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Vacuum Tests for the Main Ring

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A series of tests performed in an attempt to better understand what methods might be used to lower the pressure in the Main Ring Vacuum Chamber is described in this report. Most "improvement" methods appeared to have little value, with the possible exception of a "low-temperature bake". This technique ultimately produced a level less than $2x10^{-9}$ Torr on nude ion gauges near the ion pumps in a two-magnet test ring. The question of present (Summer 1977) Main Ring vacuum levels and its relation to the tests is also discussed.

Stainless Steel Tubing Tests

The common method used to lower the pressure limit of a vacuum chamber has been to decrease the wall gassing rate by operating the chamber at an elevated temperature for a short time (i.e baking the chamber). In a conductance limited chamber, this has been almost the only method. Usually such a bake is at a relatively high temperature (200-400°C) but in the case of the Main Ring magnet chambers, shorted magnets are "burned out" at about 400°C for recycling. The epoxy in them turns brown at about 160-170°C.[1] Clearly a high-temperature bake is out of the question in a magnet that has the chamber epoxied in place, because the intimate thermal contact of the chamber with the magnet means heating the whole magnet to the bake temperature. Many reports in the literature give results of high-temperature bakes but none at 100°C or less.

A 10-ft 4-in diameter stainless steel chamber was assembled with a 30-ℓ/sec Main-Ring style ion pump, a nude ion gauge, a Partial Pressure Analyzer (PPA) head, and a valve. Figure 1 shows the pressure history of the chamber during a series of increasing temperature 24-hr bakes into the ion pump. No particular care was taken in reading the pressure points at the maximum. However, the figure still shows qualitatively the higher pressure points reached at the higher temperatures. The chamber was not let up to air between bakes so that a given temperature bake without the previous history of bakes would show a much higher initial pressure during the bake. In addition, the lower pressures reached after the higher-temperature bakes do not indicate a greater "effectiveness" of that temperature in an independent way because of the previous bakes. A series of 24-hr bakes at a single temperature would also shown some decrease after later cycles. At the baking temperature the pressure after 24 hrs had not yet reached a constant value. During the test series, the PPA scans showed the gas composition to be changing as expected. Mass 18 (water) went down faster than mass 28 (CO), which in turn went down faster than mass 2(H_2). The curves on the right show a bottoming out of the glass gauge used. A nude gauge was installed and a 24-hr 400°C bake produced the pressure marked by the X.

The interest in the point reached after a 24-hr 400°C bake is in the gassing rate of the walls. The published value[3] after such a bake of stainless steel is 10^{-12} Torr liter per cm^2 sec. The scale on the right of Fig. 1 is generated by taking the product of the nude gauge reading and the published pumping speed of the pump for nitrogen at that pressure and dividing by the surface area of the chamber. This is obviously incorrect for the true gassing rate of the walls, because the gas is not nitrogen and in fact is changing. Correcting the gauge and pumping speed for an assumed 100% hydrogen would lower the scale by a factor of 6. For the present purpose, it seems reasonable to take the "Nitrogen" value to compare one chamber to another. The gas composition is observed to change qualitatively in the same manner in similar chambers.

The next question was, what base pressure can be obtained from a 60°C bake if you bake until the pressure at that temperature reaches a more or less constant minimum or base pressure level. Baking that long meams you have obtained almost all the short term gain.

Figure 2a shows by X's the initial pump down of a new piece of tubing. The large change on the 20th day was caused by a room-temperature change. After letting the chamber up to air for a few hours, the dotted curve was observed. The 60°C bake was continued until the 12th day. The temperature of the chamber did not come down to normal room temperature for a few days after the bake because of its proximity to another bake experiment. After both were off, the chamber pressure dropped to 10^{-9} Torr. An 80° bake was tried, but did not come as low. A 24-hr high-temperature bake was then tried to see if it would give the nominal 10^{-12} $T\ell/m^2$ sec gassing rate level expected from a hard bake. Since it did not, it seemed clear that we were having some problem. Another high-temperature bake of the entire system including ion pump into a roughing pump did succeed in bringing the pressure down to the nominal level. Baking into the ion pump was apparently the problem. Removing the gas entirely from the system is obviously better.

Two tubing chambers were vacuum degassed at 1000°C and 10^{-5} Torr. Figure 2b shows their pump down. Note the lower level on basic pump down. A 75°C bake for one week was done on one chamber while flowing N_2 from a liquid source and into a roughing pump on the other. The dots are the N_2 flow and X's are vacuum bake. Note also that it was considered fair to bake pumps, PPA heads and other such exposed pieces at higher than the "low" bake temperature, 200°C minimum was used on pumps.

The result appeared to be that the nitrogen flow did not do as well but a similar vacuum bake and change of ion gauge controller revealed a problem in the controller. The nitrogen flow comparison was not valid. However both chambers finally gave very similar results. The initial 1000°C vacuum degassing lowered the initial in-situ unbaked base, as well as giving a lower value after the low temperature bake.

Main-Ring Magnet Chamber Tests

Two shorted MR magnets that had been open to air for about a month were set side by side. Six-inch diameter "U" shaped chambers that contained nude ion gauges, a PPA, valves etc. were used to connect the ends of the magnets into a small ring. By using one MR ion pump at each end, one simulates a MR chamber, but with approximately 48% of the surface area of a dipole chamber in the end connection (a value higher than in the real ring). After some difficulty trying to use C-ring seals, Viton "O" rings were used to connect the "U" chamber to the magnets. For initial pump down, the "U" chamber had been cleaned with acetone/alcohol and baked. The magnet chamber was not touched. Interest was primarily in lowering the base pressure. Base pressure level is defined as a reasonably constant level within a period of days.

The test sequence with rough base-pressure measurements was as follows:

Test Sequence	Base Pressure (Torr)
1. Initial pumpdown	$5\text{-}7\times10^{-8}$
2. Chamber cleaning (acetone/alcohol)	$6\text{-}8\times10^{-0}/7\text{-}8\times10^{-8}$
3. Argon glow discharges	$6\text{-}8\times10^{-8}$
4. 60°C Bake	$5\times10^{-8}/7\text{-}8\times10^{-8}$
5. "O" ring removal	3×10^{-8}
6. 75°C Bake	2×10^{-9}
7. Magnet warm up to Operating levels	3×10^{-9}
8. Rate of rise/pumps off 3 days	
9. Titanium sputtering	Unsuccessful
10. Final pump down	3×10^{-9}

1. Initial pumpdown went to about 8×10^{-8} in about 10 days according to the ion pump currents. Ion gauges were then turned on and are used in the plots hereafter. Ion-gauge degassing occurred on the 38th day, causing the pressure increase shown in Fig. 3a.

2. After opening the chambers, clean swabs soaked with acetone were pulled through until no evidence of "dirt" could be seen. Several swabs of alcohol were then pulled through, always in the same direction. The first few showed considerable evidence of a black material. However, the chamber bottomed out at about $6\text{-}7\times10^{-8}$ Torr in about 10 days and remained there (more or less) for another 10 days (Figure 3b). Lewin[2] quotes the gassing rate for an unbaked metal surface to be 10^{-10} Torr-liter per cm^2 sec after 50 to 100 hours of evacuation and only a slow decrease thereafter. This value is for reasonably clean surfaces and largely determined by desorption of gases from the wall surfaces. In the test chamber, the 10^{-10} gassing level corresponds to a pressure of 10^{-7} Torr. It therefore seems clear that the pressure limit is due to desorption of gas from the chamber walls and not to

extraneous material in the chamber. In that event, opening the chamber for several hours to "clean" it simply sets one back in the time of the cleaning being done by the ion pumps. Gas adsorbed on the walls is not affected by mechanical cleaning in air.[3] We have found no author in the vacuum literature who claims any lowering of the gassing rate below the 10^{-10} to 10^{-11} Torr-liter per cm^2-sec by mechanical or chemical cleaning methods.

3. Reported reduction[4,5] of ion desorption coefficients using Argon glow discharge cleaning has stimulated much interest in this old, but newly popularized cleaning method. Fischer[2] reports using a 300°C bake before and after the glow discharge. He does not claim a reduction in the thermal desorption rate, but in the ion-impact desorption rate.

Using Fischer's number for the surface bombardment level, a glow discharge with 90% Argon and 10% oxygen gas was set up in the magnets. The results, shown in Fig. 3c, were somewhat confused by the arrival of hot weather and a delay in operation of the room airconditioner. Another glow discharge was tried with a 20-times higher integrated bombardment level. The pressure leveled out in one week to a $7\text{-}8 \times 10^{-8}$ Torr level, as can be seen in Fig. 3c, an insignificant change. Dean of SLAC also reports[6] little improvement in the thermal gassing rate from a glow discharge. The PPA showed an Argon component after the tests as expected, but not at a level that would significantly increase the pressure. Lewin remarks[7] about gas-discharge cleaning, "For efficient cleaning, a substantial fraction of the desorbed surface gas must be removed before it is reabsorbed by the clean, highly active surface. This requires high pumping speed with continuous gas flow". In the long, narrow magnet chambers, one must not be able to meet those conditions, as evidenced by the lack of improvement.

4. In the first attempt to heat the magnets, a hot-water system was used to put 180°F water through the cooling passages in the magnet coils. The outside of the magnets reached 60°C in a few days and was held there for 7 days. After the heat was turned off, the pressure dropped to 5×10^{-8} in one day and stayed there as though it was being maintained by something, as shown in Fig. 4a. Nevertheless, this was a recovery from the previous tests. Obvious errors in this attempt were the use of only a partial bake and having "O" rings in the system with limited pumping.

5. After letting the system up to dry nitrogen, the "O" rings were snipped and removed. The chamber was welded together. In a more normal pumpdown curve, shown in Fig. 4b, the pressure went below 3×10^{-8} in 2 weeks and was still slowly decreasing.

6. After insulation was put over the magnets and end pieces the outside magnet surface reached 75°C approximately, with hot-water heating. The ion pumps were also wrapped with heater tape and insulated. Their temperature was near 200°C. The end chambers were near 100°C. Dry nitrogen from a liquid supply was bled through the chamber for four days in an atmospheric pressure Nitrogen bake as recommended by Dean[9]. A turbo-cart roughing system was used to maintain the chamber near 10^{-5} Torr for four more days of bake. After shutting off the bake and starting the ion pumps, the ring was below 3×10^{-9} Torr in a week and continued down to below 2×10^{-9} Torr in 2 weeks, as can be seen in Fig. 4c. This level is consistent with the level reached in the tubing tests, 1×10^{-9} Torr. The magnets have more than a factor of two more surface area per unit pumping speed than the tubing does.

7. Normal MR magnet operating temperatures are near 35°C rather than 22°C room temperature. To obtain that temperature, the magnets were heated to above 35°C. During the slow cooldown the pressure was approximately 3×10^{-8} with the outside of the magnets at 33°C; we refer the reader to the 18th day in Fig. 4c.

8. Interest was expressed by the Main-Ring Group in a rate of rise measurement in these magnets. Figure 5 shows by X's the pressure as a function of time after the ion pumps are turned off. A constant gassing rate at the base pressure gassing rate would put the pressure orders of magnitude above the observed 1-hr level. A constant rate selected by the pressure at 1 hr produces a curve below the measured curve in the 0 to 1 hr range. The circles show calculated values, where the gassing rate is assumed to vary as $1/p^n$ with n calculated to fit the 1-hr level. This curve fits the data reasonably well except for the long time value. The degassing rate is seen to be consistent with a pressure dependence as might have been expected. Gas molecules hit the walls at a very fast rate compared with this time scale and can stick again. The system shifts to a new equilibrium. The gas composition observed qualitatively shifted with the mass 2(H_2) and 28(CO) fraction growing at the expense of the 18(H_2O) component. The observed high 64-hr level is not explained. A small, hard-baked chamber starting at one decade lower in pressure has shown a curve of similar shape, rising two orders of magnitude in a few hours initially but then remaining at that level for several days.

Turning the pumps on after 5.5 hours brought the pressure back to 1.8×10^{-9} Torr in two hours.

9. The wire used for the glow discharge was made of Titanium hoping to try reverse glow-discharge Titanium sputtering.[8] This

attempt was unsuccessful because of sparking near the springs holding the wires to the feedthroughs. In the reverse-voltage case, the electrons must come from the wire. Arching was observed before enough voltage could build up to start the gas discharge. The sputtering technique could provide a significantly lower pressure than even the 2×10^{-9} Torr with the high pumping speeds reported. It, however, would be an expensive technique to implement in the Main Ring.

10. The pumpdown time after the system is let up to 10^{-2} Torr of Argon is of interest. The ion pumps were baked for 3 days into the turbocart pump. Pumpdown time to the low 10^{-9} Torr level was still a few weeks as shown in Fig. 4d, as compared with 2 hours with no opening. This aspect is of interest in MR operation. If a sector is let up to N_2 after being in the low 10^{-9} Torr level, taking two weeks to go back to the low 10^{-9} Torr level could be a serious operational problem.

Present MR Pressure

Current interest in the Main Ring vacuum has been stimulated by visions of colliding beams of one kind or another. Rumors of real and imagined disasters have created the impression that the Main Ring has a "poor" vacuum system. Hence there are worries about the future of colliding beams.

As mentioned before, Lewin[2] quotes the gassing rate of an unbaked metal surface to be 10^{-10} Torr liter per cm^2 sec after 50 to 100 hrs of evacuation. This is for reasonably clean surfaces and is due to the desorption of gases on the surface of the walls. With approximately a 2×10^4 cm^2 surface area per MR dipole and 30 ℓ/s pump the corresponding pressure is 6×10^{-8} Torr. The original design value of the MR vacuum was 10^{-7} Torr, a value consistent with the expected 100-hr gassing rate.

Figures 6,7 and 8 show ion-pump current readouts taken in July 1971 of a "good", "average" and "bad" sector. Figure 9 shows a plot of sector-average values for the whole Main Ring taken in July 1977. Except for a few sectors that contain special devices with apparent high gas loads, sector averages are in the mid 10^{-8} range. Figure 10 shows sector F3. Note that many pumps read in the low 10^{-8} range. Remembering that the MR ion pump readout circuit stops at 10 μA ($1x10^{-8}$ Torr) with a ±50% repeatability and, with a single bit change in the computer being $2x10^{-8}$, the readout is at the lower limit. A check of pump currents in the service building of F3 showed a significant number of currents in the 5 μA range. According to an extension of the calibrated pump current such current levels correspond to mid 10^{-9} Torr pressure levels if there are no leakage currents. This pump current calibration is consistent with our observations in the two-magnet ring. With long-term operation of the ion pumps, higher leakage levels can be expected. It appears then that the vacuum levels have improved generally in the ring over the past 6 years and in fact include a number of areas in the mid 10^{-9} Torr range. The gassing rate has decreased with long-term pumping.

Conclusion

The relation of the low-temperature bake to MR vacuum is uncertain at this time. Pump currents in the baked magnets were at the 2 μA level, apparently lower than any in the MR. At operating temperature these levels became 3μ amp, a level not unheard of in the Main Ring. It appears that the low-temperature bake enabled one to reach levels in several weeks only slightly below that obtained by long-term MR pumping. Comparison of the two cases is difficult, because in the tests it was necessary to look at relatively short-term values (time constants of the order of a week). The observed Main-Ring levels come from a history of years and, even with magnet changes, time constants are still months. It is not known if the level of less than 2×10^{-9} Torr in the two-ring test would go lower on a time scale of months. A low-temperature bake capability in the Main Ring might be desirable as a more rapid way of recovering from being up to air or cleaning special problem areas. Operational problems are not answered and are difficult.

The main conclusion from our effort is that the Main Ring has places with lower pressures than generally realized, and that the present effort in the Main Ring should be in checking pump-current leakage for a better measurement of the ring pressure, in general upgrading the maintenance effort for more uniformity (finding vacuum leaks), and in finding ways to improve levels in special-device regions that are in the 10^{-7} Torr range. After this effort one can again ask what improvement might a low-temperature bake bring and is it worth the cost?

Acknowledgements

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References

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3. R.S. Barton and R.P. Govier, Vacuum 20, 1 (1970)

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5. H. Hartwig, J. Kouptsidis, "New Techniques for the PETRA Vacuum System", 1248, 1977 Particle Accelerator Conference

6. Norm Dean, SLAC, private communication

7. Gerhard Lewin, Fundamentals of Vacuum Science and Technology, p. 72

8. D. Blechschmidt, "New Vacuum Techniques for Small Aperture Proton Storage Rings", p. 1379, Proceedings of the 1977 Particle Accelerator Conference

9. $150^{o}C$ Nitrogen bakes are used at SLAC according to Norm Dean. Nitrogen bake for exceptionally dirty chambers was also recommended by John O'Meara of Fermilab.

Figure 1 Tubing Tests

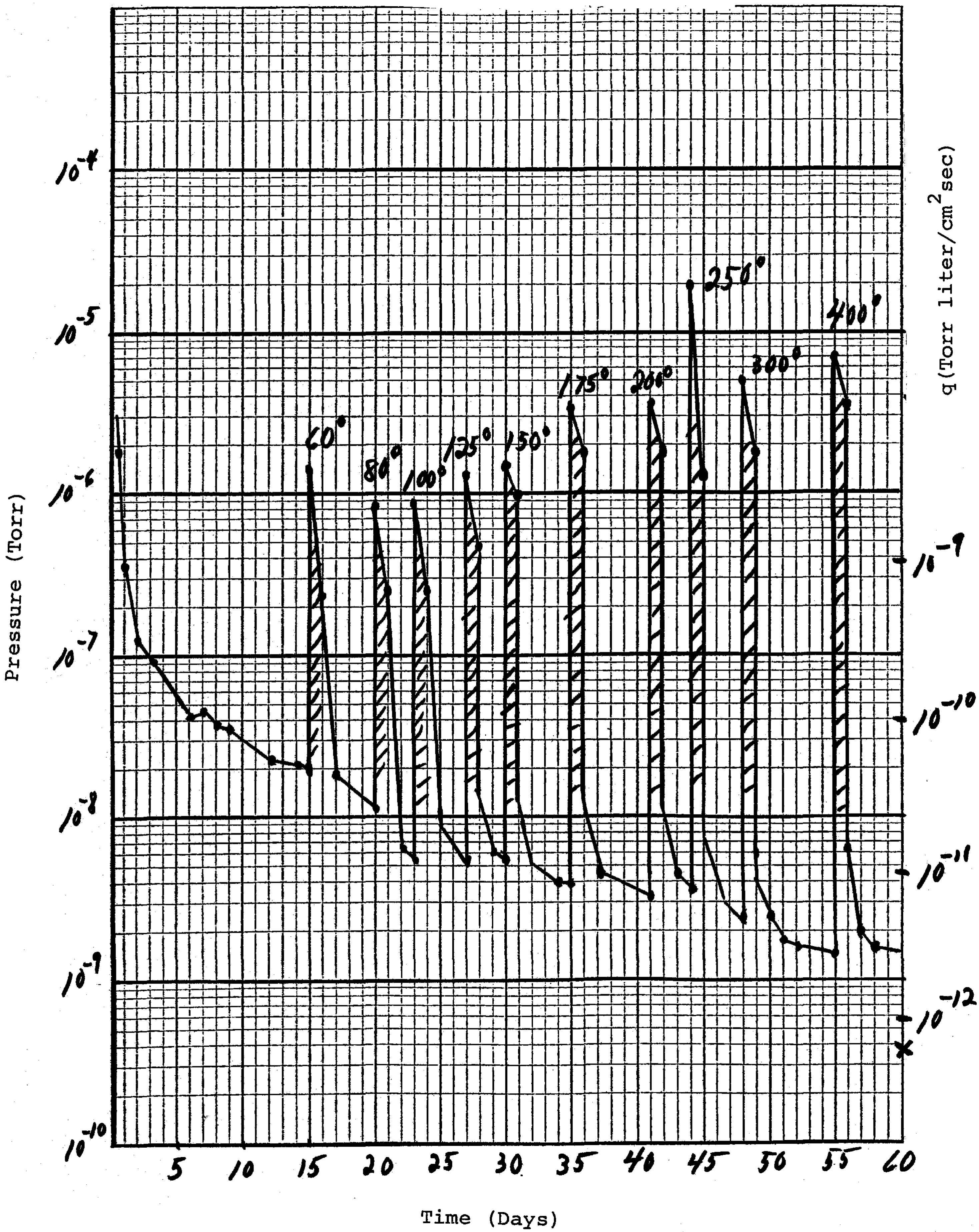

Figure 2 Tubing Tests

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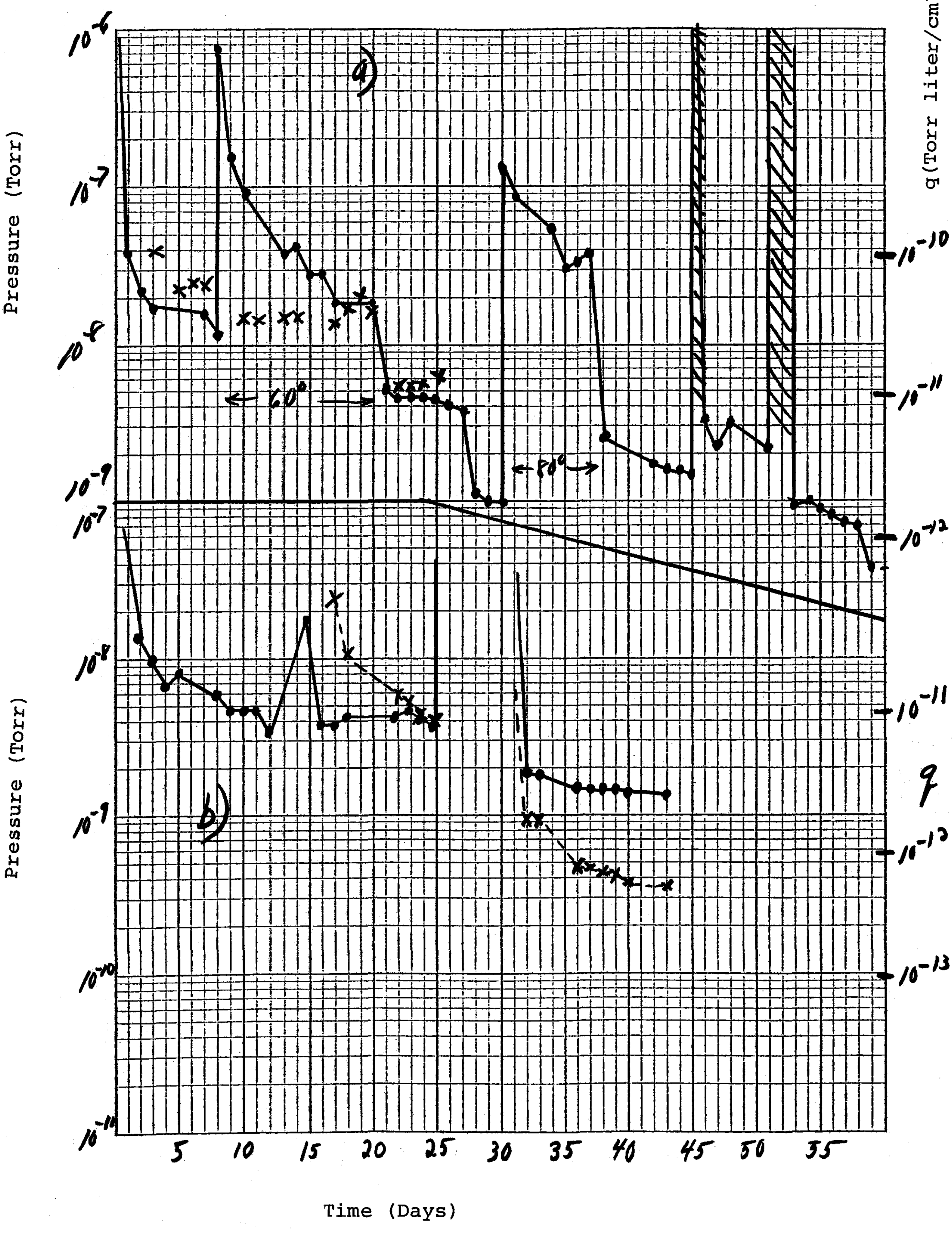


Figure 3 Magnet Test Ring

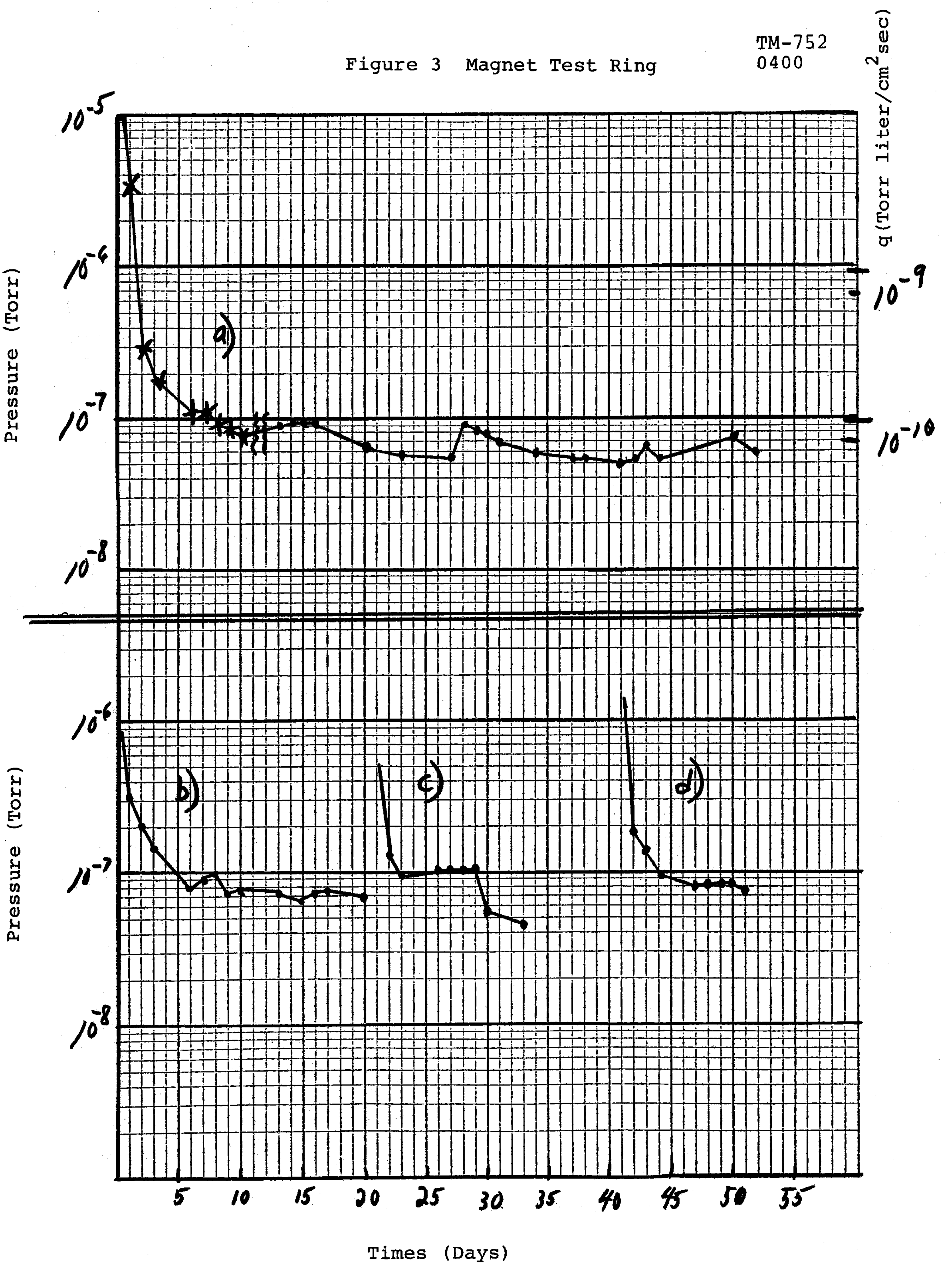


Figure 4 Magnet Test Ring

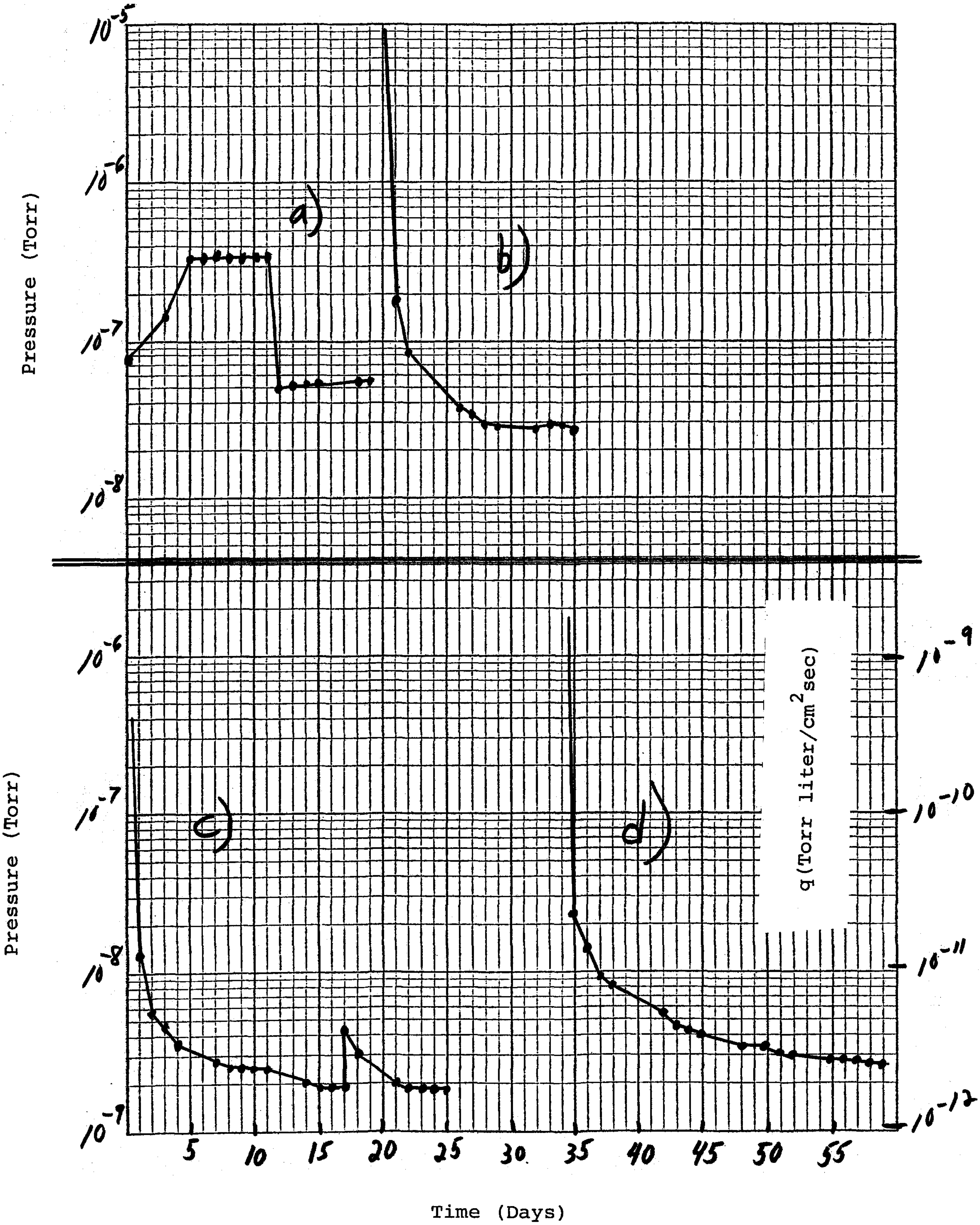
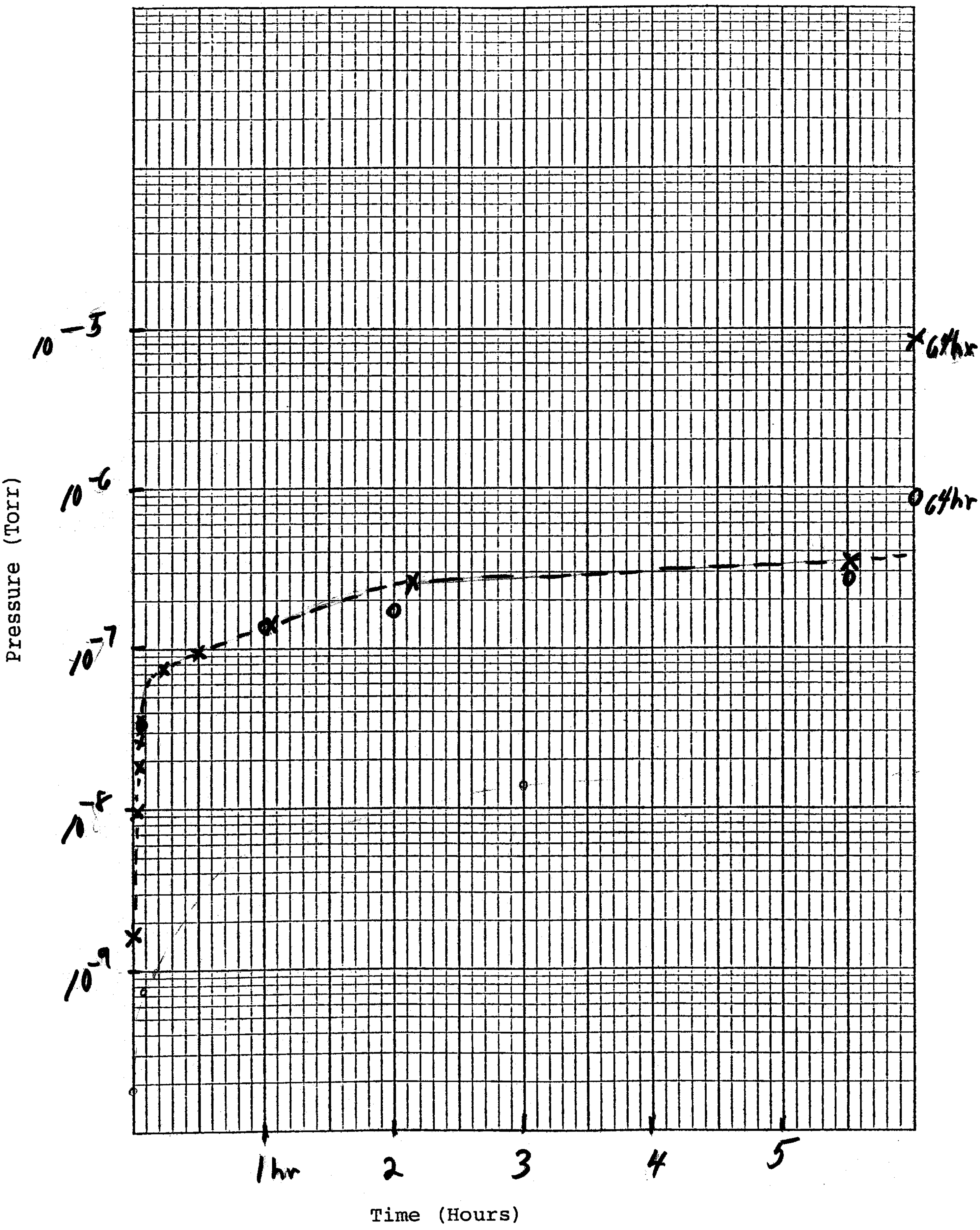


Figure 5 Test Ring Rate of Rise



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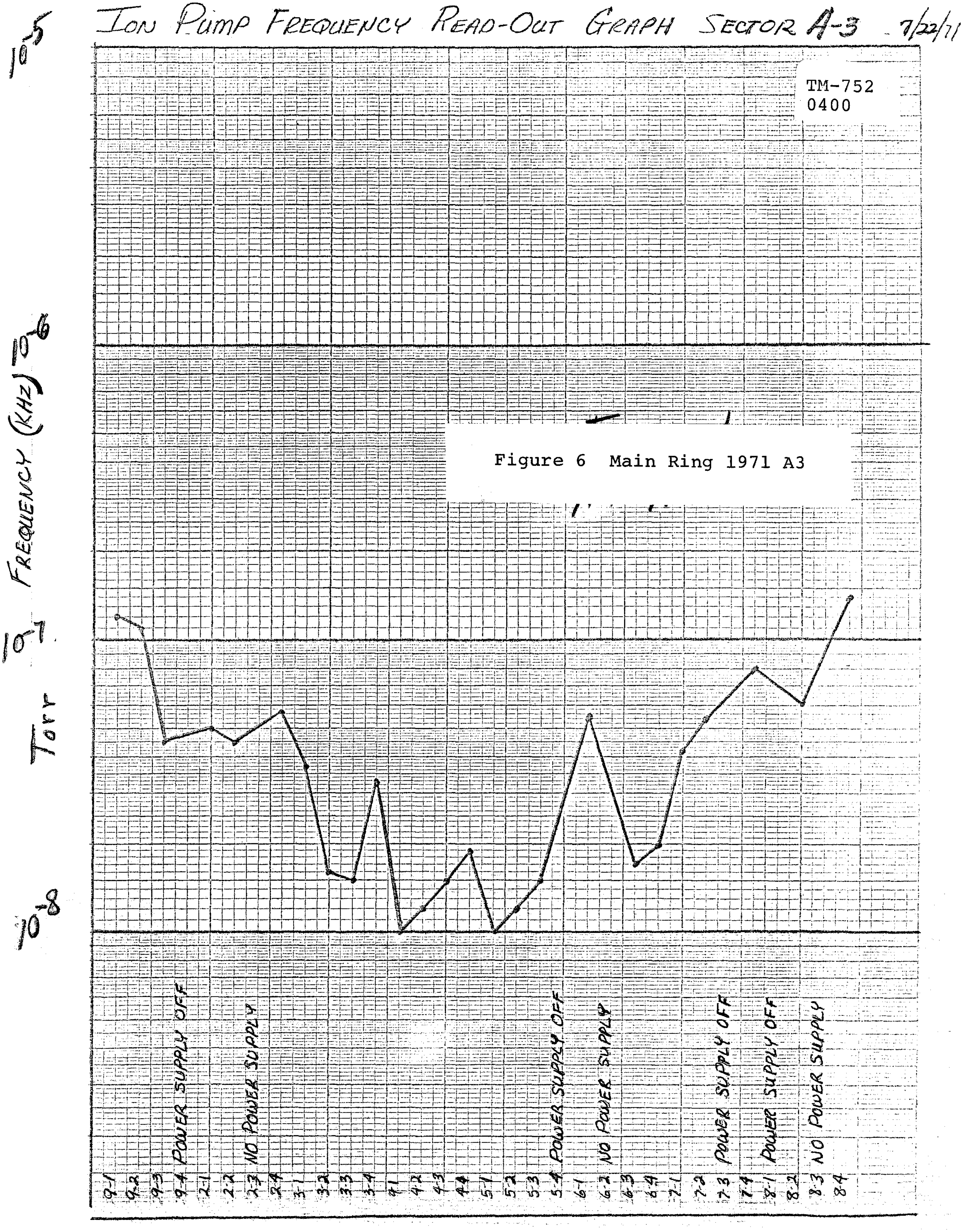

Figure 6 Main Ring 1971 A3

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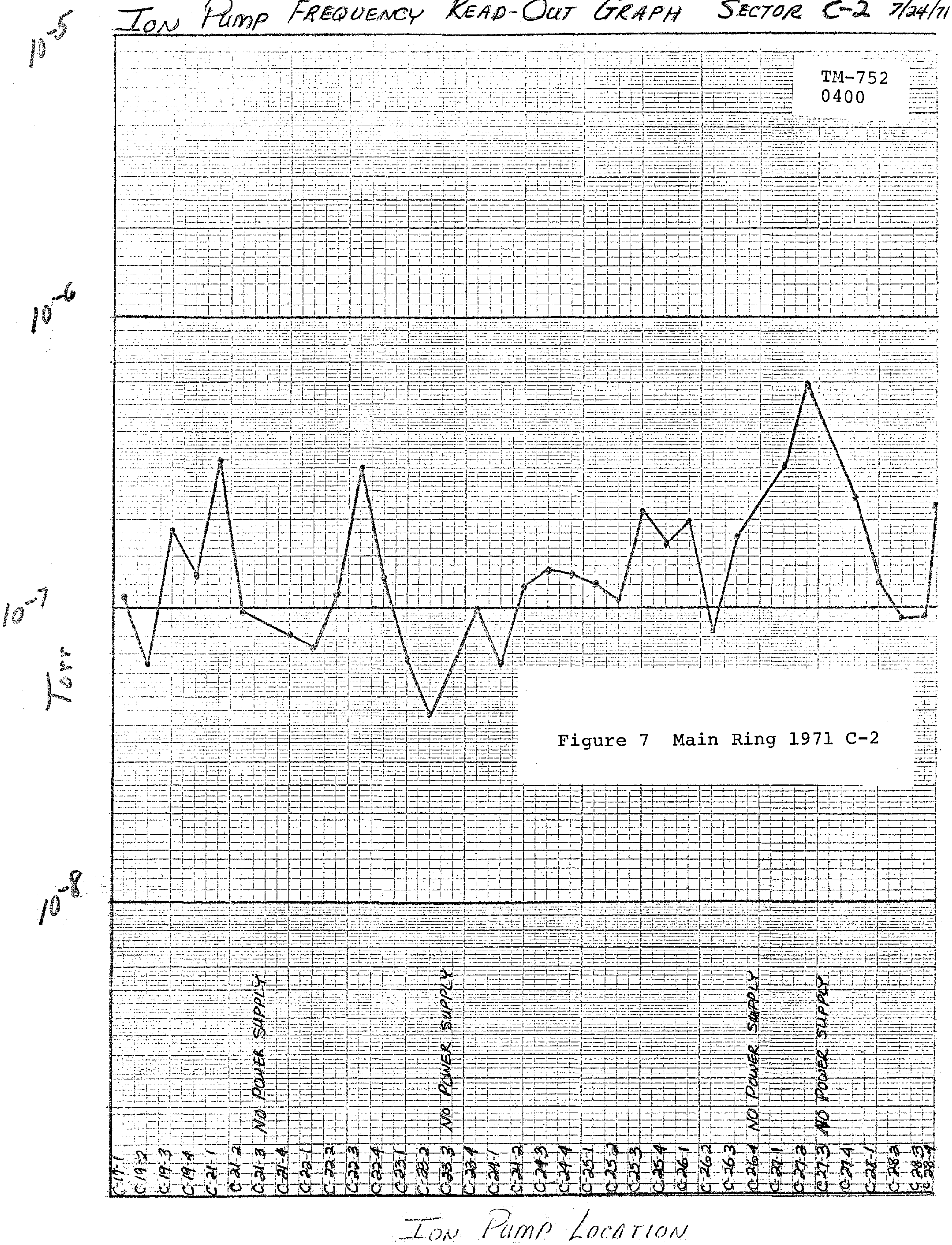

Figure 7 Main Ring 1971 C-2

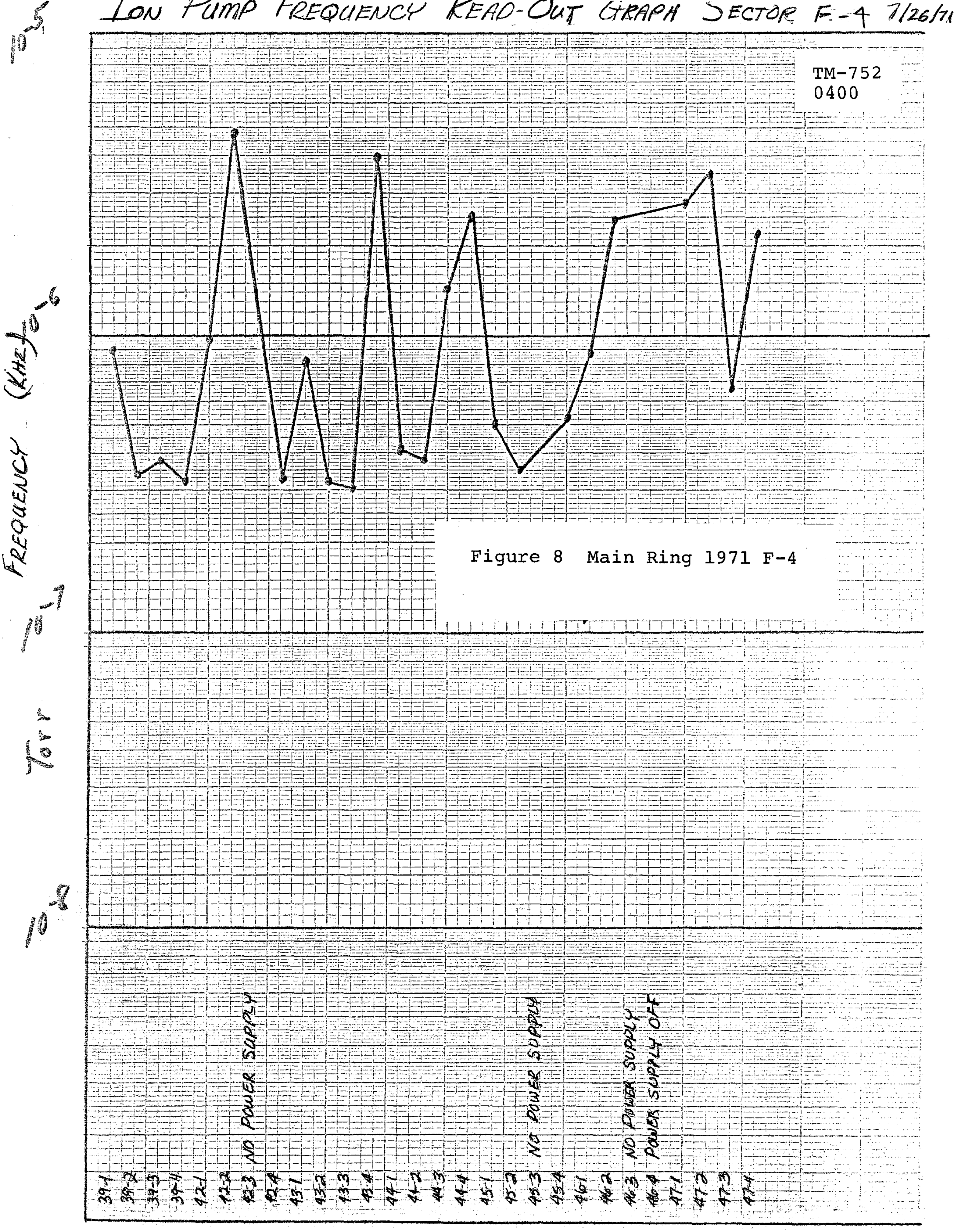

Figure 8 Main Ring 1971 F-4

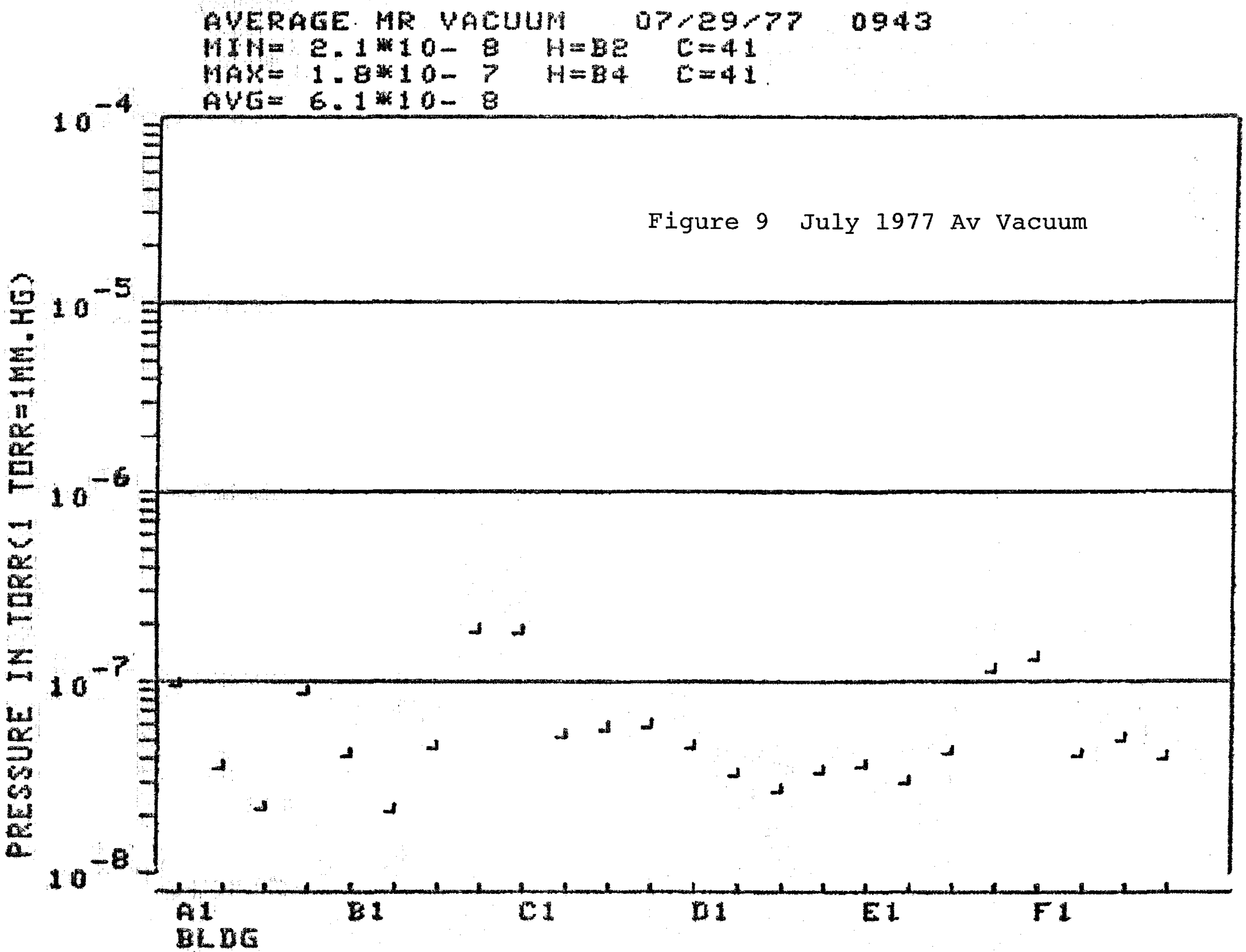

Figure 9 July 1977 Av Vacuum

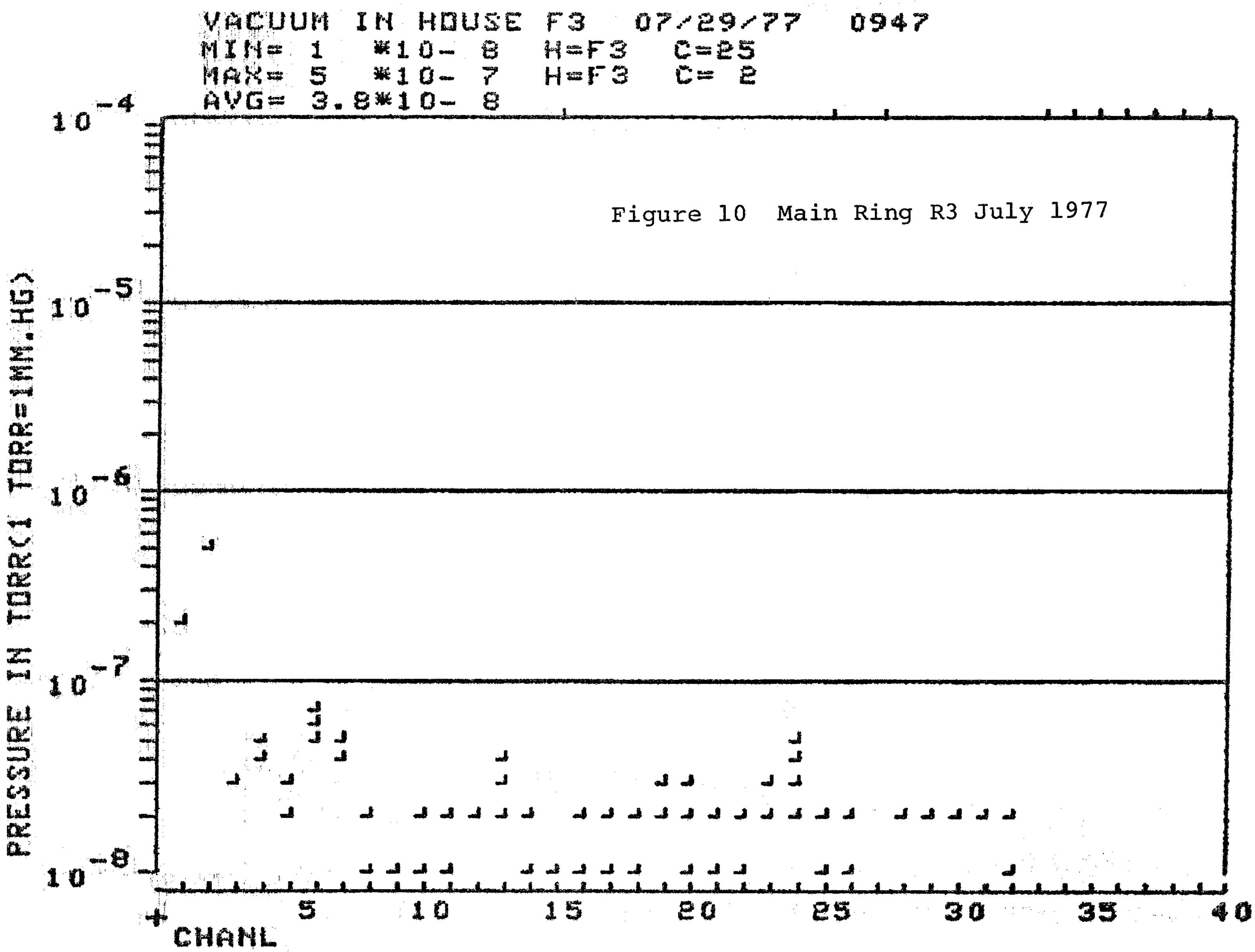

Figure 10 Main Ring R3 July 1977

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