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MEMORANDUM**

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MEASUREMENT OF A TITANIUM TRITIDE TARGET  
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**AN X-RAY MONITOR FOR MEASUREMENT OF A  
TITANIUM TRITIDE TARGET THICKNESS**

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ABSTRACT

An X-ray device capable of measuring titanium tritide film thickness from 0.1 to 30  $\mu\text{m}$  has been built and tested. The monitor was designed for use in a rotating target system which used thick targets and incorporated a sputtering electrode to remove depleted layers from the target surface. The thickness measurement can be done in the presence of an intense background of bremsstrahlung and characteristic titanium X-radiation. A measurement can be accomplished in situ in two hours with reasonable accuracy.

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## SUMMARY

An X-ray device capable of measuring titanium tritide film thicknesses from 0.1 to 30  $\mu\text{m}$  has been built and tested. The monitor was designed for use in a rotating target system which used thick targets and incorporated a sputtering electrode to remove depleted layers from the target surface. The thickness measurement can be done in the presence of an intense background of bremsstrahlung and characteristic titanium X-radiation. A measurement can be accomplished in situ in two hours with reasonable accuracy.

## INTRODUCTION

The measurement of a thin film thickness on a substrate has been accomplished for a variety of materials by the excitation of the characteristic X-rays of the substrate with a radioactive source.<sup>1, 2, 3</sup> However, these materials were not radioactive. The measurement of the thickness of a titanium tritide film, used as a target for neutron production, is complicated by the X-ray background created by the radioactive decay in the film itself.

The need for a monitor to measure the thickness of a tritide film followed the development, by these authors, of a rotating high-yield neutron target (HYNT) for a 300 kV and 30 mA deuteron accelerator. The accelerator utilized the  $\text{T}(d, n)\text{He}^4$  reaction for the production of neutrons, and incorporated a sputtering electrode in the target design which provides for the use of tritide targets up to 30  $\mu\text{m}$  thick. These targets had a thickness about ten times the range of a 300-keV deuteron. After a target film has been

depleted of tritium to about a 3  $\mu\text{m}$  depth (the range of the deuteron), the excess titanium is sputtered off in situ. A new target surface is thus exposed. By this technique the lifetime of a target surface can be extended considerably. The thickness monitor provides an accurate measurement of the amount of titanium removed.

## THE THICKNESS MONITOR

### Principle of Operation

The titanium tritide thin film target is formed on a copper substrate. The characteristic 8 keV (1.28 fJ) K X-rays of the copper substrate are excited by 24 keV (3.84 fJ) X-rays emitted by a cadmium-109 source. These 8 keV X-rays are attenuated by passing through the titanium tritide film. The X-rays then pass through the collimator system and are counted by a proportional counter which is coupled to a pulse-counting system. The copper filter over the face of the proportional counter passes the 8 keV X-rays while absorbing the majority of the background X-rays. The ratio of the 8 keV X-ray beam intensity transmitted through the titanium tritide film to the intensity of the X-ray beam from the uncoated target substrate is a measure of the film thickness.

### Description

The monitor is constructed mostly of 6061-T6 aluminum alloy. This material is best suited for use in the high neutron environment of the accelerator target. The aluminum rapidly loses its activity following neutron activation, thus minimizing the gamma ray background of the proportional counter within the monitor.

Figure 1 shows a drawing of the thickness monitor. Figure 2 shows a photograph of the monitor attached to the HYNT that illustrates its size. The monitor uses two collimator discs, one fixed and one rotatable. Two small stepper motors rotate and align, separately, the rotatable collimator and shutter. A cadmium-109 source is attached to the underside of the rotatable collimator and faces the target. Figure 3 shows a photograph of the 2.5 cm

diameter portion of the monitor that inserts into the HYNT port. The cadmium-109 source can be seen surrounding the aperture. The end window of a 0.63 cm diameter Xenon-filled proportional counter faces the aperture of the upper collimator. A 0.005 cm thick copper filter covers the window of the proportional counter. The proportional counter, electrical leads, and triax feedthrough connector are electrically insulated from the target housing. Panel lights, connected to position indicator wafer switches attached to the stepper motor shafts, show the position of the rotatable collimator and shutter.

### Background Radiation

The background radiation consists of the 4.5 keV characteristic titanium K X-rays and bremsstrahlung spectra, both excited by the tritium beta radiation. The beta radiation has a maximum energy of 18 keV. In addition to the reduction in background caused by the copper filter, a further reduction is possible by discriminating against all but the 8 keV X-rays by selective pulse sorting. Figure 4 shows the pulse height spectra from the proportional counter after pulse shaping and amplification. Pulses below 1 volt were eliminated by a threshold discriminator.

### A Thickness Measurement

A film thickness measurement can best be illustrated by use of figure 5, which shows the four rotatable collimator and shutter positions 1 through 4. Three separate counts are required for each thickness measurement. A fourth count, obtained by monitor configuration 1 as shown in figure 5(a), is the isolated counter background count. This count,  $I_1$ , is independent of the cadmium-109 or tritium sources and, therefore, this measurement is not necessary for every thickness determination. The background count is a function of the gamma activity of the surrounding accelerator environment. Thickness measurements are usually made several hours after an accelerator run when the residual background activity is at a relatively constant value. One  $I_1$  measurement can then be made prior to a series of thickness measurements.

Figure 5(b) shows monitor configuration 2 which is used for measurement of the tritium caused background intensity  $I_2$ . Note that the source is isolated so that only tritium caused X-rays are counted.

Figure 5(c) shows monitor configuration 3 which is used for measurement of count rate  $I_3$ . The rotatable shutter, when positioned beneath the source and counter, provides a copper reference surface for generation of 8 keV X-rays. The 8 keV X-ray source intensity,  $I_{OS}$ , due to the cadmium-109 source is then calculated from the equation:

$$I_{OS} = I_3 - I_1 \quad (1)$$

Source intensity  $I_{OS}$  is related to the intensity of X-rays from the uncoated copper target cylinder,  $I_{OT}$ , by a constant factor of 1.46. This factor is a function of the different source-counter geometry involved for the intensity measurements of  $I_{OS}$  and  $I_{OT}$ . The factor was measured as a ratio of  $I_{OS}/I_{OT}$  for the uncoated copper HYNT cylinder and copper reference shutter of the monitor.

Finally, figure 5(d) shows monitor configuration 4 for obtaining a count rate  $I_4$ . Substraction of the tritium caused background  $I_2$  from  $I_4$  results in the number of 8 keV X-rays transmitted through the film,  $I_X$ , where:

$$I_X = I_4 - I_2 \quad (2)$$

The fraction of 8 keV X-rays transmitted through a titanium tritide film is:

$$\left. \begin{aligned} \frac{I_X}{I_{OT}} &= \left( \frac{I_X}{I_{OS}} \right) \left( \frac{I_{OS}}{I_{OT}} \right) \\ \frac{I_X}{I_{OT}} &= \left( \frac{I_X}{I_{OS}} \right) 1.46 \end{aligned} \right\} \quad (3)$$

where

$I_X$  intensity of 8 keV X-rays transmitted through a titanium layer of thickness  $X$

$I_{OT}$  8 keV X-ray intensity from uncoated copper target cylinder

$I_{OS}$  8 keV X-ray intensity from rotatable copper shutter

The fraction of 8 keV X-rays transmitted through the tritide film can be related to the thickness of the film by the exponential equation:

$$\frac{I_X}{I_{OT}} = e^{-kX} \quad (4)$$

where

$k$  the absorption coefficient for the film,  $\text{cm}^{-1}$

$X$  the thickness of the film

Use of the reference shutter count permits determination of both  $I_X$  and  $I_{OT}$  for each thickness measurement. Therefore, changes in counting system amplifier gain, source decay, and other long-term variables are eliminated.

## EVALUATION AND TESTING

### Calibration

The thickness monitor was calibrated for the measurement of titanium thicknesses by use of a 3.45  $\mu\text{m}$  titanium-coated HYNT target cylinder. The cylinder was coated by a radiofrequency sputtering technique. The thickness of the titanium film was measured by an interferometer.

By use of equation (4) of the previous section, a one-point measurement of the intensity ratio,  $I_X/I_{OT}$ , for the 3.45  $\mu\text{m}$  titanium thickness will define a calibration line that can be used for the measurement of other titanium thicknesses. The transmission measurement for  $X = 3.45 \mu\text{m}$  corresponds to an  $I_X/I_{OT}$  ratio of 0.785. The calibration plot of  $I_X/I_{OT}$  as a function

of titanium thickness,  $X$ , is shown in figure 6. The absorption coefficient calculated from equation (4) is  $702 \text{ cm}^{-1}$ . Other titanium film thicknesses are determined by measuring the ratio  $I_X/I_{OT}$  then obtaining the corresponding thickness from figure 6. This figure was also used for the titanium deuteride and titanium tritide film thickness measurements that are done in the next section. Because our  $3.45 \mu\text{m}$  standard calibration film is titanium, our titanium deuteride and titanium tritide thickness measurements are slightly in error. The error, however, is small and can be neglected because the mass absorption coefficient of hydrogen (deuterium or tritium) for 8 keV X-radiation is much smaller, by a factor or more than 1000, than the coefficient for titanium.<sup>4</sup>

#### Target Film Thickness Measurement

The thickness monitor was used to measure titanium deuteride film thicknesses during an experiment with the HYNT. The measurements were made with the monitor attached to the HYNT and the deuteron accelerator beam tube as shown in figure 2. An entire  $3.45 \mu\text{m}$  thick titanium deuteride film was removed in situ, in approximately  $0.1$  to  $0.8 \mu\text{m}$  increments, by a sputtering electrode. Following the removal of each increment, the thickness of deuteride remaining was measured with the thickness monitor by the method explained in "The Thickness Monitor" section and by use of figure 6. Details of the experiment are reported in reference 5.

The thickness of a commercially-made titanium tritide target was also measured. The target was in the form of a  $4.6 \text{ cm}$  diameter disc with a tritium activity of approximately  $0.7 \text{ Ci/cm}^2$ . A special target holder, shown in figure 7, was made to accommodate the flat disc target for the measurement. A thickness of  $3.5 \mu\text{m}$ , by figure 6, was indicated. For this measurement, the tritium caused background intensity,  $I_2$ , was approximately 60 percent of the  $I_{OS}$  intensity.



## DISCUSSION OF COUNTING ERRORS

A 1 mCi (37 M dis/s) cadmium-109 source was used for the above thickness measurements. The  $I_{OS}$  intensity was 3000 counts per minute. This intensity was too low to make an accurate thickness measurement in a practical time period. For example, to resolve a  $0.1 \mu\text{m}$  change in a  $3.5 \mu\text{m}$  thick titanium tritide film to an accuracy of  $\pm 30$  percent requires on the order of 20 hours of counting time. We, therefore, recommend the use of a 100 mCi cadmium-109 source. The area of our cadmium-109 source is about  $1 \text{ cm}^2$ . A 100 mCi source can be electroplated onto this area by using carrier-free cadmium-109.

We performed an error analysis based on our measured absorption coefficient of  $702 \text{ cm}^{-1}$  and an  $I_{OT}$  count of  $10^7$  and determined how accurately an incremental  $\Delta X$  film thickness could be resolved for various titanium tritide total film thicknesses to  $30 \mu\text{m}$ . The statistical standard deviations in the X-ray intensity counts of equations (1) through (3) were used. The calculated percent error is plotted as a function of total film thickness in figure 8. For example, a  $0.2 \mu\text{m}$  change in a  $15 \mu\text{m}$  thick film can be measured to  $\pm 10$  percent, whereas a thicker  $0.5 \mu\text{m}$  increment is required for the same  $\pm 10$  percent accuracy at a  $30 \mu\text{m}$  film thickness. The error associated with the isolated counter background count,  $I_1$ , was assumed to be negligible in comparison with the other counts and was neglected in this analysis.

The 100 mCi source will cause an  $I_{OS}$  count rate of  $3 \times 10^5$  counts per minute. Assuming use of this source to accumulate an  $I_{OT}$  of  $10^7$ , a thickness measurement to the accuracy shown in figure 8 can be made in about 2 hours.

## CONCLUSIONS

The X-ray thickness monitor described herein is capable of measuring the thickness of a titanium tritide film on a copper substrate to a thickness of  $30 \mu\text{m}$ . The monitor is designed for use in a rotating target system that uses very thick titanium tritide films. Its purpose is to measure the thickness of depleted titanium layers removed by a sputtering electrode in the target system.

The thickness measurement can be done in the presence of the intense background of bremsstrahlung and characteristic titanium X-radiation that results from the tritium beta radiation. A measurement can be accomplished in 2 hours with reasonable accuracy. For example, a  $0.2 \mu\text{m}$  change in a  $15 \mu\text{m}$  thick film or a  $0.5 \mu\text{m}$  change in a  $30 \mu\text{m}$  thick film can be measured to an accuracy of  $\pm 10$  percent in that time.

#### REFERENCES

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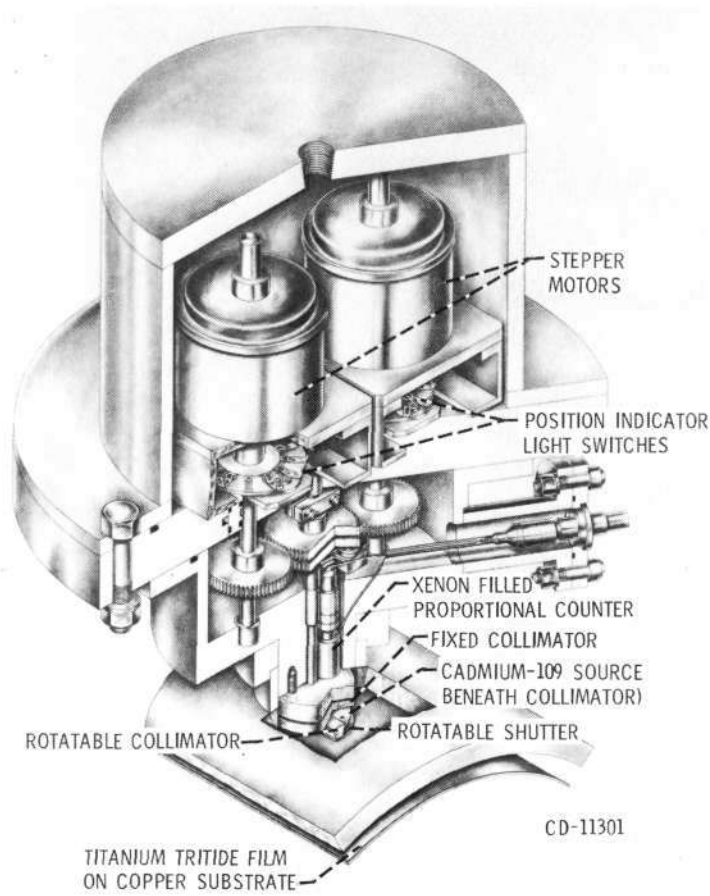


Figure 1. - The X-ray thickness monitor.

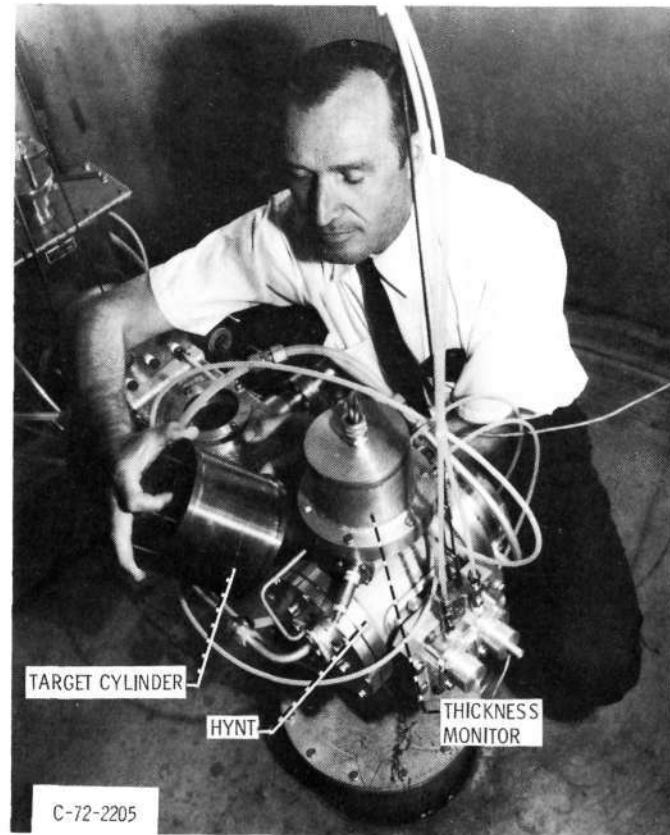
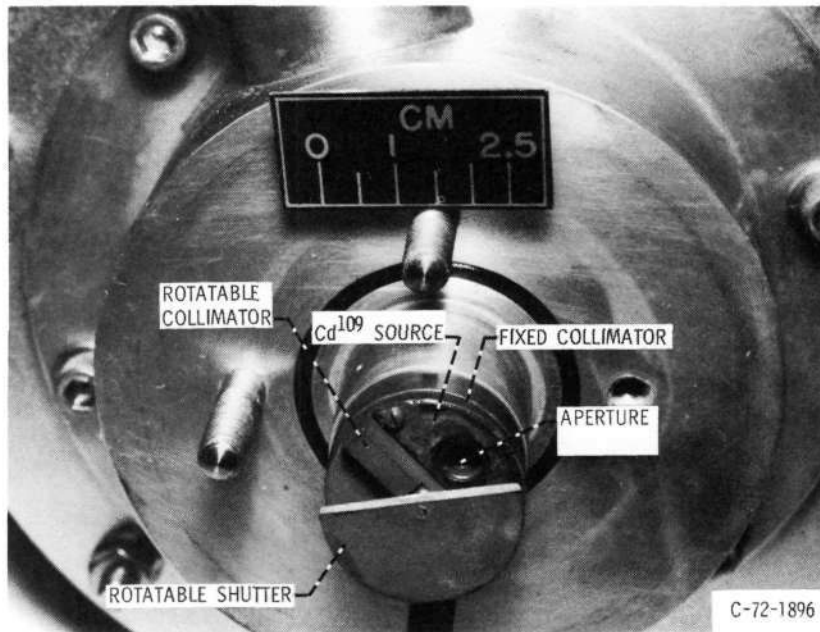
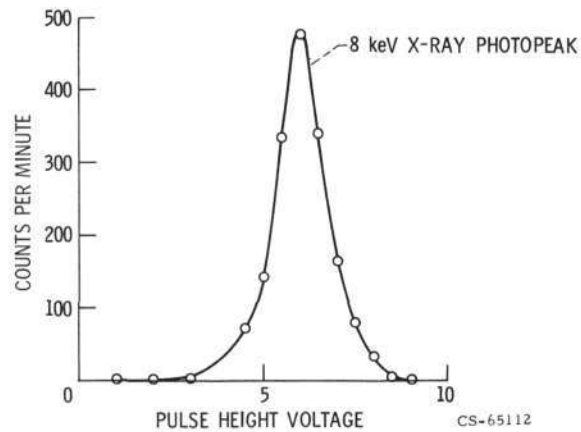


Figure 2. - The HYNT with the thickness monitor attached.



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Figure 3. - X-ray thickness monitor.

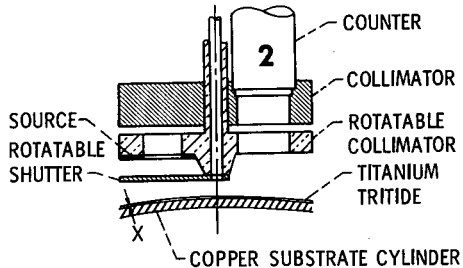
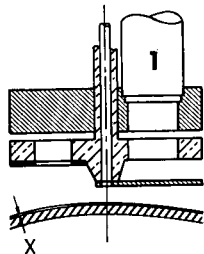


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Figure 4. - Pulse height spectra of X-radiation passing through the 0.005 cm thick filter from a titanium deuteride target with copper substrate. Excitation by 24 keV cadmium -109 X-rays.

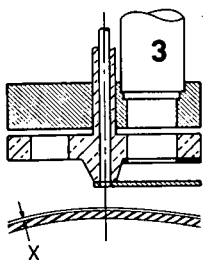
1. ISOLATED COUNTER BACKGROUND

2. TRITIUM CAUSED BACKGROUND

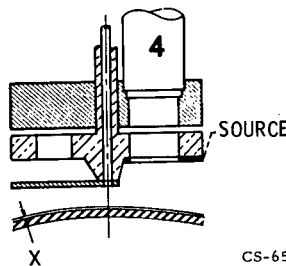


(a) ISOLATED COUNTER BACKGROUND,  $I_1$

(b) TRITIUM CAUSED BACKGROUND,  $I_2$



(c) REFERENCE COUNT,  $I_3$



(d) TOTAL COUNT,  $I_4$

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Figure 5. - The four rotatable collimator and shutter configurations, 1 to 4, of the thickness monitor.

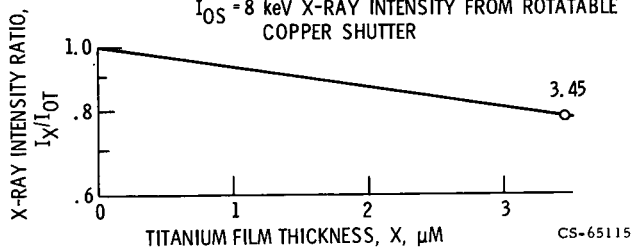
$$\frac{I_X}{I_{OT}} = \left( \frac{I_X}{I_{OS}} \right) \times 1.46$$

WHERE

$I_X$  = INTENSITY OF 8 keV X-RAYS AS ATTENUATED BY A TITANIUM LAYER OF THICKNESS X

$I_{OT}$  = 8 keV X-RAY INTENSITY FROM UNCOATED COPPER TARGET SURFACE

$I_{OS}$  = 8 keV X-RAY INTENSITY FROM ROTATABLE COPPER SHUTTER



CS-65115

Figure 6. - X-ray intensity ratio,  $I_X/I_{OT}$ , as a function of titanium film thickness.

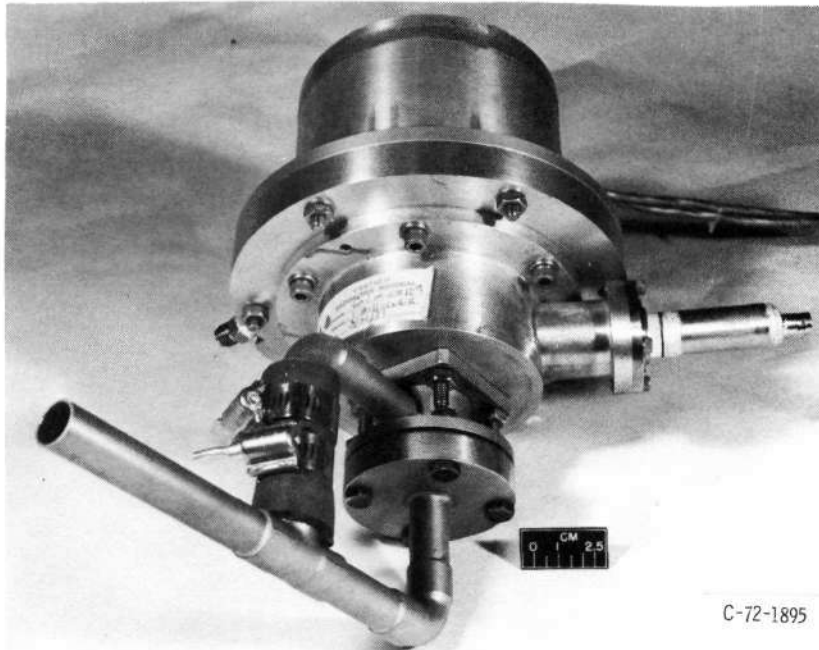


Figure 7. - The thickness monitor with adapter for measuring the thickness of a titanium tritide disc target.

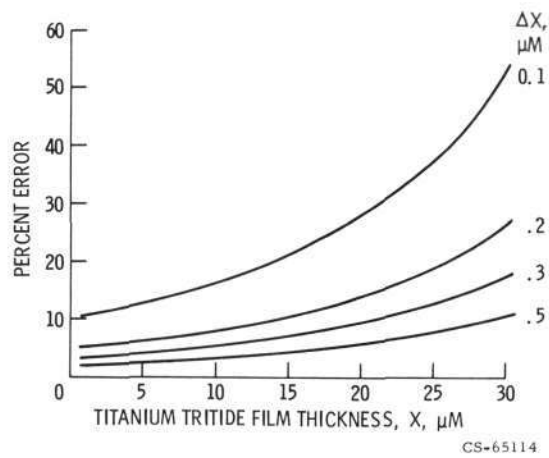


Figure 8. - Percent error in measurement of an incremental film thickness,  $\Delta X$ , as a function of total film thickness for  $I_{0T} = 10^7$  counts.