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DEVELOPMENT OF A POSITION SENSITIVE X-RAY DETECTOR FOR USE IN A LIGHT WEIGHT X-RAY DIFFRACTOMETER

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Final Report for the Period August 17, 1972 through June 17, 1973

FOREWORD

This is the final report under Contract No. NASW-2382 covering the period from August 17, 1972 through June 17, 1973. This project is a continuation of work initiated under contract NASW-2111.

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DEVELOPMENT OF A POSITION SENSITIVE X-RAY DETECTOR FOR USE IN A LIGHT WEIGHT X-RAY DIFFRACTOMETER

INTRODUCTION AND SUMMARY

This project continues work on a curved position sensitive x-ray detector with primary emphasis on the analysis of operation in a focusing diffraction geometry. Previous counters have been operated successfully in Debye powder diffraction cameras and are described in Reference 1.

A curved counter with an entrance window spanning about 80° and with a 5-in. radius to match the JPL focusing diffractometer test bed has been designed and constructed in this program. Completion of the testing as a sealed counter in a focusing geometry has not been possible because of persistent leaks in the epoxy seal around the beryllium window. The present recommendation is to remove the epoxy sealing material and reinstall the beryllium window using a glass powder seal.

Counter Design and Construction

The basic construction drawings for the counter constructed in this program are included in Appendix A. The counter is shown in Figure 1 and is similar to previously constructed curved counters except (1) the radius is 5-in. to permit tests in the JPL diffractometer, (2) the cathode diameter is 1/4-in. instead of 1/2-in., and (3) the detector is sealed.

Construction of this counter body was difficult compared to previous counters because of the special setup work required to do machining on a curved surface. Entirely different machining techniques were necessary because of the desire to seal this unit rather than operate it as a flow counter.

Although the anode wire length is the same as in the last counter, the present counter could not be operated with a free standing anode as was previously possible. Three supports were

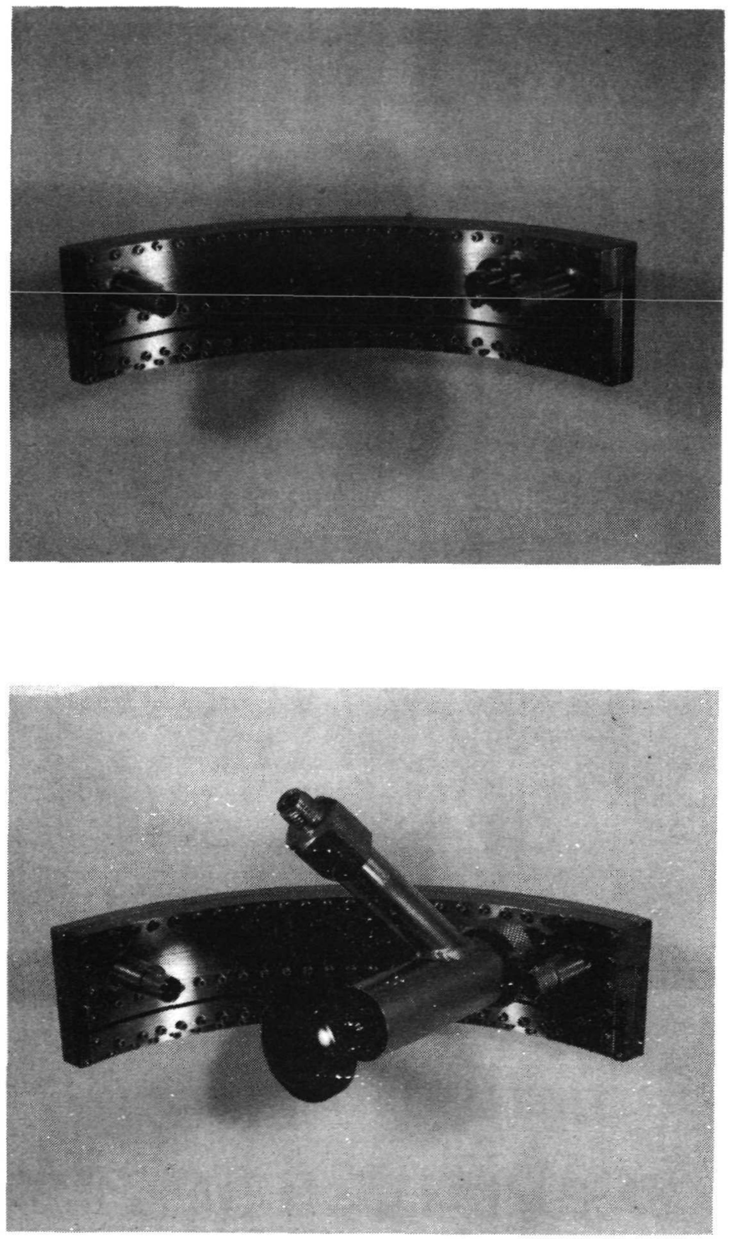


Figure 1 Photographs of the curved counter with and without the filling valve attached

installed after a single midpoint support also proved inadequate. The supports consist of a 0.5 mil nylon fiber across the diameter of the detector. The fiber is knotted around the anode and epoxied to the walls of the detector.

The beryllium window installation turned out to be particularly difficult. Initial plans called for a powder glass seal installed by LND, Inc. A later decision to install the window at IITRI using a low vapor pressure epoxy proved to be unfortunate. Although epoxy seals have been used successfully in other counters, a leaktight seal could not be established in this counter.

The leaks around the beryllium window have prevented filling the detector with Xenon and completing the testing in a focusing geometry.

<u>Fiber Development</u>

The original program anticipated two sources of resistive anode fiber: quartz fibers coated with pyrolytic carbon from the General Electric Company and glass fibers coated with tin oxide prepared by the Optics Section of IITRI. The General Electric Company has since ceased manufacturing the carbon coated fiber and has no stock on hand. Commercially prepared carbon-coated fibers do not appear to be available from any alternate source.

The preparation of resistive coatings on glass proved difficult and led to a trial of a thin aluminum coating. Initially, metal coatings were largely discounted because of the very thin layer required to give appreciable resistance (see Figure 2). The experience with aluminum, however, was generally favorable.

The first batch of special anode fibers made from 2.6 mil diameter glass was coated with aluminum to a thickness of 75-100 Å. The resistance was 9,600 ohm/cm. Tests in a position sensitive counter indicated minor irregularities although

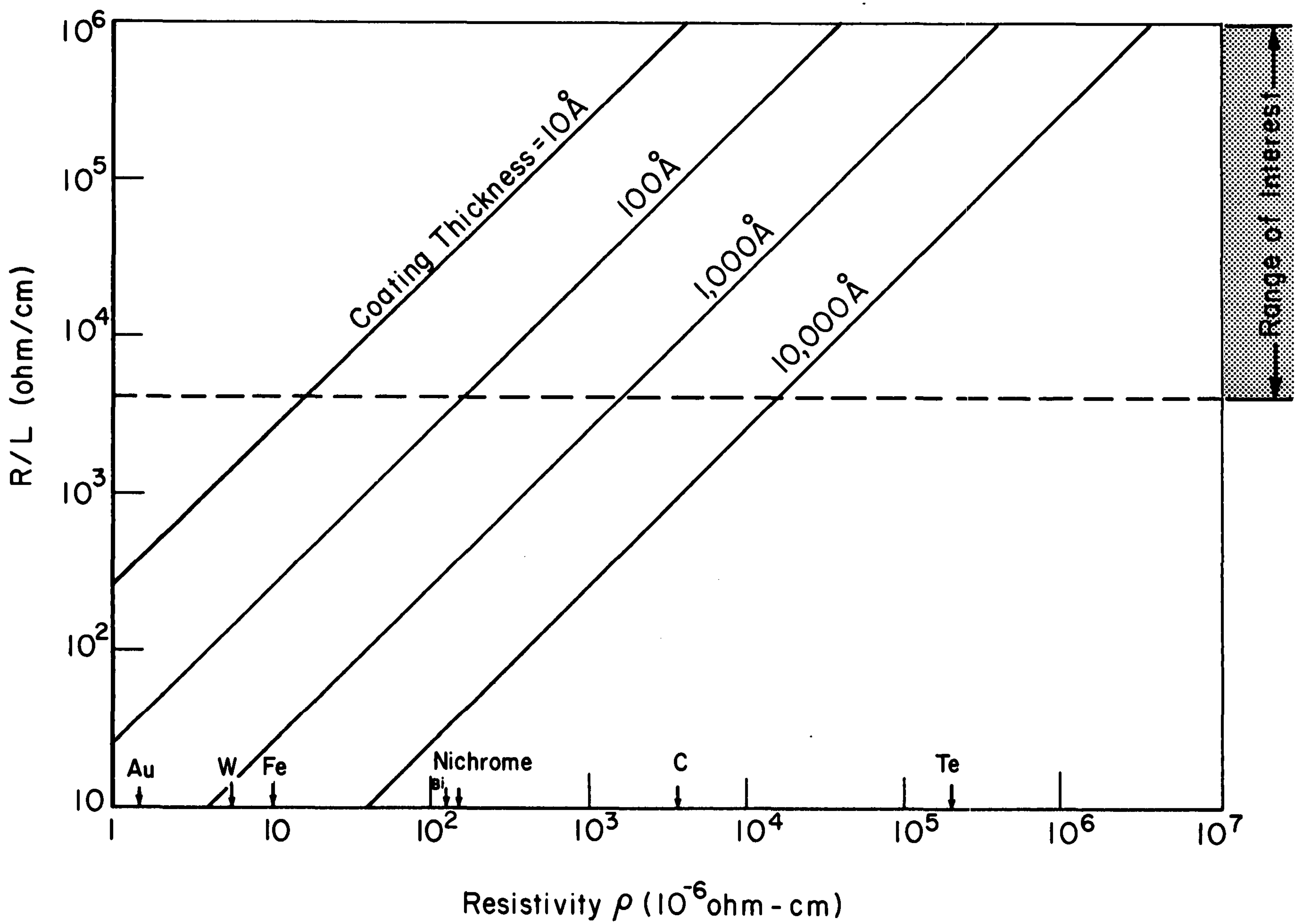


Figure 2 Resistance per unit length as a function of resistivity for a variety of coating thicknesses on a 5 mil diameter base fiber

basic operation as a position sensitive counter was generally satisfactory. The energy resolution was only fair (about 20-22% FWHM for Fe-55) but the position resolution was within 5% of that obtained from a 1 mil quartz fiber coated with pyrolytic carbon.

Subsequent work using fibers with the desired 5 mil diameter, however, produced coatings with substantial irregularities in the resistivity. These proved un-useable for proportional counter work.

The best available fiber, therefore, was the limited quantity of 5 mil quartz coated with pyrolytic carbon which was on hand from previous work. This fiber has been used in the detector.

Electronics

The electronic system for detecting and comparing the pulses at each end of the position sensitive x-ray counter is essentially a re-creation of the system used in Reference 1 but which had been dismantled during lapses in support.

While assembling the system, some consideration was given to changing the preamplifiers in order to improve the signal-to-noise ratio. The major changes involved isolation of the preamplifiers by increasing the input capacitance. Although improved isolation was achieved, the accompanying decrease in signal amplitude resulted in a net loss rather than a net gain in signal-to-noise ratio. The use of current sensitive preamplifiers to improve the signal-to-noise ratio was recently reported.[2]

Minor modifications were made to the "backup" analogue divider. This unit is now electrically interchangeable with the primary unit although the speed and accuracy remain inferior.

Hadamard Transform Spectroscopy

Hadamard transform spectrometry[3] is a technique for detecting any type of line spectra, including X-ray spectra. Because of its possible use of a curved proportional counter and its potential for X-ray spectrometry, a brief analysis was made of the technique and its capabilities.

Generally, the predicted results were found to be slightly worse than those expected from a single scanning slit although the entire spectrum is viewed simultaneously so that variations in X-ray source intensity do not result in ficticious local variations in the output spectrum. No speed advantage, however, is obtained when the Hadamard technique is applied to X-ray spectroscopy because the uncertainty in the raw data is determined by counting statistics rather than background noise. A more complete discussion with sample spectra is contained in Appendix B.

REFERENCES

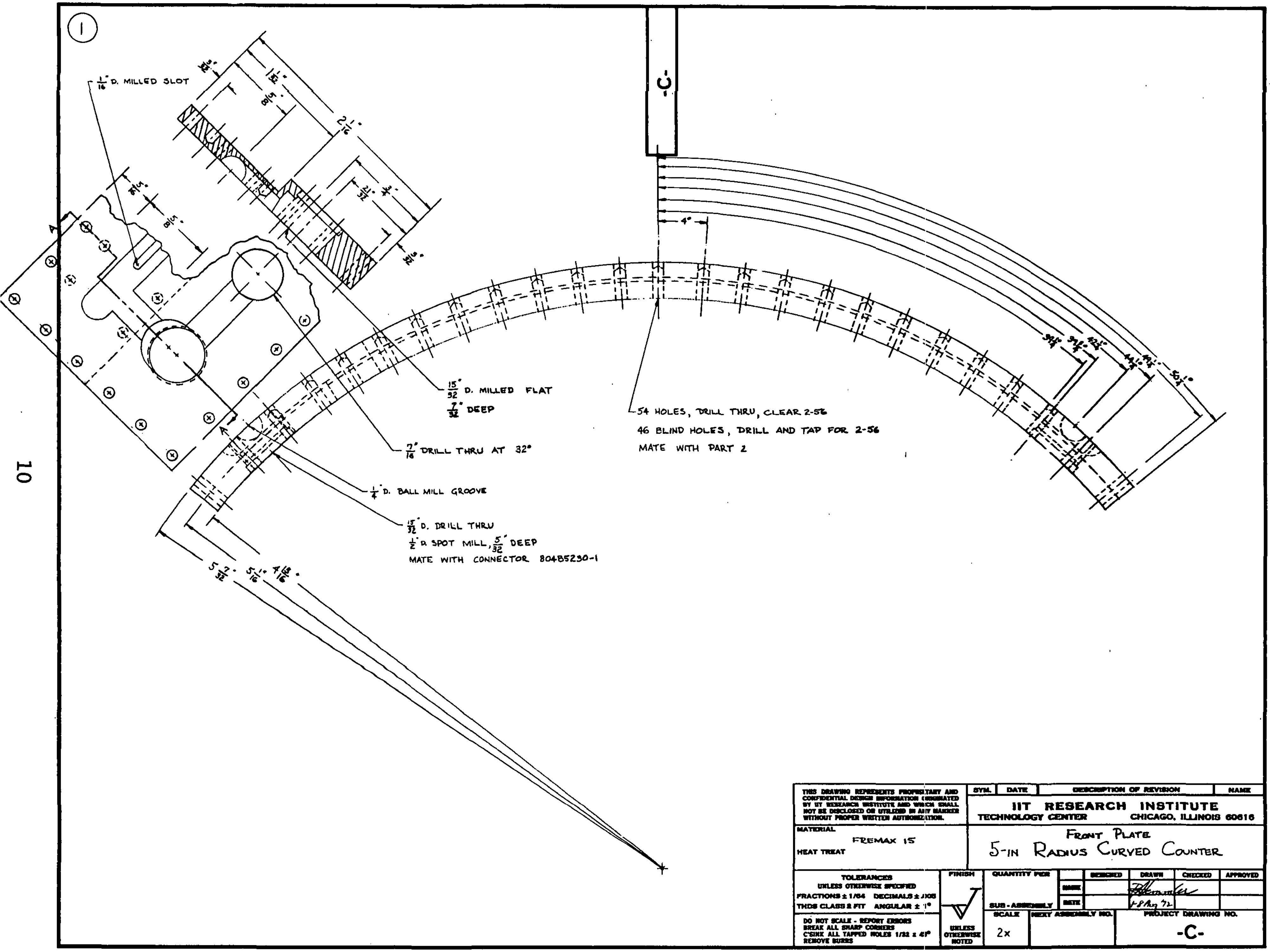
1. R. A. Semmler, "Development of a Position Sensitive X-Ray Detector for Use in a Light Weight X-Ray Diffractometer," Report No. IITRI-V6112-4 (July 1971). NASA Accession No. N72-13366. Available from NTIS.

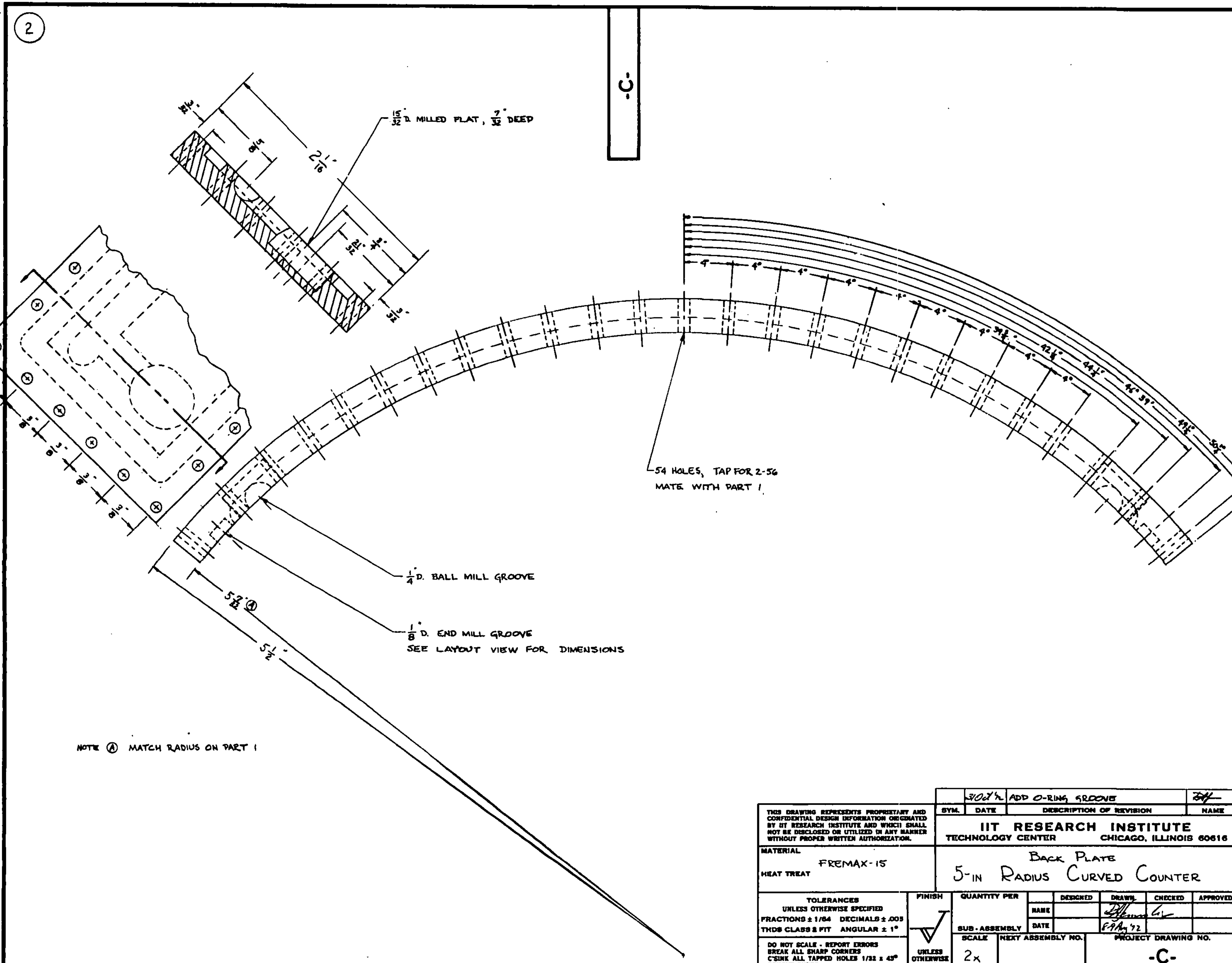
2. G. D. Westphal, "On The Resolution of a Position Sensitive $^{10}BF_3$ Proportional Counter," Nucl. Inst. and Methods 106, 279-283 (1973).

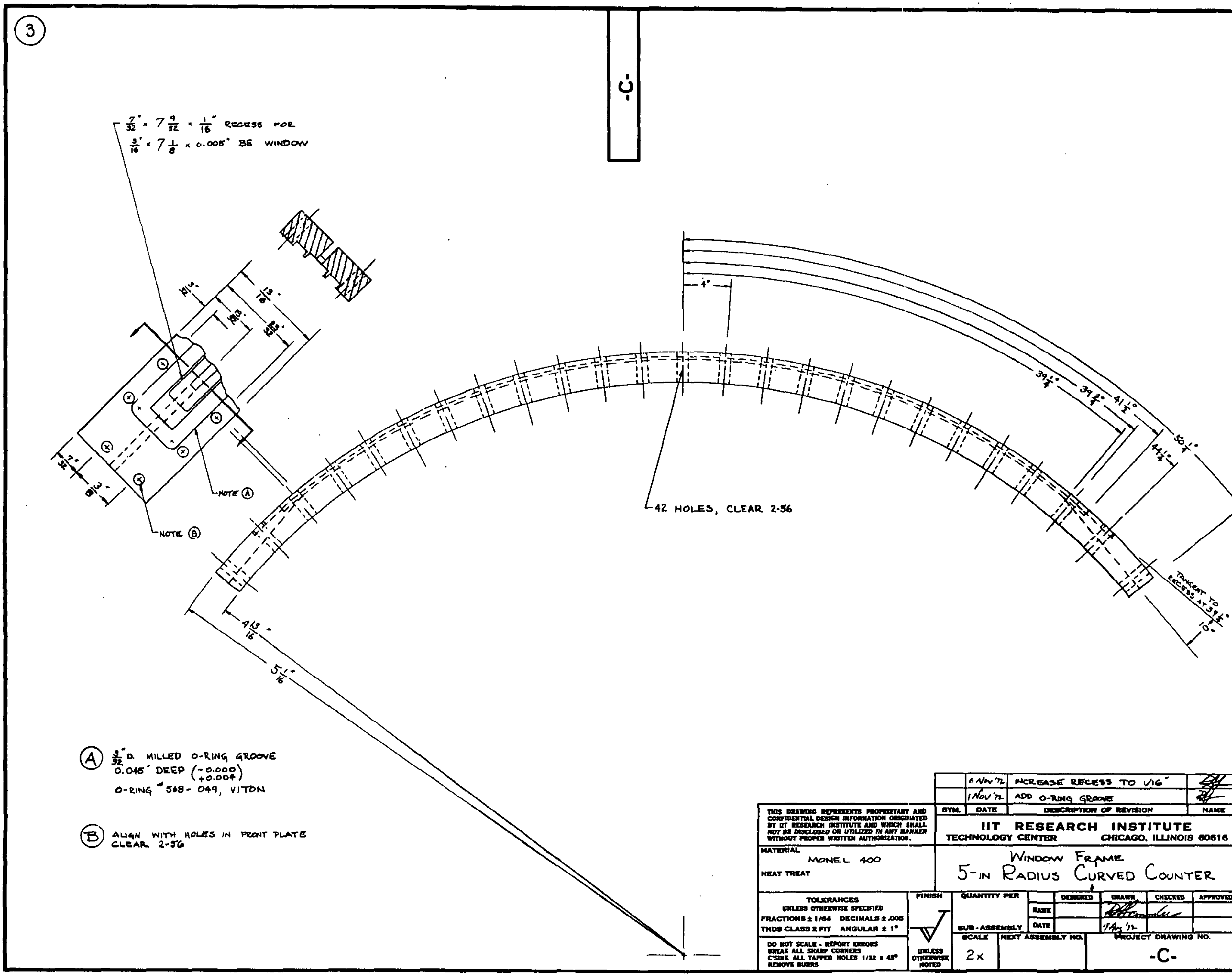
3. E. D. Nelson and M. C. Fredman, "Hadamard Spectroscopy," J. Opt. Soc. Amer. 60, 1664-1669 (December 1970).

APPENDIX A

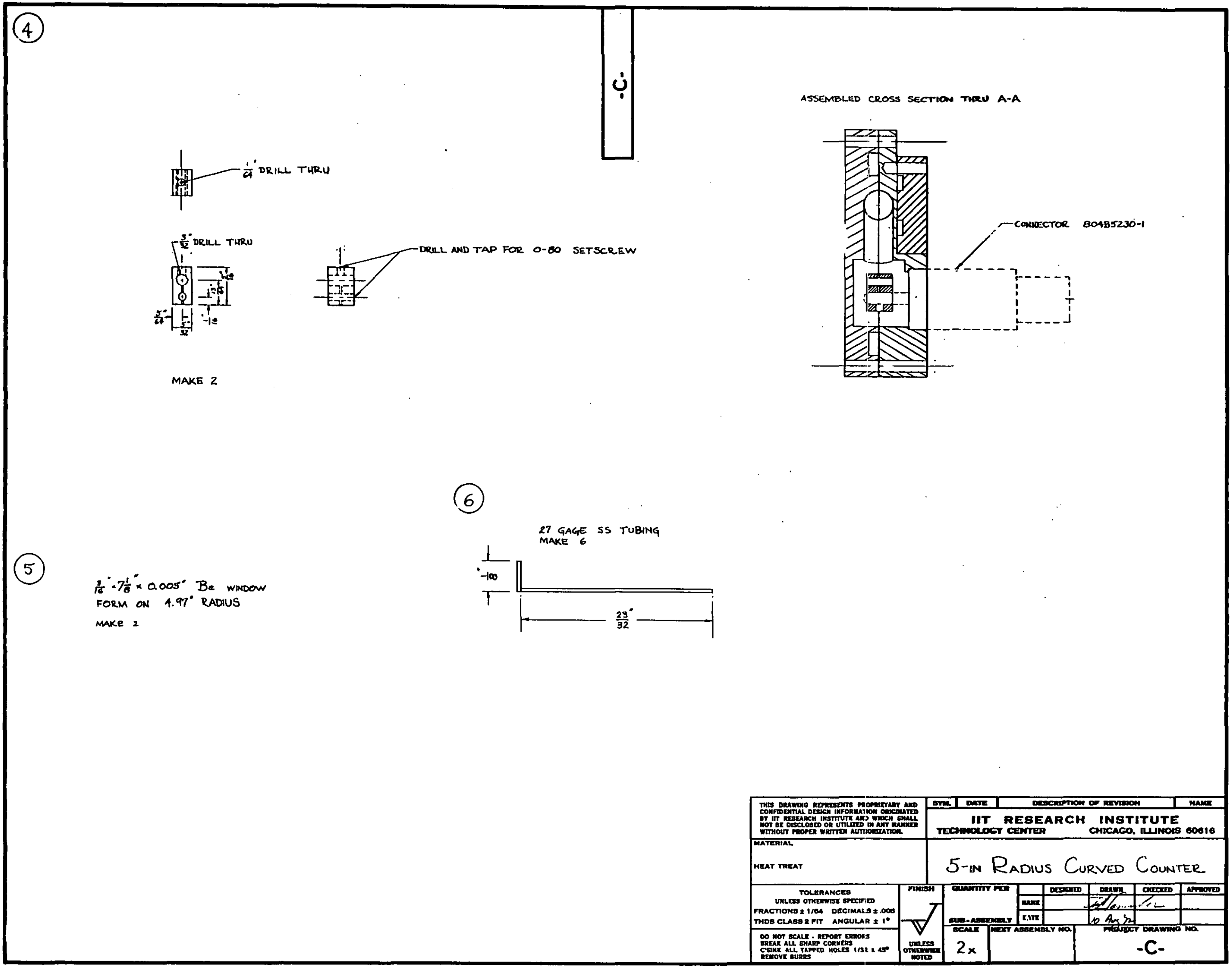
CONSTRUCTION DRAWINGS


-C-
1/16" D. MILLED SLOT
15/32" D. MILLED FLAT
7/32" DEEP
7/16" DRILL THRU AT 32°
1/4" D. BALL MILL GROOVE
15/32" D. DRILL THRU
1/2" D. SPOT MILL, 5/32" DEEP
MATE WITH CONNECTOR 804B5230-1
54 HOLES, DRILL THRU, CLEAR 2-56
46 BLIND HOLES, DRILL AND TAP FOR 2-56
MATE WITH PART 2
4°
THIS DRAWING REPRESENTS PROPRIETARY AND CONFIDENTIAL DESIGN INFORMATION ORIGINATED BY IIT RESEARCH INSTITUTE AND WHICH SHALL NOT BE DISCLOSED OR UTILIZED IN ANY MANNER WITHOUT PROPER WRITTEN AUTHORIZATION.
MATERIAL FREMAX 15
HEAT TREAT
IIT RESEARCH INSTITUTE
TECHNOLOGY CENTER CHICAGO, ILLINOIS 60616
FRONT PLATE
5-IN RADIUS CURVED COUNTER
TOLERANCES UNLESS OTHERWISE SPECIFIED
FRACTIONS ± 1/64 DECIMALS ± .005
THDS CLASS 2 FIT ANGULAR ± 1°
DO NOT SCALE - REPORT ERRORS
BREAK ALL SHARP CORNERS
C'SINK ALL TAPPED HOLES 1/32 x 45°
REMOVE BURRS
FINISH
UNLESS OTHERWISE NOTED
SCALE 2x
PROJECT DRAWING NO. -C-

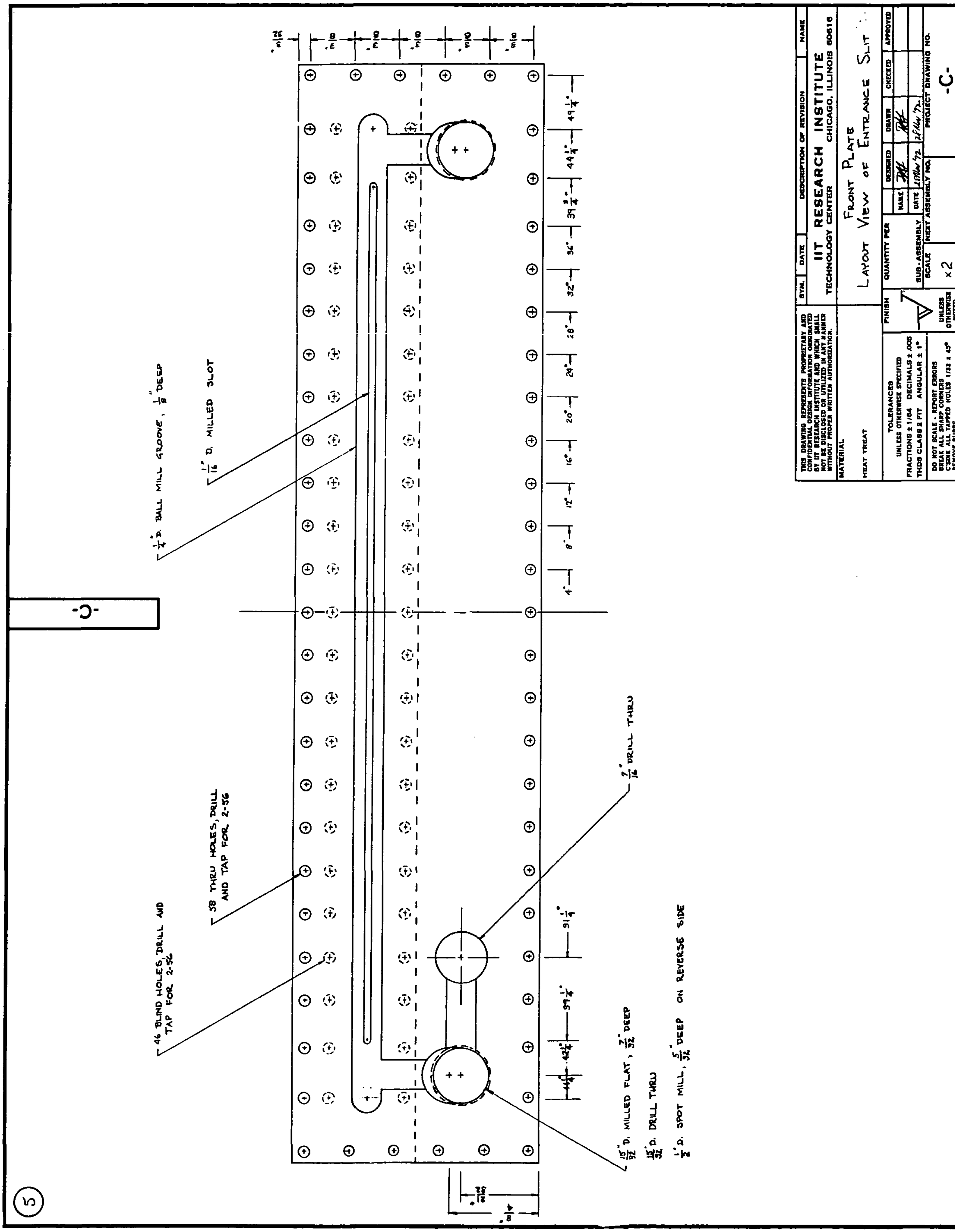

2
-C-
15/32" D. MILLED FLAT, 7/32" DEEP
2 1/16"
5/8"
3/32"
21/32"
3/4"
54 HOLES, TAP FOR 2-56
MATE WITH PART 1
1/4" D. BALL MILL GROOVE
1/8" D. END MILL GROOVE
SEE LAYOUT VIEW FOR DIMENSIONS
5 7/32" (A)
5 1/2"
NOTE (A) MATCH RADIUS ON PART 1
ADD O-RING GROOVE
SYM. DATE DESCRIPTION OF REVISION NAME
THIS DRAWING REPRESENTS PROPRIETARY AND CONFIDENTIAL DESIGN INFORMATION ORIGINATED BY IIT RESEARCH INSTITUTE AND WHICH SHALL NOT BE DISCLOSED OR UTILIZED IN ANY MANNER WITHOUT PROPER WRITTEN AUTHORIZATION.
IIT RESEARCH INSTITUTE
TECHNOLOGY CENTER CHICAGO, ILLINOIS 60616
MATERIAL FREMAX-15
HEAT TREAT
BACK PLATE
5-IN RADIUS CURVED COUNTER
TOLERANCES
UNLESS OTHERWISE SPECIFIED
FRACTIONS ± 1/64 DECIMALS ± .005
THDS CLASS 2 FIT ANGULAR ± 1°
DO NOT SCALE - REPORT ERRORS
BREAK ALL SHARP CORNERS
C'SINK ALL TAPPED HOLES 1/32 x 45°
REMOVE BURRS
FINISH
UNLESS OTHERWISE NOTED
QUANTITY PER
SUB-ASSEMBLY
DESIGNED DRAWN CHECKED APPROVED
NAME
DATE
SCALE 2x
NEXT ASSEMBLY NO.
PROJECT DRAWING NO. -C-

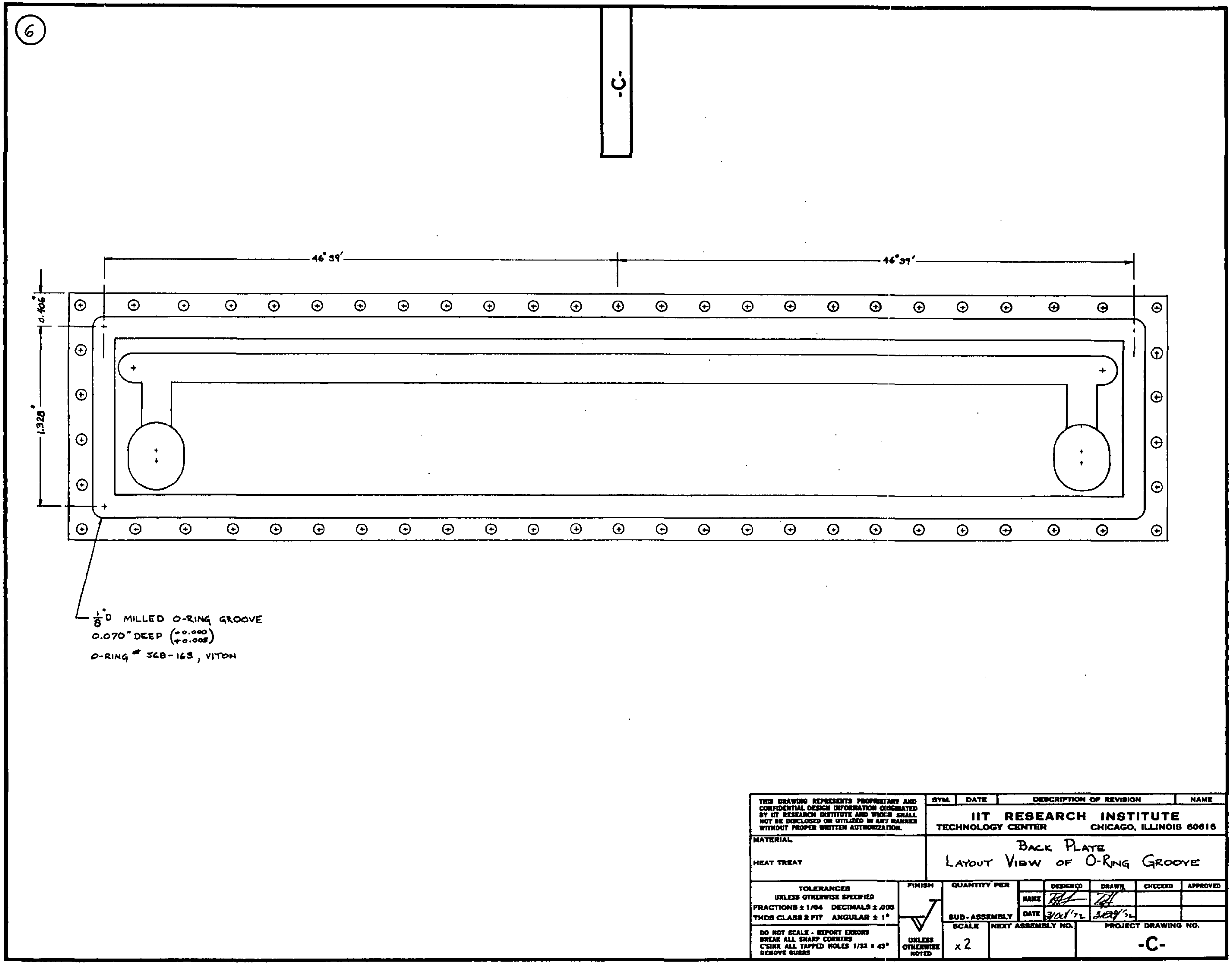


3
-C-
7/32" × 7 9/32 × 1/16" RECESS FOR
3/16" × 7 1/8 × 0.005" BE WINDOW
42 HOLES, CLEAR 2-56
NOTE (A)
NOTE (B)
4 13/16"
5 1/16"
4°
39 1/4°
39 3/4°
41 1/2°
50 1/4°
44 1/4°
TANGENT TO RECESS AT 39 1/4°
10°
(A) 3/32" D. MILLED O-RING GROOVE
0.045" DEEP (-0.000 +0.004)
O-RING # 568-049, VITON
(B) ALIGN WITH HOLES IN FRONT PLATE
CLEAR 2-56
6 Nov '72 INCREASE RECESS TO 1/16"
1 Nov '72 ADD O-RING GROOVE
SYM. DATE DESCRIPTION OF REVISION NAME
THIS DRAWING REPRESENTS PROPRIETARY AND CONFIDENTIAL DESIGN INFORMATION ORIGINATED BY IIT RESEARCH INSTITUTE AND WHICH SHALL NOT BE DISCLOSED OR UTILIZED IN ANY MANNER WITHOUT PROPER WRITTEN AUTHORIZATION.
IIT RESEARCH INSTITUTE
TECHNOLOGY CENTER CHICAGO, ILLINOIS 60616
MATERIAL MONEL 400
HEAT TREAT
WINDOW FRAME
5-IN RADIUS CURVED COUNTER
TOLERANCES
UNLESS OTHERWISE SPECIFIED
FRACTIONS ± 1/64 DECIMALS ± .005
THDS CLASS 2 FIT ANGULAR ± 1°
DO NOT SCALE - REPORT ERRORS
BREAK ALL SHARP CORNERS
C'SINK ALL TAPPED HOLES 1/32 × 45°
REMOVE BURRS
FINISH
UNLESS OTHERWISE NOTED
QUANTITY PER
SUB-ASSEMBLY
DESIGNED DRAWN CHECKED APPROVED
NAME
DATE
SCALE 2×
NEXT ASSEMBLY NO.
PROJECT DRAWING NO. -C-



-C-
4
1/64" DRILL THRU
3/32" DRILL THRU
DRILL AND TAP FOR 0-80 SETSCREW
MAKE 2
ASSEMBLED CROSS SECTION THRU A-A
CONNECTOR 804B5230-1
5
3/16" × 7 1/8" × 0.005" Be WINDOW
FORM ON 4.97" RADIUS
MAKE 2
6
27 GAGE SS TUBING
MAKE 6
.100
23/32"
THIS DRAWING REPRESENTS PROPRIETARY AND CONFIDENTIAL DESIGN INFORMATION ORIGINATED BY IIT RESEARCH INSTITUTE AND WHICH SHALL NOT BE DISCLOSED OR UTILIZED IN ANY MANNER WITHOUT PROPER WRITTEN AUTHORIZATION.
SYM. DATE DESCRIPTION OF REVISION NAME
IIT RESEARCH INSTITUTE
TECHNOLOGY CENTER CHICAGO, ILLINOIS 60616
MATERIAL
HEAT TREAT
5-IN RADIUS CURVED COUNTER
TOLERANCES
UNLESS OTHERWISE SPECIFIED
FRACTIONS ± 1/64 DECIMALS ± .005
THDS CLASS 2 FIT ANGULAR ± 1°
FINISH
UNLESS OTHERWISE NOTED
QUANTITY PER
SUB-ASSEMBLY
DESIGNED DRAWN CHECKED APPROVED
NAME
DATE
10 Aug 72
DO NOT SCALE - REPORT ERRORS
BREAK ALL SHARP CORNERS
C'SINK ALL TAPPED HOLES 1/32 x 45°
REMOVE BURRS
SCALE
2x
NEXT ASSEMBLY NO.
PROJECT DRAWING NO.
-C-


IIT RESEARCH INSTITUTE
TECHNOLOGY CENTER
CHICAGO, ILLINOIS 60616
FRONT PLATE
LAYOUT VIEW OF ENTRANCE SLIT
SCALE x2
PROJECT DRAWING NO. -C-
1/4" D. BALL MILL GROOVE, 1/8" DEEP
1/16" D. MILLED SLOT
58 THRU HOLES, DRILL AND TAP FOR 2-56
46 BLIND HOLES, DRILL AND TAP FOR 2-56
7/16" DRILL THRU
15/32" D. MILLED FLAT, 7/32" DEEP
15/32" D. DRILL THRU
1/2" D. SPOT MILL, 5/32" DEEP ON REVERSE SIDE
-C-
5


6
-C-
46° 39′
46° 39′
0.406"
1.328"
$\frac{1}{8}$" D MILLED O-RING GROOVE
0.070" DEEP $\binom{-0.000}{+0.005}$
O-RING # 568-163, VITON
THIS DRAWING REPRESENTS PROPRIETARY AND CONFIDENTIAL DESIGN INFORMATION ORIGINATED BY IIT RESEARCH INSTITUTE AND WHICH SHALL NOT BE DISCLOSED OR UTILIZED IN ANY MANNER WITHOUT PROPER WRITTEN AUTHORIZATION.
SYM. DATE DESCRIPTION OF REVISION NAME
IIT RESEARCH INSTITUTE
TECHNOLOGY CENTER CHICAGO, ILLINOIS 60616
MATERIAL
HEAT TREAT
BACK PLATE
LAYOUT VIEW OF O-RING GROOVE
TOLERANCES
UNLESS OTHERWISE SPECIFIED
FRACTIONS ± 1/64 DECIMALS ± .005
THDS CLASS 2 FIT ANGULAR ± 1°
DO NOT SCALE - REPORT ERRORS
BREAK ALL SHARP CORNERS
C'SINK ALL TAPPED HOLES 1/32 x 45°
REMOVE BURRS
FINISH
UNLESS OTHERWISE NOTED
QUANTITY PER
SUB-ASSEMBLY
SCALE
x 2
NEXT ASSEMBLY NO.
DESIGNED DRAWN CHECKED APPROVED
NAME
DATE
PROJECT DRAWING NO.
-C-

APPENDIX B

HADAMARD TRANSFORM SPECTROMETRY AS A DETECTION TECHNIQUE FOR X-RAY DIFFRACTION ANALYSIS

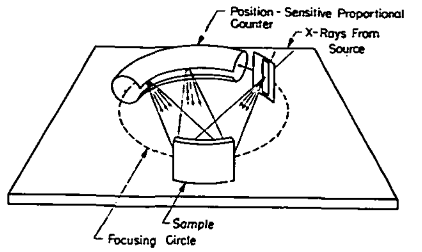
APPENDIX B

Hadamard transform spectrometry is a technique for detecting any line spectra although the description here emphasizes the application to X-ray diffraction analysis. A quick comparison of the Hadamard Technique with other detection techniques is given in Figure 1. The geometry is basically similar to a single scanning slit except that the mask contains thousands of slits rather than one slit. Approximately 50% of the mask is transparent.

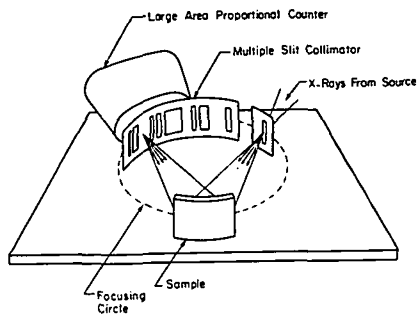
In use, the mask with multiple slits scans across the spectrum while a single stationary detector located behind the entire mask measures the total rate of radiation passing through the mask. No position information for individual events is measured. The only data recorded is the gross X-ray counting rate as a function of mask position. The data collection process is therefore similar to simple recording on a strip chart.

Of course the raw data does not "look" like a line spectrum. But after computer processing, the original spectrum can be reconstructed from the X-ray counting rate. The reconstruction is similar, in principle, to the solution of a set of simultaneous equations. In particular, there are no end effects and no smearing introduced by the reconstruction since it is an analytic solution.

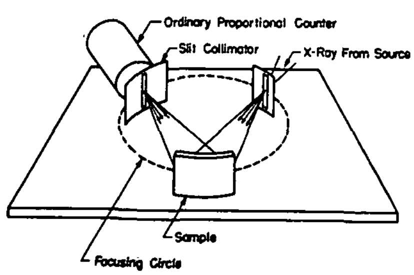
As an aid in visualizing the data transformations used in the Hadamard technique, Figure 2 compares the output of 2 hypothetical instruments - a single scanning slit and a Hadamard transform spectrometer. The same amount of time is spent collecting data in each case although the total counts collected with the Hadamard spectrometer is greater because of the multiple slits. The reconstructed spectrum is also shown for comparison.



a. Position sensitive detector in a focusing geometry. Diffraction at all angles is detected with high efficiency.



b. Hadamard transform spectrometer. Diffraction at all angles is detected simultaneously.



c. Conventional slit detector in a focusing geometry. Diffraction at only one angle is detected but with good efficiency.

Figure 1 Schematic diagram comparing a position sensitive detector with a Hadamard transform spectrometer and a single slit spectrometer.

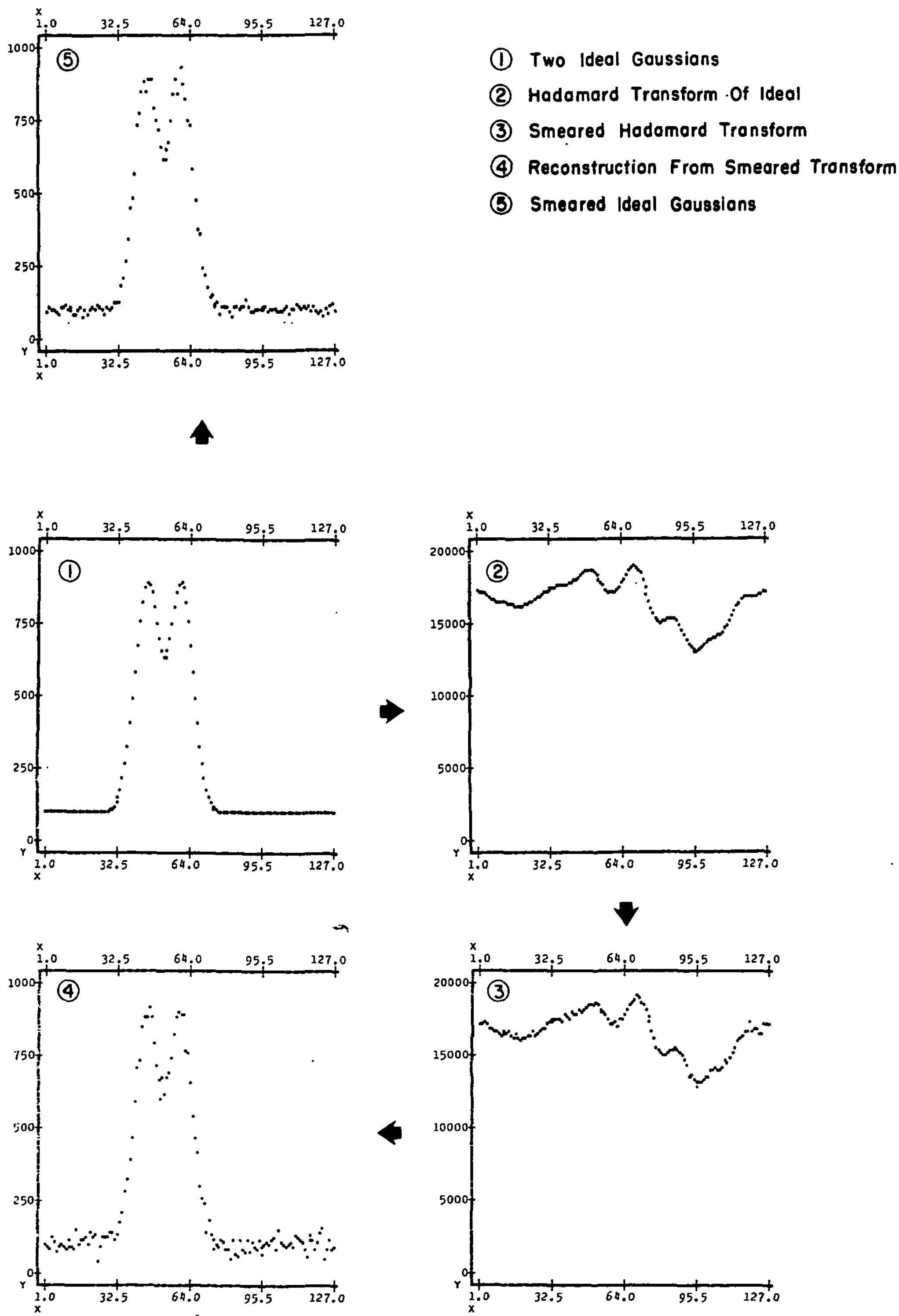


Figure 2 Comparison of output of a Hadamard transform spectrometer, (4), with a single slit spectrometer, (5). Measurement times are equal and noise (random error) is due to counting statistics only. The ideal gaussians contain 10,000 counts each on a background of 100 counts/channel. The peaks are located at x = 45 and 60 with FWHM = 12.

The features of the Hadamard technique compared to other techniques are as follows:

1. Sensitivity. The sensitivity is uniform across the entire spectrum. There are no dead regions because of, say, anode support posts for the X-ray detector anode wire. There is no variation in response introduced by fluctuations in the X-ray source intensity because the entire spectrum is being measured at any given time.

2. Signal-to-Noise. The multi-slit arrangement increases the signal strength by several orders of magnitude compared to a single slit instrument but the background noise increases only with the detector size. Although a larger detector is needed, the increase in size is not as great as the increased signal level so that the signal-to-background ratio is improved.

3. Resolution. The angular resolution is determined by the mask and is equivalent to a single slit instrument using only one of the thousands of slits. The resolution does not depend on any detector characteristics. There is no loss of resolution because of X-ray slant paths in the detector.

4. Data Collection. Only gross counting of the X-ray pulses as a function of mask position is required. Pulse height analysis and storage of individual events in real time is not required as with a position sensitive detector. A multichannel analyzer is a convenient data storage device if it is used in a multiscaling mode.

5. Ruggedness. The X-ray detector for the Hadamard technique can be built for maximum ruggedness and rigidity. A free standing anode wire is not required. The Hadamard technique does require, however, physical

movement of a mask which is not necessary with a position sensitive detector.

6. Data Reduction. Conversion of raw data to an X-ray diffraction spectrum requires computer analysis while a scanning slit or position sensitive detector will generate the diffraction spectrum directly.

7. Statistical Fluctuations. Although the high detection efficiency of the Hadamard spectrometer produces large counting rates and therefore small statistical counting errors, the conversion of the raw data into an actual spectrum amplifies the apparent statistical fluctuations. Assuming the same collection time, the spectrum from a Hadamard spectrometer contains fluctuations which are comparable to, though slightly worse than, those in a spectrum from a single scanning slit.

The last item - the effect of noise - is particularly important since the usual benefit (Fellgett advantage) of a Hadamard spectrometer is the improvement in signal-to-background ratio. If the system is limited by background noise, then significant improvements are possible compared to a single slit. If the system is limited by statistical fluctuations, then the system is very similar to a single scanning slit.

The errors in a system that is count-rate limited can be calculated approximately as follows:

For a single slit instrument, assume that n counts are collected at a typical position. The uncertainty in this directly measured spectrum value is

$$\sigma^2 = n \qquad (1)$$

In a Hadamard spectrometer, the spectrum is reconstructed as a linear combination of all measurements N_i, e.g.,

$$y = a \Sigma c_i N_i \tag{2}$$

where c_i is $\pm$ 1. The uncertainty, σ_y^2, in this indirectly measured value is given by

$$\sigma_y^2 = a^2 (\sigma_{N_1}^2 + \sigma_{N_2}^2 + \ldots.) \tag{3}$$

Since the response of the Hadamard spectrometer is relatively constant, the uncertainty in each measurement is approximately the same and is given by

$$\sigma_{N_1}^2 \approx \sigma_{N_2}^2 \approx \ldots.\sigma_N^2 = N \tag{4}$$

where N is the number of counts collected at each position. For a Hadamard spectrometer with P mask elements and about 50% mask transmission, this is approximately (P/2)n where n has the same meaning (counts/slit) as in Eq. (1). Also, the value of (a) depends only on the number of measurements and is given by 2/(P+1). Inserting these values into Eq. (3) gives

$$\sigma_y^2 = (\frac{2}{P+1})^2 \frac{P^2 n}{2} \approx 2n \tag{5}$$

Comparison of Eq. (5) with Eq. (1) shows that the variance in the value reconstructed from the Hadamard spectrometer is about double the variance of a value measured directly with a single slit.