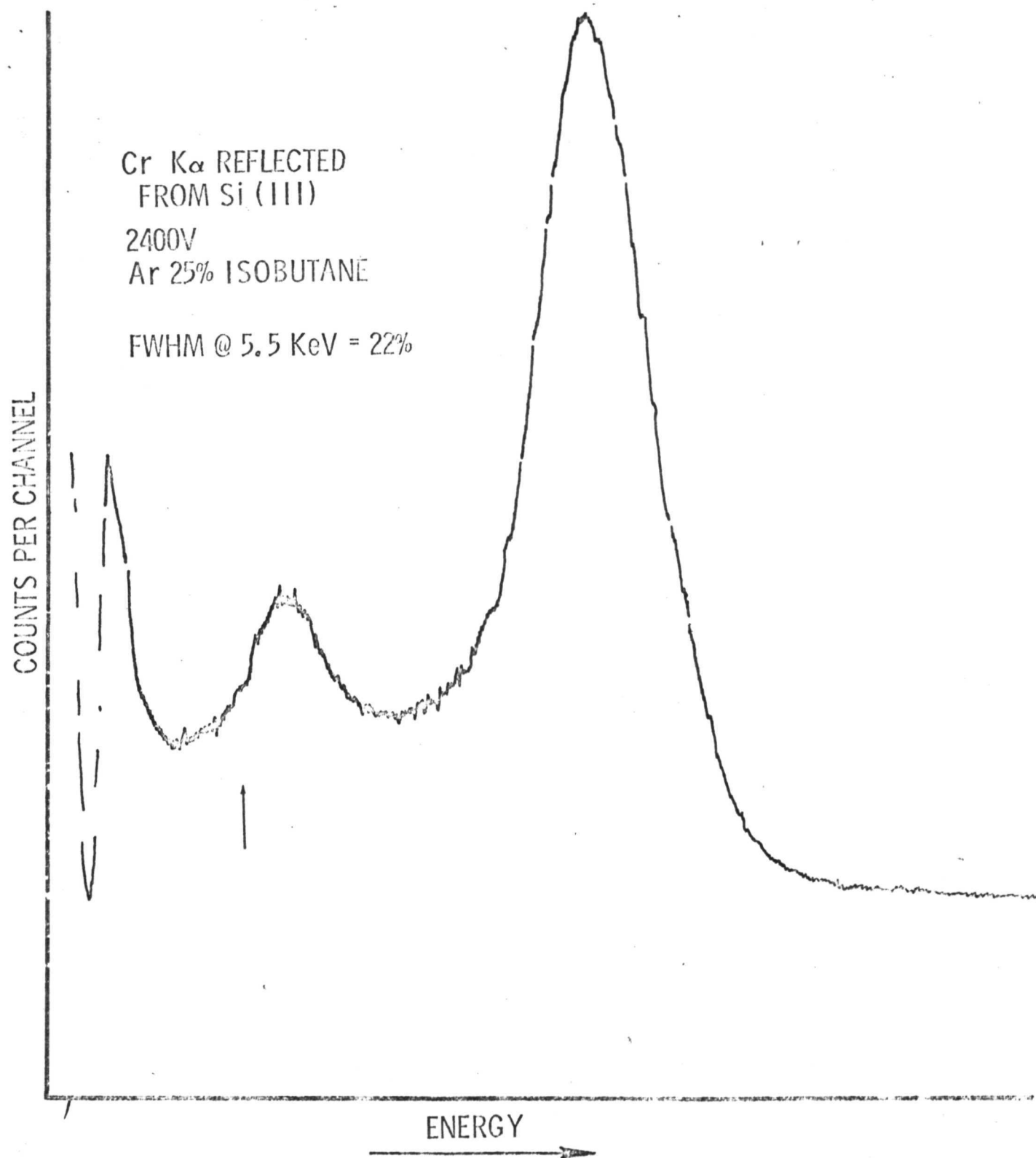
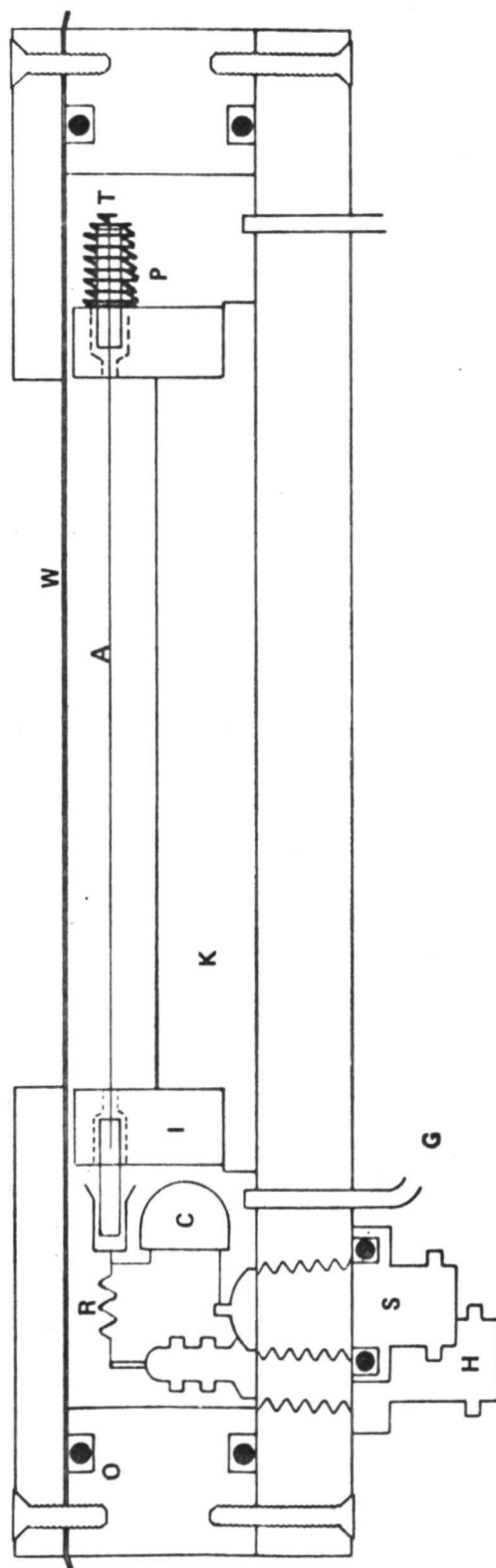


FIGURE 12 ENERGY SPECTRUM OF 5.5 keV PHOTONS IN THE 1mm. WIRE SPACING DIFFRACTOMETER TEST COUNTER.

CROSS SECTION OF SMALL MULTIWIRE COUNTER



LEGEND

- | | |
|----------------------------------|--|
| A - ANODE WIRES (2 MIL) | O - O RING SEAL |
| C - HV COUPLING CAPACITOR | P - ANODE POSITIONING PIN |
| G - GAS CONNECTIONS | R - LOAD RESISTOR |
| H - HIGH VOLTAGE CONNECTOR (MHV) | S - SIGNAL CONNECTOR (BNC) |
| I - INSULATOR BLOCK (KEL F) | T - ANODE TENSION SPRING |
| K - CATHODE BLOCK | W - WINDOW (ALUMINIZED MYLAR OR AL FOIL) |

FIGURE 13

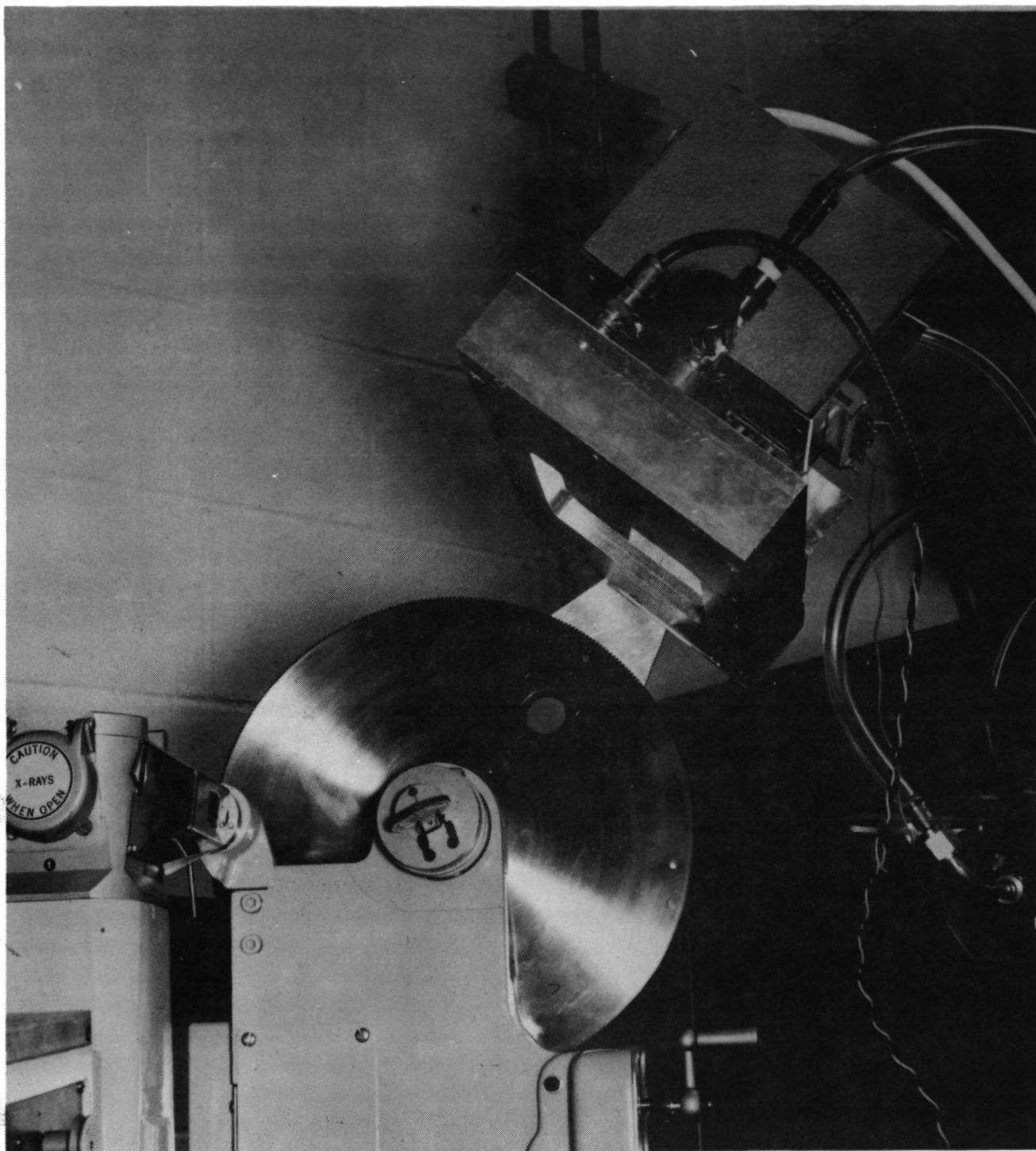


FIGURE 14

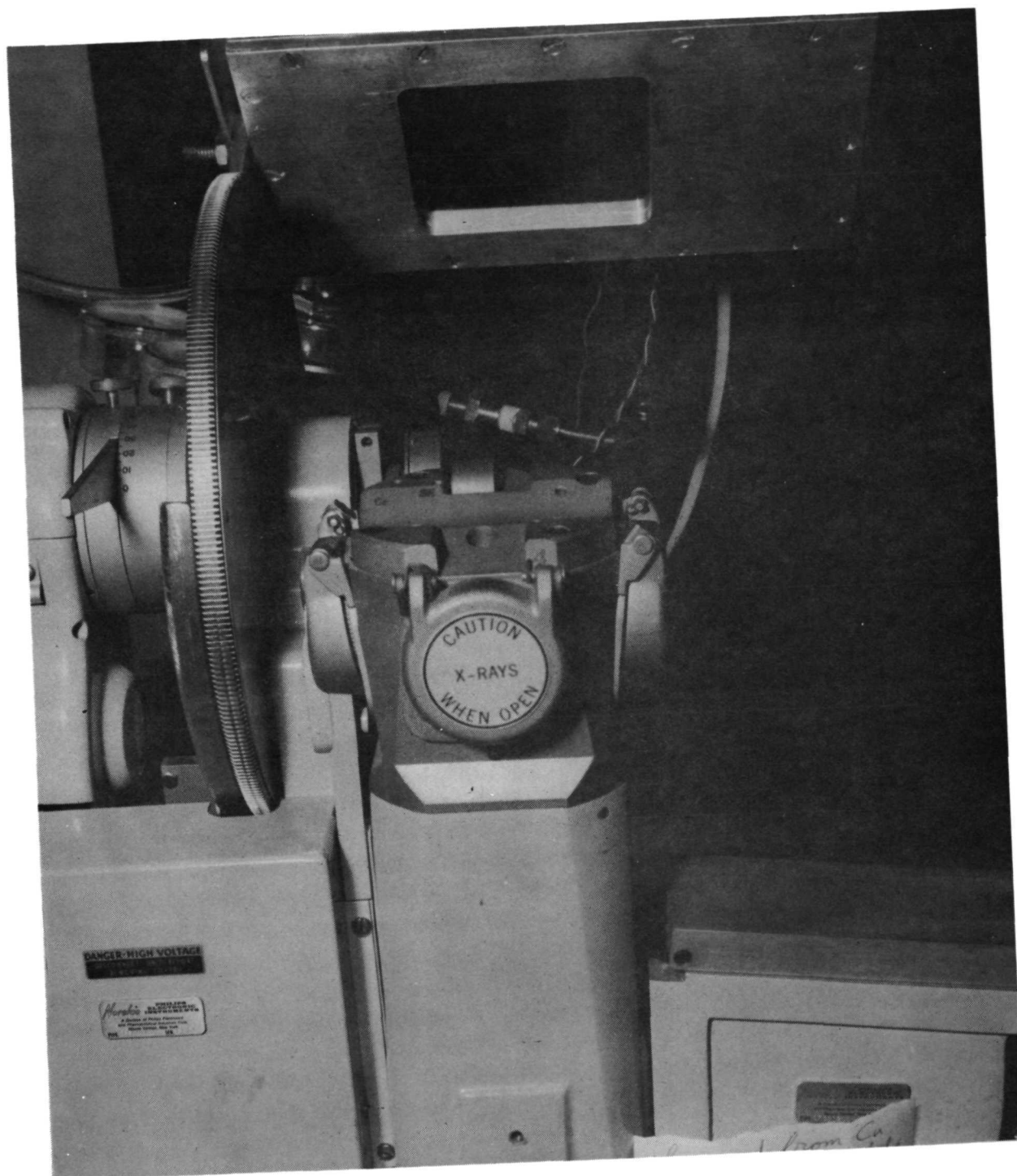


FIGURE 15

described by the detectors was 17.0 cms, and thus attainable angular resolution was not quite so good as in the Seeman-Bohlin diffractometer previously described.

A package of 10 preamplifier/single channel analyzers was attached to the back of the proportional counter and the outputs operated 10 scalars directly. With this set-up two experiments were performed: (a) with only one wire being used, the counter was scanned in 0.2 degrees 2θ increments, and (b) with all 10 wires used, count rates were recorded with the counter in a fixed position. Experiment (a) is analogous to the conventional goniometer operation and demonstrates the position sensitivity of the technique and the electrical collimation effect of the multiple wires. The results are shown in detail for the Si (III) reflection in Figure 16 (A), and over a large range of 2θ in Figure 17. Experiment (b) represents more closely the conditions obtained in the planetary diffractometer.

The set of 10 counting wires was aligned with a diffraction peak and the count rate on each recorded. The whole counter was then moved by 3.3 degrees and the process repeated. The results are shown in Figure 16(B), where individual wire positions and count rates are indicated, and over a large range of 2θ in Figure 18.

The deterioration of angular resolution of line-width as the beam incidence on the counters diverges from normal was discussed above from the design standpoint, and is shown as an experimental result in Figure 19. The Si (III) reflection at normal incidence to the counter is shown compared with the same reflection obtained when the counter was rotated 40° about the axis of the central anode of the array. Although the effect is ameliorated at low angles by displacement of the detectors from the focusing circle, it still prevents resolution of weaker peaks in this angular region.

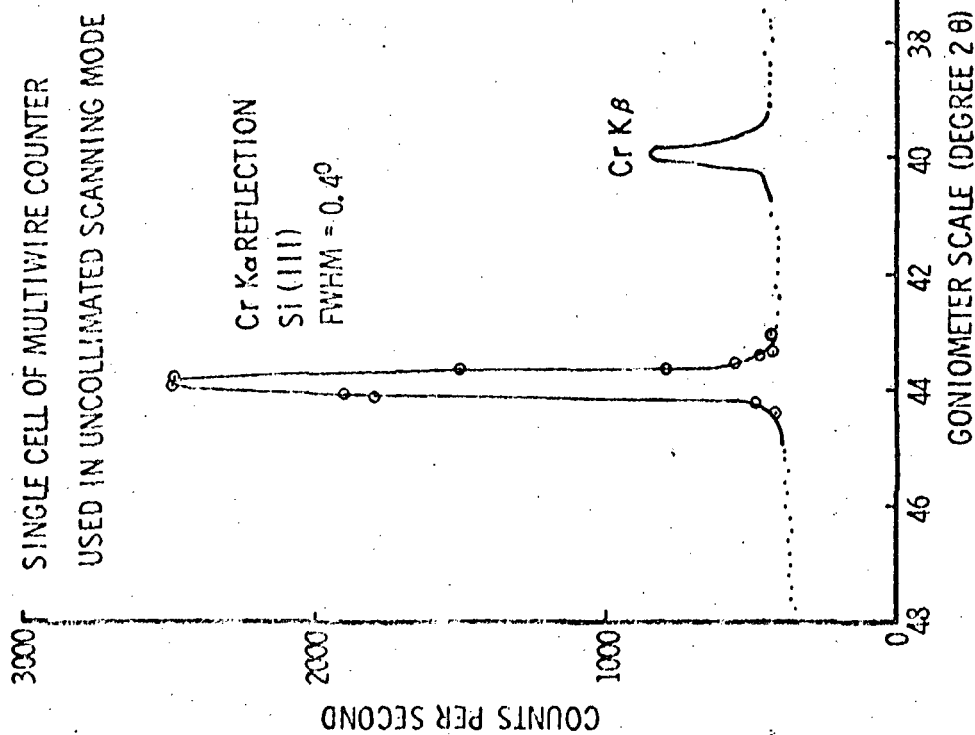


FIGURE 16 (A)

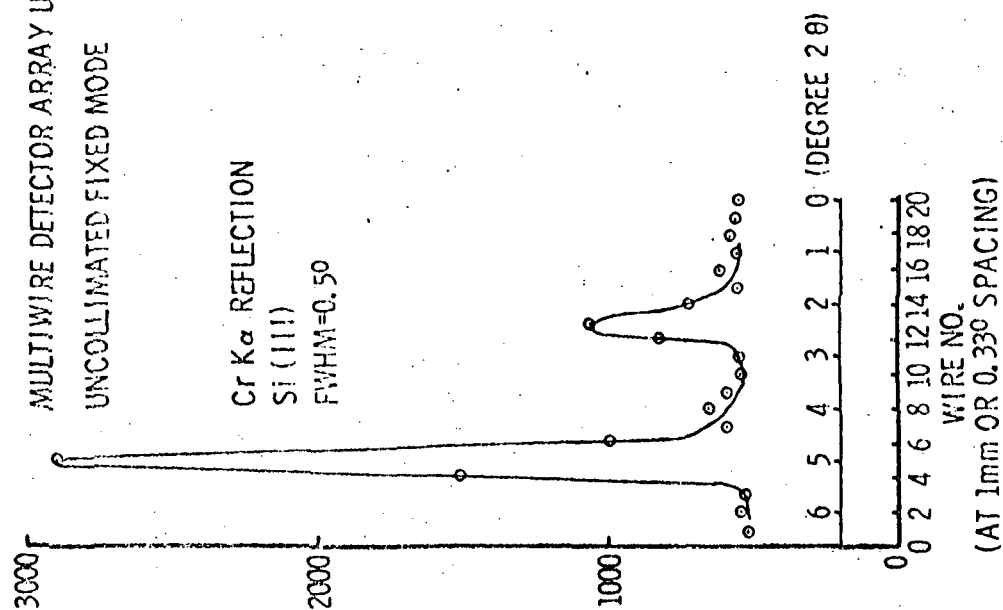


FIGURE 16 (B)

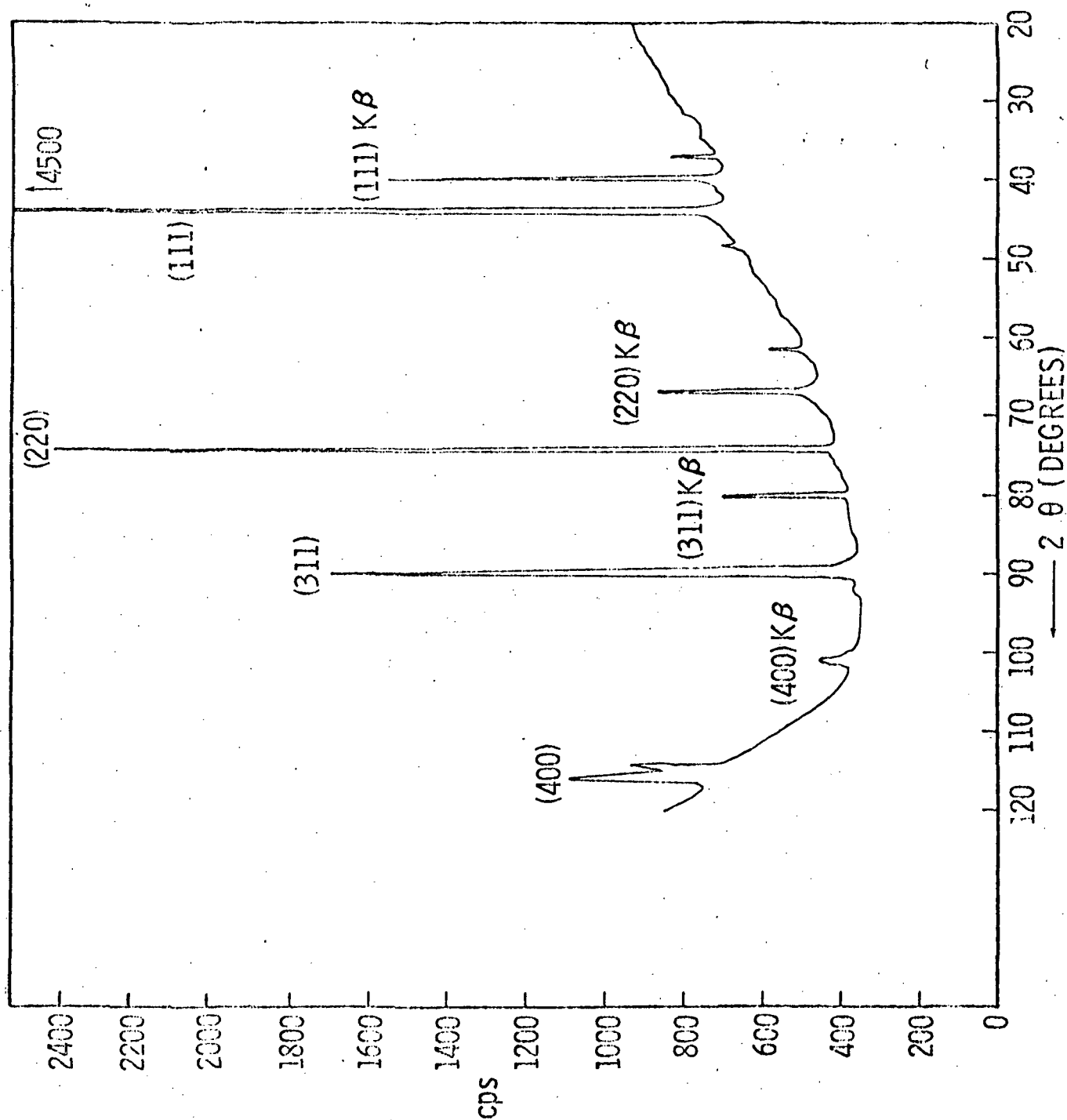


FIGURE 17. DIFFRACTION PATTERN OF SILICON POWDER AND UNFILTERED CHROMIUM RADIATION USING SINGLE WIRE IN THE SCANNING MODE.

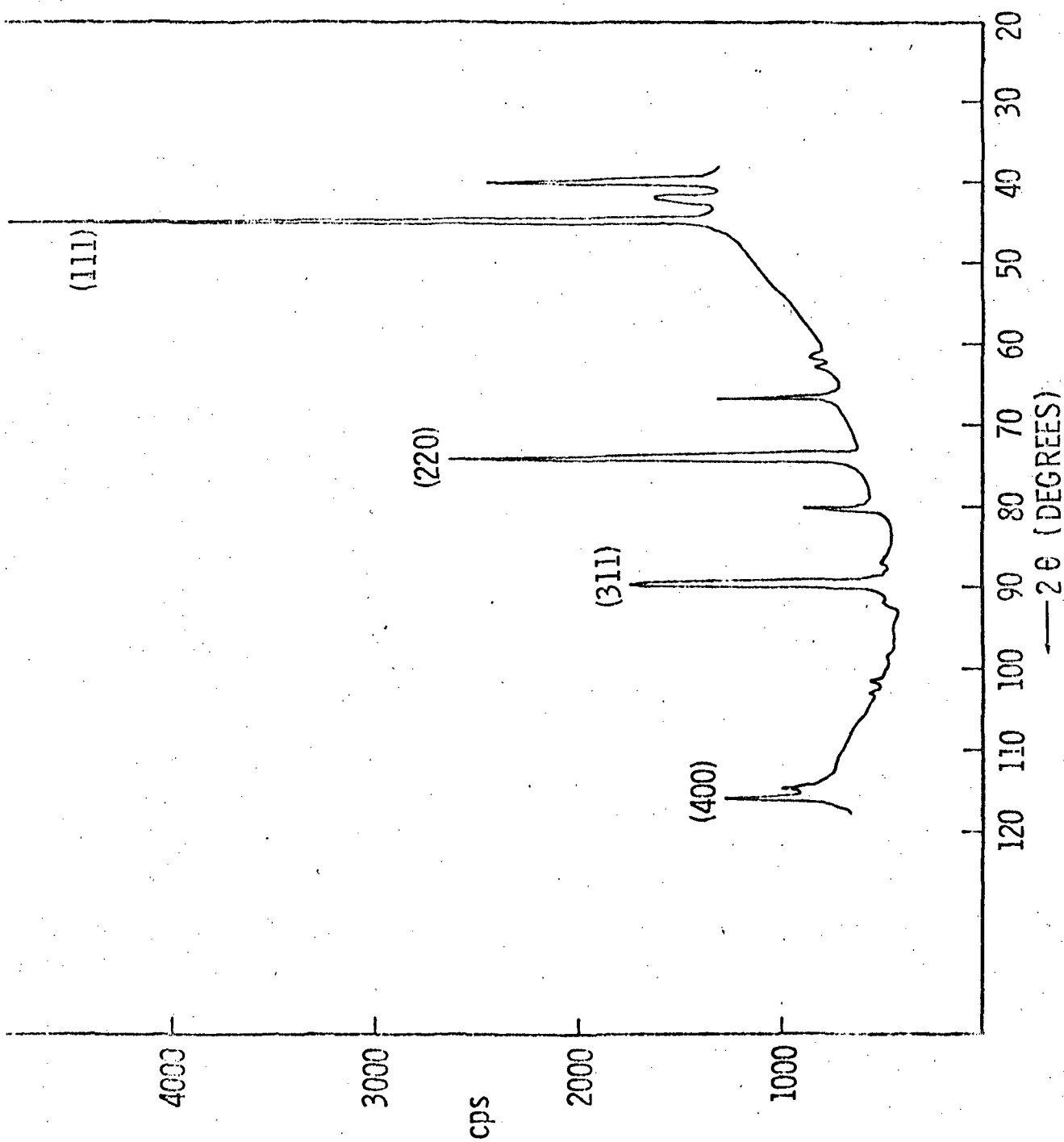


FIGURE 18 DIFFRACTION PATTERN OBTAINED WITH A SET OF 10 WIRES IN A SERIES OF FIXED POSITIONS TO SIMULATE A FIXED ARRAY OF 600 COUNTERS. SILICON POWDER WITH UNFILTERED CHROMIUM RADIATION WAS USED.

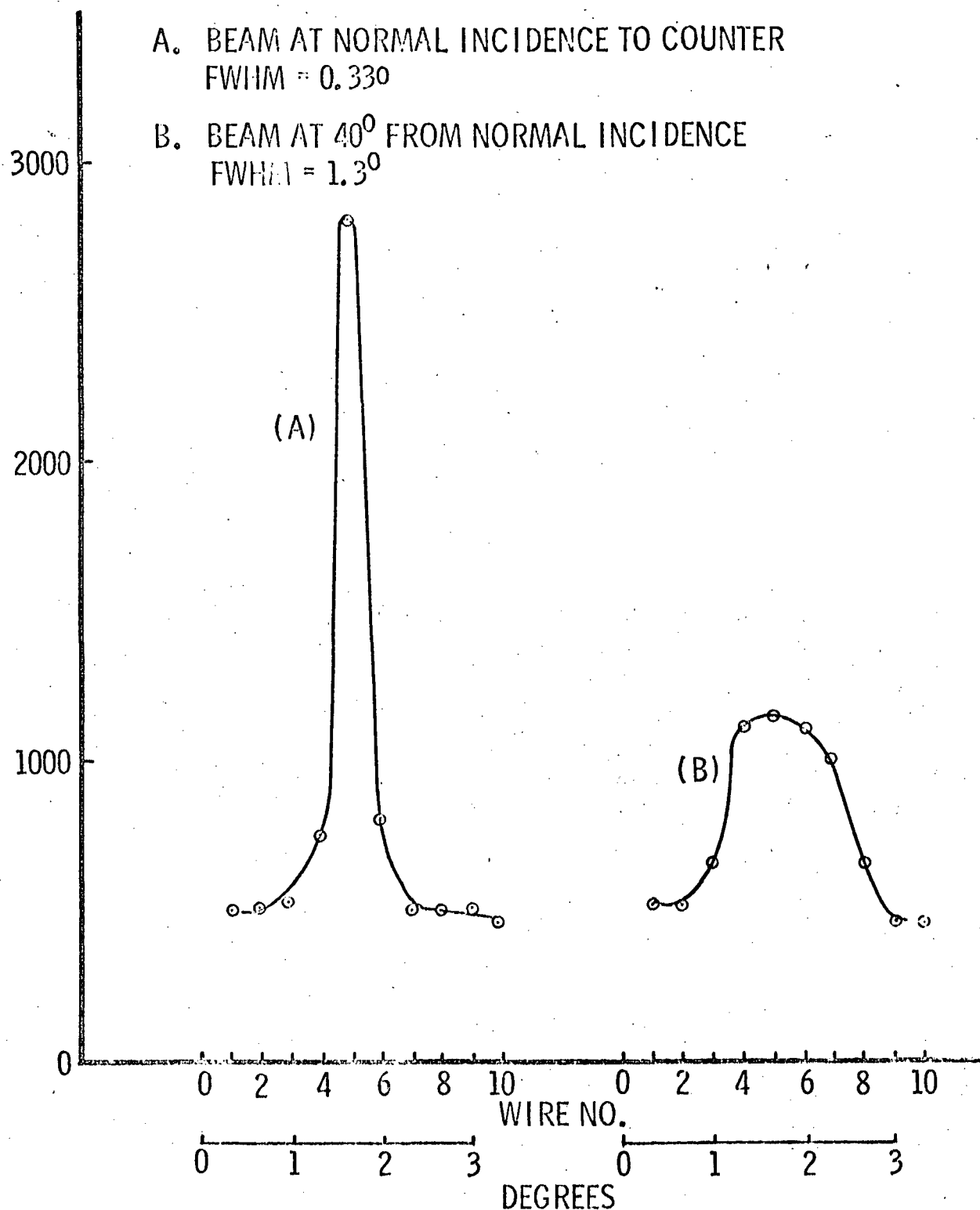


FIGURE 19 EXPERIMENTAL RESULTS DEMONSTRATING THE EFFECT OF X-RAY BEAM INCIDENCE ON THE COUNTERS AT ANGLES FAR FROM NORMAL

CONCLUSIONS

The laboratory experiments described above demonstrate the feasibility of thin multiwire proportional counters as detectors for a lightweight low power x-ray diffractometer. Until other detector systems of greater position and energy resolution and lesser thickness become available, the multiwire proportional counter array represents a good compromise of characteristics for a fixed detector array.

An instrument with a power requirement of less than 5 watts has been designed which incorporates these detectors into a lightweight diffractometer capable of performing mineralogical analysis of a rock mixture.

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