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AGS DIVISION TECHNICAL NOTE

No. 50

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AERO VAC AVA-1 RESIDUAL GAS ANALYZER OPERATING PROCEDURE AND GENERAL NOTES ON MASS SPECTROMETRY

Operate only if pressure at F-7 is below 1×10^{-4} Torr.

1. Switch on power to recorder, control and remote amplifier.

2. a) Wait for equipment to stabilize (2 minutes) and switch on filament.

   NOTE: When filament is turned on or off, the emission control should first be turned down to a low value.

   b) Set filament current to 8.0 microampere.
   NOTE: This current will change with system pressure changes and should be adjusted as required.

3. Select desired mass range: Low 2-11 (H_2, He)
   High 12-70 (CH-, H_2O, N_2 A)

   NOTE: When switching from one range to another, allow 30 sec for magnet shunt to change position, thus changing ranges.

4. Select Pressure Range: $5 \times 10^{-9/-11}$ Torr
   $5 \times 10^{-7/-9}$ Torr
   (approximate partial pressures may be read directly off chart)

5. To record residual gas spectrum:

   a. Set recorder chart speed to 360 in/hr.
   b. Switch on toggle switch for automatic scan.
   System will continue to rescan until switch is turned off.

6. To monitor a single mass peak, manually set the ion volts control to the proper voltage and start the recorder.

   NOTES 1. This residual gas analyzer can be operated while the AGS is operating, however, the noise pick-up will obscure most of the signal and much of the scan will be lost. It is recommended that the instrument be operated while the AGS is off.

2. The recorder is a three decade log-linear model. Its purpose is to expand the smaller peaks thus increasing sensitivity. This point should be kept in mind when comparing peak heights.

3. To find a particular peak set voltage as follows:

Mass Number	Voltage F-10 Remote	Voltage F-10 Local	Voltage F-20 Local
40	100		140
32	125	110	175
28	140	125	205
20		175	290
19	200		
18	208	200	330
17	220	210	355
16	233	225	380
15		240	410
14	268	260	450
13	290	280	
12	310	310	
4	162		190
3	220		
2	323	315	390

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I. MASS SPECTROSCOPY

The Aero Vac Vacuum Analyzer AVA1 combines the functions of an ionization gauge and a mass spectrometer into one instrument. Electrons emitted thermionically from the filament are accelerated sufficiently to ionize atoms of residual gases present in the vacuum system. The resulting positive ions are accelerated and focused down a tube. The ions then pass through a magnetic field where the amount of deflection depends on the ion velocity. Emerging from the magnetic field, ions of the same mass are focused on one point down the tube, a point which differs from the points of focus for ions of other masses.

Since the ion velocity depends on the accelerating electric field, ions of any mass desired within the range of the instrument can be focused on the collector at the end of the tube and measured as an electric current. If the accelerating voltage is increased linearly, the collector current will vary according to the amount of ions of decreasing mass number present. For constant magnetic field, the mass specie collected is inversely proportional to the ion accelerating voltage. Consequently, an X-Y or T-Y plot shows mass number varying hyperbolically, not linearly.

In a mass spectrometer, sensitivity and resolution are the principal performance determinants. Sensitivity is the instrument's ability to detect small quantities of gas present and depends on the efficiency with which ions are formed, focused, and collected. Resolution is the effectiveness with which different masses are separated. There are two principal limitations on resolution. One is that the ions have a distribution of velocities prior to acceleration. Consequently, the velocity of all ions of a given mass is not the same when they are deflected and separated by the magnetic field. However, the greater the acceleration by the electric field, the smaller will be the percentage variation in velocities of ions of a given mass. The other limitation is that the percentage separation by mass is much larger for low mass numbers than high ones. Consequently, resolution falls off at high mass numbers. In addition, some atoms may be doubly ionized and experience twice the acceleration of singly ionized atoms of the same mass number. Some particles will be singly-ionized molecules (e.g. N_2) and others singly-ionized atoms (e.g., N). These effects are not serious but must be considered in interpreting mass spectra.

Interpretation of mass spectra requires experience, but is facilitated by familiarity with the patterns produced by common residual gases. Typical spectra are included in the Appendix.

MASS SPECIE IDENTIFICATION

The following table indicates typical gas residuals associated with appearance of mass specie in Vacuum Analyzer spectra. Determination of which particular gas is the cause of an indicated residual depends on information as to system history, applied leaks, type of test specimens, etc. In some cases, gas residuals not listed may be present.

Mass Number	Typical Gas Residual
2	Hydrogen (H_2^+)
4	Helium (He^+)
12	Carbon (C^+)
13	Radical (CH^+)
14	Nitrogen (N^+)
	Radical (CH_2^+)
15	Radical (CH_3)
16	Oxygen (O^+)
	Methane (CH_4^+), Sulfur (S^+)
17	Radical (OH^+)
18	Water (H_2O^+)
19	Fluorine (F^+), Heavy Water (HDO^+)
20	Argon (Ar^{++}), Neon ($_{22}Ne^+$)
21	
22	Carbon Dioxide (CO_2^{++})
	Neon ($_{22}Ne^+$)
23	Sodium (Na^+)

Mass	Residuals
24	Radical (C_2^+)
25	Radical (C_2H^+)
26	Radical ($C_2H_2^+$)
27	Radical ($C_2H_3^+$)
28	Nitrogen (N_2^+)
	Carbon Monoxide (CO^+)
	Radical ($C_2H_4^+$)
29	Radical ($C_2H_5^+$)
30	Radical ($C_2H_6^+$)
31	Phosphorus (P^+)
32	Oxygen (O_2^+), Sulfur (S^+)
33	
34	
35	Chlorine ($_{35}Cl^+$)
36	
37	Chlorine ($_{37}Cl^+$)
38	
39	Potassium (K^+)
40	Argon (Ar^+), Calcium (Ca^+)
41	
42	
43	
44	Carbon Dioxide (CO_2^+)

Organic compounds and radicals occur throughout the spectrum. Many of these are grouped around multiples of carbon (12-15 AMU, 24-30 AMU, etc.)

Several graphical constructions are useful in identifying the mass numbers of a peak produced by recording a mass spectrum. All methods are dependent on prior identification of at least two peaks, preferably well separated in mass number. Accuracy of the methods decreases as distance from the known peaks increases.

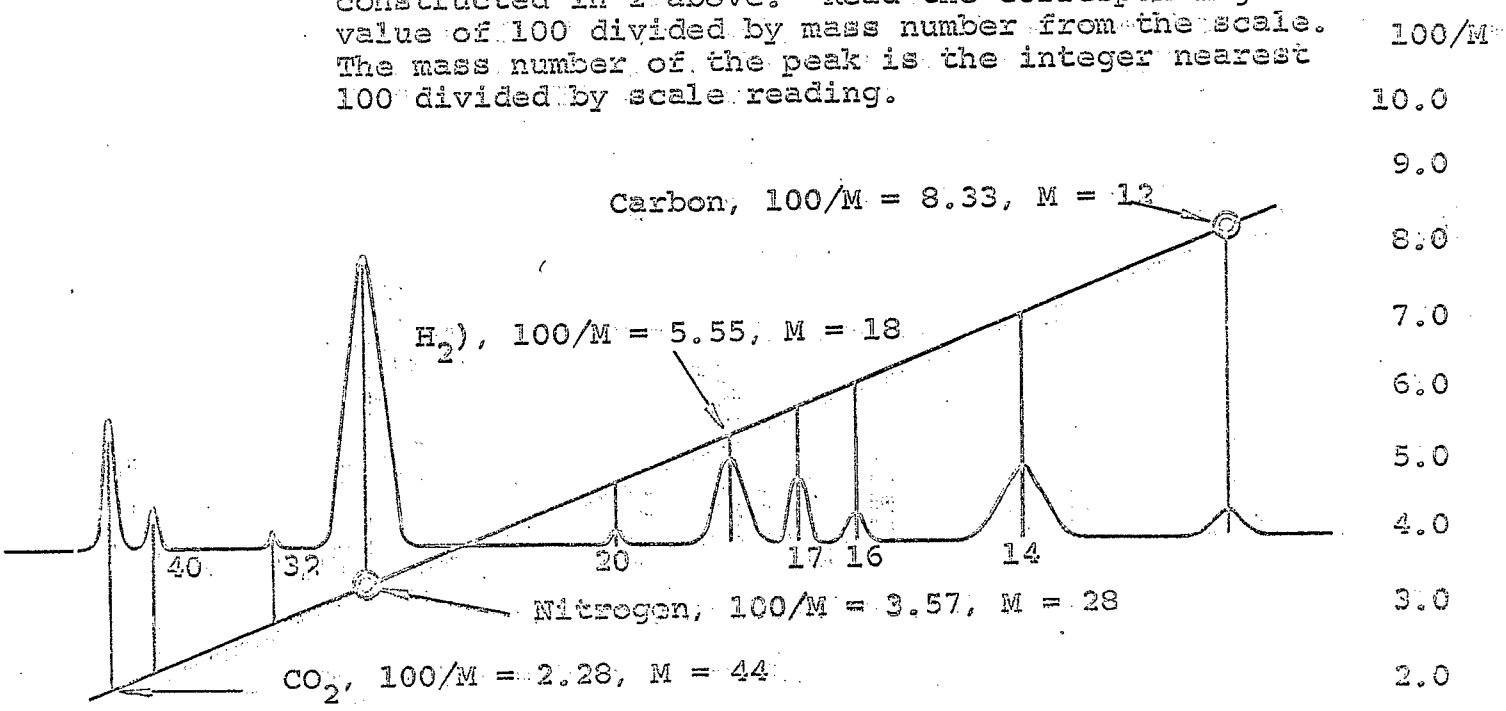
In the method illustrated below, a scale of K divided by mass number is set up along the partial pressure (peak amplitude) axis (K is any convenient constant, e.g., 100) using any convenient scale and zero point. Two or more known peaks are then plotted, determining a line that is approximately straight. All other peaks must correspond with the line. The intersection of the center of the peak with the constructed line gives 100/M read from the scale. The mass number of the peak is then 100 divided by the scale reading.

PROCEDURE:

1. Set up a scale from 0 to 10 on the sheet on which the spectrum is recorded. Zero may be at any point and any convenient interval may be used.

2. (For AMU 2-11 spectra, use 10 divided by mass number in the following. For AMU 12-70 spectra use 100 divided by mass number). Identify two peaks. Calculate 100 divided by the mass number for each peak. Plot the result as a point at the intersection of the axis of the peak and the calculated 100 divided by mass number on the scale set up, for each peak. Draw a straight line through the two points.

3. To identify a peak note the intersection of a line through the center of the peak and the line constructed in 2 above. Read the corresponding value of 100 divided by mass number from the scale. The mass number of the peak is the integer nearest 100 divided by scale reading.



II. CALIBRATION OF AVA-1

(Aero Vac)

1. Reduce system pressure to 1.0×10^{-6} torr or lower.

2. Admit the gas to be calibrated, through a leak, so that the total pressure rises at least one decade (e.g. 1.0×10^{-5} torr).

3. Note the total pressure increase by using an absolute pressure gauge such as a McLeod Gauge.

4. Tune to the peak due to the gas admitted and note the partial pressure increase.

5. Calculate $K = \dfrac{P_{Total}}{P_{Partial}}$

   K is the calibration constant.

6. If, for example, nitrogen was used, the result might be:

$$P_{Total} = 1.0 \times 10^{-5} \text{ torr}$$

$$P_{28} = 1.0 \times 10^{-7} \text{ torr}$$

Then: $$K = \frac{1.0 \times 10^{-5}}{1.0 \times 10^{-7}} = 100.$$

In this example, nitrogen pressure is calculated by multiplying indicated mass 28 partial pressure by 100.

M/e	Residual Air	Nitrogen	CO_2	Argon	Methane	Oxygen	Room Air	Ethane	Water
12			20.0		5.9			3.3	
13					11.8			6.7	
14	25.0	12.8	5.0		23.6	4.9		6.7	
15			5.0		91.5				
16	16.6	1.9	30.0		100.0	19.6	6.3		6.6
17	16.6				5.9				29.3
18	33.3		5.0			4.9	2.1	3.3	100.0
20				27.1					
22			5.0						
25								6.7	
26								23.3	
27								36.7	
28	100.0	100.0	25.0	6.2		34.2	100.0	100.0	
29								23.3	
30								23.3	
32	25.0		5.0	3.1		100.0	23.0		
40				100.0		4.9	4.2		
44		3.9	100.0	6.2		4.9	4.2		
K_{gas}	77.0	85.0	129.0	117.0	61.0	72.0	67.0	68.5	
$\sim K_{N_2}$	.9	1.0	1.5	1.4	.7	.85	.8	.8	

$P_{T_{gas}} = 5 \times 10^{-5}$

$P_{T_i} = 2 \times 10^{-6}$

Per R. Lang AIL

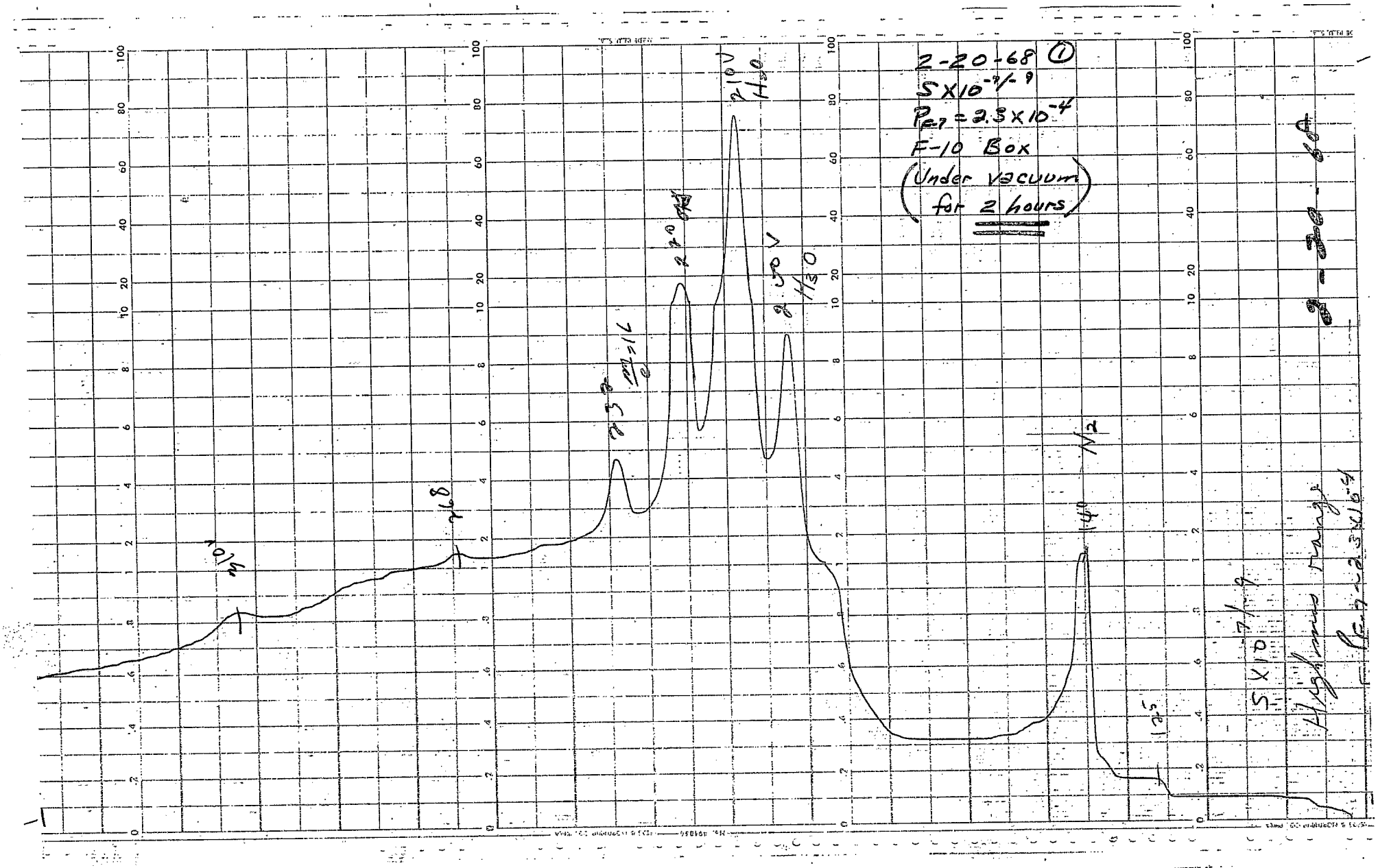


FIG. 1
F-10 Box, under vacuum for 2 hours, high mass range, scale $10^{-7}/^{-9}$

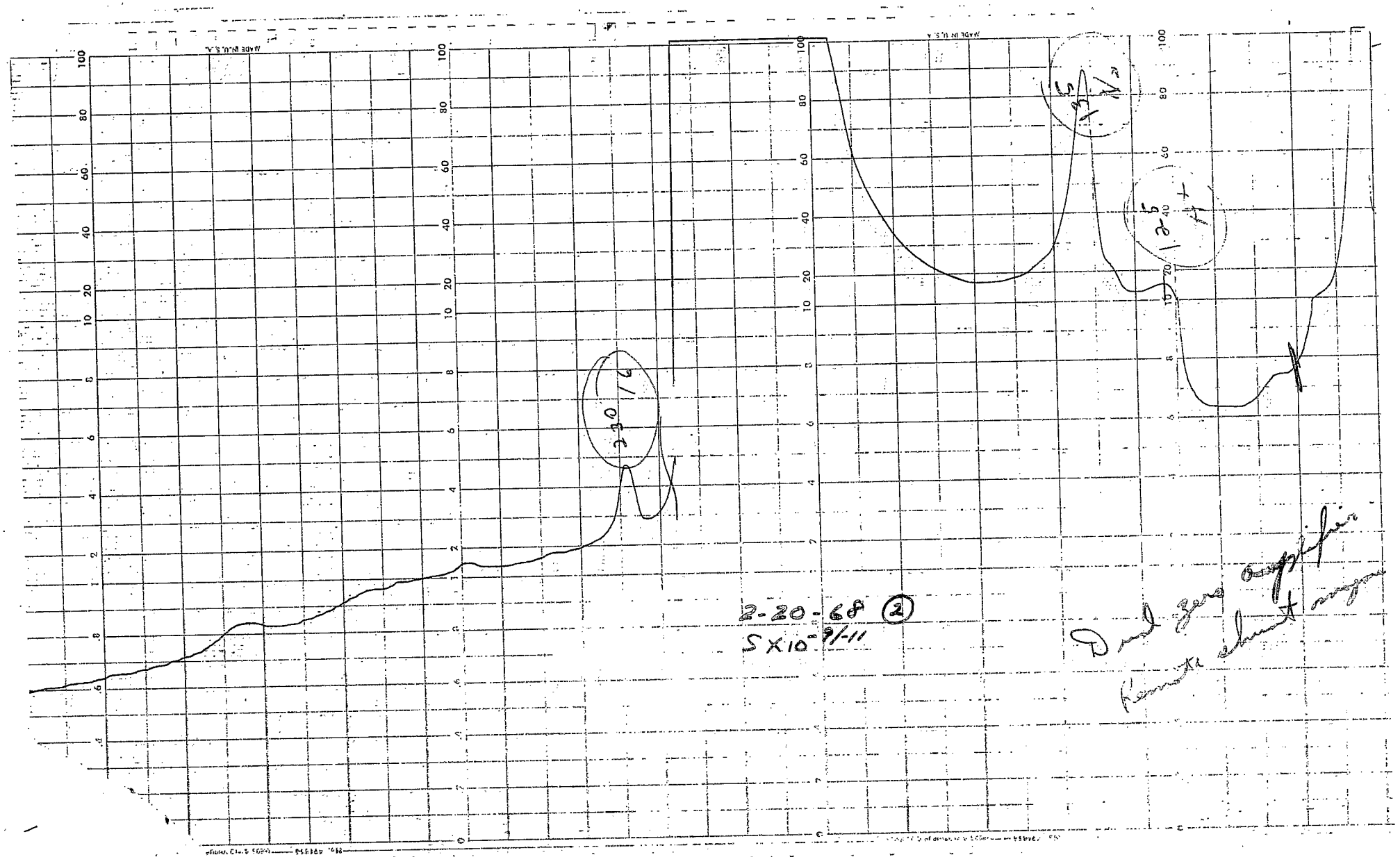


FIG. 2

F-10 Box, under vacuum for 2 hours, high mass range, scale $10^{-9}/-11$

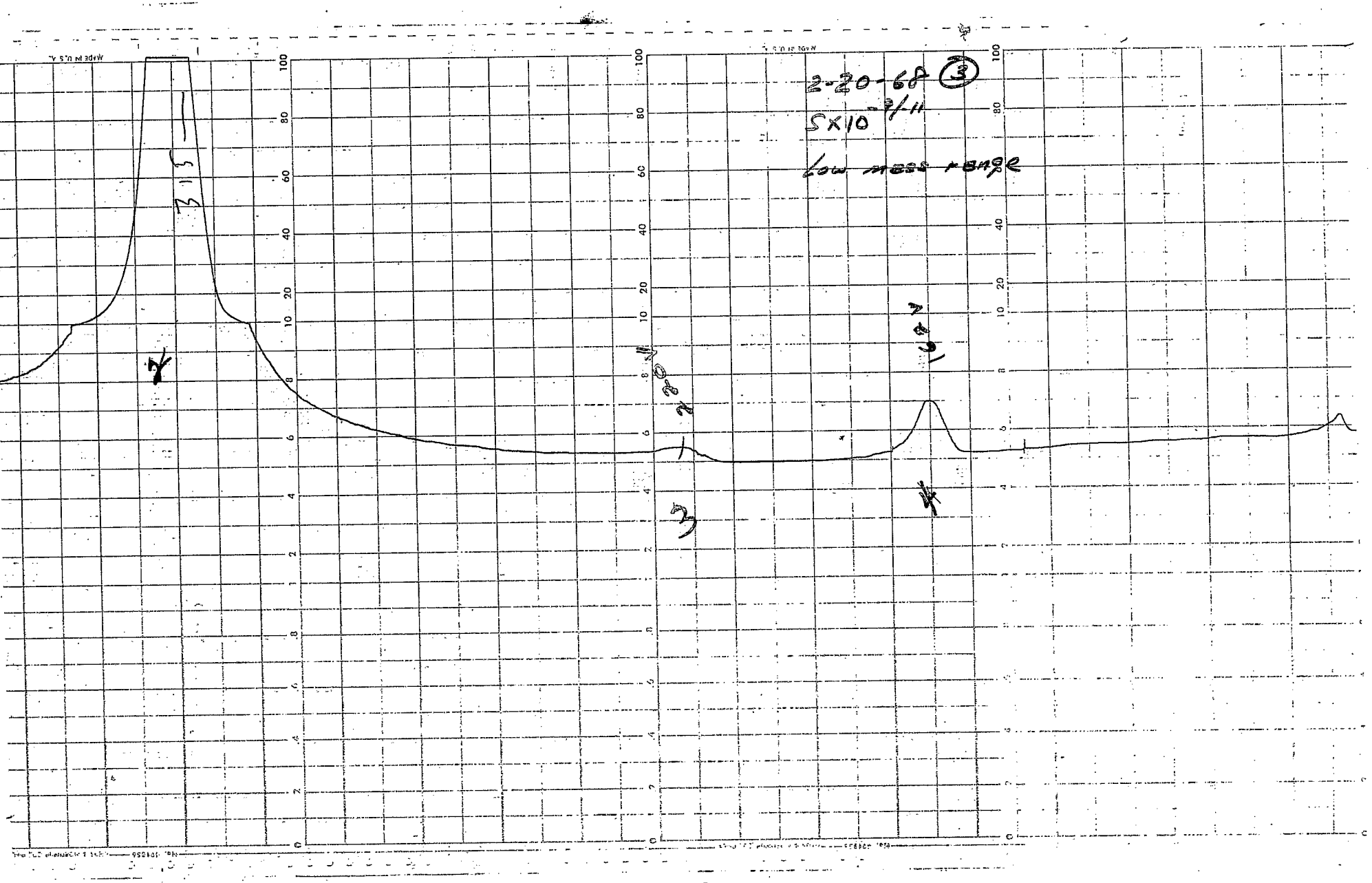


FIGURE 3
F-10 Box under Vacuum for 2 hours, low mass range, scale $10^{-9}/-11$

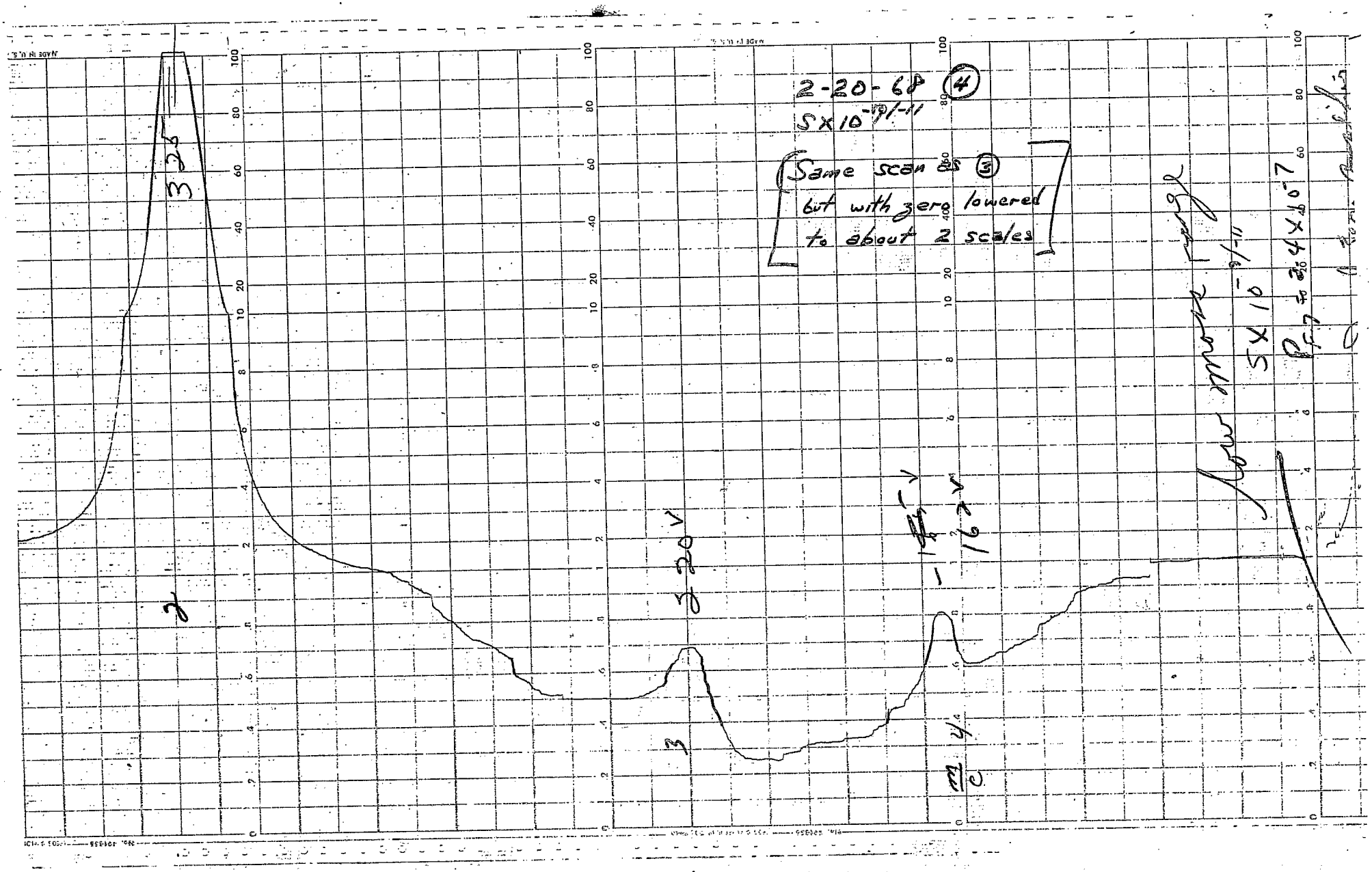


FIGURE 4

Same as Figure 3 with zero lowered.

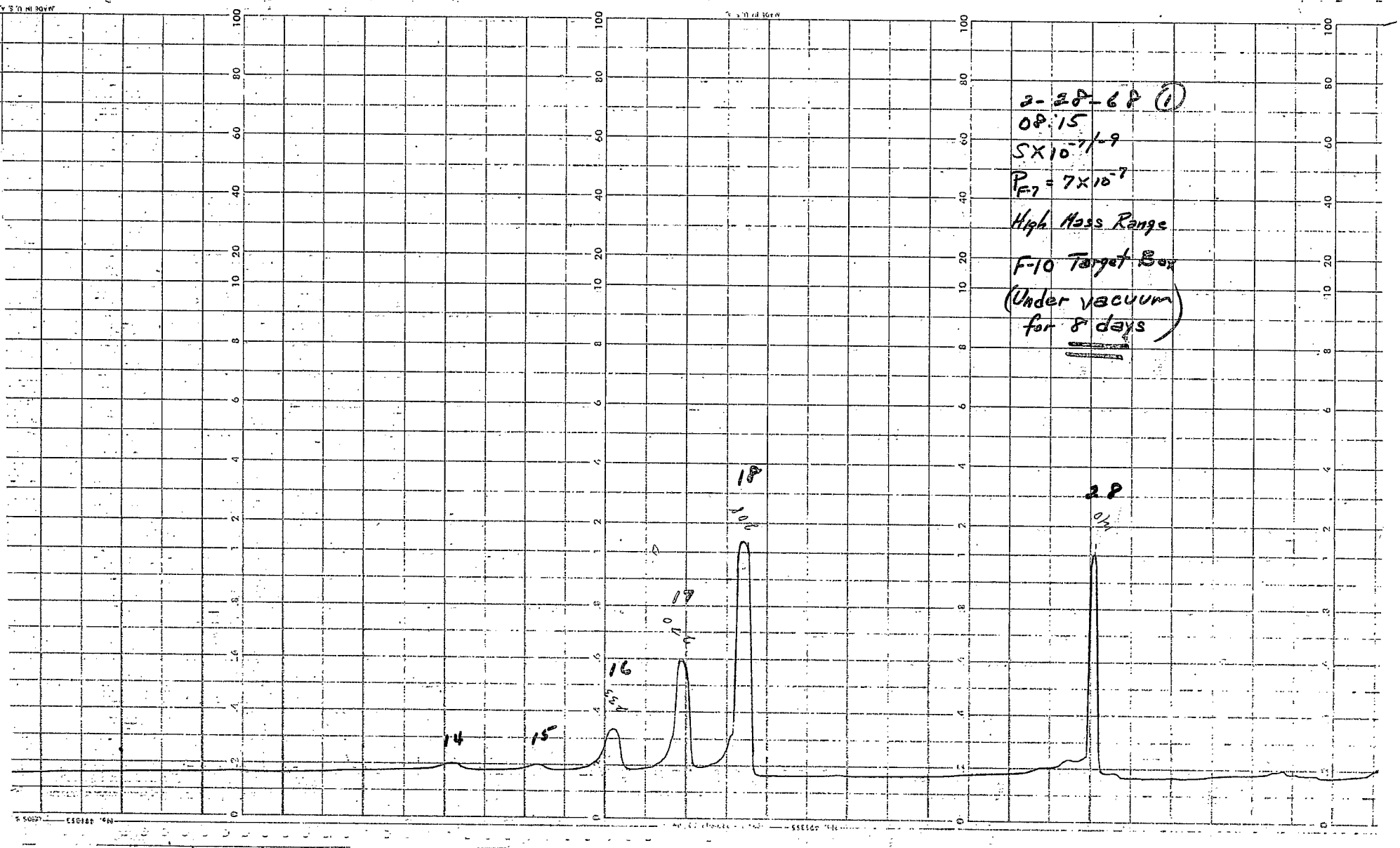


FIG. 5

F-10 Box under Vacuum for 8 days, high mass range, scale $10^{-7}/-9$

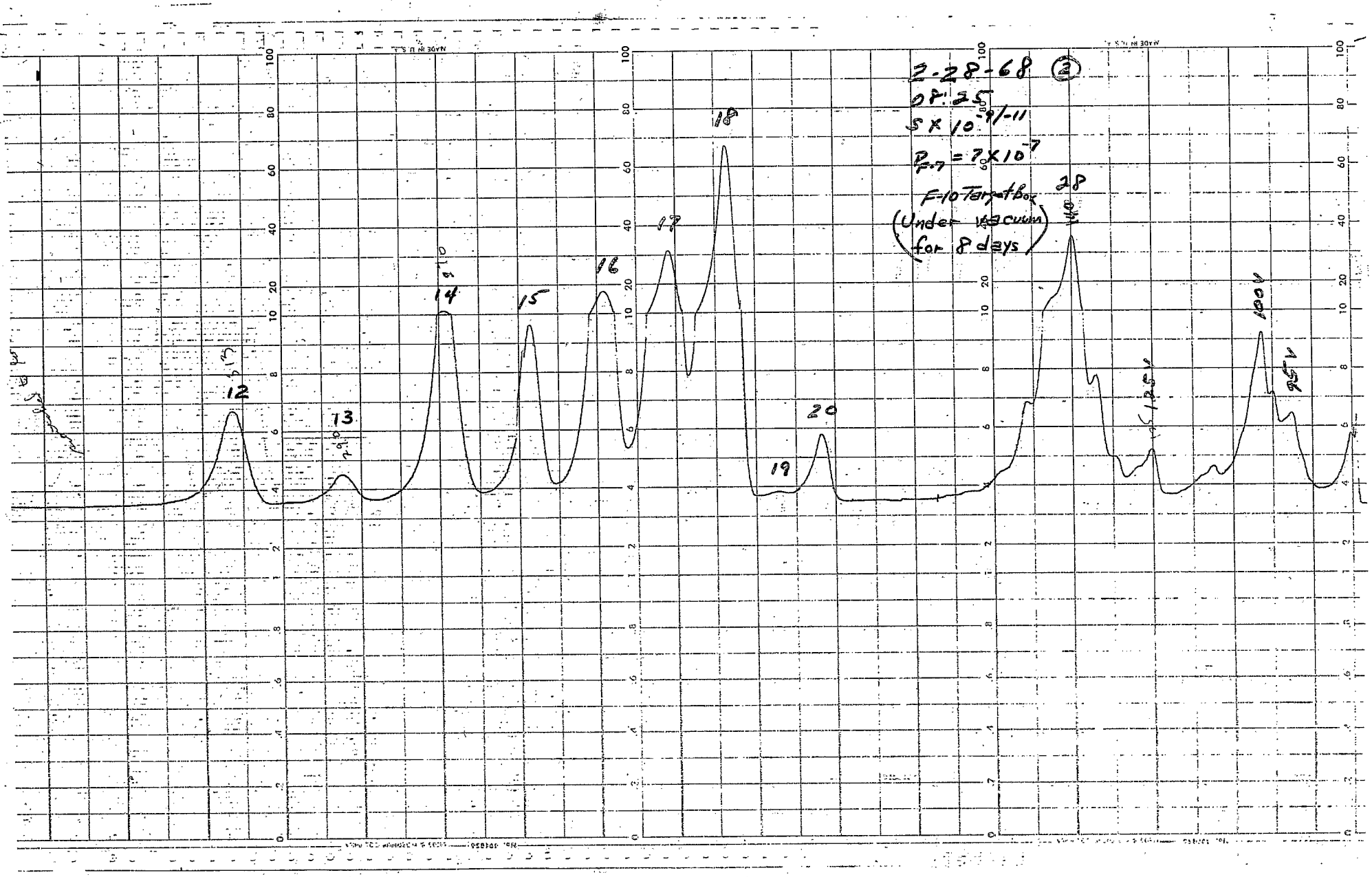


FIG. 6

F-10 Box under vacuum for 8 days, high mass range, scale 10-9/-11

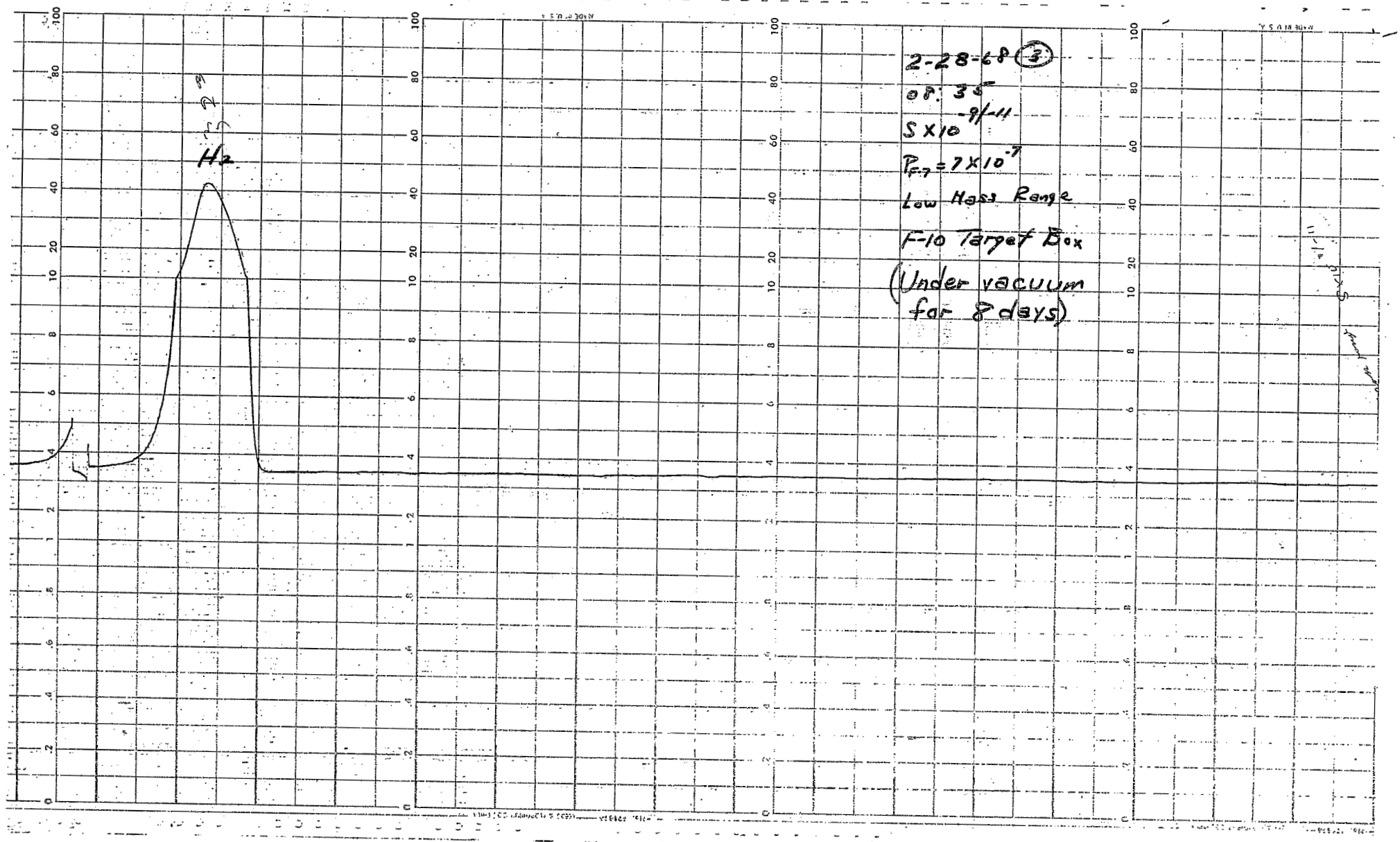


FIG. 7

F-10 Box under Vacuum for 8 days, low mass range, scale $10^{-9}/-11$

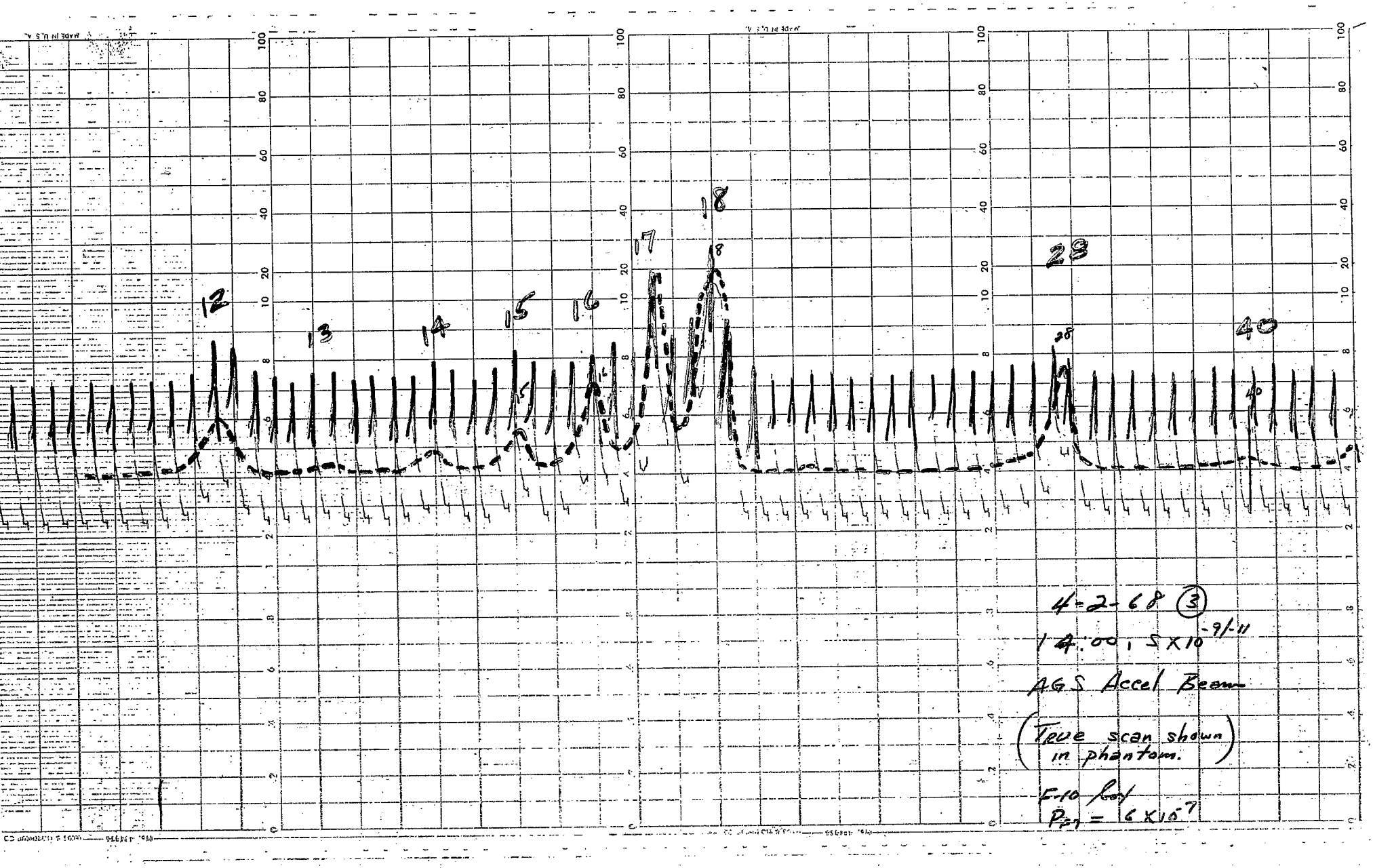


FIGURE 8
F-10 Box, AGS Accelerated Beam

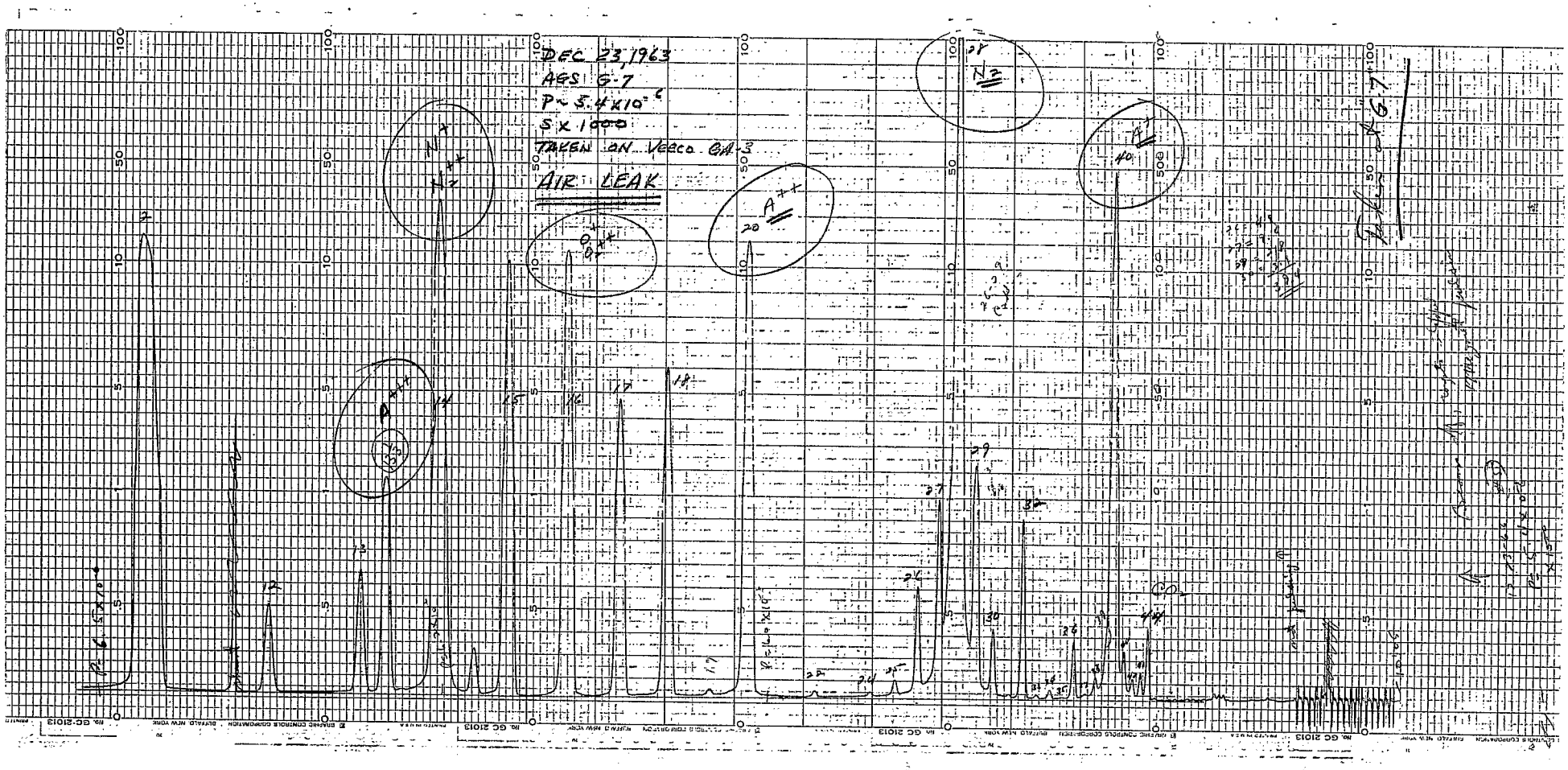


FIGURE 9

Typical Air Leak (taken on Veeco GA-3)

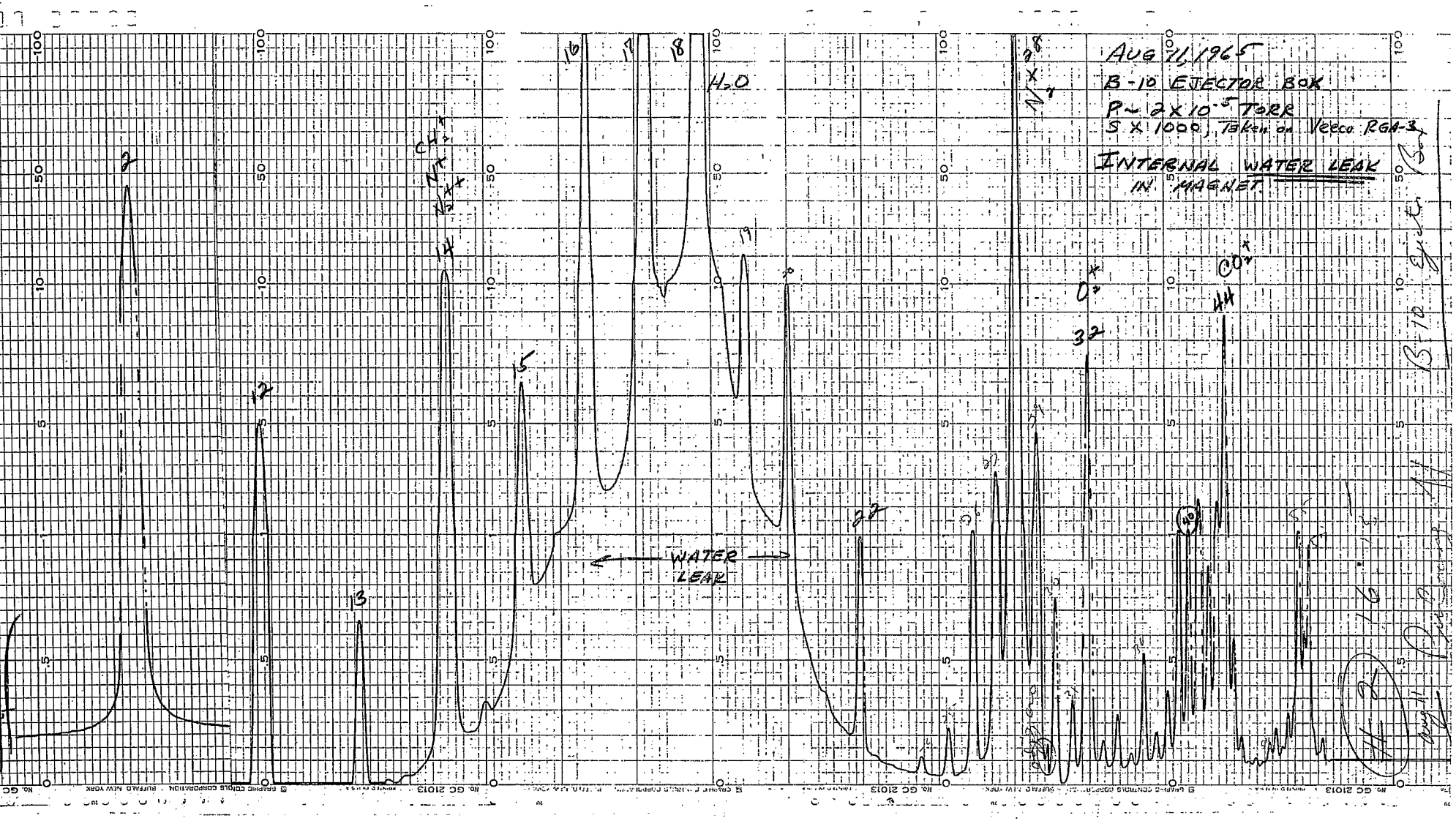


FIGURE 10

Typical Water Leak (taken on Veeco GA-3)

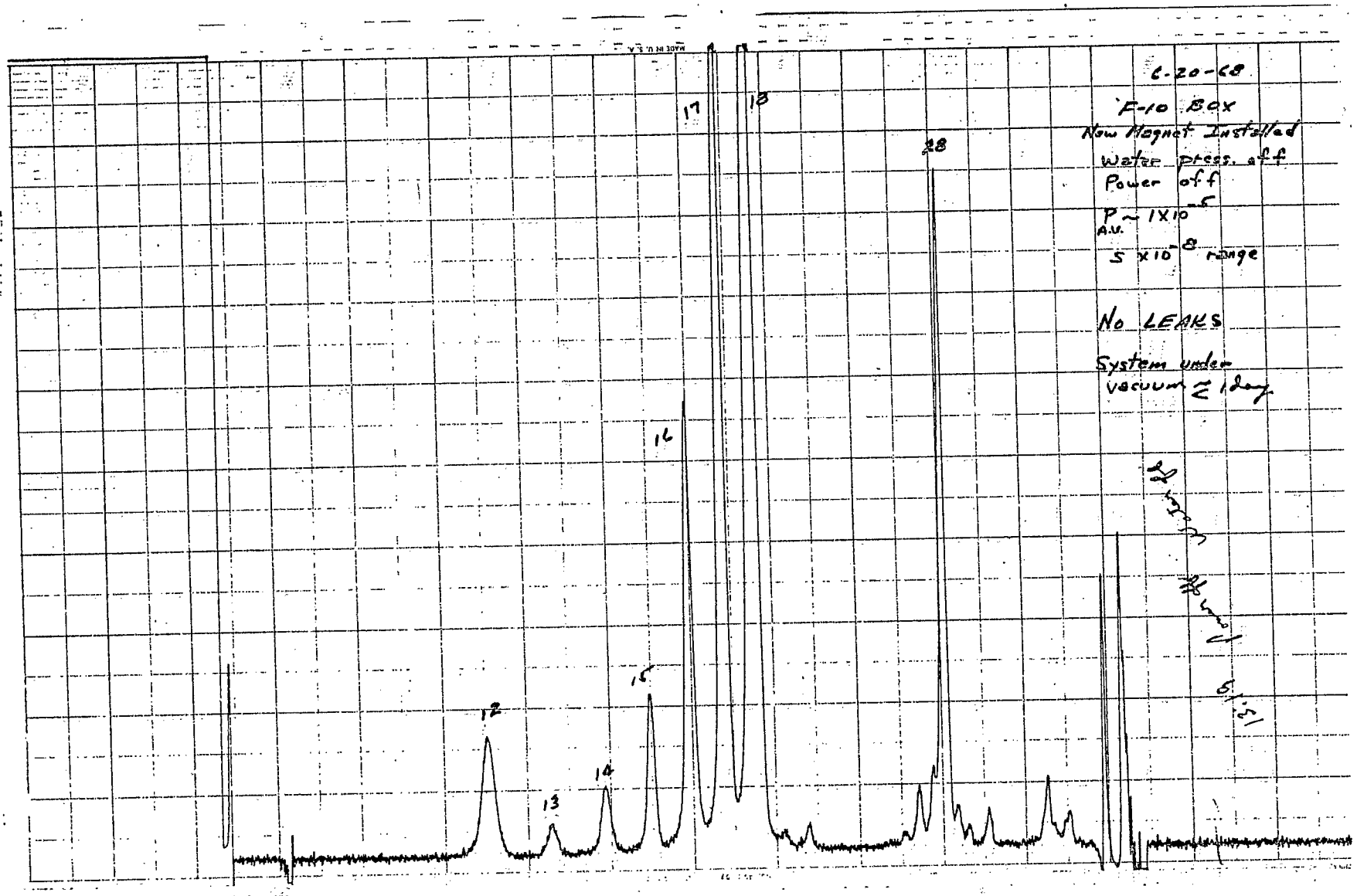


Fig. 11

F-10 Box Typical Scan after One Day of Pumping (linear scale chart paper.)

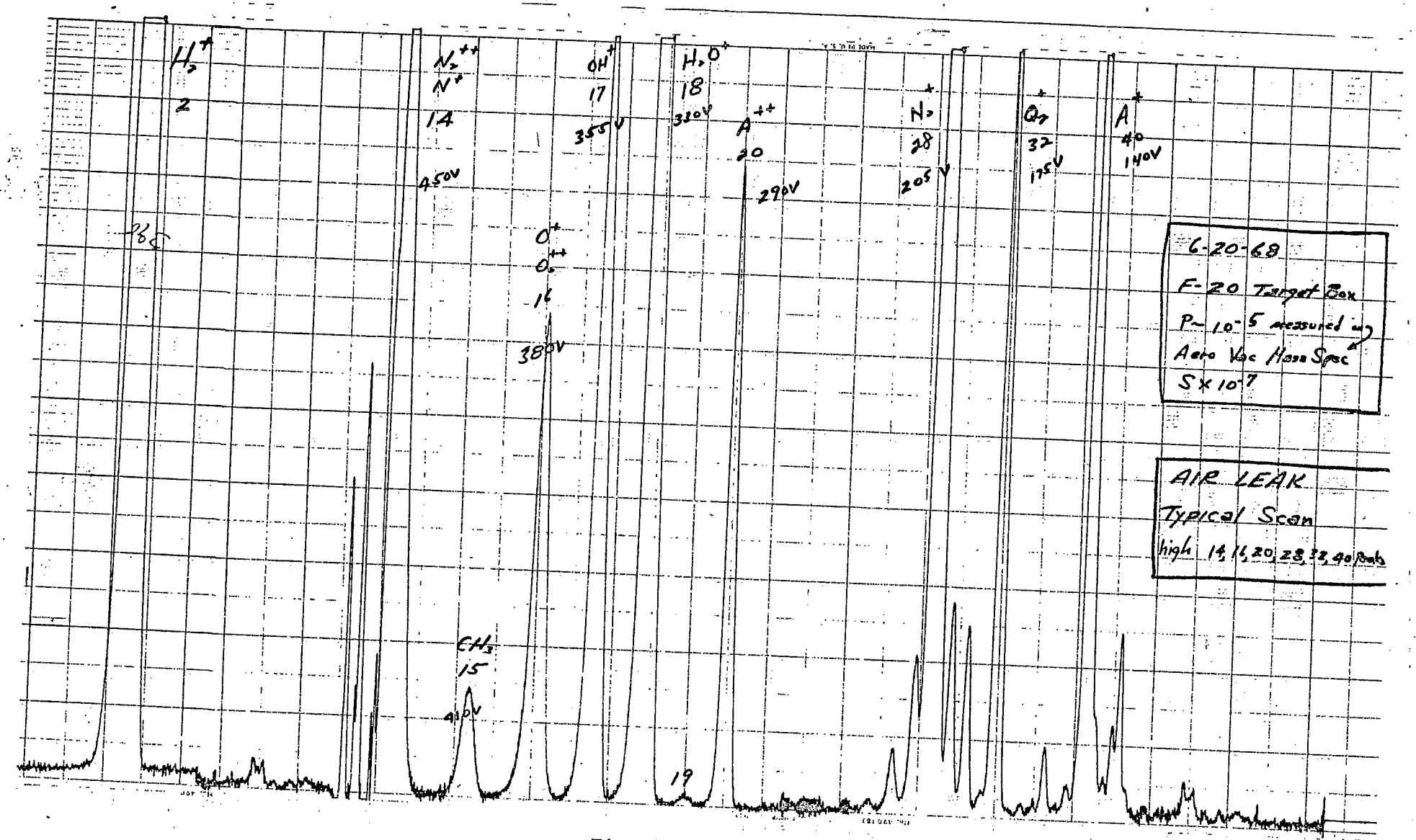


Fig. 12

F-20 Target Area, Air Leak (linear scale chart paper).

IV. Leak Detection

The nature of a vacuum system leak can be determined generally by examining a spectrum obtained as in Section 3, Fig. 9 For example, a large nitrogen signal not accompanied by a significant oxygen signal would indicate a leak in the nitrogen bleed or vent system, whereas the additional presence of oxygen would indicate an atmospheric leak. Failure to pump down to a sufficiently low pressure may be caused by a high outgassing rate. Such a failure may appear to be a leak. A large water (AMU 18) signal, for example, would probably indicate high outgassing of water from paints or other surfaces in the system.

The indication that an atmospheric leak exists is the presence of an air signal (peaks at 32, 28, 16, 14 AMU. See Typical Spectrum - Air in Appendix). Leak location can be determined by probing with a tracer gas. The most common are indicated below, along with the indication as the leak is approached with the probe gas.

PROBE GAS	PEAK OBSERVED	INDICATION
Helium (4)	Helium (4)	Increase
Helium (4)	Nitrogen (28)	Decrease
Argon (40)	Argon (40)	Increase
Argon (40)	Nitrogen (28)	Decrease

The advantage in observing the nitrogen peak is that the location of this peak has already been found in the spectrum that indicated an air leak. Argon has the advantage of greater sensitivity than helium (about 8 : 1). Helium is least likely to be confused with other peaks of the same mass number.

Normally, the most sensitive partial pressure scale (10^{-10}), should be used until sufficient signal is obtained to allow switching to a less sensitive scale. If the means exist on the vacuum system being used, leak test sensitivity can be increased by throttling down the pumping speed. This has the disadvantage of increasing the time constant, however (time from application of gas to leak until meter indicates 63% of maximum indication for the leak).

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