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# THERMAL DESORPTION ANALYSIS APPLIED TO MATERIALS CHARACTERIZATION

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MASTER

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# INTRODUCTORY REMARKS FOR "THERMAL DESORPTION ANALYSIS APPLIED TO MATERIALS CHARACTERIZATION"

by T.K. Mehrhoff
GEND

(To be presented 10/24/79, GE CRD, Schenectady)

One of GEND's more important products is the neutron generator (Figure 2). This device is a vacuum tube which is capable of producing neutrons. Deuterium ions are produced at the source end by striking an arc between two erbium di-deuteride films. The deuterium ions from the arc plasma are then accelerated to the target end which is composed of a thin film of erbium hydrided with tritium. High energy collisions between deuterium ions and tritium in the target produce neutrons via the reaction:

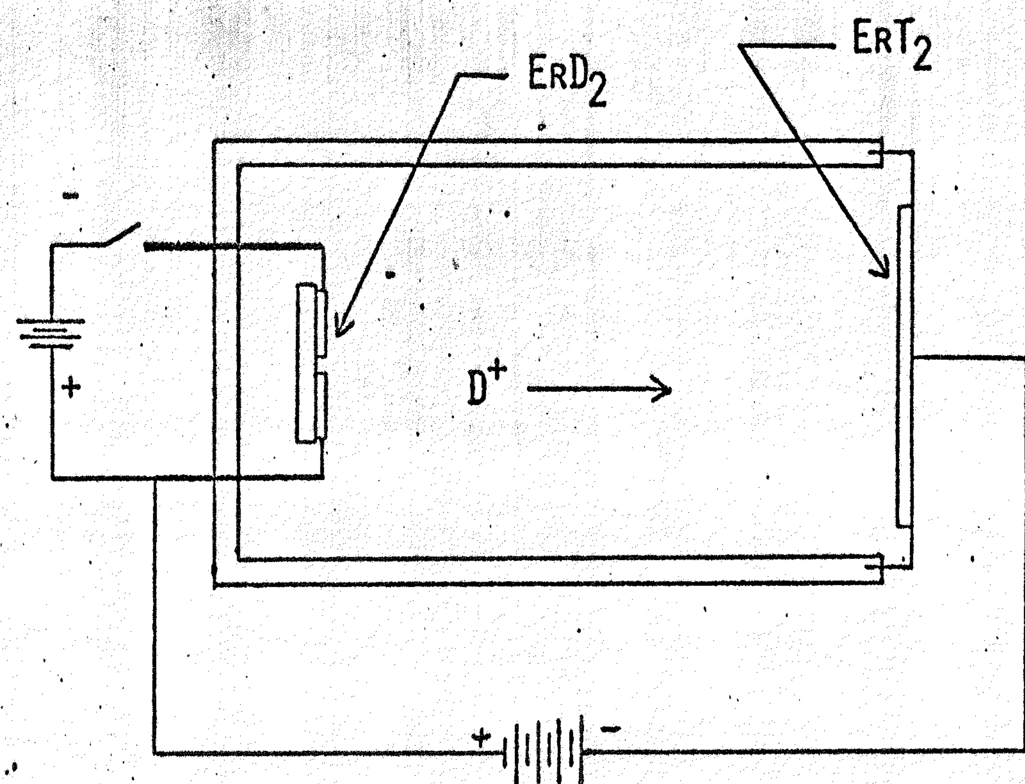
$$D + T \longrightarrow N + \alpha$$

Hydrogen isotopes in the tube must be carefully controlled. Tritium is particularly critical since it will decay to helium-3. The helium-3 will accumulate in the erbium film and release to the tube vacuum via grain boundary diffusion. Loss of vacuum in the tube results in lower neutron output and may eventually lead to high voltage breakdowns during tube operation.

Loss of vacuum in the neutron generator can also be a result of outgassing of materials used in the construction of the vacuum envelope. Thermal desorption analysis is used to characterize the outgassing properties of both vacuum envelope materials and the hydrided sources and targets in neutron generators.

FIGURE 2

$$D + T \longrightarrow He^4 + N$$




TYPICAL NEUTRON GENERATOR

THERMAL DESORPTION ANALYSIS
APPLIED TO MATERIALS CHARACTERIZATION

One of GEND's major products is the neutron generator. The sources and targets for these neutron generators are composed of hydrogen isotope loaded metal hydrides in thin film form on a metallic substrate. Measurements of the hydrogen isotope content of the thin films is an important part of materials characterization analysis for neutron generators. For erbium and scandium occluders the total hydrogen isotope content can be measured by thermally decomposing the hydride at 900°C and examining the evolved gases via high resolution mass spectrometry. A temperature of 600°C is used for titanium occluders. Both the ratio and the quantity of hydrogen isotopes are measured. Hydrogen, itself, is generally considered to be an impurity if present in the film. Ramp heating of the sample using rapid scan low resolution mass spectrometry differentiates hydrogen which comes from the film as opposed to hydrogen which is evolved from the substrate. Gas evolution vs. temperature plots also provide a method for detection of the trihydride phase in erbium samples. The dihydride decomposition pattern is used to detect surface diffusion barriers often associated with heavily oxidized films.

Ramp heating has also been used for detection of reactions between unloaded occluders and reactive gases such as $CO_2$. These measurements are helpful in understanding film degradation processes which occur during hydriding of air exposed targets.

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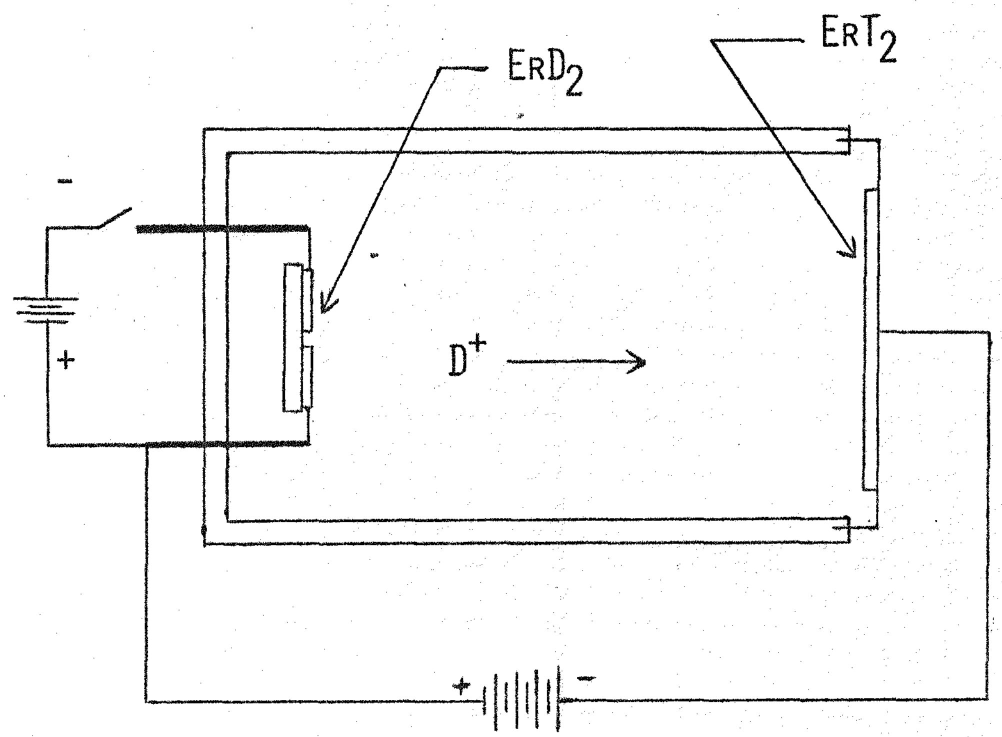
# THERMAL DESORPTION ANALYSIS
# APPLIED TO MATERIALS CHARACTERIZATION

$$D + T \longrightarrow He^4 + N$$




TYPICAL NEUTRON GENERATOR

## SOME GAS-METAL REACTIONS WHICH AFFECT NEUTRON GENERATOR PERFORMANCE

$$ErT_2 \xrightarrow{\text{DECAY}} Er + He^3 + \beta^-$$

$$Er + D_2 \longrightarrow Er - D_2$$

$$Er + CO_2 \longrightarrow Er - CO_2$$

$$Er + CO \longrightarrow Er - CO$$

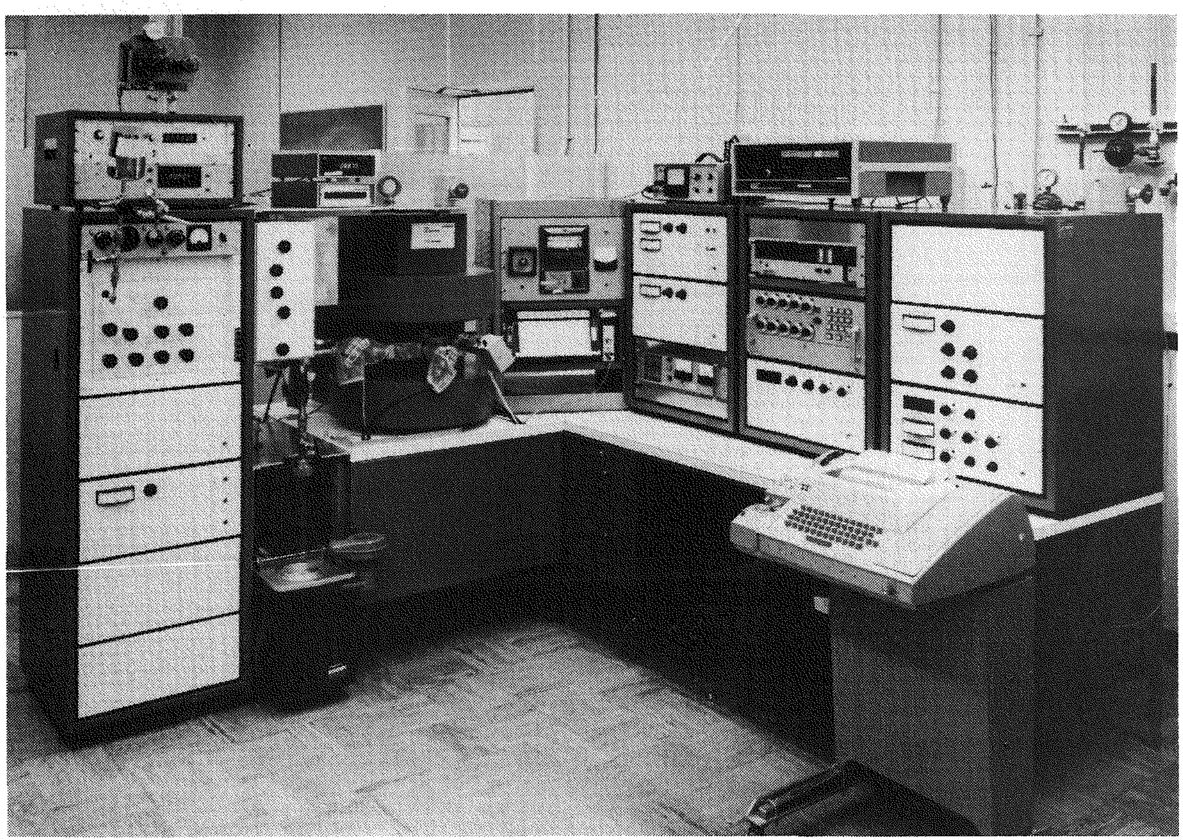
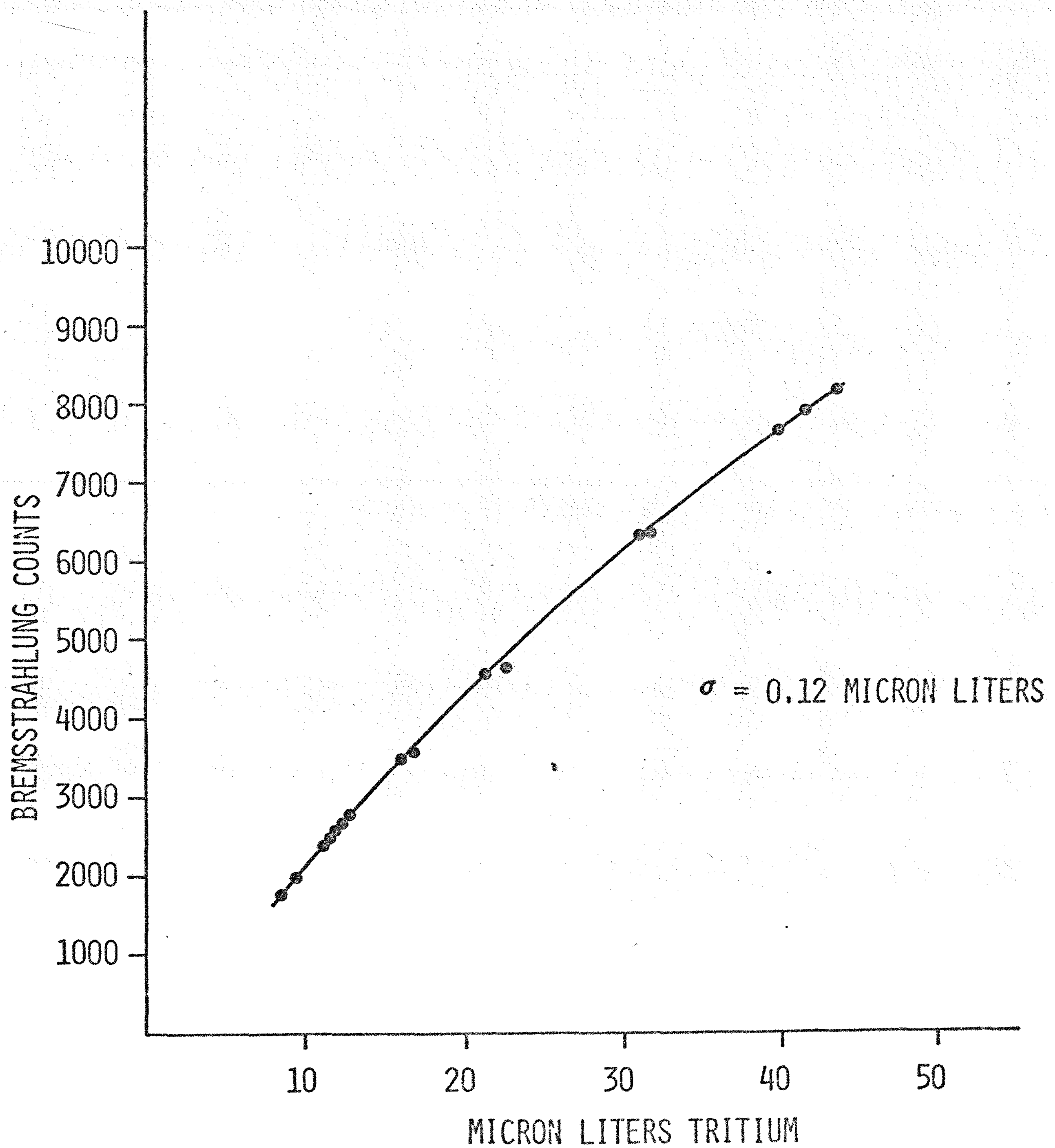
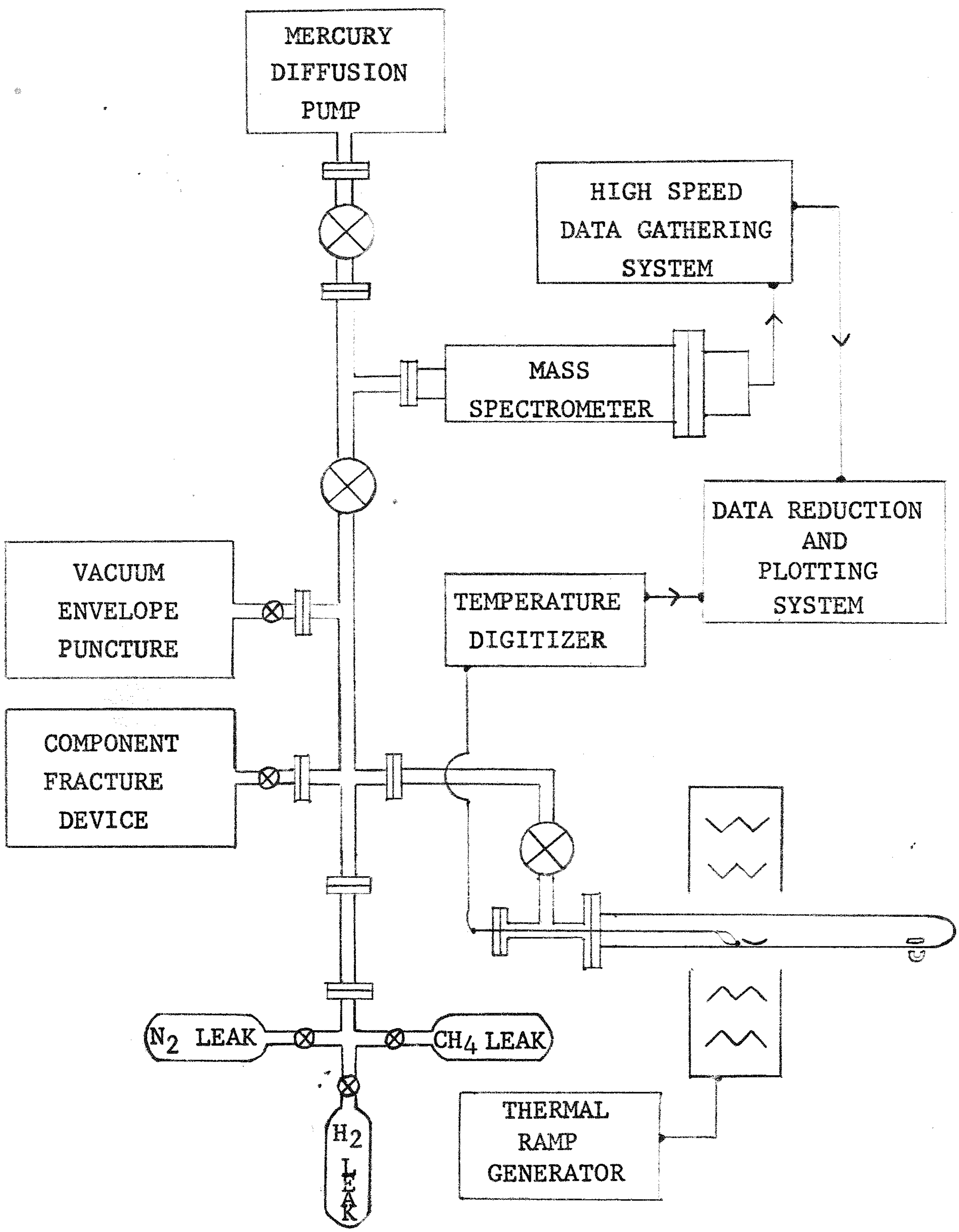


Figure 2. Du Pont 21-104 Mass Spectrometer Modified for Hydrogen Isotope Analyses

ERBIUM FILMS OUTGASSED FOR ONE MINUTE, 15 SECONDS AT 900°C



## VALVING LAYOUT FOR THERMAL DESORPTION ANALYSIS

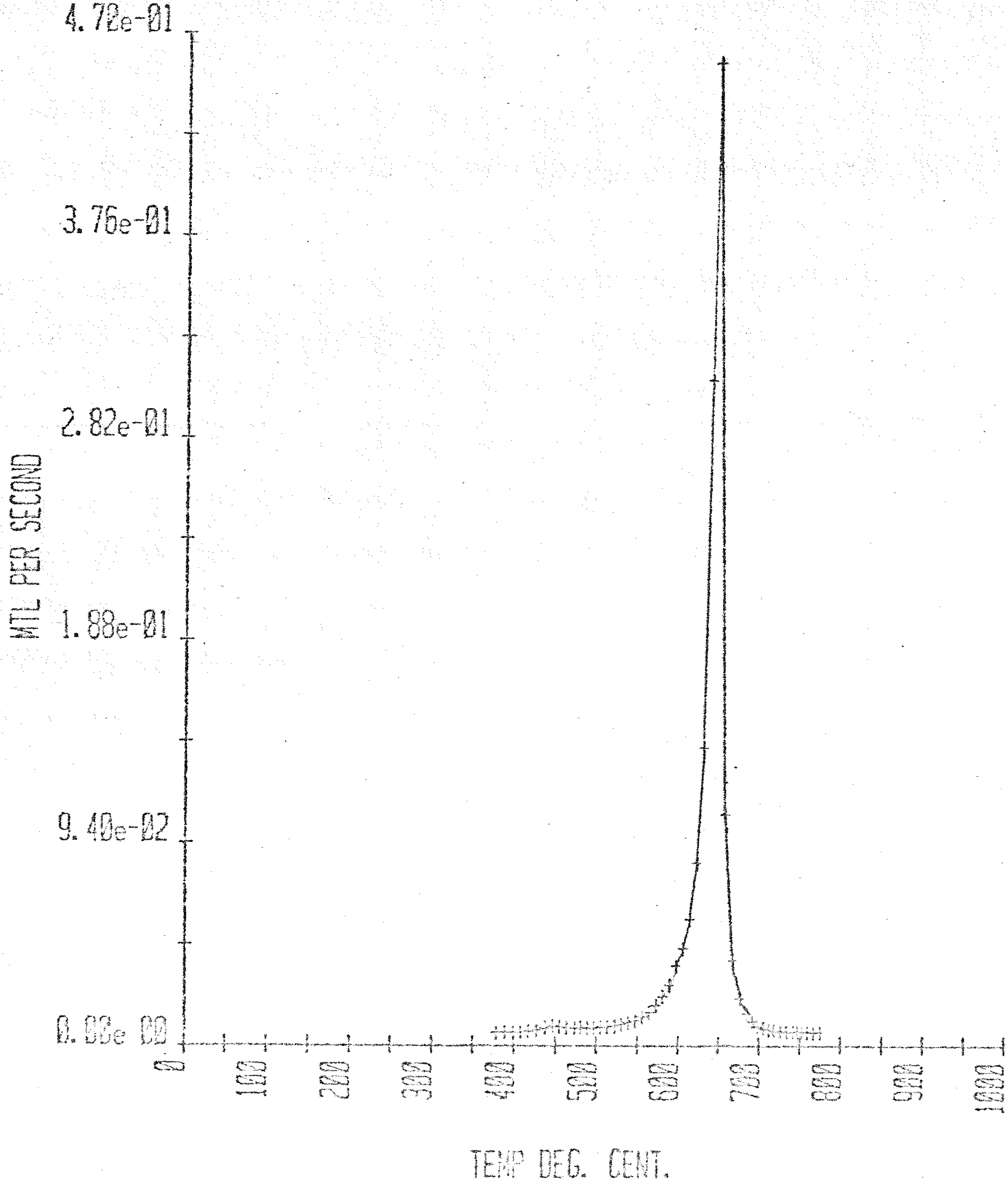


ERBIUM DI-HYDRIDE

DATE: 3- 27-79

IN SITU LOADED

+ MASS 4    1.20e 01 MTL evolved  blank subt.



ERBIUM TRI-HYDRIDE

DATE: 4- 20-79

IN SITU LOADED

+ MASS 4    1.97e 01 MTL evolved

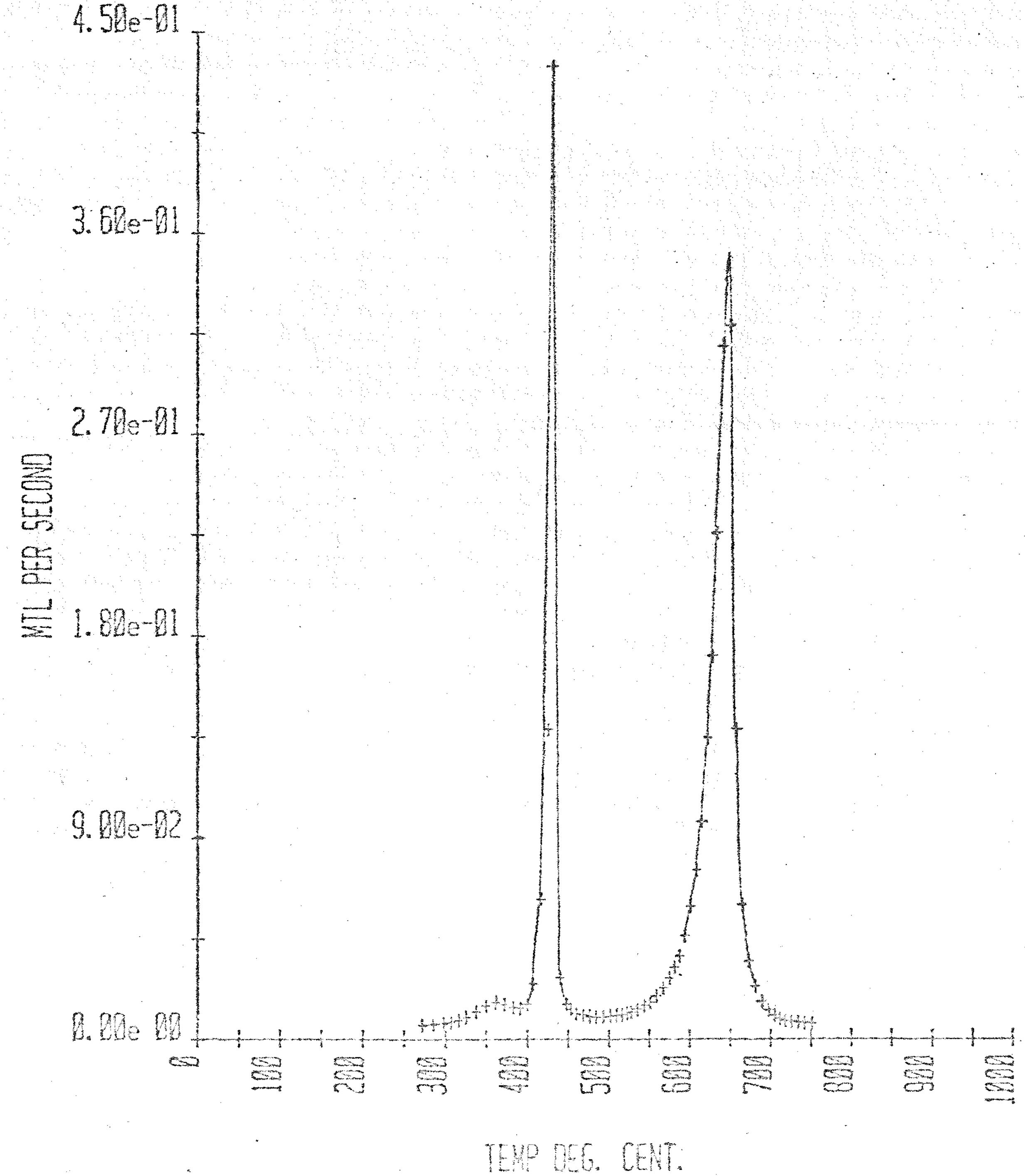


Figure 4

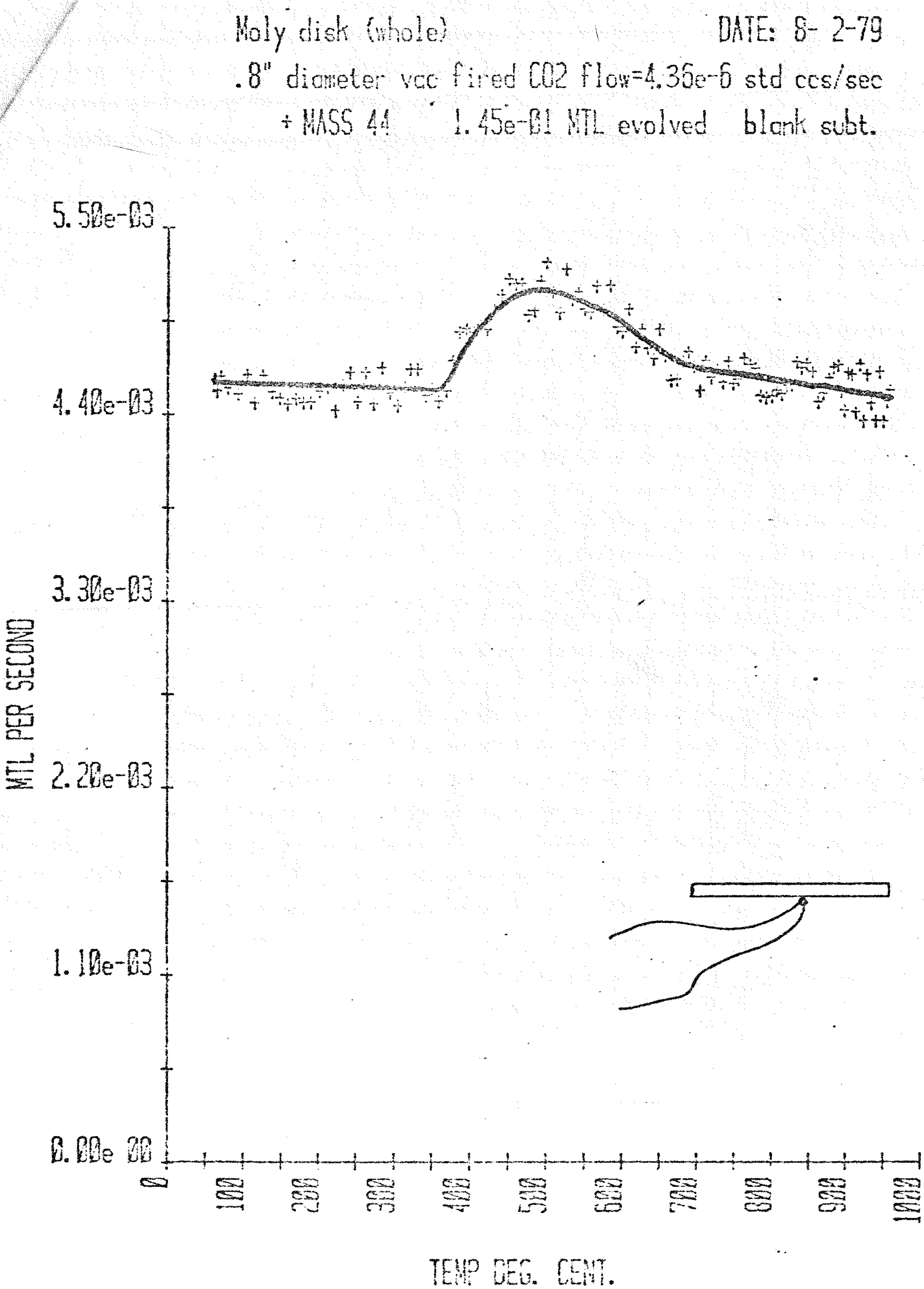

Moly disk (whole)
DATE: 8- 2-79
.8" diameter vcc fired CO2 flow=4.36e-6 std ccs/sec
+ MASS 44
1.45e-01 MTL evolved
blank subt.
5.50e-03
4.40e-03
3.30e-03
2.20e-03
1.10e-03
0.00e 00
MTL PER SECOND
0
100
200
300
400
500
600
700
800
900
1000
TEMP DEG. CENT.

Figure 5

Er/Mo disk .8" diameter DATE: 8- 7-79
CO2 flow = 4.36e-6 ccs/sec
+ MASS 44 5.35e-03 MTL evolved blank subt.

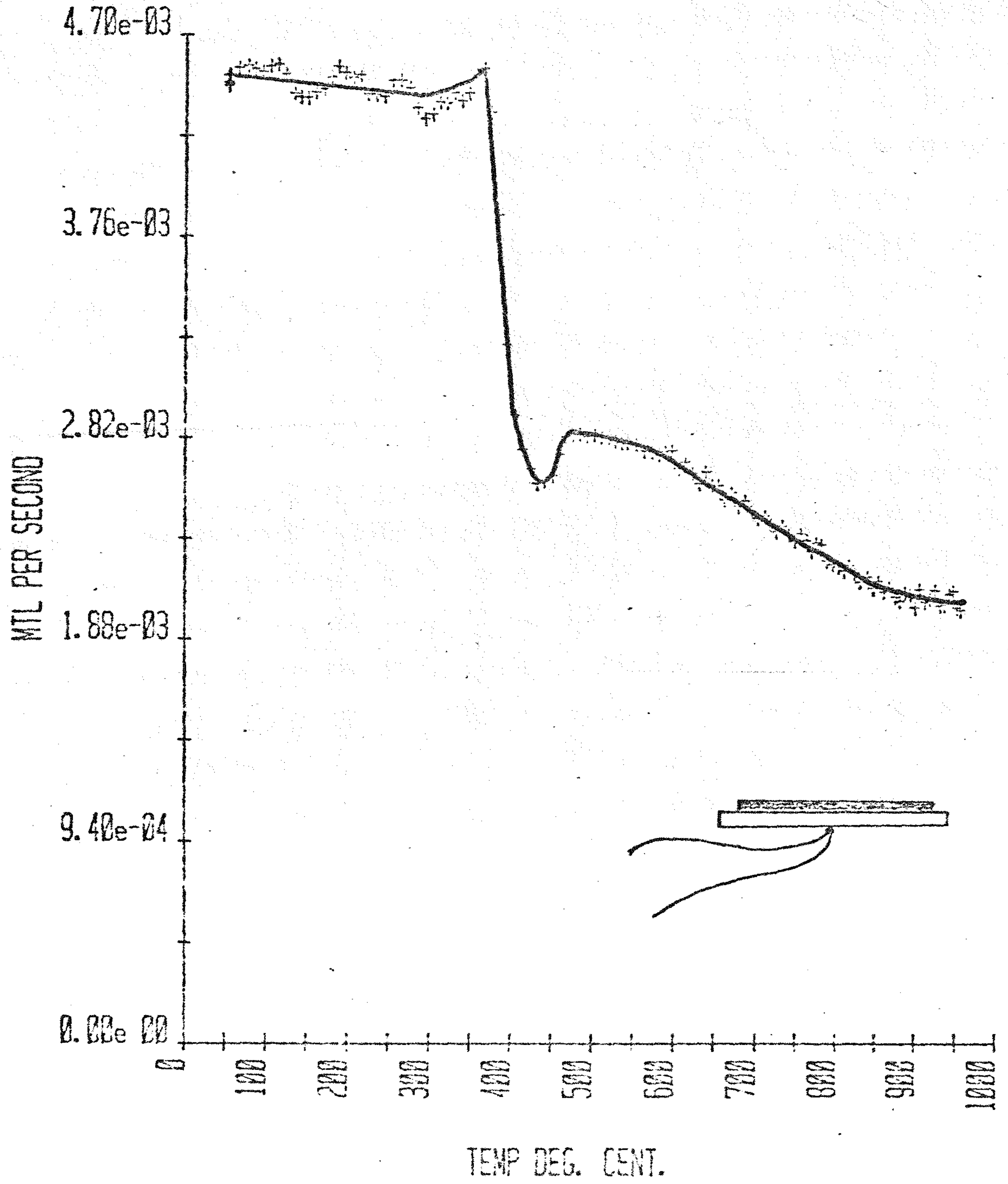
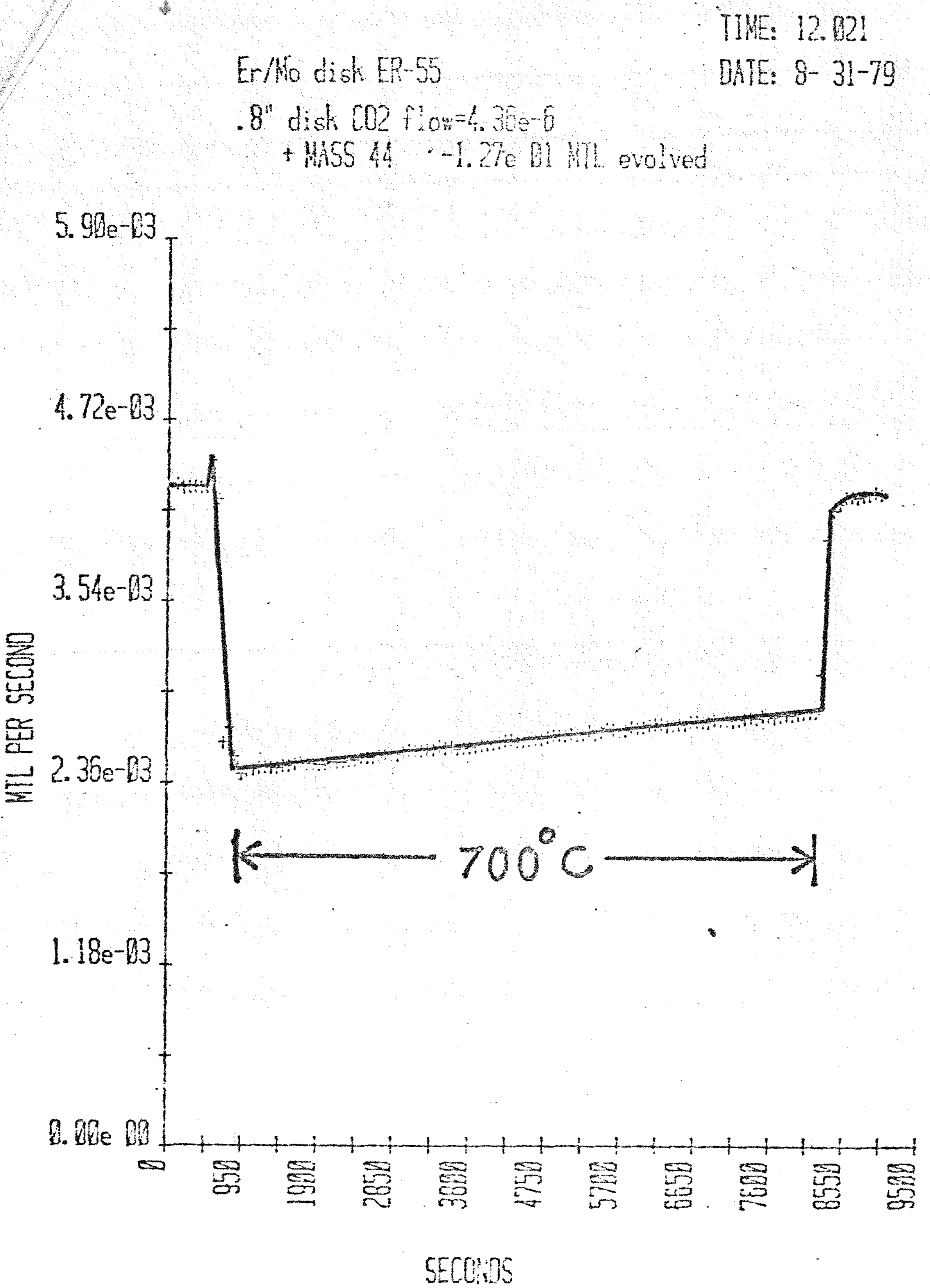
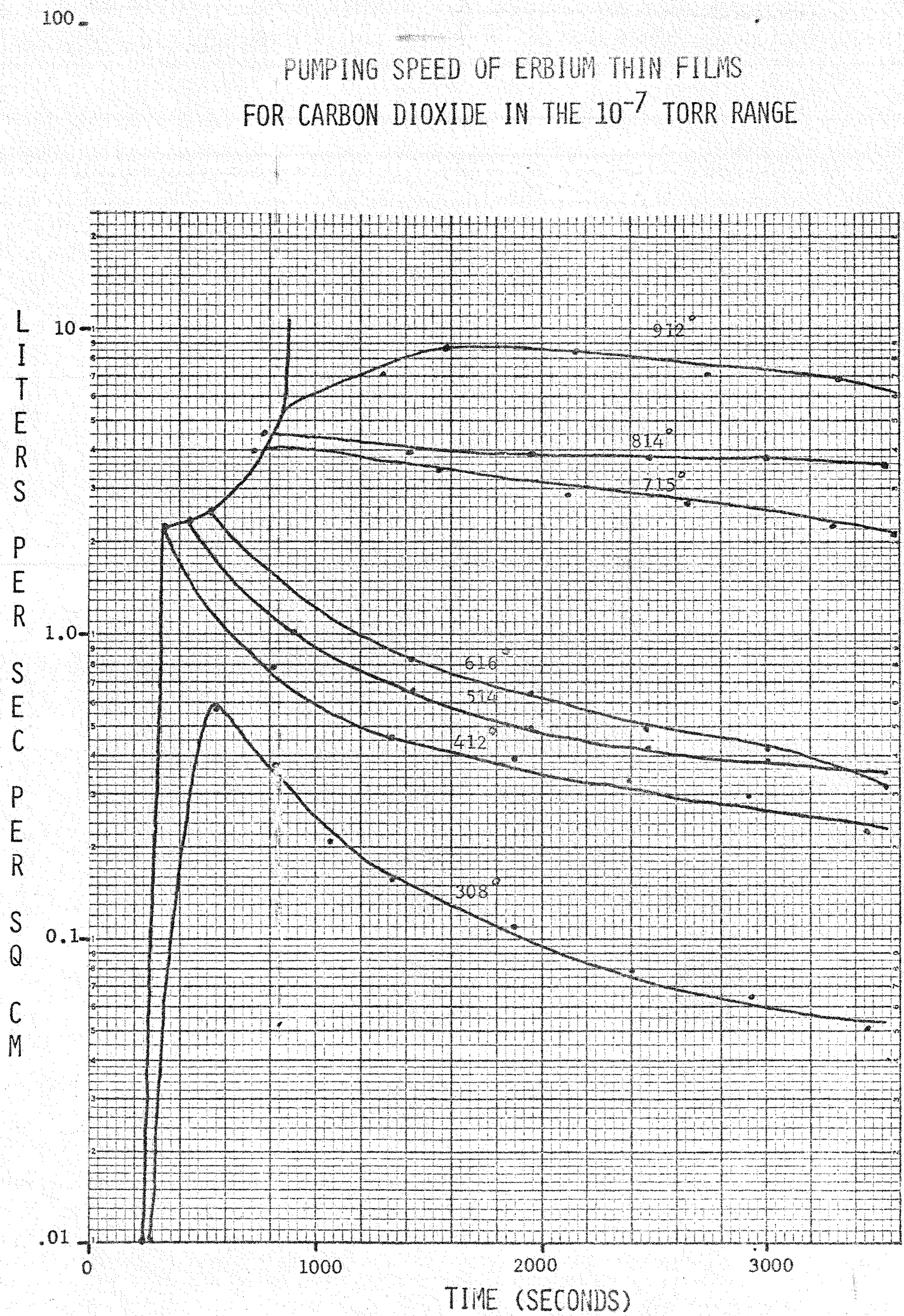


Figure 10



TIME: 12.021
DATE: 8- 31-79
Er/Mo disk ER-55
.8" disk CO2 flow=4.36e-6
+ MASS 44 -1.27e 01 MTL evolved
5.90e-03
4.72e-03
3.54e-03
2.36e-03
1.18e-03
0.00e 00
MTL PER SECOND
0
950
1900
2850
3800
4750
5700
6650
7600
8550
9500
SECONDS
700°C


PUMPING SPEED OF ERBIUM THIN FILMS
FOR CARBON DIOXIDE IN THE 10^-7 TORR RANGE
LITERS PER SEC PER SQ CM
100
10
1.0
0.1
.01
0
1000
2000
3000
TIME (SECONDS)
912°
814°
715°
616°
514°
412°
308°

# CONCLUSIONS

(1) STATIC THERMAL DESORPTION MEASUREMENTS OF TRITIUM IN NEUTRON GENERATOR TARGETS IS NECESSARY TO INSURE EXTENDED SHELF LIFE OPERATION WITH MINIMAL HELIUM-3 RELEASE TO THE TUBE VACUUM.

(2) DYNAMIC THERMAL DESORPTION METHODS ARE USEFUL IN THE FOLLOWING AREAS:

(A) DETERMINATION OF GASES GIVEN OFF BY MATERIALS WITHIN THE TUBE VACUUM ENVELOPE.

(B) CHARACTERIZATION OF GAS-METAL INTERACTIONS WITHIN THE NEUTRON GENERATOR TUBE.

(C) DETECTION OF TRI-HYDRIDE ERBIUM PHASE WHICH MUST BE AVOIDED TO INSURE MINIMAL HELIUM RELEASE.