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EFFECTS OF STERILIZATION AND VACUUM EXPOSURE ON POTENTIAL HEAT SHIELD MATERIALS FOR UNMANNED MARS MISSIONS

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Prepared by
SOUTHERN RESEARCH INSTITUTE
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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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ABSTRACT

Sixteen candidate heat shield materials were subjected to a screening program consisting of dry heat sterilization in nitrogen, ethylene oxide decontamination, and exposure to vacuum of 10^{-7} to 10^{-8} torr for two weeks. Flexural tests were made on as-received and exposed specimens at temperatures ranging from 70°F to -200°F in order to evaluate the effects of sterilization, vacuum, and low temperatures on the mechanical properties. Thermogravimetric data were also obtained on as-received and exposed specimens. Of the 16 materials evaluated, five survived all flexural tests without a single failure in the as-received and exposed conditions. The flexural strengths and moduli of most of the materials were reduced significantly by the sterilization-vacuum exposures.

INTRODUCTION

Heat shield materials for an unmanned Mars landing vehicle must withstand a number of severe environmental conditions (1). Among these are temperatures ranging from -200°C to +100°C, vibrations imposed by the booster during launch, interplanetary radiation and micrometeroid bombardment, and entry heating. In addition, since one of the objectives of a Mars mission is the identification of life forms on the planet, it is necessary that the landing vehicle contain no living organisms if positive identification is to be made. Thus the vehicle must be sterilized prior to launch and kept sterile during transit to Mars.

To meet the performance requirements of the mission, the heat shield must possess certain unique physical characteristics. Among these are low thermal conductance and/or good ablative properties (char strength and retention) and RF transparency. Also, the material must possess sufficient plasticity or ductility at low temperatures to absorb the environmental loads. Since the material's properties may be affected by the aforementioned environmental conditions, it is necessary to investigate the effects of these singly or collectively. Those conditions studied in this investigation were sterilization, vacuum and low temperature as they affect flexural response.

Prior to launch, sterilization of the Mars landing vehicle will be accomplished in two steps. The first is a thermal process in which the vehicle is exposed to dry nitrogen at 275°F (408°K) for 92 hours in several cycles. The second is a chemical process consisting of several exposure cycles to a mixture of 12 wt % ethylene oxide (ETO)-88 wt % Freon 12 at 35 to 55% relative humidity at a temperature of 122°F (323°K). To prevent recontamination, the vehicle will be enclosed in a sterilization cannister on the launch pad and during the initial part of the flight (Reference 1).

Space vacuum between earth and Mars ranges from 10^{-12} to 10^{-16} torr. Pressures seen by the heat shield may be considerably higher, depending on when the sterilization cannister is opened and the rate of gas leakage prior to opening.

The object of the investigation described in this report was to evaluate the combined effects of heat sterilization, ETO decontamination and prolonged exposure to high vacuum on the low temperature flexural properties of candidate heat shield materials. The test specifications called for the materials to be subjected to 92 hours of dry heat sterilization at 275°F (408°K) followed by 28 hours' exposure to 12 percent ETO-88% Freon 12 at 122°F (323°K), and finally two weeks in a vacuum of 10^{-7} to 10^{-8} torr at 150°F (329°K). At the conclusion of the vacuum exposure, flexural tests were to be performed on the specimens at temperatures of -100°F (200°K), -150°F (172°K), and -200°F (144°K) while still maintaining vacuum. All phases of the test sequence were to be performed without exposing the test specimens to ambient conditions between phases. For comparison, flexural tests were performed on as-received specimens at room temperature, -100°F, -150°F and -200°F at atmospheric pressure. Thermogravimetric analysis (TGA) data were also obtained on both as-received and exposed specimens.

THE MATERIALS

A total of 16 materials was evaluated in the program. For test purposes these were separated by major constituents into four groups. Detailed compositions of the materials are not known, in most cases these being proprietary. A summary of the available information on the materials is presented in Table 1. It can be seen that the majority of the materials were silicone elastomers, the others being of various types. Most were processed in some manner to reduce the density. Some were foamed while some contained fillers of phenolic or glass Microballoons.

APPARATUSES AND PROCEDURES

Sterilization-Vacuum Apparatus

The sterilization-vacuum apparatus contained all the components necessary to permit maintenance of the specified temperatures, pressures and environments throughout the test cycle and to permit testing of the specimens after the run. Figure 1 is a photograph of the complete system. For purposes of description the system can be divided into three major subsystems: the vacuum system, the environmental control system and the flexural test system.

Vacuum System. - This system was the heart of the apparatus and served as the housing for the environmental control and test systems. The vacuum system was equipped with three high vacuum pumping mechanisms; namely, titanium sublimation, cryogenic pumping and ion pumping. The largest of these was a titanium sublimation pump having a pumping speed of about 5000 liters/sec. The sublimation pump consisted of four titanium filaments surrounded by a water cooled copper housing. The housing was located in a sump below the bell jar. The filaments were operated continuously or cycled manually or automatically by means of a controller. The housing could be water cooled or, by use of suitable external connections, it could be cooled with liquid nitrogen to provide auxiliary cryogenic pumping. For some runs trichloroethylene chilled with dry ice to a temperature of approximately -100°F (200°K) was pumped throughthe system and recirculated through an external pump and reservoir. This provided an economical auxiliary pumping capacity when the specimen outgassing load was greater than the combined pumping speeds of the ion and sublimation pumps but was not large enough to require the use of liquid nitrogen.

The ion pump was a triode type rated at 500 liters/sec. This pump was located adjacent to the sump and connected to it through a valve and right angle elbow.

A mechanical pump rated at 7 liters/sec was used for roughing the system. To prevent backstreaming of oil vapor, this pump was connected to the bell jar through a foreline trap filled with molecular sieves. A sorption pump was added after the first sterilization-vacuum run to increase the rough pumping capacity of the system.

The specimens, the environmental control system and the flexure-test apparatus were contained in a bell jar 20 inches in O.D. x about 13 inches high. The bell jar was provided with rotary and linear motion feedthroughs to permit operation of the flexural apparatus. Twelve feedthroughs, installed in a ring below the bell jar, provided access for the necessary electrical power instrumentation and plumbing circuits.

Pressure was measured by two means: First the ion pump current, being proportional to pressure, provided a direct measurement of pressure in the pump and an indication of pressure in the system. Second, the pressure in the bell jar was measured with a Bayard-Alpert type ionization gage. Due to conductance losses, the pressure in the ion pump generally ranged from $\frac{1}{2}$ to 1 order of magnitude lower than the pressure in the bell jar.

Environmental Control System. - This system included all the plumbing and electrical circuits and controls required to maintain the specified atmospheric environment in the bell jar. Temperatures inside the bell jar were controlled by means of external heaters wound around the bell jar and sump and with heating and cooling units inside the bell jar. The internal heating and cooling sections are shown in the assembly drawing in Figure 2. These enclosed the specimens (Part No. 302) which were located on a rotary turntable. By this means it was possible to rotate each specimen under the loading ram (Assy 3B) and perform flexural tests. The heating and cooling sections were rectangular in cross section with the cooling section enclosing the heating section. (See Appendix A, Figure A1, for a drawing of these sections.) One of the advantages of this design was that it permitted the heating sections to be energized while cooling sections were being chilled with liquid nitrogen without requiring excessive LN_2 or electrical power. This was believed to be necessary in order to maintain the specimens within the required tolerances of $\pm$ 10°F while performing the flexural tests at -100, -150, and -200°F. Both the heating and cooling sections were made in three sections enclosing $\frac{1}{3}$ of the circumference of the turntable. Each 120° segment consisted of a three-sided box with a removeable lid. The heating section was made of copper plate in which circumferential grooves were machined for installation of electrical heaters. The heating circuits consisted of 20 gage Kanthal wire insulated with aluminum oxide beads. To permit precise temperature control, a total of six circuits was used, the top plate and three-sided bottom sections of each 120° segment being wrapped individually. Power was controlled manually using variable voltage transformers (Powerstats). The cooling sections were constructed of copper plate to which circumferentially wound copper coils were silver

soldered. Three separate cooling circuits were employed, one to each of the 120° segments, the coils on the top and bottom of each segment being connected with sections of flexible brass tubing. Both the heating and cooling sections werenickel plated to prevent reaction with ethylene oxide (it was later concluded that this precaution was unnecessary). In operation, it was found that the heating and cooling arrangement would easily permit control of temperatures above ambient within the specified tolerance of ± 4°F. It was also found that simultaneous use of the heating and cooling sections was not required at cryogenic temperatures. However, the cooling rates were very slow, due to the radiation shielding effect of the heating sections and the low emittance of the nickel plating (about 0.03). Therefore, to decrease cool down times and reduce the consumption of liquid nitrogen, the following modifications were performed prior to the third sterilization-vacuum run: the heating sections were removed and the inner surfaces (facing specimens) of the cooling sections were coated with a high absorptance black paint (Sherwin-Williams F-65-B2 Flat Black Enamel) and the heating coils were clamped directly to these surfaces. With this modified system, cool down times were redueed by a factor of about three without having any adverse effect on temperature control.

A schematic of the flow system is shown in Figure 3. For heat sterilization, gaseous nitrogen was withdrawn from the cylinder, metered through a flowmeter into the bell jar and exhausted to the atmosphere. Valves (D) in the vent lines were adjusted to maintain a pressure of about 1 psig in the bell jar for a flow rate of 8-10 SCFH. The nitrogen was preheated to 275°F by means of the auxiliary heater. The system was plumbed so that gaseous nitrogen could be withdrawn from either the gas space in the LN_2 tank or from compressed gas cylinders; however, since analysis of the gas from the LN, tank showed it contained more than the allowable impurity content of 50 ppm, only the cylinders of prepurified gaseous nitrogen were used.

For ETO decontamination, the mixture of 12%ETO-88% Freon (8812) was withdrawn from a commercial cylinder, in which it was stored as a liquid at 70 psig, throttled through valve C to a water bath heat exchanger where it was vaporized. From the heat exchanger it passed through the flowmeter and auxiliary heater, where it was further heated to 122-126°F, and into the bell jar. By means of the throttling valves (D) in the vent line, the pressure was controlled at the level corresponding to an ETO concentration of 650 ± 50 mg/l. The control pressure ranged from 3.3 to 5.9 psig depending on the % ETO in the commercial cylinder. A certified analysis was furnished by the cylinder supplier.

It has been established that for ETO decontamination to be most effective the environment must be maintained at a controlled relative humidity, generally in the vicinity of 50 percent (Ref. 1). However, humidity control was not made a requirement of the program since it was not thought to be sufficiently important to the screening studies to warrant the increased cost that it would entail.

For cooling the specimens to cryogenic temperatures for flexural tests, liquid nitrogen was piped from the supply tank through separate circuits to each cooling section and the cryopanel. Flow rates to the cooling sections were throttled through a common valve in the inlet line or by individual valves in the vent lines.

Flexure-Test Apparatus. - An assembly drawing of the flexure-test apparatus is shown in Figure 2. The main parts of this apparatus were a rotary turntable, load ram and specimen support. The turntable, which contrained slots for 30 specimens 1 inch wide x $2\frac{1}{4}$ inches long x $\frac{1}{4}$ inch thick, was supported by three split rods (Part No. 303 in Figure 2) connected by turnbuckles (No. 304) to permit height and level! adjustment. The rods were connected to a driven member (Assy 4B) which was supported by a chain from a tripod (Assy 4A). A vertical rod on 4B extended through a slot in the tripod assembly (4A) (this slot covered an arc of approximately 300") into an eccentric hole in Part No. 408 which was connected to the rotary feedthrough (104). With this arrangement it was possible to rotate 25 specimens under the loading ram for testing. After the chain failed during the flexure tests at the conclusion of Run 2, it was replaced with a $\frac{3}{4}$ inch diameter rod and spherical ball which rotated in a Teflon socket.

The specimens rested on a support (301), which in turn rested on thin stainless steel tabs welded to the bottom of the turntable at each end of the slot. The tabs acted as springs which could be deflected downward against the load support (Assy 3A) during testing, providing a slight clearance which would permit free rotation of the turntable at other times. A load of approximately 2 to 5 pounds was required to completely deflect these springs against the support. The load ram and support were designed in accordance with ASTM D 797-64 to provide three point loading over a 2 inch span. The load ram was instrumented with strain gages to permit measurement of load. These gages were open faced 0.250 inch gage length with an epoxy backing. A phenolic adhesive was used to adhere the gages to the thin walled load ram after a thorough chemical and solvent cleaning. Eight gages were installed, four to monitor axial strain and four to monitor circumferential strain

(temperature compensation). Two axial gages and two circumferential gages were connected in a Wheatstone bridge circuit, thus there were two complete load measuring circuits, one being intended for a backup. Output from either bridge was recorded on the Y axis of an $X-Y$ recorder.

The load ram was actuated externally through a linear motion feedthrough. Deflection of the specimen was measured with a differential transformer on the top of the bell jar with its stylus bearing on the feedthrough. The differential transformer can be seen in Figure 1. The output of this transformer was rectified by a demodulator and this signal plotted on the X scale of the $X-Y$ recorder versus load on the Y scale. The output signal was calibrated using a dial gage (also shown in Figure 1). Locating the transformer outside the bell jar provided the advantages of ease of access and freedom and eliminated problems which may have resulted from deterioration by temperature changes and from the ETO and vacuum environments; however, a marked disadvantage of this arrangement was that deflections of the specimen could not be distinguished from other deflections in the load train. Since the loading ram and specimen support had to be constructed of thin wall tubing to provide a sufficient strain gage signal and reduce heat leaks at cryogenic temperatures, these extraneous deflections were not insignificant and could cause an error of as much as -30 percent in the measured modulus of the stiffer specimens. To correct for this error, the apparatus was calibrated with steel and aluminum specimens to determine the inherent deflection of the system, and these corrections were applied to the measured load-deflection curves for the plastic specimens. To determine the correction in the measured deflections, the measured load versus deflection curves for the steel specimens were compared with the theoretical curve for a modulus of 30×10^6 psi. The difference in the theoretical and measured deflections was plotted versus load and this curve was used to correct the load-deflection curves obtained on the plastic specimens (which had maximum moduli in the vicinity of 300,000 psi). The correction procedure was verified by applying it to curves obtained on aluminum specimens (modulus = 10×10^6 psi). A typical load deflection curve, showing the corrected plot, is presented in Figure A2, Appendix A.

Test Procedures

The test specifications called for the specimens to be placed in the bell jar and subjected to the dry heat sterilization, ETO sterilization, vacuum exposure and flexural tests without being reexposed to the ambient

environment during the sequence. The detailed test specifications for a complete run are presented in Table 2. Four runs were performed, one on each group of specimens. The following test procedures were employed although, as will be discussed later, equipment problems caused deviations in some of the runs:

1. The specimens were weighed, placed in the bell jar, and the system was evacuated three to four times (using the mechanical pump only) and backfilled with dry nitrogen.

2. The specimens were heated to 275°F and maintained at this temperature for 92 hours. During this period the bell jar was purged with dry nitrogen at a rate of approximately 10 SCFH.

3. The specimens were allowed to cool to 122°F, the system was evacuated to 60 ± 5 torr, then pressurized with a gaseous mixture of 88% Freon-12% ETO. The "8812" mixture was throttled through Valve C (see Figure 3) and vaporized in the water bath heat exchanger. The system pressure was maintained at a slight pressure, usually about 5.9 ± 0.4 psig, corresponding to the specified ETO concentration of 600 ± 50 mg/liter. This condition was maintained for 28 hours ± 15 minutes. During this time a low flow rate (3-8 SCFH) of the 8812 mixture was maintained.

4. The system was evacuated 3 to 4 times using the mechanical pump and backfilled with nitrogen.

5. The specimens were heated to 150°F, the system was evacuated to the lowest vacuum obtainable with the mechanical pump, usually about 10 microns or lower, and the sublimation pump was turned on. When the system pressure stabilized at its lowest value, the roughing valves (VS in Figure 3) were shut off and the ion pump started. Although the ion gage was rated for service at up to 10^{-4} torr, it was found that the filaments burned out quickly at higher pressures. Therefore, the ion gage was usually not turned on until the system pressure was below about 10^{-6} torr.

6. After the system pressure had decreased to about 10^{-6} torr (usually within about two hours after the ion pump was started) the specimens were heated to 200°F and maintained at this temperature for about 24 hours. This step was incorporated in the first run in order to "bake out" the specimens, thereby reducing the outgassing rate to a level which the pumps could handle at a pressure of 10^{-7} torr.

7. The specimen temperature was reduced to 150°F, causing the system pressure to decrease as the outgassing rate fell off. The two weeks' vacuum exposure was measured from the time the system pressure, as measured by the ion gage, decreased to 10^{-7} torr. The specimens were maintained at 150 ± 4°F at the lowest attainable pressure for two weeks.

8. The heaters were shut off after two weeks and the specimens cooled to room temperature using the liquid nitrogen cooled heat sink.

9. Flexural tests were performed on five specimens of each material. The specimens were deflected 0.021 inch (5.33×10^{-4} m) or until failure, whichever occurred first.

10. The specimens were further cooled to -100°F (200°K) and tested in flexure by deflecting them 0.021 inch or to failure, whichever occurred first. Specimen temperatures were maintained at -100 ± 10°F during these tests by manually shutting off or throttling the flow of liquid nitrogen through the cooling sections.

11. The specimens were cooled to -150°F (172°K) and deflected 0.021 inch or to failure, whichever occurred first.

12. The specimens were cooled to -200°F (144°K) and deflected to 0.042 inch, or 1500 lb load or to failure, whichever occurred first.

13. The system was allowed to warm up to room temperature and backfilled with dry nitrogen. The specimens were removed, weighed and sealed in double-walled plastic bags filled with an inert gas (helium or argon).

TGA Apparatus and Procedures

Weight losses were determined on as-received and exposed specimens by thermogravimetric analysis, using a Robert L. Stone Model TGA 3C apparatus. This apparatus, which was used in conjunction with a differential thermal analysis (DTA) apparatus, has a temperature range from ambient to 750°C. A photograph of the apparatus is presented in Figure B1 of Appendix B. The TGA apparatus is a spring suspension system with the specimen container maintained at a null position, The other end is attached to a transducer which provides the read-out signal. Positioning of the

tranducer is done by a servomotor which receives its signal from the null-position device. The electrical signal from the transducer is amplified by means of a dc amplifier and recorded on a 1 to 10 mv recorder. Temperature is increased at a constant rate by means of a programmer. A switch is provided which marks the chart on the recorder at temperature intervals of 100°C.

The specimen container, shown in Figure B2, is an Inconel cup, 0.5 inch diameter x 1.0 inch long, that has a porous disc in the base to permit an inert gas through the sample. This space between the oven and the sample is also purged by an inert gas to carry away evolved vapors and to prevent condensation on the weighing suspension system.

The thermocouple bead is located just above the specimen container.

The apparatus is designed to require a total weight of specimen plus inert diluent of 2.000 $\pm$ 0.075 grams. The powdered aluminum oxide used as the diluent provides a porous structure so that there is free passage of inert gas around the polymer particles, thereby aiding the removal of volatile components given when polymer specimens melt during thermogravimetric analyses.

The specimens were prepared by mixing exactly 50 mg of sample with sufficient aluminum oxide to obtain a total weight of 2 g $\pm$ 50 mg. Although the apparatus is designed to utilize a 100 mg sample, it was necessary to use a lighter sample in these measurements due to the low densities of some of the specimen materials. The full range of the recorder was utilized in order to detect an apparent weight gain at the lower temperatures which resulted from buoyancy effects. Nitrogen was used as the purge gas in the oven, and prepurified nitrogen as the purge gas in the sample container. All determinations were made at atmospheric pressure at a flow rate of 0.05-0.10 SCFH of dynamic gas. The samples were heated at a rate of 18°F/minute to the maximum temperature obtainable in the apparatus of 750°C. Most of the samples were reweighed on an analytical balance at the completion of each run, and the weight loss compared with that recorded on the instrument. In most cases the values agreed within a few percent. Where there was a significant difference, the determination was repeated.

The inherent accuracy of the instrument using a 50 mg sample is 0.4 percent. The accuracy of the analytical balance is also 0.4 percent. An error introduced by buoyancy is often as great as 3 percent. Because of this, the total uncertainty in the determinations cannot be considered less than 3 percent.

EXPERIMENTAL DATA

Flexural Test Data on As-Received Specimens

Control data on as-received specimens were obtained by testing the specimens in the environmental chamber at a pressure of one atmosphere in helium at temperatures of -100, -150, and -200°F, in that order. Data were also obtained at room temperature on most materials. At least four specimens of each material were tested at each temperature level. The specimens were deflected 0.021 inch at room temperature, -100 and -150. At -200°F the specimens were deflected 0.042 inch or to failure, whichever occurred first.

The data are presented in Tables 3 through 5 in the order obtained. The minimum flexural elastic modulus (MOE) which could be detected within the sensitivity of the load cell was approximately 10,000 psi. Consequently, values less than 10,000 psi are so noted in the tables. From the tables it can be seen that the modulus increased as the test temperature decreased, with the largest increase occurring between -150 and -200°F. The moduli of three of the materials, McDonnell B45RF, Avco Mod 5 and Avco Mod 7, increased by a factor of three or more between room temperature and -200°F. The smallest change and lowest values were exhibited by the Avco Teflon foam (0.54 gm/cm^3) and Martin SLA-561. Of the 16 materials evaluated, 11 had one or more failures at -200°F. Only the NASA 50-50 low-density phenolic-nylon specimens failed at higher temperatures. Three of these specimens failed at -150°F and the remaining two failed at -200°F. The McDonnell B45RF specimens, which exhibited the highest MOE of all 16 materials at -200°F, did not fracture at any temperature. The Avco 0.54 gm/cm^3 Teflon foam, which exhibited the lowest MOE of the 16 materials, also survived all the tests without a single failure. The Martin SLA-561 material exhibited the second lowest MOE at -200°F; however, all four specimens tested at this temperature failed at an average outer fiber stress (MOR) of 271 psi.

Results of Sterilization-Vacuum Runs

Run 1. - The first run was performed on specimens from Group 4 (listed in Table 1).

A plot of average specimen temperature versus time for the run is presented in Figure 4. Temperatures were measured with chromel/alumel thermocouples in three specimens located approximately 120" apart on the turntable. The thermocouples were installed in the ends of two of the specimens and in the middle and the end of the third specimen to monitor axial gradients within the specimen. (The third specimen was a dummy specimen and was not tested in flexure.) Additional thermocouples were installed on each of the six heating sections surrounding the specimens and on the loading ram and specimen support.

The sequence in making the run was as follows: Flexural tests were performed on all specimens at room temperature. The system was then evacuated using the mechanical pump to 15-30 microns and backfilled to about 4 psig with dry nitrogen gas. This sequence of evacuation and backfilling was performed four times, the pressure was reduced to approximately atmospheric, and a continuous purge of dry nitrogen at about 7-10 SCFH was maintained. After starting the purge, the internal heaters were energized and the temperature of the specimens was raised to 275°F. The nitrogen gas was also preheated at the inlet to 275°F. The specimens were maintained at this temperature under a constant nitrogen purge for 92 hours. No difficulty was experienced in maintaining temperatures of the specimens within the required tolerance of ± 4°F.

Following the gaseous nitrogen heat sterilization phase, the temperature of the specimens and bell jar was lowered to 122°F, the system evacuated to 60 torr, and the mixture of 12 wt % ethylene oxide (ETO) and 88 wt % dichlorodifluoromethane (Freon 12) was admitted to the bell jar. The system pressure was increased to 6.5 psig to maintain the required concentration of ethylene oxide of 650 ± 50 mg/l. This pressure at a temperature of 122°F was maintained with a slight purge of the "8812" mixture for a period of 28 hours.

Following the ETO exposure, the system was evacuated 3 times to pressures of 300, 100, and 30 microns, respectively, and backfilled with dry nitrogen to a pressure of 5 psig. Following the third evacuation-backfill cycle, the system was backfilled again with nitrogen and the temperature was raised to 150°F. The system was then roughed down to a pressure of about 7 microns using the mechanical roughing pump. These evacuation-backfill cycles required 2 hours and 20 minutes. After $2\frac{1}{2}$ hours, the high vacuum valve to the ion pump was opened and within about 20 minutes the pressure dropped to about 3×10^{-5} torr (as indicated by the ion pump

current). Figure 5 shows a plot of pressure versus time for the first 14 hours of pumpdown. A plot of the complete vacuum exposure is shown in Figure 6. After about 8 hours the system pressure had dropped to about 2×10^{-6} torr, where it leveled out. After an additional 12 hours of pumping with no significant decrease in pressure, it was concluded that an additional "bakeout" of the specimens at a higher temperature was probably necessary in order to reduce the outgassing load to a level which the pumps could handle at a lower pressure. After discussing this idea with the NASA contract monitor and obtaining his approval, the temperature of the specimens was raised to 200°F and held at this level for about 24 hours. As can be seen in Figure 6, the temperature rise caused an almost immediate rise in pressure to about 1×10^{-5} torr; however, after about 13 hours of pumping the pressure began to fall, and 23 hours after heatup it had dropped to 4×10^{-6} torr. At this time the temperature was reduced to 150°F and the sublimation pump was set on continuous operation. The pressure fell rapidly into the low 10^{-7} range, where it remained for about 3 days. About 4 days after the cooldown, the pressure dropped into the 10^{-8} torr range and remained there for the rest of the vacuum cycle.

After two weeks the system was cooled to -100°F using the liquid nitrogen cooling coil inside the bell jar. At this point one of the cooling sections was found to be leaking, causing a sharp pressure spike. In order to maintain vacuum this section was valved off from the others and a rough vacuum pulled on it through the mechanical pump. However, after about 30 hours of cooling the specimen temperature had dropped to only about -30°F and to accelerate cooldown, the leaking section was put back into use. When the temperature reached -100°F, a rough vacuum was pulled on this section and the pressure inside the bell jar dropped to 4×10^{-9} torr. This procedure of utilizing Section 1 for cooldown and then evacuating it was employed for the subsequent tests at -150°F and -200°F. By this means it was possible to maintain pressures in the 10^{-8} and 10^{-9} torr ranges during testing. Temperatures were held within $\pm$ 10°F of the nominal test temperature except at -200°F when due to a shortage of liquid nitrogen, the temperature rose to -170°F before completion of the tests. The Avco 5026-99 and NASA 50-50 phenolic-nylon specimens were tested at average temperatures between -190 and -200°F; however, test temperatures for the Avco 5026-39 specimens ranged from -170 to -190°F.

Results of the flexural tests are presented in Table 6. At -100°F and -150°F the specimens were deflected 0.021 inch. At -200°F the Avco 5026-99 specimens were deflected to 0.042 inch or failure, as required.

Due to an erroneous indication of deflection, none of the Avco 5026-39 and NASA 50-50 phenolic-nylon specimens were deflected more than about 0.025 inch. This may account for the fact that none of these specimens failed.

Table 7 shows a comparison of the average flexural strengths and moduli of the Group 4 materials in the as-received condition and after exposure to the sterilization-vacuum environment. Observe that with one exception the moduli of the exposed specimens were lower at each respective temperature of -100, -150, and -200°F. The ultimate strengths, where comparative data were obtained, were also lower. Fewer failures occurred for the exposed specimens.

Weight loss data for the specimens are presented in Table 8. The weights were determined on an analytical balance before and after the run. Average percent weight losses ranged from 0.73 percent for the NASA 50-50 phenolic-nylon to 3.39 percent for the Avco 5026-99.

Run 2. - Following Run 1, the leak in one of the cooling sections was found and repaired, the vacuum system was baked out and Run 2 was initiated. This run was performed on the silicone materials in Group 2. A complete plot of the average specimen temperature versus time is shown in Figure 7. Figure 8 shows pressure versus time during the vacuum exposure portion of the run, and Figure 9 shows the initial pumpdown to high vacuum on an expanded time scale.

As shown in Figure 9 the system was rough pumped for slightly over six hours using a mechanical pump and a sorption pump. (The sorption pump was added after the first run to increase the rough pumping capacity.) At a pressure of about 7 microns (7×10^{-3} torr) the roughing pumps were valved off and the ion pump and titanium sublimation pump were used to pump the system down to high vacuum. Within about 20 minutes, the pressure dropped to 3×10^{-5} torr then dropped at a slower rate to about 5×10^{-6} torr. After argon processing the ion pump the pressure leveled out near 1×10^{-5} torr. After nearly 10 hours of pumping the specimens were heated to 200°F to accelerate outgassing and permit obtaining the required vacuum of 10^{-7} to 10^{-8} torr. The specimen temperatures were maintained at 200°F for about 24 hours then reduced to 150°F where they were kept for two weeks. As shown in Figure 8, the pressure had dropped to 10^{-7} torr after about 60 hours and continued to decrease gradually during the remainder of the 150°F vacuum exposure.

After two weeks at 150°F, the specimens were cooled to room temperature and flexural tests performed. The specimens were then cooled to -90°F and again tested in flexure, all the while maintaining a vacuum of 10^{-7} to 10^{-8} torr. Cooldown to -90°F was slower than anticipated, however, requiring about 51 hours and about 600 gallons of liquid nitrogen. The cooldown rate was retarded by the very low absorptance (about 0.03) of the nickel plated cooling sections. Since the cooldown curve was exponential (see Figure 7) it was evident that even longer times and correspondingly larger expenditures of liquid nitrogen would be required to cool to -150°F and -200°F if the specimens were cooled solely by radiation. In order to reduce the consumption of LN, we decided to raise the system pressure to about 10^{-2} torr (10 microns) to obtain additional heat transfer by gas conduction. Before cooling to -150°F, however, the system was backfilled to 10^{-2} torr with dry nitrogen, reevacuated to about 10^{-7} torr and several specimens were retested at temperatures ranging from -105°F to -115°F. This step was added in order to assess the effect that increasing the pressure would have on the flexural properties of the specimens. The pressure was again increased to 10^{-2} torr. From this point cooldown proceeded rapidly, the average specimen temperature decreasing from -114°F to -149°F in 2 hours and 15 minutes.

After reducing the pressure to 10^{-7} torr, the flexural tests were initiated. After 7 of the 25 specimens were tested, the chain supporting the specimen turntable broke. At this point it was necessary to warm the specmens to room temperature, break vacuum and make repairs before completing the remaining tests.

Prior to the run, the Avco Mod 5 and Avco 893-150 specimens were tested at room temperature to complete the as-received tests on these materials. The NASA E6A7 silicone-phenolic specimens were also tested. Results of these tests are presented in Table 9. The modulus of elasticity was less than 10,000 psi for all specimens tested except one; however, two of the NASA E6A7 specimens failed—one at a stress of 50 psi (about 1 lb load), the other under no measurable load. These results confirmed prior data on this material.

The data for all specimens at room temperature and -90°F after two weeks at 10^{-7} to 10^{-8} torr are presented in Table 10. Recall that these tests were made prior to increasing system pressure to accelerate cooldown. All five materials exhibited low moduli although the values were generally higher at -90°F. The NASA E6A7 material, which was the weakest of the five materials at room temperature, exhibited the highest average modulus, 51,000 psi, at -90°F. One of these specimens failed at an MOR of 360 psi.

A summary of all tests is shown in Table 11. The results are presented chronologically from left to right across the table,

Plots of the data for each material in the as-received and exposed conditions are presented in Figures 10 through 14. The average strengths and moduli at room temperature, -100, -150, and -200°F are tabulated in Table 12. Where the tests on the exposed specimens were not made at these exact temperatures the data were interpolated from the plots (Figures 10 through 14). When the values obtained after opening the bell jar to repair the turntable support were higher than the prior data, they were not used in computing the averages.

The following comparisons can be made between the properties of the as-received and exposed specimens:

Avco Mod 5 (Figure 10) - The modulus of the exposed specimens was slightly higher at room temperature and lower at all other temperatures than the modulus of the as-received specimens. At -200°F the average ultimate strength of the exposed specimens was about 20 percent lower than that of the as-received specimens.

Avco Mod 7 (Figure 11) - The moduli of the as-received and exposed specimens were about the same. There appeared to be no effect on the moduli as a result of backfilling the system prior to test. There was one anomaly in the data at -190°F. The stress-strain curves of all specimens tested indicated an abrupt 10 percent decrease in load at deflections of about 0.040 inch. However, post-test inspections of these specimens and under the microscope revealed no cracks or other evidence of failure.

Avco Mod 20 (Figure 12) - The average modulus of the exposed specimens was generally lower than that of the as-received specimens at each temperature. The average ultimate strength at -200°F was about the same before and after exposure.

Avco 893-150 (Figure 13) - At -100°F the modulus was about 25 percent lower after exposure. At -150°F, the modulus was slightly higher after exposure; however, this measurement was made after opening the bell jar and may not be truly representative of the post-vacuum condition.

NASA E6A7 Silicone-Phenolic (Figure 14) - The average modulus of the exposed specimens was higher than the modulus of the as-received specimens at -100°F, about the same at -150°F, and considerably lower at -200°F. The

values obtained after opening the bell jar were distinctly higher than the prior data, as shown in Figure 14; however, backfilling to 10^{-2} torr for cooldown appeared to have no effect on the properties.

This material was quite fragile at room temperature. One specimen tested in the as-received condition failed at a stress of 50 psi and one failed with no measurable indication of load. Recall that similar results were obtained in prior tests on as-received specimens. After exposure, a third specimen failed at -90°F and the remaining two failed at -200°F.

Weight loss data are presented in Table 13. Average weight losses ranged from 1.92 percent for the Avco Mod 5 material to 2.49 percent for the NASA E6A7 material.

Following Run 2, the turntable support was repaired using a solid rod instead of the chain for greater strength. The temperature control apparatus was modified by removing the heating sections and coating the inner surfaces of the cooling sections with a high absorptance black paint. The cooling sections were then baked out at 500°F under vacuum. Two bakeouts were necessary in order to reduce the evolution of oil from the paint and achieve a vacuum approaching 10^{-8} torr. Several dummy specimens were cooled to -150°F in slightly over eight hours at a vacuum of about 4×10^{-7} torr. This represented a significant decrease in cooldown time when compared with the prior time of about 50 hours required to cool the specimens from room temperature to -90°F.

Run 3. - The third run was performed on the five silicone materials in Group 1. Plots of temperature and pressure versus time for the run are presented in Figures 15 and 16, respectively. A plot of pressure versus time during the initial portion of the vacuum exposure is presented in Figure 17.

No problems were encountered in maintaining temperatures within the specified tolerance of ± 4°F with the modified temperature control sections. Recall that prior to the run the temperature control sections were coated with a high absorptance black paint to accelerate specimen cooldown. As shown in Figure 15, approximately 21 hours were required to cool the specimens from 150°F and perform flexural tests at room temperature, -100, -150 and -200°F. During cooldown, it was observed that the cooling rate of the Martin SLA-561 specimens lagged that of the other four materials by about 2 to 4 hours, apparently due to the low emittance of this material.

During the vacuum portion of the run the pressure only fell below 10^{-7} torr for a few short intervals, and generally averaged about 10^{-6} torr; see Figure 16. The ultimate pressure could have been limited by (1) further evolution of oil from the cooling sections, although these were twice baked out in vacuum prior to the run; (2) a high gas evolution rate from the specimens; or (3) a leak, although none could be detected. Because of the high pumping speed required, all four filaments of the sublimation pump burned out early in the run, and it was necessary to break vacuum and replace them. While performing this operation, the bell jar was purged with gaseous nitrogen to prevent the influx of air. During the remainder of the run it was necessary to use the sublimation pump only sparingly to prevent burning out the filaments. This limited the minimum ultimate pressure attainable to about 10^{-6} torr.

The flexural test data are presented in Table 14. A comparison of the flexural strengths and moduli in the as-received and exposed conditions is presented in Table 15. In general, the strengths and moduli were lowered by exposure to the sterilization-vacuum cycle, and those materials which exhibited one or more failures at -200°F in the as-received condition exhibited about the same number of failures after exposure. McDonnell B45RF was an exception. None of these specimens failed in the as-received condition, while three of five specimens tested failed after exposure. No specimens of material GE1004 (0.5 gm/cm^3) failed, either before or after exposure.

Weight loss data for the Group 1 materials are presented in Table 16. Percentage weight losses ranged from 0.64 percent for GE1004 (0.5 gm/cm^3) to 3.03 percent for Martin SLA-561.

Run 4. - This run, performed on the materials from Group 3, was completed without mishap and all requirements of the test specifications were met. A pressure of about 3 x 10^{-8} torr was maintained during the two week vacuum exposure. Complete plots of temperature and pressure histories for this run are presented in Figures 18 and 19, respectively.

Flexural test results for the Group 1 materials after exposure are presented in Table 17. Table 18 presents a comparison of the average strengths and moduli in the as-received and exposed conditions. At -200°F, all the Boeing Carborazole specimens failed at an average stress of 3350 psi and an average deflection of 0.070 inch. None of the Avco Teflon foam specimens failed at any temperature although they were deflected more than 0.070 inch at -200°F. However, these specimens did take on a

a permanent deflection or bow, as well as being indented in the center by the load ram. Note that three tests were performed at -200°F on the 0.75 gm/cm^3 Avco Teflon foam specimens without a failure. A review of Table shows that the moduli of all three materials were generally reduced by exposure to the sterilization-vacuum sequence. The moduli of the Teflon foams increased less than a factor of two between room temperature and -200°F. The Boeing Carborazole became much more brittle, the modulus increasing from less than 10,000 psi at room temperature to 136,000 psi (after exposure) at -200°F. This material had about a 30 percent higher strength after exposure.

Weight loss data for this group are presented in Table 19. Note that the weight losses for the Teflon foams were the smallest of any of the materials tested, being 0.01 and 0.02 percent, while the Boeing Carborazole exhibited the highest weight loss (7.21 percent) of the 16 materials,

Thermogravimetric Analyses on All Materials

Table 20 presents a summary of the data. TGA curves for all runs are presented in the Appendix. There were no significant differences in the weight losses of the as-received and exposed specimens from Group 1; however, some significant differences were observed within the other groups. Some materials lost more weight in the as-received condition than after exposure to the sterilization-vacuum sequence. This might be attributed to additional outgassing under vacuum. Materials which exhibited this behavior were Avco Mod 5 and 893-150 in Group 2, Boeing Carborazole in Group 3 and Avco 5026-39 and 5026-99 in Group 4.

Avco Mod 20 and NASA E6A7 silicone-phenolic in Group 2, and the two Avco Teflon samples in Group 3 show a greater weight loss in the exposed condition. For some runs on the Teflon samples the apparent weight loss of the exposed specimens exceeded 100 percent, possibly due to the formation of volatile aluminum fluorides through reaction of Teflon with aluminum oxide. An analogous reaction between Teflon and silicon dioxide in glass was reported by Madorsky (Ref. 2). In that case, silicon tetrafluoride was observed in pyrolyzate products of Teflon above 500°C. In fact, at 538°C, silicon tetrafluoride comprised 3.2 mole percent of the pyrolyzate.

SUMMARY AND DISCUSSION

A summary of the effects of low temperatures and the sterilization-vacuum environments on the 16 materials is presented in Table 21. These data should provide some indication of the materials' potential usefulness for the intended mission.

Certainly it would be desirable for the materials to survive the flexural tests without failure, since such a failure might indicate marginal or unsatisfactory performance under flight conditions. Only five materials met this criterion in both the as-received and exposed conditions. These included the silicone elastomers GE 1004 (35 lb/ft^3) and Avco 893-150, the two Avco Teflon foams, and the Avco 5026-39.

In order that failure not occur due to stresses, the material probably should exhibit only a moderate change in modulus at low temperatures. The allowable change would be a function of the particular application and material. On a comparative basis, a moderate change in modulus has been designated in Table 21 as less than a factor of two. Note that for some of the materials the modulus changed by as much as 40 times between room temperature and -200°F. Three of the materials exhibited changes in modulus of less than 200 percent; namely, Martin SLA-561, Avco Teflon foam (0.54 gm/cm^3), and the NASA 50-50 low-density phenolic-nylon. Of these, only the Teflon foam survived all the flexural tests without a failure.

The moduli and strengths of most of the materials were significantly reduced by the sterilization-vacuum exposures. Four of the materials, Martin SLA-561, Avco 893-150, Avco Teflon foam (0.54 gm/cm^3), and the Boeing Carborazole were only slightly affected. Curiously, the Boeing Carborazole was the only material for which a higher strength was measured after exposure. Moderate (<30%) decreases in modulus were exhibited by the GE 1004 (35 lb/ft^3), Avco Mod 7, Avco Teflon foam (0.75 gm/cm^3), and the three materials in Group 4.

Weight loss may be an important selection criterion if the outgassed products are condensables which may coat thermal control or optical surfaces. It has been stated that of the materials tested the silicone elastomers lose the most weight under space conditions (Ref. 1). The results of this program bear this out. In Run 3, performed on the Group 2 silicones, an oily film was observed on the sight window after several hours of pumping, and the pressures in this run generally ran about 10^{-6} torr. Of the silicones,

weight losses were lowest for the GE1004, at less than 1 percent. As previously mentioned, the Avco Teflon foams had the lowest weight losses at 0.01 and 0.02 percent. Barton and Govier (Ref. 3) reported that the gas evolved from polytetrafluoroethylene (Teflon) at pressures ranging from 10^{-7} to 10^{-9} torr consisted of 16 percent water, 80 percent carbon monoxide, and the remainder carbon dioxide.

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TABLE 1

MATERIALS EVALUATED IN THE PROGRAM

	Average bulk density gm/cm^3	
Group 1 Silicone Elastomers		
1. GE 1004 AP-35 lb/ft^3	0.50	Foamed
2. GE 1004 AP-20 lb/ft^3	0.35	Foamed
3. McDonnell B47RF	0.39	Foamed
4. McDonnell B45RF	0.60	Syntactic (Glass)
5. Martin SLA-561	0.19	Cork
Group 2 Silicone Elastomers		
1. Avco Mod 5	0.72	Syntactic (Phenolic)
2. Avco Mod 7	0.73	Syntactic (Phenolic)
3. Avco Mod 20	0.69	Cork
4. Avco 893 High Cork	0.44	Cork
5. NASA E6A7 Silicone-Phenolic	0.61	Syntactic (Phenolic)
Group 3		
1. Avco Teflon Foam - 43.7 lb/ft^3	0.75	Foamed
2. Avco Teflon Foam - 32.7 lb/ft^3	0.54	Foamed
3. Boeing Carborazole	0.56	Foamed
Group 4		
1. Avco 5026-39	0.61	Syntactic (Phenolic)
2. Avco 5026-99	0.39	Syntactic (Phenolic and Glass)
3. NASA 50-50 Low Density Phenolic Nylon	0.59	Syntactic (Phenolic)

TABLE 2

SPECIFICATIONS FOR STERILIZATION-VACUUM EXPOSURE

1. Place the specimens into an atmosphere of dry nitrogen at 275 ± 4°F (408 ± 2°K) and maintain them there for 92 hours ± 15 minutes.

2. Sterilize the specimens for 28 hours ± 15 minutes at 122 ± 4°F (323 ± 2°K), using a solution of 12% ethylene oxide and 88% Freon 12 with an ETO concentration of 600 mg/liter in accordance with JPL Specification VOL-50503-ETS, dated January 12, 1966.

3. Maintain the specimens in a vacuum of 10^{-7} to 10^{-8} torr for two weeks at 150°F (339°K).

4. Reduce the specimen temperature to -100 ± 10°F (200°K) while maintaining the high vacuum and perform flexural tests on five (5) samples of each material, deflecting until failure or to 0.021 inch, whichever occurs first.

5. Reduce the specimen temperature to -150" ± 10°F (172°K) while maintaining the vacuum and perform flexural tests on the same specimens, deflecting until failure or to 0.021 inch, whichever occurs first.

6. Reduce the specimen temperature to -200 ± 10°F (144°K) while maintaining the vacuum and perform flexural tests on the specimens, deflecting until failure or to 0.042 inch, whichever occurs first.

7. In order to ascertain the effects of the heat sterilization, ethylene oxide decontamination and vacuum exposure on the mechanical properties of the materials, comparison tests shall be performed on five (5) specimens of each material under ambient conditions at each of the three specified temperatures.

TABLE 3

FLEXURAL DATA ON AS-RECEIVED SAMPLES
RUN 1 (4229-37)

Material group	Specimen identification	-100°F MOE in 10^3 psi	-100°F MOR psi	-160°F MOE in 10^3 psi	-160°F MOR psi	-200°F MOE in 10^3 psi	-200°F MOR psi
4	NASA 60-60 Low-Density Phenolic Nylon-13	210	-	220	-	210	1500
	NASA 50-50 Low-Density Phenolic Nylon-12	180	-	200	1490	-	-
	NASA 50-50 Low-Density Phenolic Nylon-11	220	-	190	1440	-	-
	NASA 50-50 Low-Density Phenolic Nylon-10	210	-	210	-	195	1820
	NASA 50-50 Low-Density Phenolic Nylon-9	200	-	230	1630	-	-
	Averages	204	-	210	1520	202	1660
4	Avco 6026-39-8	230	-	270	-	310	-
	Avco 5026-39-7	260	-	290	-	350	
	Avco 5026-39-6	270	-	300	-	310	-
	Avco 5026-39-5	-	-		-	370	-
	Avco 5026-39-4	270	-	300	-	310	-
	Averages	258	-	290	-	330	-
4	Avco 5026-99-3	110	-	106	-	131	1750
	Avco 5026-99-2	120	-	96	-	81	1570
	Avco 5026-99-1	125	-	86	-	150	1490
	Avco 5026-99-30	140	-	110	-	135	1490
	Avco 5026-99-29	120	-	130	-	123	1630
	Averages	124	-	105	-	124	1590
2	Avco 893-150 High Cork-28	50	-	57	-	100	-
	Avco 893-150 High Cork-27	47	-	38	-	100	
	Avco 893-150 High Cork-26	46	-	52	-	83	-
	Avco 893-150 High Cork-25	52	-		-	100	-
	Avco 893-150 High Cork-24	60	-	61	-	105	
	Averages	51	-	52	-	98	-
2	Avco Mod 5 Silicone Elastomer-23	130	-	180	-	360	3600
	Avco Mod 5 Silicone Elastomer-22	140	-	180	-	320	4100
	Avco Mod 5 Silicone Elastomer-21	130	-	190	-	300	3700
	Avco Mod 5 Silicone Elastomer-20	110	-	180	-	280	4200
	Avco Mod 5 Silicone Elastomer-19	- [1]	-	—	-	- [1]	- [1]
	Averages	128	-	182	-	315	3900

1 Not tested

TABLE 4

FLEXURAL DATA ON AS-RECEIVED SAMPLES
RUN 2 (4229-42)

Material Group	Specimen Identification	Room Temperature MOE in 10^3 psi	Room Temperature MOR psi	-100°F MOE in 10^3 psi	-100°F MOR psi	-150°F MOE in 10^3 psi	-150°F MOR psi	-200°F MOE in 10^3 psi	-200°F MOR psi
3	Boeing Carborazole-19	*		142	-	167	-	164	2680
	Boeing Carborazole-20	*		128	-	147	-	154	2550
	Boeing Carborazole-21	*		128	-	167	-	178	2620
	Boeing Carborazole-22	*		154		167	-	212	
	Boeing Carborazole-23	*		116		114	-	154	2520
	Average		-	134	-	152	-	172	2600
2	Avco Mod 7 Silicone Elastomer -24	10	-	18	-	58	-	308	
	Avco Mod 7 Silicone Elastomer -25	13	-	28	-	68	-	332	
	Avco Mod 7 Silicone Elastomer -26	12	-	20	-	41	-	319	3940
	Avco Mod 7 Silicone Elastomer -27	12	-	20	-	51	-	332	-
	Average	12	-	22	-	54	-	323	3940
3	Avco Teflon Foam 0.54 gm/cm^3 - 29	*	-	*	-	*	-	14	
	Avco Teflon Foam 0.54 gm/cm^3 - 30	*	-	*	-	*	-	*	
	Avco Teflon Foam 0.54 gm/cm^3 - 1	*	-	*	-	*	-	12	
	Avco Teflon Foam 0.54 gm/cm^3 - 2	*	-	*	-	*	-	13	
	Avco Teflon Foam 0.54 gm/cm^3 - 3	*	-	*	-	*	-	*	
	Average		-		-		-	13	
2	Avco Mod 20 Silicone Elastomer -4	*		52	-	86	-	189	2590
	Avco Mod 20 Silicone Elastomer -5	*		45	-	86	-	189	2420
	Avco Mod 20 Silicone Elastomer -6	*		46	-	85	-	216	2560
	Avco Mod 20 Silicone Elastomer -7	*		43	-	74	-	204	2280
	Avco Mod 20 Silicone Elastomer -8	*		44	-	84	-	190	2380
	Average		-	46	-	83	-	196	2450
1	Martin SLA-561 Silicone Elastomer -9	*	-	*	-	*	-	19	168
	Martin SLA-561 Silicone Elastomer-10	*	-	*		*	-	23	272
	Martin SLA-561 Silicone Elastomer-11	*	-		-	*	-	24	269
	Martin SLA-561 Silicone Elastomer-12	*	-	*	-	*	-	18	144
	Martin SLA-581 Silicone Elastomer-13	*	-	*	-	*	-	27	240
	Average		-		-		-	22	271

* Denotes MOE less than 10×10^3 psi

TABLE 5

FLEXURAL DATA ON AS-RECEIVED SAMPLES
RUN 3(4229-45)

Material Group	Specimen Identification	Room Temperature MOE in 10^3 psi	Room Temperature MOR psi	-100°F MOE in 10^3 psi	-100°F MOR psi	-150°F MOE in 10^3 psi	-150°F MOR psi	-200°F MOE in 10^3 psi	-200°F MOR psi
2	NASA E6A7 Silicone-Phenolic - 1	[1]	[1]	-	-	-	-	-	-
	NASA E6A7 Silicone-Phenolic - 2	*	84	-	-	-	-	-	-
	NASA E6A7 Silicone-Phenolic - 3	[2]	[2]	58	-	91	-	200	2600
	NASA E6A7 Silicone-Phenolic - 4	[2]	[2]	58	-	85	-	214	2540
	Average		84	58	-	88	-	207	2570
1	McDonnell B47RF - 5	*	-	*	-	24	-	109	1730
	McDonnell B47RF - 6	*	-	*	-	18	-	109	2060
	McDonnell B47RF - 7	*	-	*	-	19	-	96	1680
	McDonnell B47RF - 8	*	-	*	-	23	-	109	1820
	Average		-		-	21	-	106	1820
1	McDonnell B45RF-9	*	-	80	-	147	-	376	-
	McDonnell B45RF-10	*	-	55	-	151	-	400	-
	McDonnell B45RF-11	*	-	76	-	159	-	434	-
	McDonnell B45RF-12	*	-	70	-	138	-	376	-
	McDonnell B45RF-13	*	-	78	-	130	-	432	-
	Average		-	72	-	145	-	404	-
1	G≤1004 0.50 gm/cm^3 - 19	*	-	*	-	21	-	103	-
	G≤1004 0.50 gm/cm^3 - 20	*	-	*	-	*	-	103	-
	G≤1004 0.50 gm/cm^3 - 21	*	-	*	-	36	-	103	-
	G≤1004 0.50 gm/cm^3 - 22	*	-	*	-	27	-	124	-
	Average		-		-	28	-	105	-
3	Avco Teflon 0.75 gm/cm^3 - 23	*	-	24	-	48	-	90	-
	Avco Teflon 0.75 gm/cm^3 - 24	*	-	23	-	48	-	75	-
	Avco Teflon 0.75 gm/cm^3 - 25	*	-	24	-	41	-	60	-
	Avco Teflon 0.75 gm/cm^3 - 26	*	-	22	-	35	-	80	-
	Average		-	23	-	43	-	76	-
1	G≤1004 0.35 gm/cm^3 - 27	*	-	*	-	20	-	70	-
	G≤1004 0.35 gm/cm^3 - 28	*	-	*	-	16	-	130	-
	G≤1004 0.35 gm/cm^3 - 29	*	-	*	-	11	-	108	980
	G≤1004 0.35 gm/cm^3 - 30	*	-	*	-	16	-	77	-
	Ave age		-		-	16	-	96	980

Notes

* Denotes MOE less than 10 x 10^3 psi

1. Broke when zeroing ram visually - no data.
2. Did not test at room temperature.

TABLE 6

FLEXURAL DATA ON SPECIMENS IN GROUP 4 MEASURED AT CONCLUSION OF STERILIZATION - VACUUM RUN 1

Spedmen identification	Temperature							
	Room temperature[1]		-100°F		-150°F		-200°F	
	MOE in 10^3 psi	MOR psi	MOE in 10^3 psi	MOR psi	MOE in 10^3 psi	MOR psi	MOE in 10^3 psi	MOR psi
Avco 5026-39-22	41		280		210		200[5,6]	
Avco 5026-39-23	95		260		180		250[5,6]	
Avco 5026-39-24	45		360		210		280[5,6]	
Avco 5026-39-25	42		290		260		250[5,6]	
Avco 5026-39-26	45		220		220		240[5,6]	
Averages	52		280		216		245	
Avco 5026-99A-29	30		- [9]					
Avco 5026-99A-30	25		70		90		90	
Avco 5026-99A-1	17		94		90		100	1120
Avco 5026-99A-2	50		98		110		82	
Avco 5026-99A-3	30		98		110		80	1150
Averaged	32		90		100		88	1135
NASA 50-50 Low-Density Phenolic Nylon-5	140		160		147		190[5]	
NASA 50-50 Low-Density Phenolic Nylon-8	160		150		170	450[4]		
NASA 50-50 Low-Density Phenolic Nylon-9	130		130		150		150[5]	
NASA 50-50 Low-Density Phenolic Nylon-10	160		140		170		160[5]	
NASA 50-50 Low-Density Phenolic Nylon-11	180		150		150	___	140[5]	
Averages	154		146		160	450	160	

1. Room temperature determinations made prior to start of run.
2. Denote MOE $< 10^3$ psi.
3. Specimen broken while positioning loading ram.
4. Mid-point deflection at failure approximately 0.009 in.
5. Due to an erroneous deflection readout, this specimen was deflected Po only 0.025 inch at -200°F.
6. These specimens tested at temperatures ranging from -170°F to -190°F

TABLE 7

COMPARISON OF FLEXURAL PROPERTIES OF AS RECEIVED AND EXPOSED SPECIMENS FROM GROUP 4

Material	Temperature °F	Average flexural strength and modulus MOE, ir 0^3 psi		MOR, psi	
		As Received	Exposed	As Received	Exposed
Avco 5026-30	Room	52			
	-100	258	280		
	-150	290	216		
	-200	330	245		
Avco 5026-99	Room	32			
	-100	124	90		
	-150	105	100		
	-200	124	88	1590[1]	1135[2]
NASA 50-50 Low-Density Phenolic Nylon	Room	154			
	-100	204	146		
	-150	210	160	1520[3]	450[5]
	-200	202	160	1660[4]	

Notes

1. All five specimens failed at this temperature.
2. Two of four specimens failed at this temperature,
3. Three of five specimens failed at this temperature.
4. Remaining two specimens failed at -200°F.
5. Only one specimen failed at this temperature.

TABLE 8

WEIGHT LOSS DATA FOR SPECIMENS FROM GROUF 4

	Weight, grams		Percent weight loss
	Initial	Final	
Avco 5026-39 - 15	5.7780	5.6583	2.07
Avco 5026-39 - 22	5.6480	5.5186	2. 29
Avco 5026-39 - 23	5.7330	5.6123	2.10
Avco 5026-39 - 24	5.6610	5.5310	2.29
Avco 5026-39 - 25	5.7418	5.6254	2.02
Avco 5026-39 - 26	5.7453	5.6262	2.07
Average			2.14
Avco 5026-99A - 16	3.7140	3. 5921	3.28
Avco 5026-99A - 29	3.8030	3.6732	3.41
Avco 5026-99A - 30	3.7115	3. 5918	3.22
Avco 5026-99A - 1	3.6230	3.4979	3.45
Avco 5026-99A - 2	3.6440	3.5257	3. 24
Avco 5026-99A - 3	3.7560	3.6152	3.74
Average			3, 39
NASA 50-50 Low-Density Phenolic Nylon - 17			
NASA 50-50 Low-Density Phenolic Nylon - 5	5.6590	5,6097	0.87
NASA 50-50 Low-Density Phenolic Nylon - 8	5, 5827	5.5379	0. 80
NASA 50-50 Low-Density Phenolic Nylon - 9	5.3550	5.3199	0.65
NASA 50-50 Low-Density Phenolic Nylon - 10	5.5452	5.4953	0.89
NASA 50-50 Low-Density Phenolic Nylon - 11	5.3858	5,3609	0.46
Average			0.73

Notes:
1. Weights were determined before and after Sterilization - Vacuum Run 1.
2. Specimens 15, 16, and 17 were not tested in flexure (located in dead zone).

TABLE 9

ROOM TEMPERATURE FLEXURAL TEST DATA ON SPECIMENS FROM GROUP 2 SILICONES (MEASURED PRIOR TO RUN AT ATMOSPHERIC PRESSURE)

	MOE in 10^3 psi	MOR in psi
Avco Mod 5-9	See Note [1]	
-10	[1]	
-11	[1]	
-12	[1]	
-13	[1]	
Average		
Avco 893-150 High Cork -19	See Note [1]	
-20	[1]	
-21	[1]	
-22	[1]	
-23	[1]	
Average		
NASA E6A7 Silicone-Phenolic - 4	See Note [2]	
- 5	[a]	
- 6	[1]	*50*
- 7	19	
- 8'		

Notes:

1. MOE $<10^4$ psi
2. Not tested
3. Failed with no measurable indication of load.

TABLE 10

FLEXURAL TEST DATA ON SPECIMENS IN GROUP 2 MEASURED AT CONCLUSION OF STERILIZATION VACUUM RUN 2

		RT		-90°F [4]	
		MOE in 10^3 psi	MOR psi	MOE in 10^3 psi	MOR psi
Avco Mod 5-9		35	-	44	
-10		21	-	36	
-11		16	-	36	
-12		14	-	44	
-13		19	-	41	
Average		21	-	40	
Avco Mod 7-24	See Note	1		20	
-25		2		21	
-26		38		21	
-27		1		30	
-28		16		30	
Average				24	
Avco Mod 20-29		2	-	14	
-30		1	-	1	
-1		1	-	1	
-2		1	-	1	
-3		1	-	1	
Avco 893-150 High Cork -19		2	-	1	
-20		2	-	28	
-21		1	-	36	
-22		1	-	33	
-23		1	-	33	
Average					
NASA E6A7 Silicone-Phenolic-4		2		50	
-5		21		57	360
-6		3			
-7		1		47	
-8		3			
Average				51	

Notes: 1. MOE 10^4 psi
2. Not tested.
3. Failed at RT prior to run.
4. Tests at -90°F made after two weeks at vacuum of 10^{-7} to 10^{-8} torr.

TABLE 11

FLEXURAL TEST DATA FOR SPECIMENS IN GROUP 2 MEASURED AT CONCLUSION OF STERILIZATION VACUUM RUN 2

Specimen identification	Room temperature after run		-90°F before breaking vacuum		Data obtained after backfilling with GN_2 to 10^{-2} torr and reevacuating to $<10^{-7}$ torr				Data obtained after backfilling to 1 atm with GN_2, warmup to RT cooldown and reevacuation to $\leq 10^{-5}$ torr				Approximate deflection at failure in 10^{-3} inch
					-105 to -115°F		-140°F		-100°F	-150°F	-160 to -190°F		
	MOE in 10^3 psi	MOR psi	MOE in 103 ps	MOR psi	MOE in 10^3 psi	MOR psi	MOE in 10^3 psi	MO psi	MOE in 10^3 pa	MOE in 10^3 psi	MOE in 10^3 psi	MOR psi	
Avco Mod 5- 9	38		48		[a]		85		68	135	160[6]	3000	57
-10	27		48		64, 70[3], 89[4]		133		80	151	196[6]	2950	55
-11	18		43		[a]		150		93	152	210[6]	2950	45
-12	14		59		73		145		103	152	205[6]	2820	52
-13	24		47		2		134		107	169	2188	3750	60
Average	24		49		68		130		90	152	198	3090	54
Avco Mod 7-24	See Note[1]		20						[p]	2	134[1]		
-25	[a]		21						[a]	[a]	2		
-26	34		21						25	49	244[5]	2940[7]	34
-27	1		30						24	44	224[1]	2800[7]	40
-28	16		30						17	49	237[5]	3050[7]	44
Average			24						22	47	235	2930[1]	40
Avco Mod 20-29	[a]		14						22	62	124[1]	2400	65
-30	1		1						19	59	155[5]	2100	47
- 1	1		1						22	61	120[5]	2260	66
- 2	1		1						18	56	135[1]	2830	65
- 3	1		1						11	55	135	2300	65 —
Average									16	59	134	2380	62
Avco 893-150													
High Cork -19	[a]		1		[a]						[a]		
-20	[a]		38		36						76[9]		
-21	1		45		36						66[9]		
-22	1		32		[a]						78[9]		
-23	1		37		2						76		
Average											74		
NASA E6A7 Silicone Phenolic-4	2		56				85, 85[8]		98	108	152[6]	2360	
-5	24		62	160									
-6	3												
-7	1		60				84		78	97	147[5]	1820	
-8	3		—				—		—	—	—	—	
Average			59				84		88	102	150	1590	

Notes:
1. MOE $< 10^4$ psi
2. Not tested
3. Second run
4. Third run
5. Tested at -190°F
6. Tested at -170°F
7. Outer fiber stress — no visible fracture observed in post-test inspection, although load decreased sharply during test
8. Measured at -186°F, 2×10^{-8} torr
9. Tested at -160°F

TABLE 12

COMPARISON OF AVERAGE FLEXURAL STRENGTHS
AND MODULI OF AS-RECEIVED AND EXPOSED SPECIMENS FROM GROUP 2

Material	Average Test Temperature (°F) and Test Condition	MOE in 10^3 psi		MOR, psi	
		As Received	Exposed	As Received	Exposed
Avco Mod 5	Room Temperature	<10	24		
	-100	128	49		
	-150	182	152		
	-200	315		3900	3090
Avco Mod 7	Room Temperature	12	14[1]		
	-100	22	22		
	-150	54	47		
	-200	323	235	3940	2930[3]
Avco Mod 20	Room Temperature	<10	<10		
	-100	46	18		
	-150	83	59		
	-200	196	134	2450	2380
Avco 893-150	Room Temperature	10	10		
	-100	51	35[1]		
	-150	52	74		
	-200	98			
NASA E6A7 Silicone-Phenolic	Room Temperature	<10	<10	52	
	-100	58	59		
	-150	88	102		
	-200	207	90-150[2]	2570	1590

Notes:
1. Approximate average (See Table 10).
2. Extrapolated from plot (Figure 14).
3. Maximum outer fiber stress. Failure indicated by stress-strain curves but not detectable in post test inspection.

TABLE 13

WEIGHT LOSS DATA FOR GROUP 2 SILICONE SPECIMENS

Material	Initial weight grams	Final weight grams	Percent weight loss
Avco Mod 5-18	6.6620	6.5319	1.95
-13	6.8420	6.7106	1.92
-12	6.7870	6.6568	1.91
-11	6.7785	6.6467	1.94
-10	6.8020	6.6732	1.89
- 9	6.8450	6.7127	1.93
Average			1.92
Avco Mod 7-17	6.8740	6.5950	4.05
-28	6.7870	6.6824	1.54
-27	6.9210	6.7317	2.73
-26	6.8825	6.6926	2.75
-25	6.8885	6.6963	2.79
-24	6.7885	6.5990	2.79
Average			2.78
Avco Mod 20-16	6.4086	6.2910	1.83
- 3	6.4690	6.3604	1.67
- 2	6.6805	6.5591	1.81
- 1	6.4540	6.3372	1.80
-30	6.4315	6.3181	1.76
-29	6.4120	6.2951	1.82
Average			1.78
Avco 893-150-19	4.0380	3.9546	2.06
-20	5.1970	4.1041	2.21
-21	4.1400	4.0442	2.31
-22	4.0900	4.0113	1.92
-23	4.1645	4.0736	2.18
-14	4.0855	4.0031	2.01
Average			2.12
NASA E6A7 Silicone Phenolic-15	5.7485	5.6022	2.54
-8	5.7190	5.5781	2.46
-7	5.7320	5.5947	2.39
-6	5.7230	5.5838	2.43
-5	5.7280	5.5767	2.64
-4	5.8140	5.6707	2.46
			2.49

Notes: 1. Weights were measured before and after Sterilization Vacuum Run 2
2. Specimens 14 through 18 were not tested in flexure (located in dead zone).

TABLE 14

FLEXURAL TEST DATA FOR SPECIMENS FROM GROUP 1, RUN 3

Specimen identification	Modulus of elasticity (MOE) in 10^6 psi					MOR psi	Approximate deflection at failure
	RT Prior to Run	RT after[2] Run	-100°F[2]	-150°F[2]	-200°F[2]	-200°F	in 10^{-3} inch
GE1004 (0.5 gm/cm^3)-9	<10	<10	<10	[1]	94		
-10	<10	<10	13	[1]	75		
-11	<10	<10	<10	<10	97		
-12	<10	<10	<10	<10	71		
-13	<10	<10	[1]	<10	83		
		<10		<10	84		
GE 1004 (0.35 gm/cm^3)-4	<10	<10	10	<10	23	334	54
-5	<10	<10	11	<10	29		
		<10		<10	28		
		<10		<10	29		
		<10	[1]	<10	59		
		<10	14	<10	34	334	54
McDonnell B 47RF-19	<10	<10	[1]	[1]	57	090	40
-20	<10	<10	[1]	[1]	63	710	42
-21	<10	<10	<10	<10	74	990	54
-22	<10	<10	<10	<10	78	1040	46
-23	<10	<10	<10	<10	47	720	48
		<10	<10	<10	04	830	46
McDonnell B-46RF-3	<10	<10	40	133	210		
-2	10	<10	16	154	210		
-1	<10	<10	17	164	280	4560	54
-30	<10	11	20	177	340	4500	55
-29	<10	<10	23	155	320	4390	54
Average	<10	<10	24	157	272	4480	54
Martin SLA-561-24	<10	<10	[1]	<10	20	214	27
-25	<10	<10	[1]	<10	30	314	39
-26	<10	<10	<10	10	21	207	39
-27	<10	<10	<10	[1]	25	230	29
-28	<10	<10	<10	[1]	27	302	40
		<10	em	<10	20	265	35

Notes: 1. Not tested
2. Measured after sterilization–vacuum exposure at pressure of 10^{-6} to 10^{-7} torr.

TABLE 15

COMPARISON OF AVERAGE FLEXURAL STRENGTHS AND MODULI OF AS-RECEIVED AND EXPOSED SPECIMENS FROM GROUP 1

Material	Average Test Temperature °F	MOE in 10^3 psi		MOR, psi	
		As Received	Exposed	As Received	Exposed
GE 1004 (0.5 gm/cm^3)	RT	<10	<10		
	-100	<10	<10		
	-150	28	<10		
	-200	105	84		
GE 1004 (0.35 gm/cm^3)	RT	<10	<10		
	-100	<10	14		
	-150	16	10		
	-200	96	34	980[1]	334[1]
McDonnell B 47RF	RT	<10	<10		
	-100	<10	<10		
	-150	21	<10		
	-200	106	64	1820[2]	830[3]
McDonnell B 45RF	RT	<10	<10		
	-100	72	24		
	-150	145	157		
	-200	404	272		4480[4]
Martin SLA-561	RT	<10	<10		
	-100	<10	<10		
	-150	<10	<10		
	-200	22	26	271[3]	265[3]

Notes- 1. One of four specimens tested failed at this temperature.
2. All four specimens tested failed at this temperature,
3 All five specimens tested failed at this temperature.
4 Three of five specimens tested failed at this temperature.

TABLE 16

WEIGHT LOSS DATA FOR GROUP 1

Material	Initial weight grams	Final weight grams	Percent weight loss
GE1004 (0.5 gm/cm^3)-1-17	5.1052	5.0717	
- 9	4.9037	4.8714	
-10	4.6470	4.6176	
-11	4.6725	4,6426	
-12	4.8066	4.7741	
-13	4.6661	4.6371	
GE1004 (0.35 gm/cm^3)-2-15	3.4523	3.4206	
- 4	3.3185	3.2906	
- 5	3.3112	3.2801	
- 6	3.2542	3.2278	
- 7	3.3250	3.2981	
- 8	3.4154	3.3869	
McDonnell B47RF-3-14	3.8864	3.8036	
-23	3.6487	3.5693	
-22	3.7753	3.6861	
-21	3.6220	3.5373	
-20-	3.7166	3.6318	
-19	3.6804	3.5962	
McDonnell B45RF-4-18	5.9619	5.8684	1.56
- 3	5.5377	5.4494	1.59
- 2	5.6191	5.5293	1.59
- 1	5.5946	5.5069	1.56
-30	5.6557	5.5654	1.59
-29	5.5720	5.4833	1.59
			1.58
Martin SLA-561-5-16	1.9323	1.8703	3.20
-28	1.8091	1.7570	2.87
-27	1.7681	1.7173	2.87
-26	1.7132	1.6700	2.52
-25	1.7979	1.7319	3.67
-24	1.8281	1.7727	3.03
		Average	3.03

TABLE 17

FLEXURAL TEST DATA FOR SPECIMENS FROM GROUP 3, RUN 4

Specimen identification	Modulus of elasticity (MOE) in 10^3 psi					MOR' psi -200°F	Approximate deflection at failure in 10^{-3} inch
	RT prior to run	RT[6]	-100°F[6]	-150°F	-200°F		
Avco Teflon Foam (0.75 gm/cm³) -22	<10	<10	24	28	41 [2,3] 50 [2,4]		
-23	<10	<10	18	29	54 [3] 46 [2,4] 63 [2,5]		
-24	<10	<10	24	45	61 [3] 61 [4] 75 [2,5]		
-25	1	14	28	41	61 [3] 57 [2,4] 59 [2,5]		
-26	1	10	28	41	55 [2,3] 62 [2,4] 68 [2,5]		
Average	<10	—	25	37	54 [3] 55 [4] 66 [5]		
Avco Teflon Foam (0.54 gm/cm³) -1[7]	1	<10	18	17	17 [2]		
-2	1	<10	<10	<10	16 [2]		
-3	<10	<10	<10	<10	14 [2]		
-4	<10	<10	<10	<10	15 [2]		
-5	<10	<10	<10	<10	18 [2]		
Average	<10	<10	<10	<10	16		
Boeing Carborazole -7	<10	<10	194	157	160	3520	74
-8	<10	<10	118	127	125	2970	70
-9	1	1	142	142	135	2920	65
-10	1	1	161	149	148	3680	76
-11	1	1	185	163	187	3680	67
Average			140	149	151	3350	70

Notes. 1. Not tested.
2. Deflected more than 0.070 in. without failure.
3. First run. Average test temperature -190°F
4. Second run. Average test temperature -194°F
5. Third run. Average test temperature -200°F
6. Measured at conclusion of sterilization-vacuum exposure at pressure of—2 x 10^{-6} torr
7. 0.229 in. thick,

TABLE 18

COMPARISON OF AVERAGE FLEXURAL STRENGTHS AND MODULI OF AS-RECEIVED AND EXPOSED SPECIMENS FROM GROUP 3

Material	Average Test Temperature °F	MOE in 10^3 psi		MOR, psi	
		As Received	Exposed	As Received	Exposed
		<10			
	---	23	25		
	-150	43	37		
	-200	76	54		
Avco Teflon Foam (0.54 gm/cm^3)	RT	<10	<10		
	-100	<10	<10		
	-150	<10[2]	<10		
	-200	13	16		
Boeing Carborazole	RT	<10	<10		
	-100	134	140		
	-150	152	149		
	-200	172	151	2600[3]	3350[4]

Notes:
1. Three of five specimens had moduli $<10^4$ psi (see Table 4).
2. Two of five specimens had moduli <1d psi.
3. Four of five specimens tested failed at this temperature.
4. All five specimens tested failed at this temperature

TABLE 19

WEIGHT LOSS DATA FOR RUN 4, GROUP 3

Material	Initial weight grams	Final weight grams	Total weight loss	Percent weight loss
Avco Teflon Foam (0.75 gm/cm^3)				
-1-22	7.3173	7.3160	0.0013	0.02
23	7.4887	7.4875	0.0012	0.02
24	7.3618	7.3617	0.0001	-
25	7.2391	7.2386	0.0005	0.01
26	7.6196	7.6188	0.0008	0.01
	Average			0.01
Avco Teflon Foam (0.54 gm/cm^3)				
-2-1	4.8009	4.7998	0.0011	0.02
2	5.3247	5.3228	0.0019	0.04
3	5.2600	5.2592	0.0008	0.02
4	5.2206	5.2197	0.0009	0.02
5	5.2820	5.2808	0.0012	0.02
	Average			0.02
Boeing Carborazole				
-3-7	5.1877	4.7948	0.3929	7.57
8	5.0667	4.7059	0.3608	7.12
9	5.0467	4.6902	0.3565	7.06
10	5.3502	4.9501	0.4001	7.47
11	5.3265	4.9533	0.3732	7.00
15	5.0270	4.6744	0.3526	7.01
	Average			7.21

TABLE 20

WEIGHT LOSSES FROM TGA MEASUREMENTS

	Percent weight loss at 750°C		
	As Received	Exposed	Remarks
Group 1			
GE 1004 AP-20	52	50	Weight loss complete at 600°C
GE 1004 AP-35	59	58	Weight loss complete at 600°C
McDonnell B45RF	51	50	Weight loss complete at 700°C
McDonnell B47RF	70	70	Weight loss complete at 700°C
Martin SLA-561	60	60	Still losing weight at 750°C
Group 2			
Avco Mod 5	74	71	Unexposed specimen losing weight at 70°C. Weight loss complete for exposed specimen at 700°C.
Avco Mod 7	76	76	Gradually losing weight at 750°C
Avco Mod 20	70	76	Weight loss complete at 700°C
Avco 893-150	90	83	Gradually losing weight at 750°C
NASA E6A7 Silicone-Phenolic	76	88	Gradually losing weight at 750°C
Group 3			
Avco Teflon 0.75 gm/cm^3	98	100	Weight loss complete at 600°C
Avco Teflon 0.54 gm/cm^3	98	100	Weight loss complete at 700°C
Boeing Carborazole	68	64	Weight loss essentially complete at 750°C
Group 4			
Avco 5026-39	71	64	Losing weight at 750°C
Avco 5026-99	62	54	Losing weight at 750°C
NASA 50-50 Low-Density Phenolic-Nylon	80	80	Losing weight at 750°C

TABLE 21

SUMMARY OF EFFECTS OF LOW TEMPERATURE AND STERILIZATION-VACUUM ON MATERIALS

Run no.		No Failure		Increase in Modulus with Decreasing Temperature	Decrease in Modulus after Exposure	Exposure	Percent Weight Loss	Remarks
	Group 1 Silicone Elastomers							
3	1. GE 1004 AP-35 lb/ft³	0/4	0/5	Large (8-10X)	Moderate (~15%)	-	0.64	
	2. GE 1004 AP-20 lb/ft³	1/4	1/5	Large (3-10X)	Slight above - 150°F Large below - 150°F	Large (~300%)	0.86	
	3. McDonnell B47RF	4/4	5/5	Large (6-10X)	Moderate (~50%)	Large (>50%)	2.26	
	4. McDonnell B45RF	0/5	3/3	Large (27-40X)	Moderate (30-60%)	-	1.58	
	5. Martin SLA-561	5/5	5/5	Moderate (~2X)	Increased ~20%	Insignificant	3.03	
	Group 2 Silicone Elastomers							
2	1. Avco Mod 5	4/4	5/5	Large (20-30X)	Moderate (30-60%)	Moderate (25%)	1.92	
	2. Avco Mod 7	1/4	0/3 [1]	Large (20-30X)	Moderate (~25%)	Moderate (25%)	2.78	
	3. Avco Mod 20	5/5	5/5	Large (13-20X)	Moderate (30-60%)	Slight (< 5%)	1.78	
	4. Avco 893-150 High Cork	0/5	0/4 (-160°F) [2]	Large (10X)	Moderate (30%)	-	2.12	
	5. NASA E6A7 Silicone-Phenolic	3/4	3/3	Large(10-20X)	Slight to moderate	Large (40%)	2.49	Very low strength at room temperature
	Group 3							
4	1. Avco Teflon Foam - 0.75 gm/cm³	0/4	0/5	Large (5-8X)	Slight to moderate	-	0.01	
	2. Avco Teflon Foam - 0.54 gm/cm³	0/5	0/5	Moderate (30-60%)	0 to slight increase	-	0.02	
	3. Boeing Carborazole	4/5	5/5	Large (15-20X)	Slight (~ 15%)	~30% increase	7.21	
	Group 4							
1	1. Avco 5026-39	0/5	0/5 [3]	Large (~6X)	Moderate (~ 25%)	-	2.14	
	2. Avco 5026-99	5/5	2/4	Large (~4X)	Moderate (25% max)	Moderate (25%)	3.39	
	3. NASA 50-50 Low Density Phenolic Nylon	5/5	1/5 [3]	Moderate (~25%)	Moderate (~ 25%)	Large (3X)	0.73	

Notes

1. No visible evidence of failure after test although modulus decreased sharply during test.
2. Lowest test temperature.
3. Deflected only 0.025 inch at -200°F

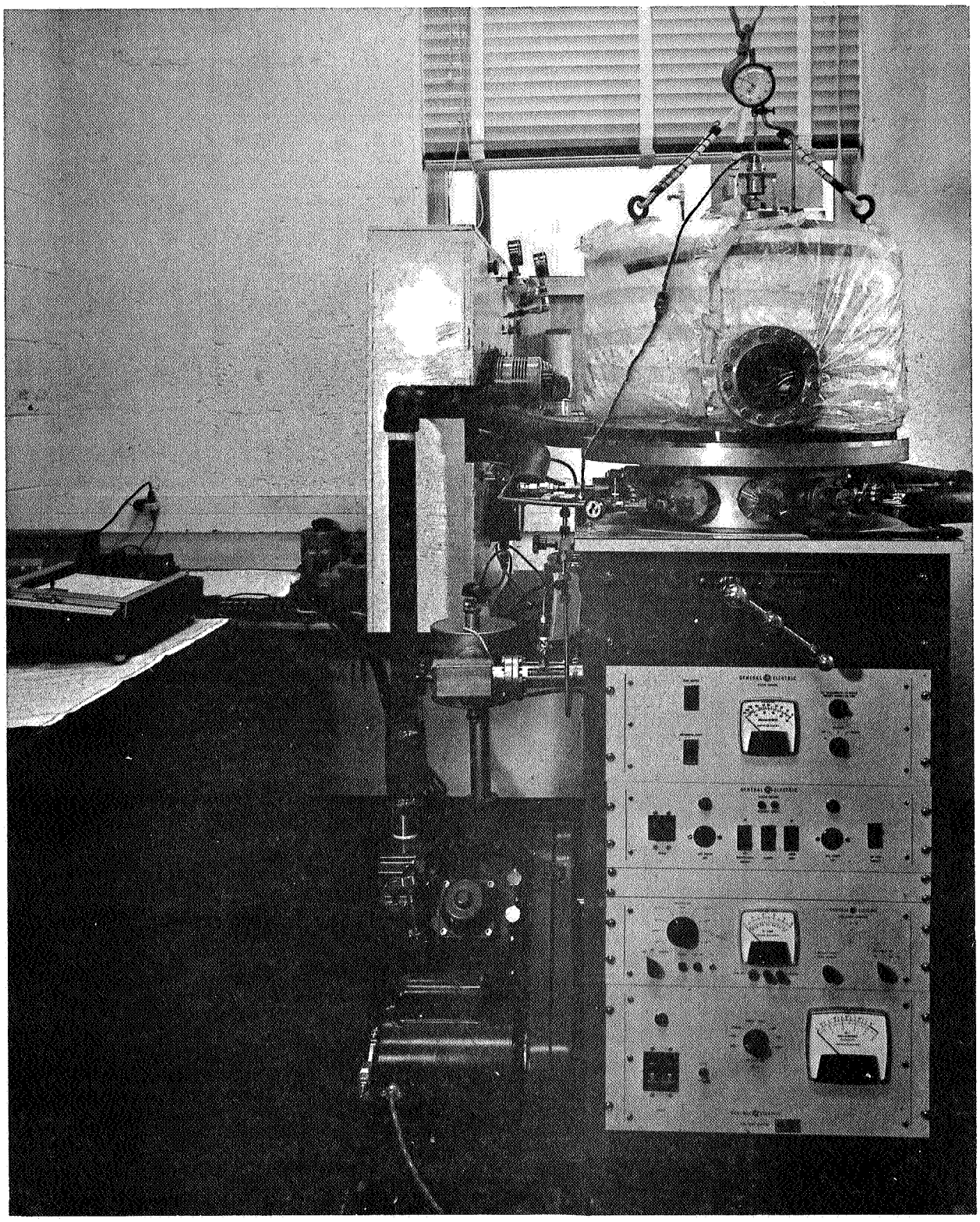


Figure 1. Photograph of sterilization–vacuum apparatus

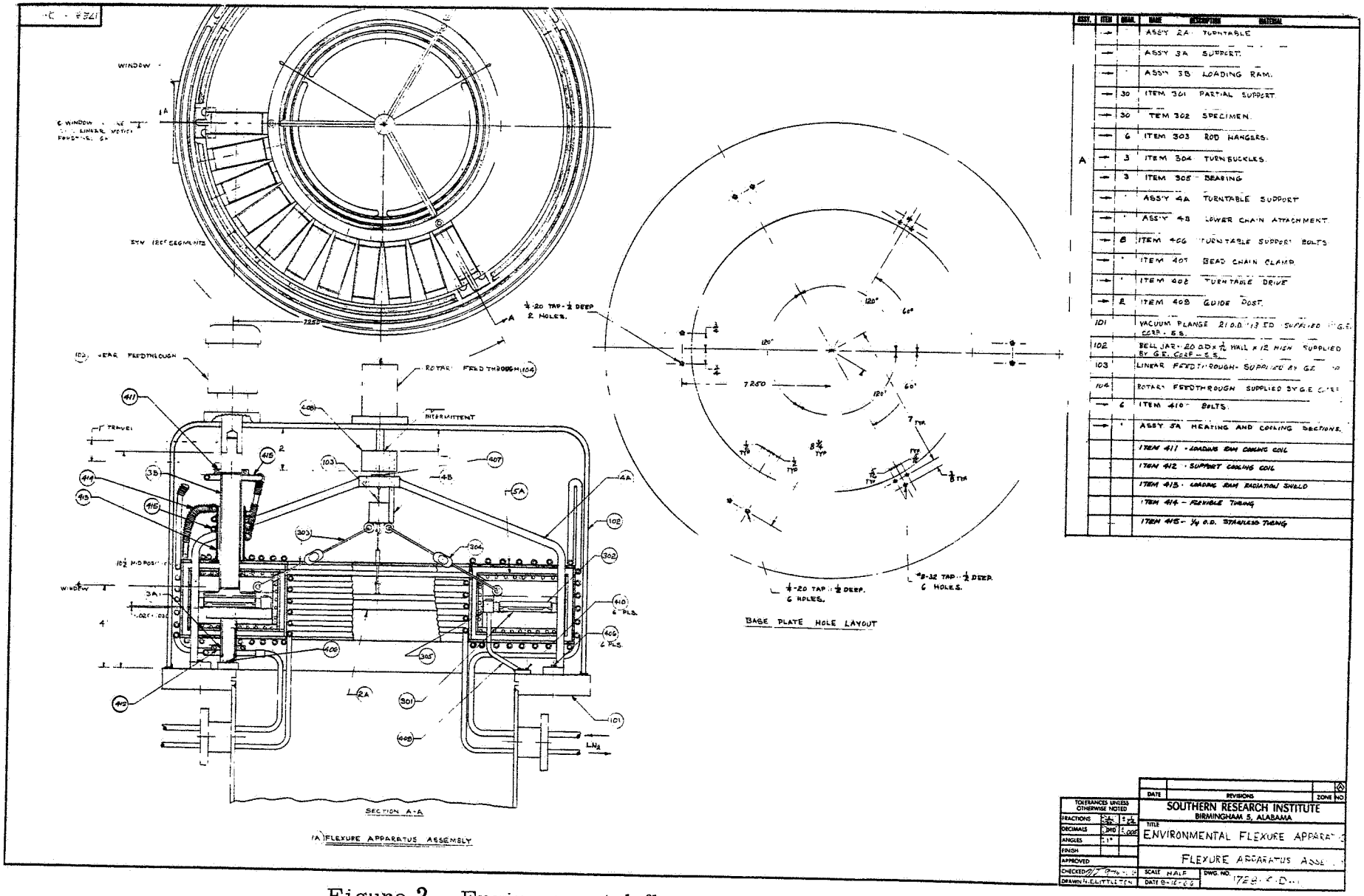


Figure 2. Environmental flexure apparatus

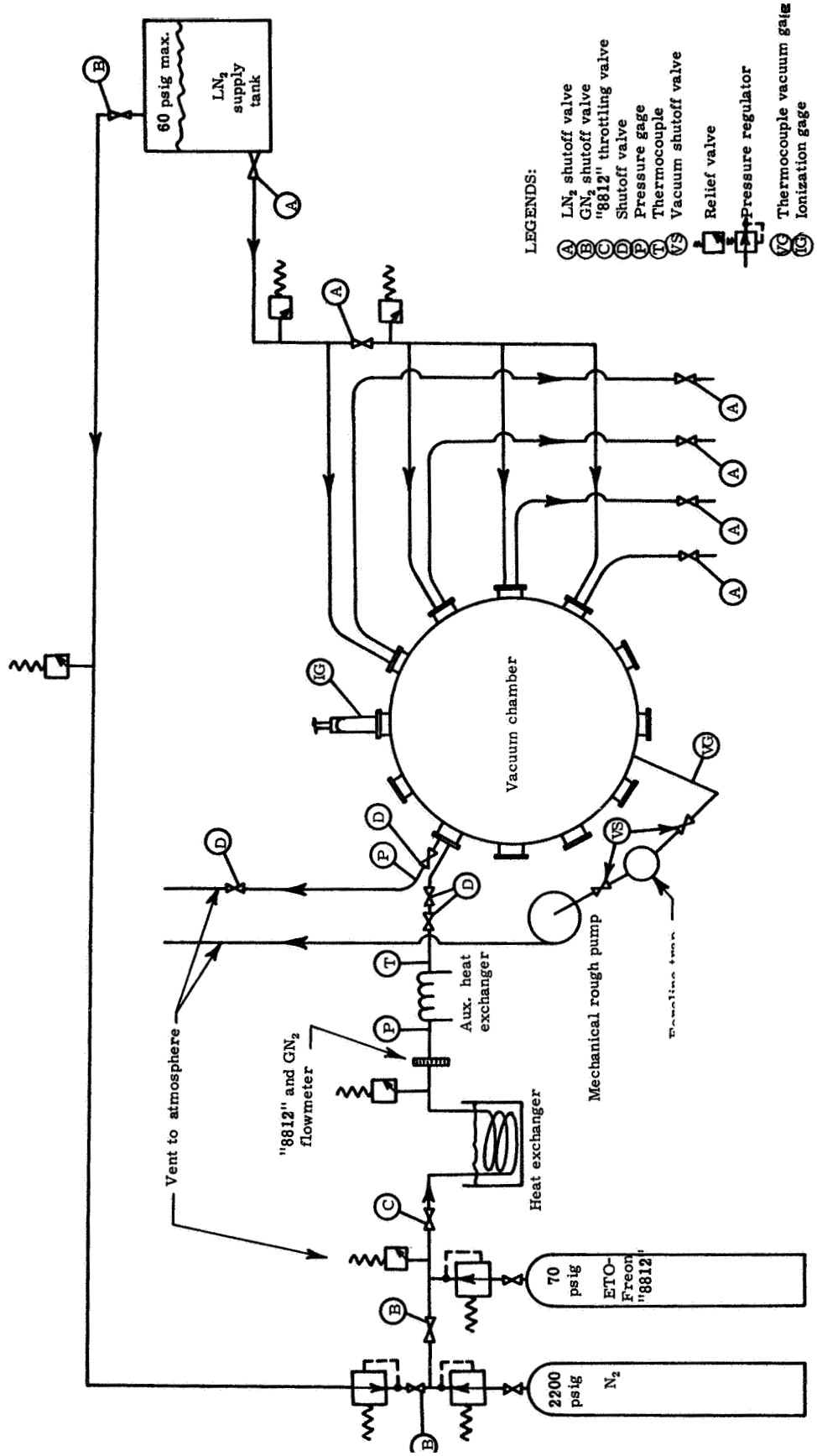


Fig. 3. Flow schematic - environmental control system

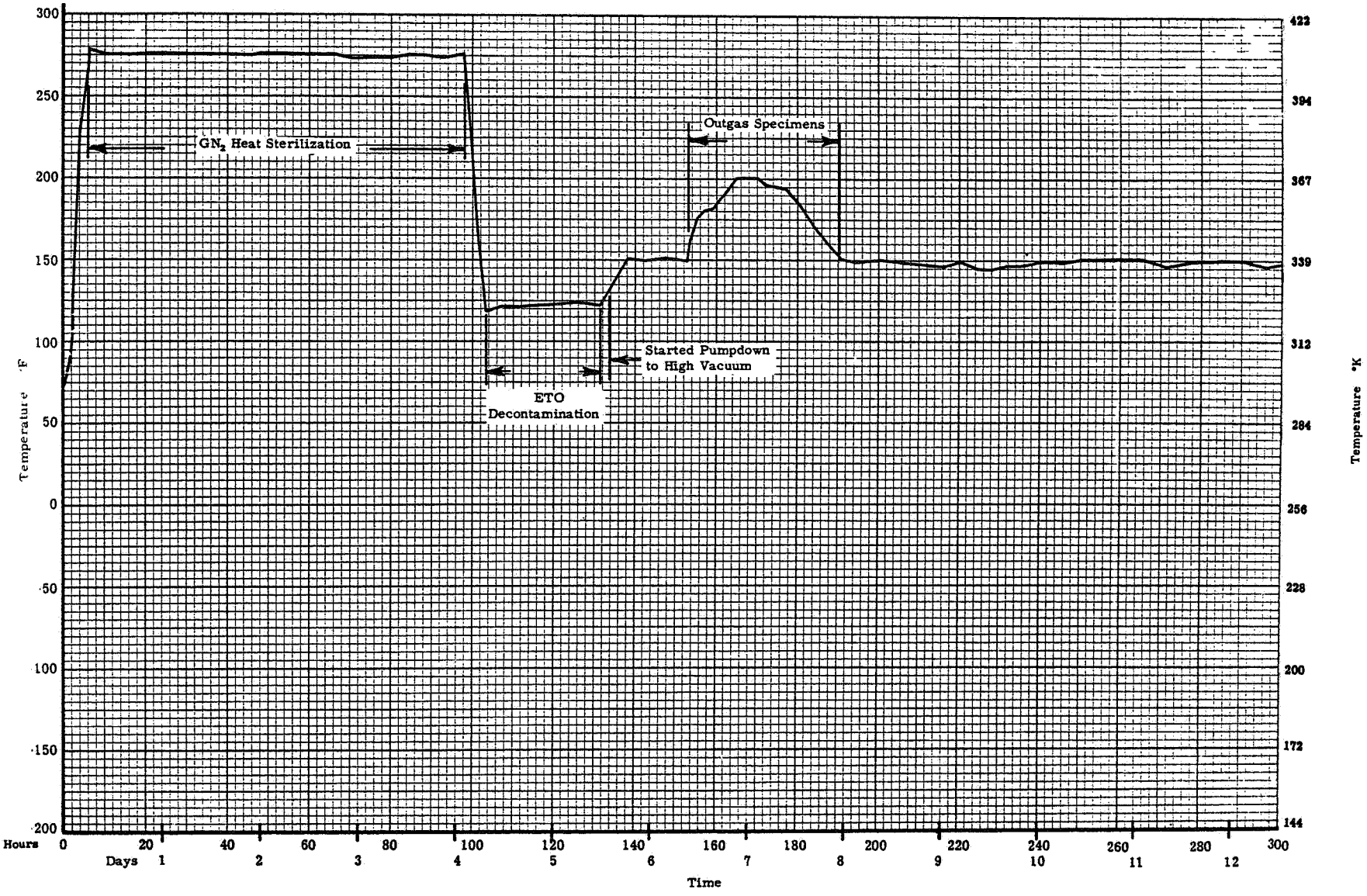


Figure 4. Plot of Average Specimen Temperature-Time History for Run 1

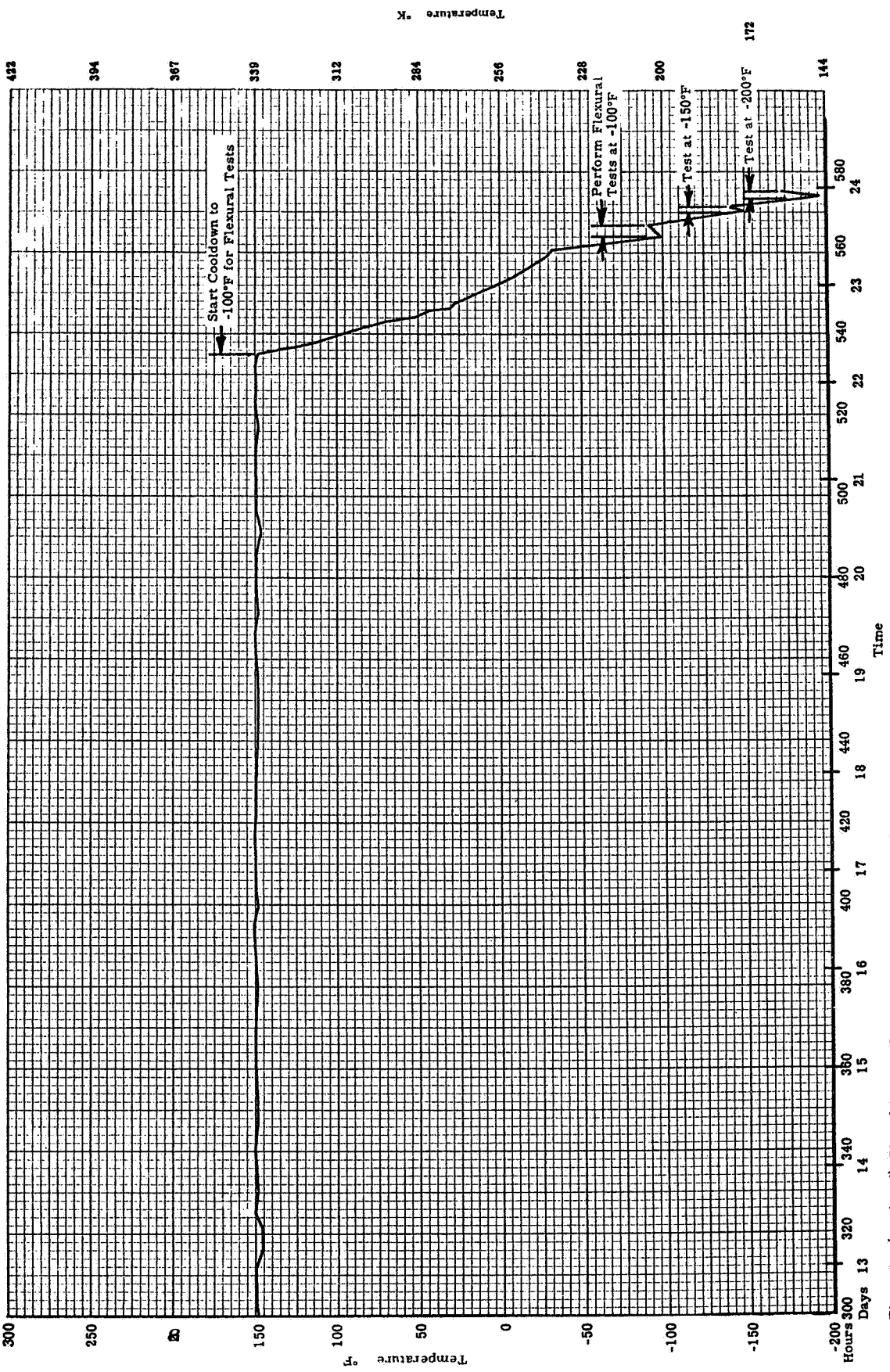


Figure 4. (continued) Plot of Average Specimen Temperature-Time History for Run 1

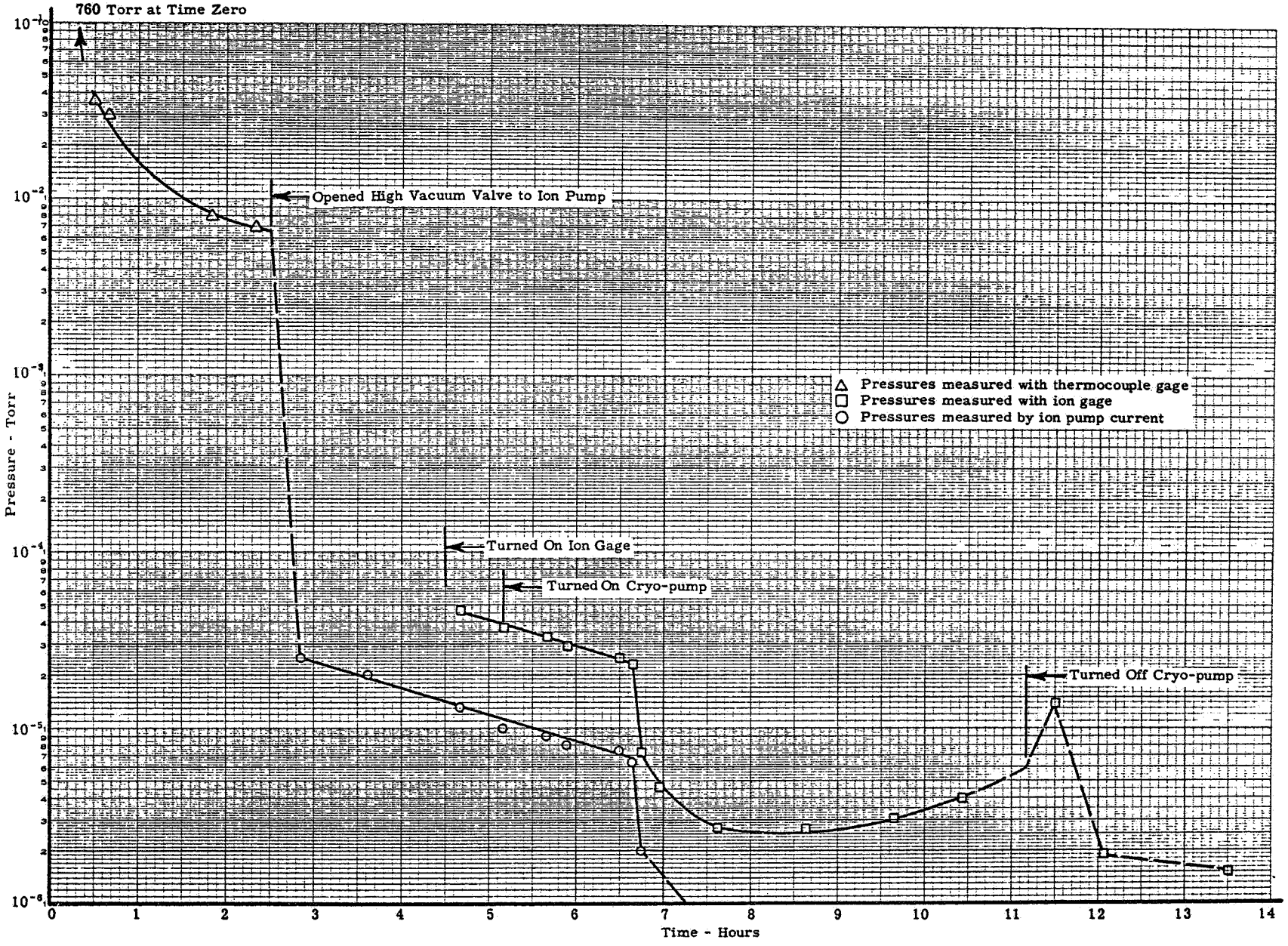


Figure 5 Pressure-Time History during Initial Portion of Vacuum Cycle - Run 1

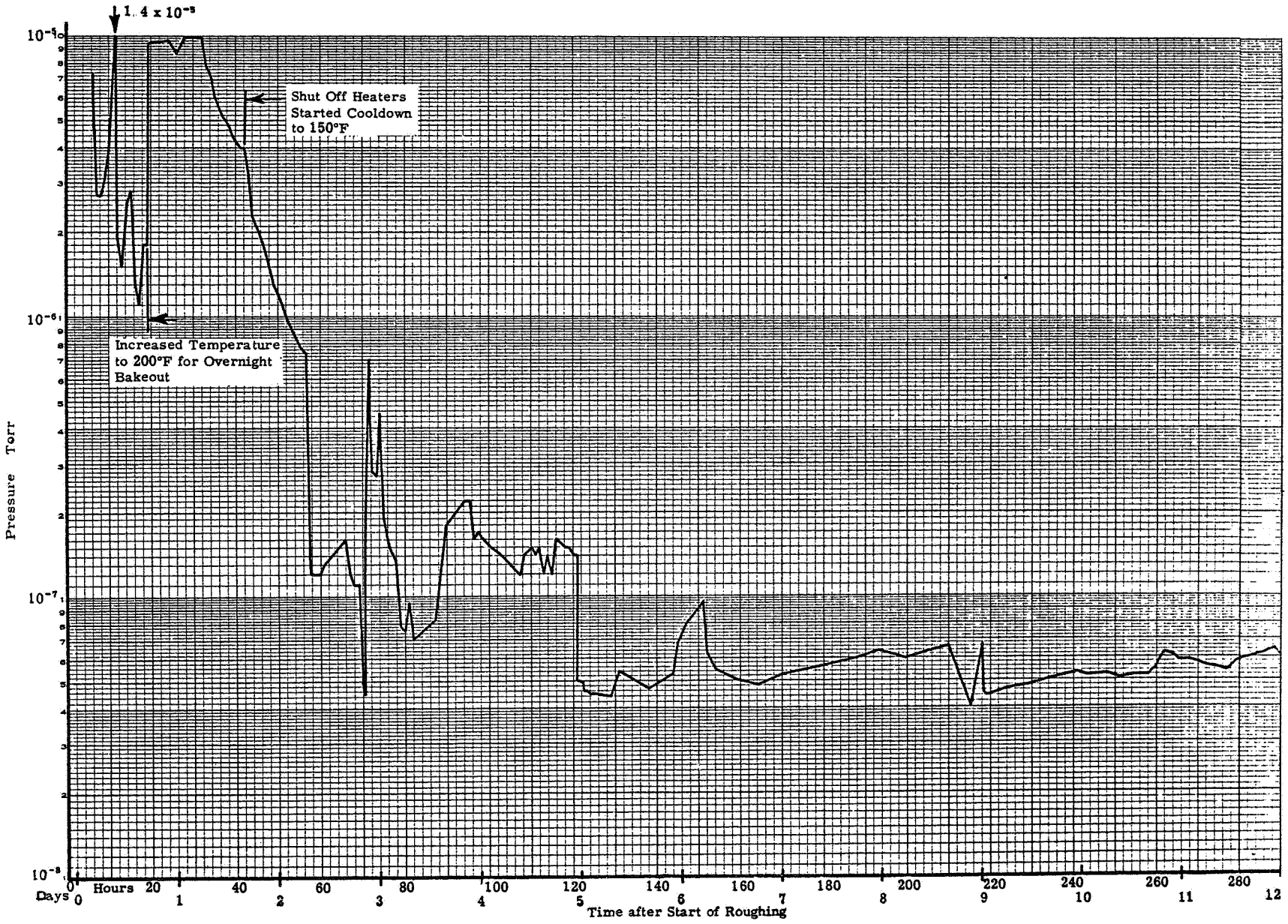


Figure 6 Pressure-Time History during [illegible] Exposure - Run 1

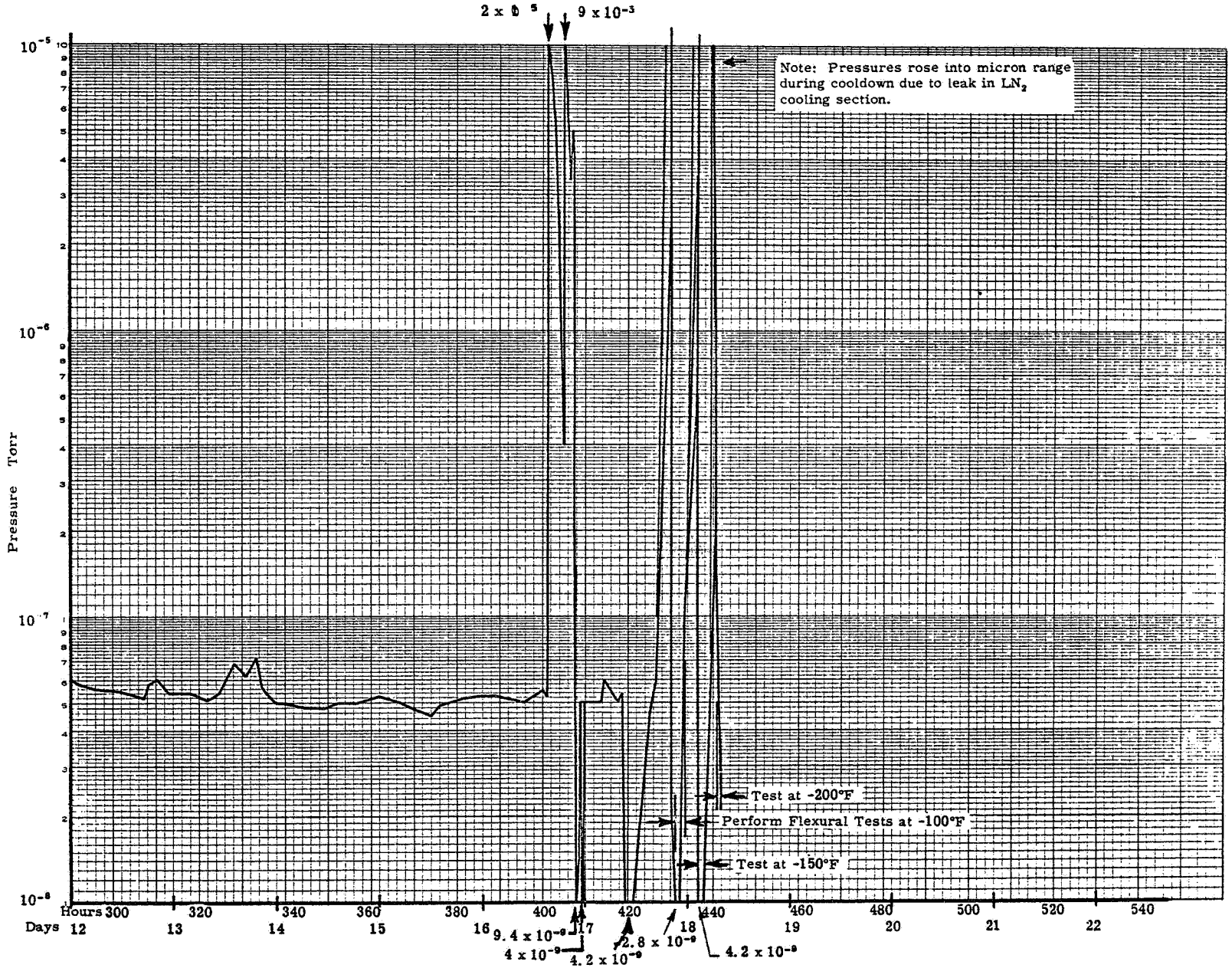


Figure 6 (continued) Pressure-Time History during Vacuum Exposure Run 1

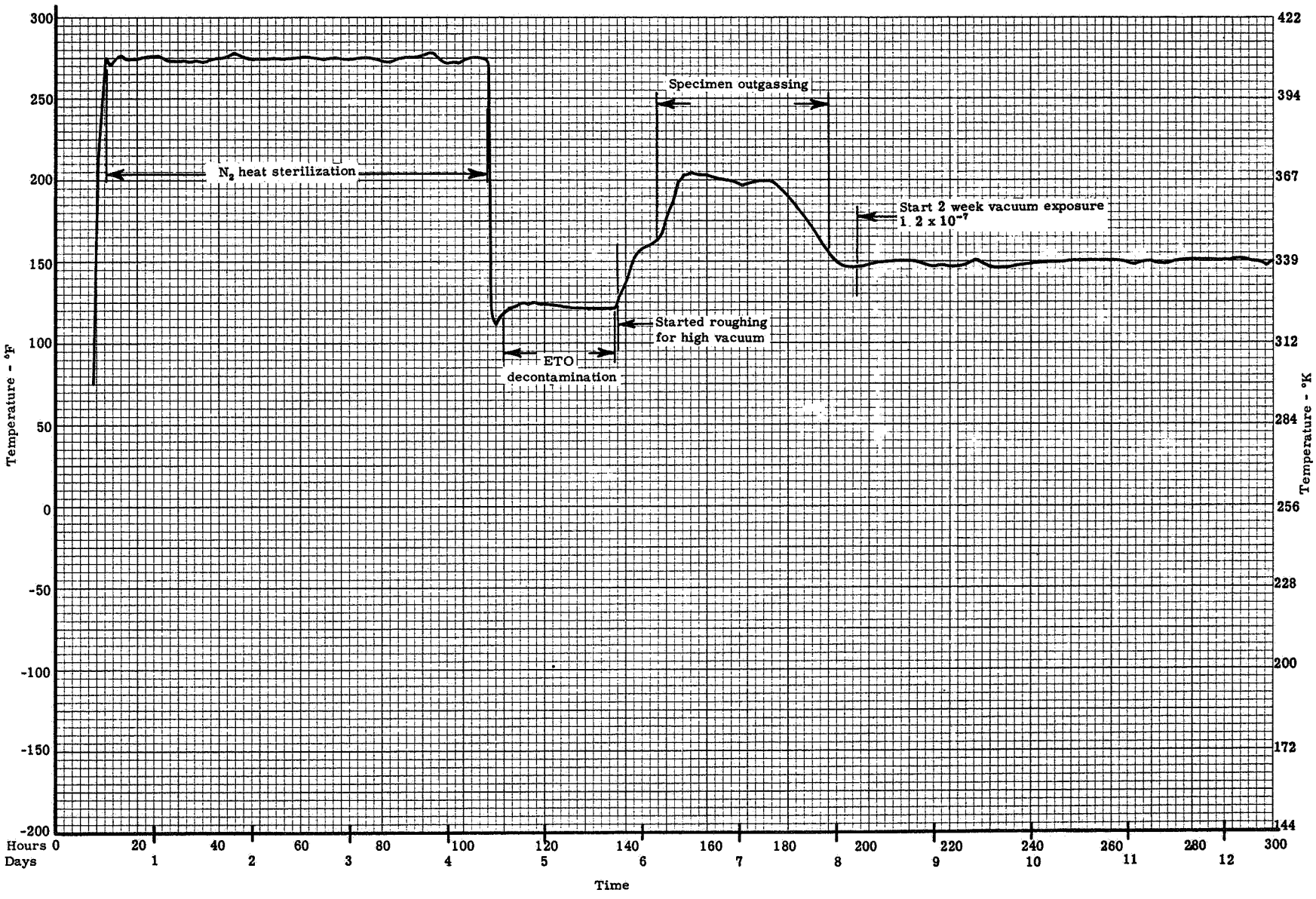


Figure 7. Plot of average spe im ∩ èmω ττκu e e ∃ ∃ time for Group 2 (silicone) specimens during Sterilization-Vacuum Run 2

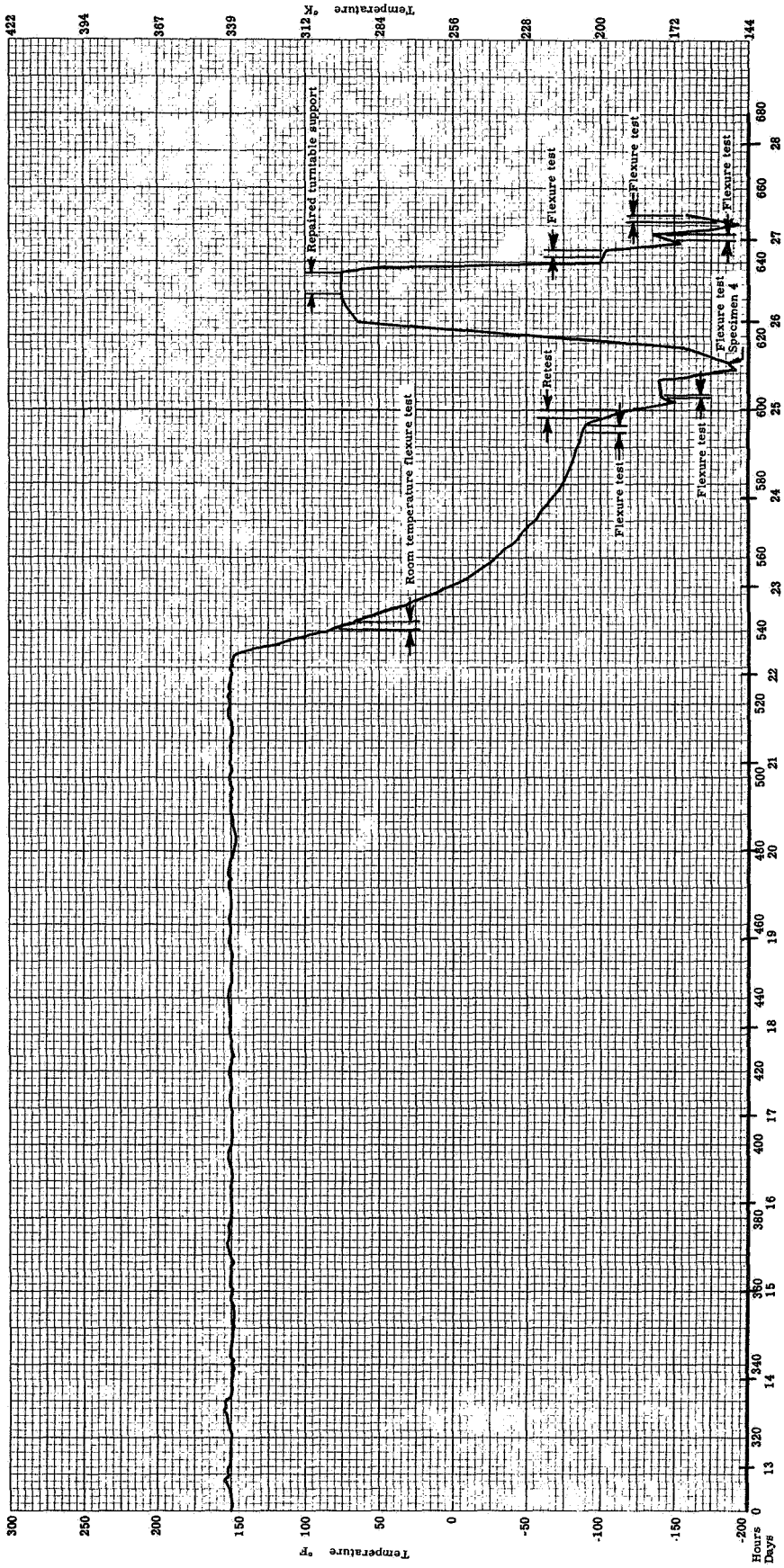


Figure 7. (continued) Plot of average specimen temperature versus time for Group 2 (silicone) specimens during Sterilization - Vacuum Run 2

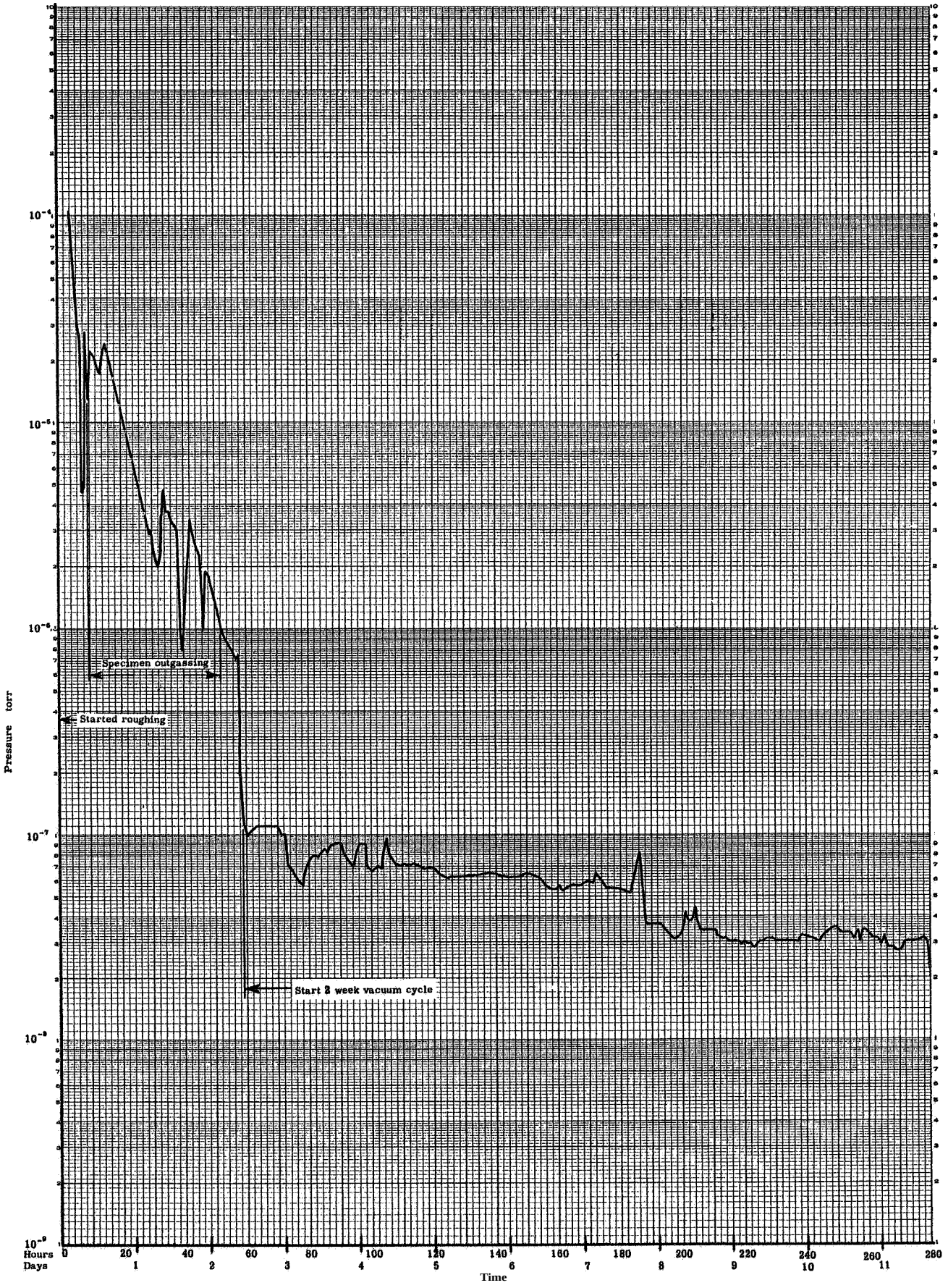


Figure 8. Pressure versus time during vacuum exposure portion of Run 2

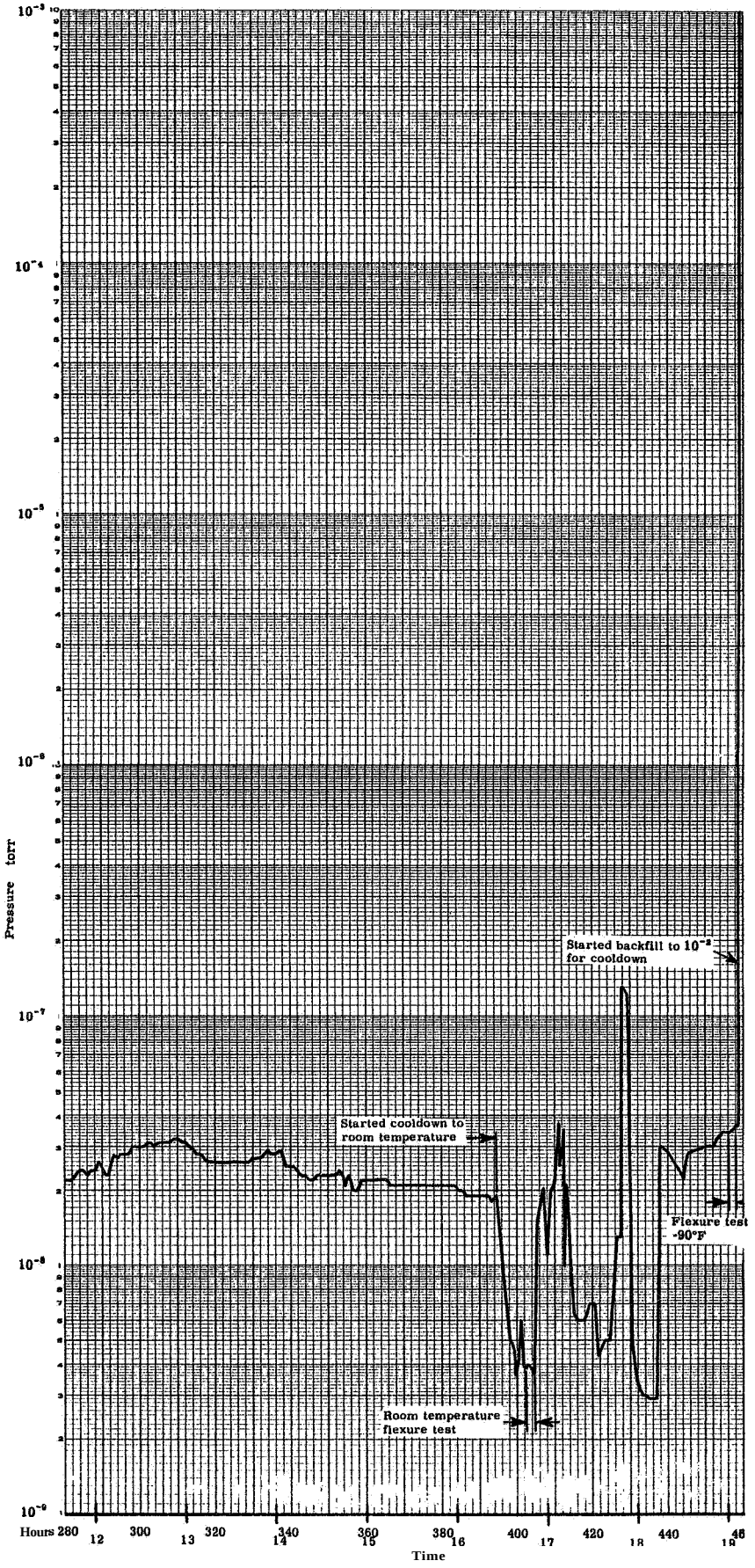


Figure 8. (continued) Pressure versus time during vacuum exposure portion of Run 2

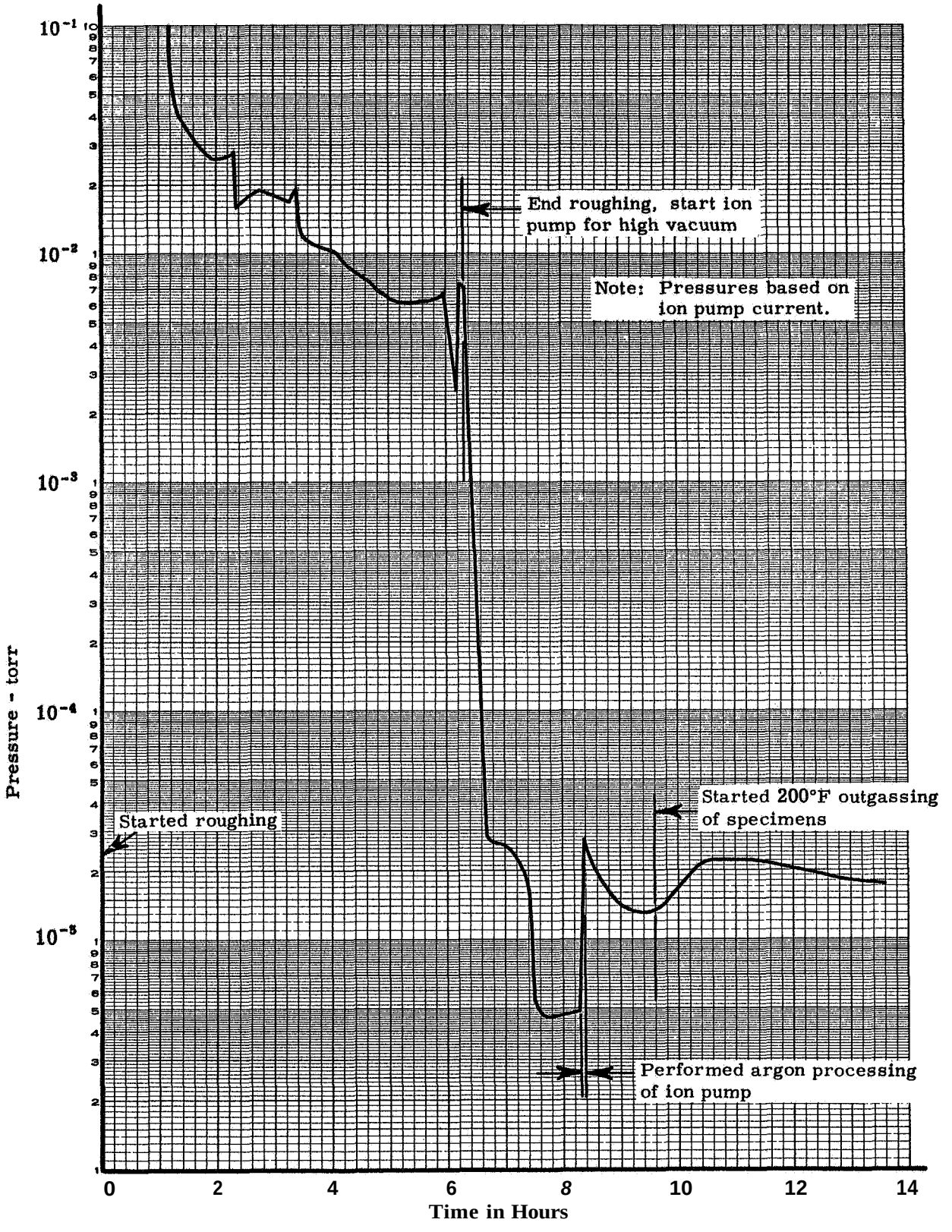


Figure 9. Pressure versus time during initial portion of Vacuum Exposure Run 2

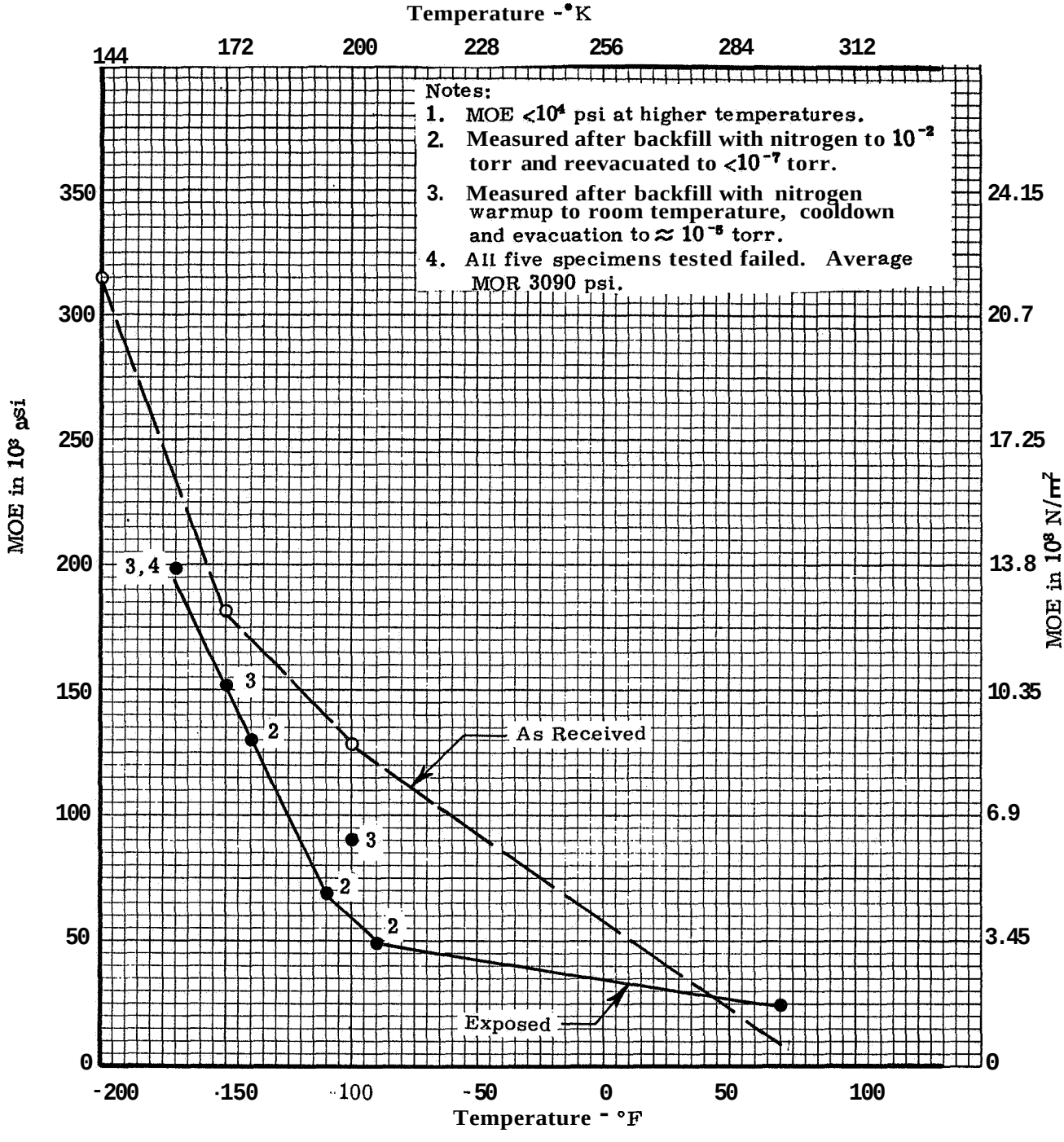


Figure 10. Comparison of flexural modulus of Avco Mod 5 before and after exposure to sterilization vacuum environment

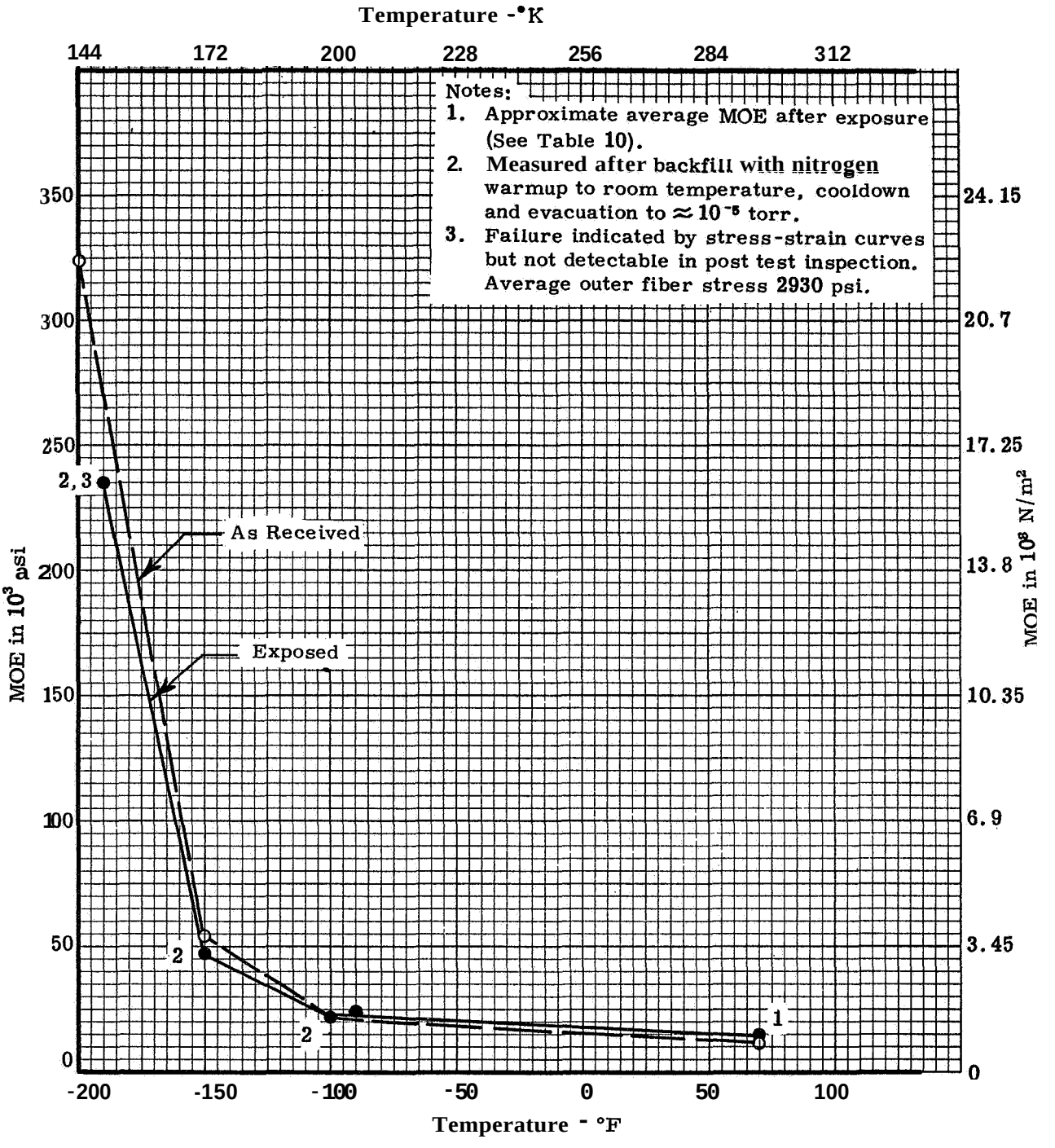


Figure 11. Comparison of flexural modulus of Avco Mod 7 before and after exposure to sterilization vacuum environment

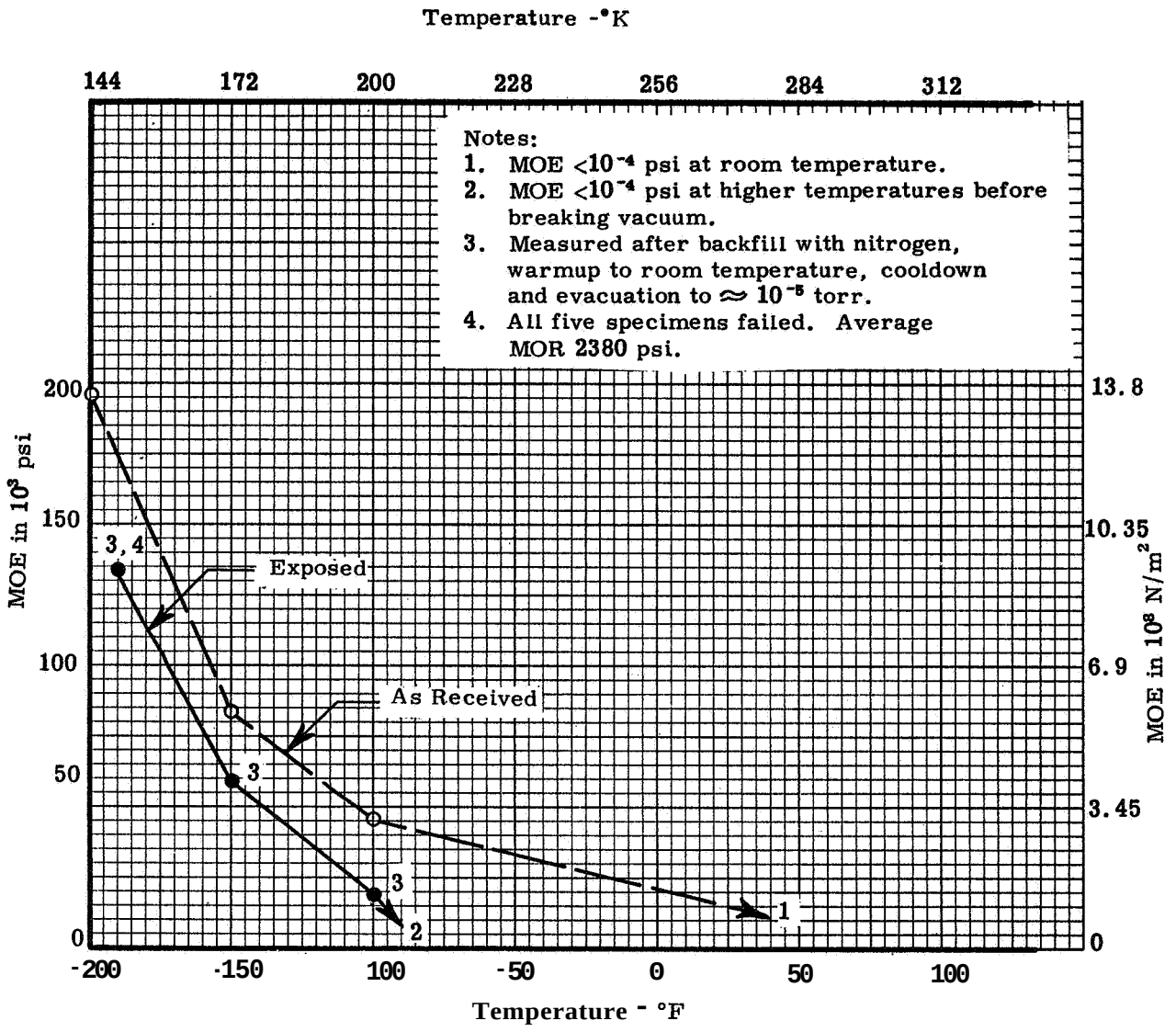


Figure 12. Comparison of flexural modulus of Avco Mod 20 before and after exposure to sterilization vacuum environment

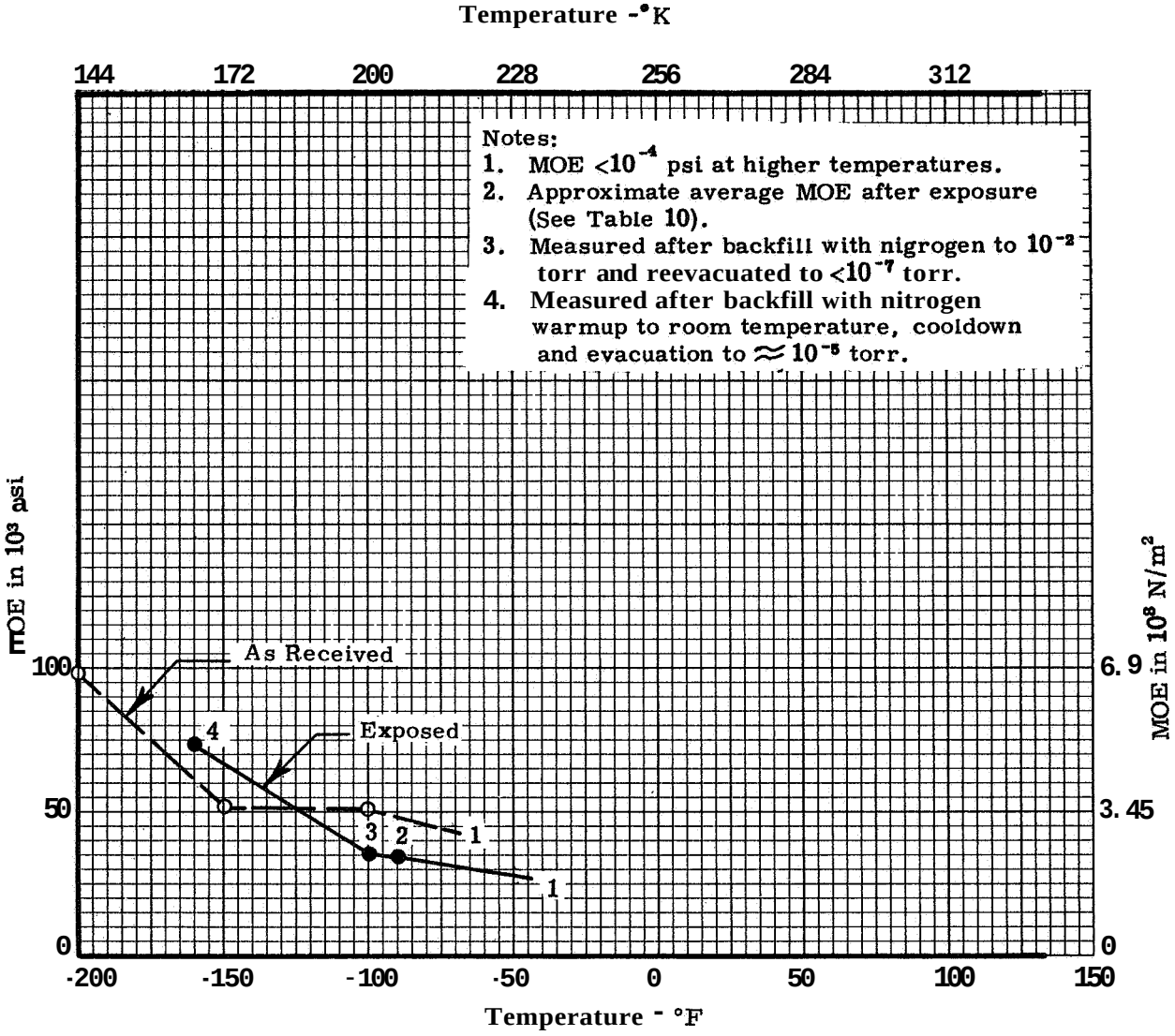


Figure 13. Comparison of flexural modulus of Avco 892-150 before and after exposure to sterilization vacuum environment

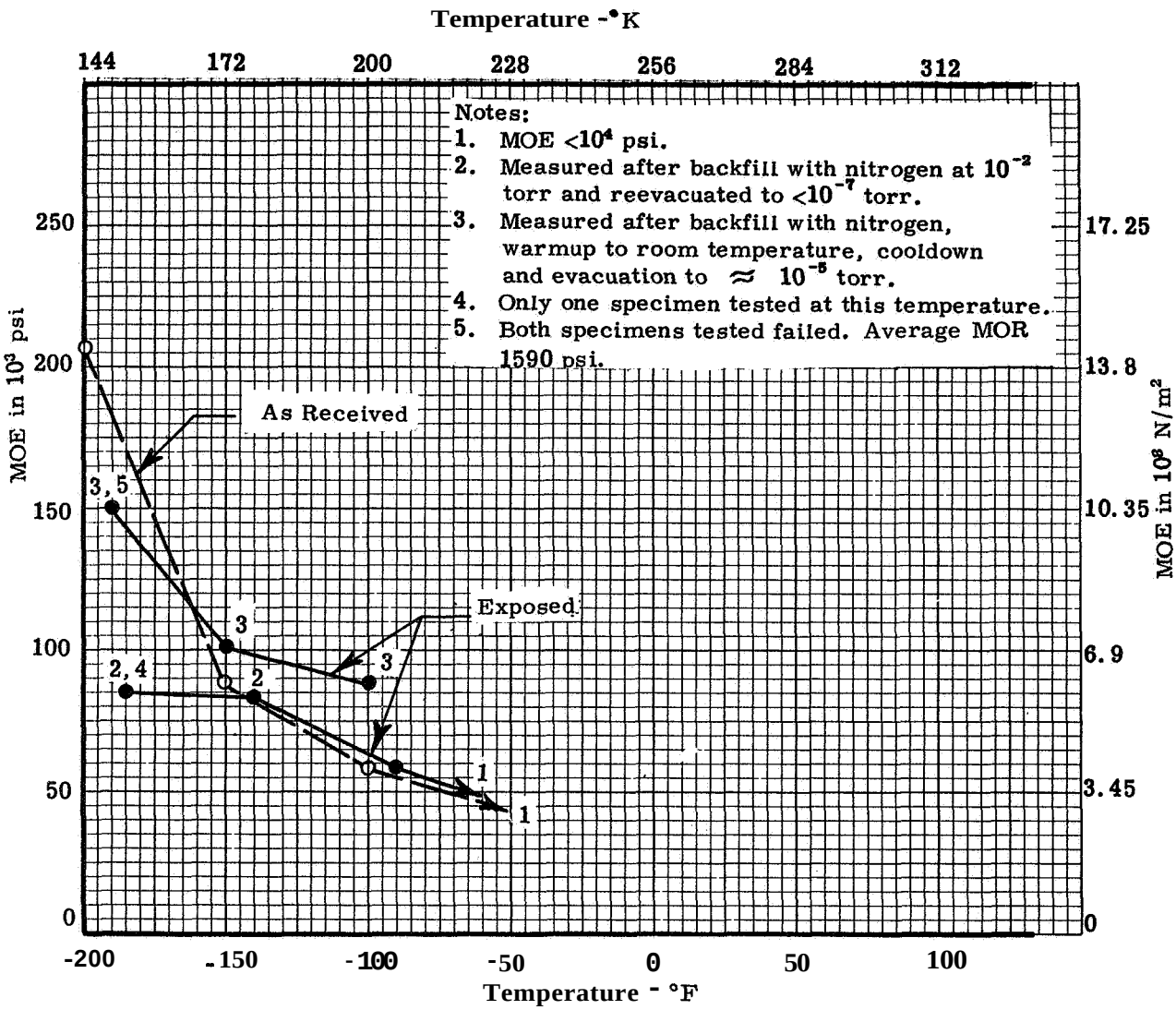


Figure 14. Comparison of flexural modulus of NASA E6A7 silicone-phenolic before and after exposure to sterilization vacuum environment

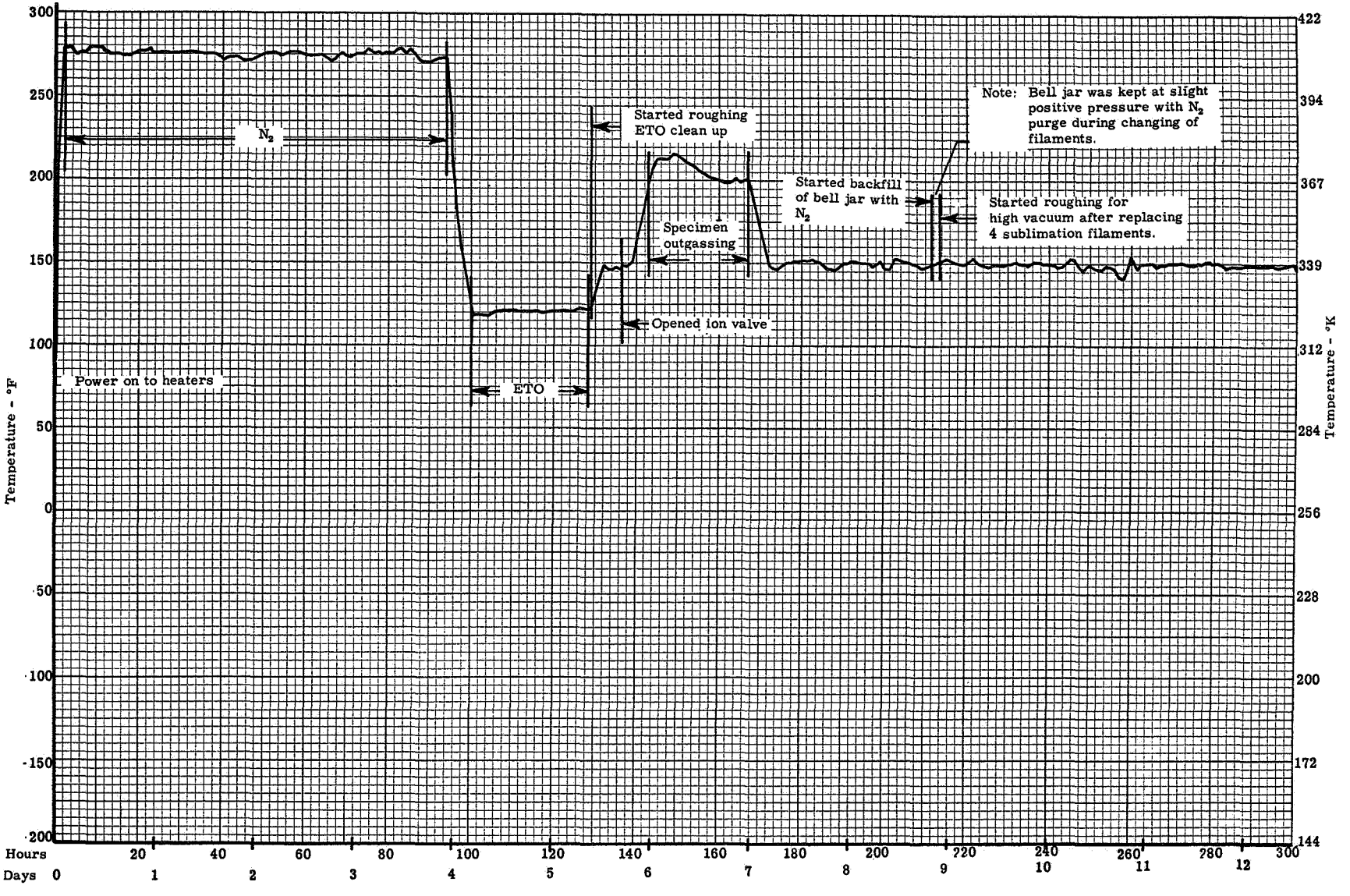


Figure 15. Plot of average specimen temperature vs time for Group 1 (Silicone) ● imms during Sterilization-Vacuum Run 3

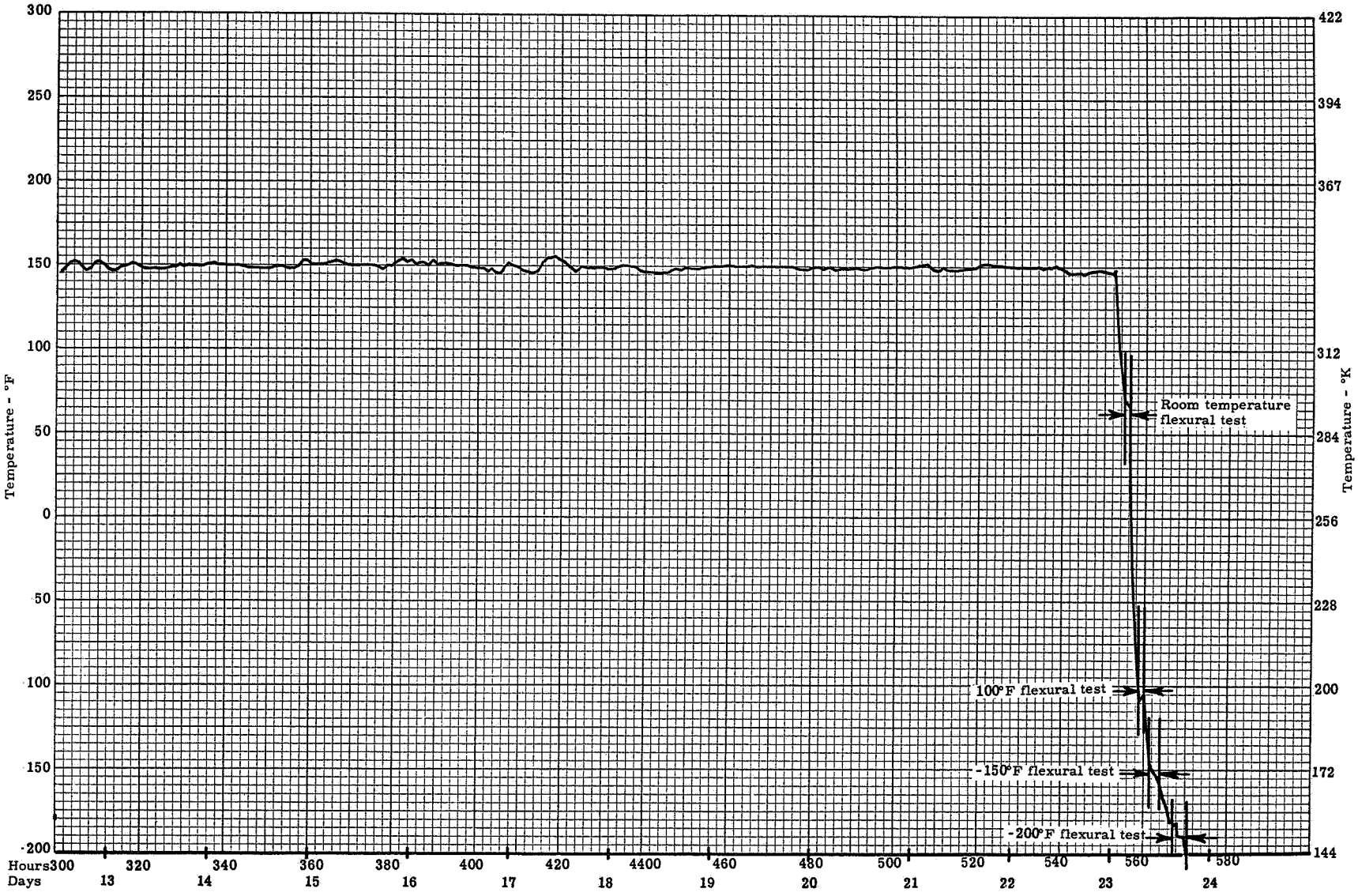


Fig 15. (continued) Plot of average specimen temperature vs time for Group 1 (Silicone) specimens during Sterilization-Vacuum Run 3

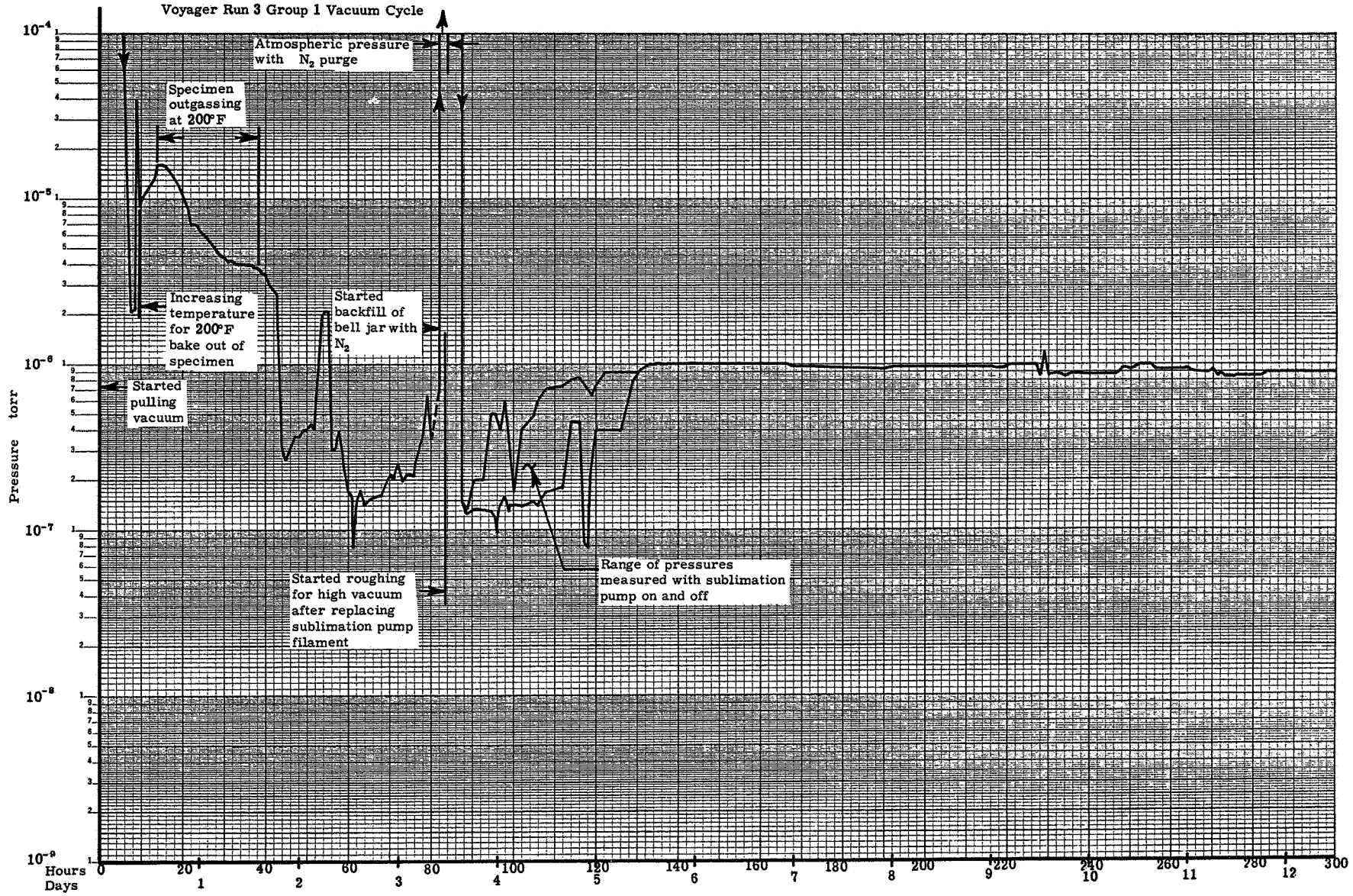


Figure 16. Pressure versus time during vacuum exposure portion of Run 3

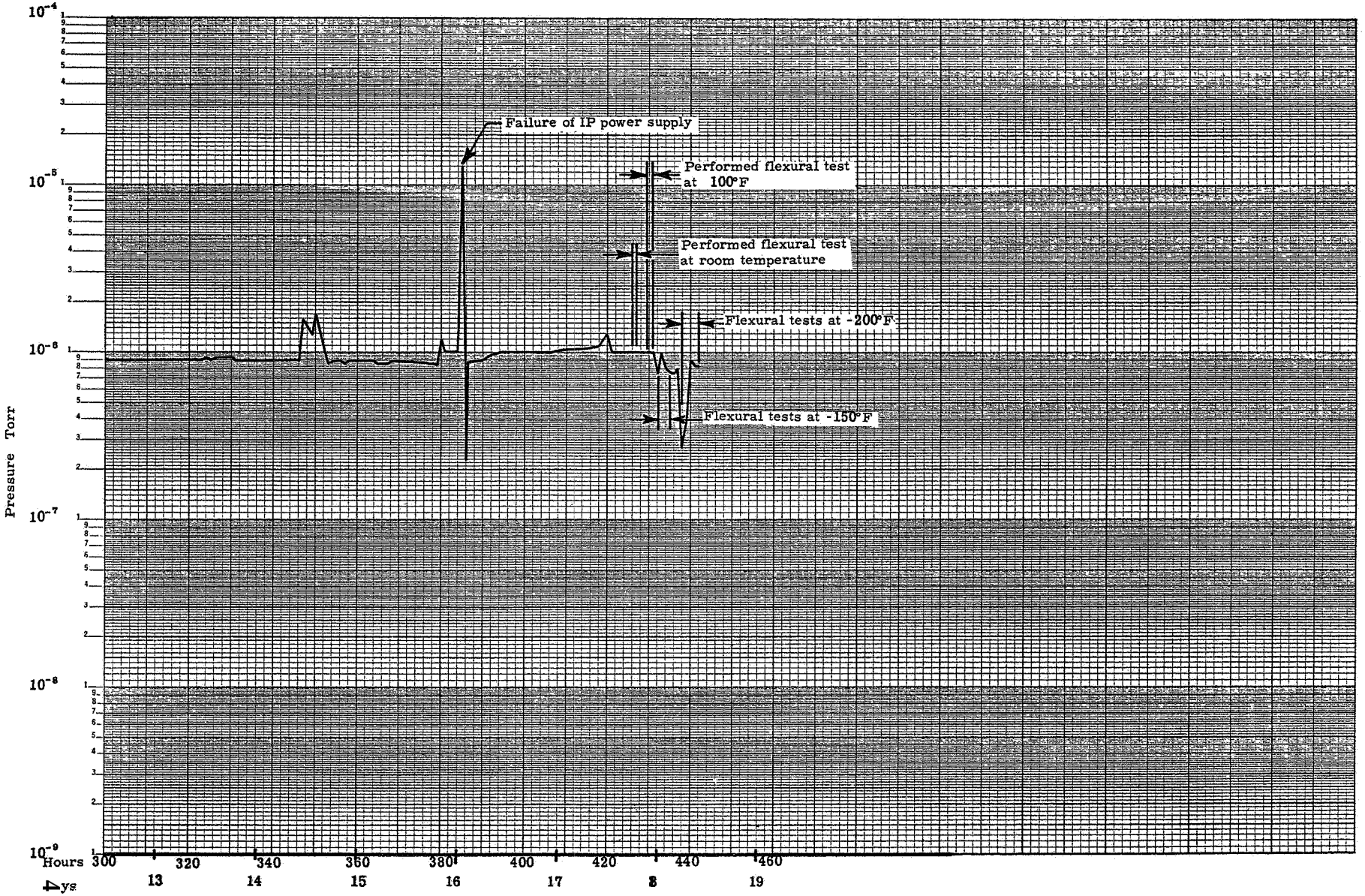


Figure 16. (continued) Pressure versus time during vacuum exposure portion f Run 3

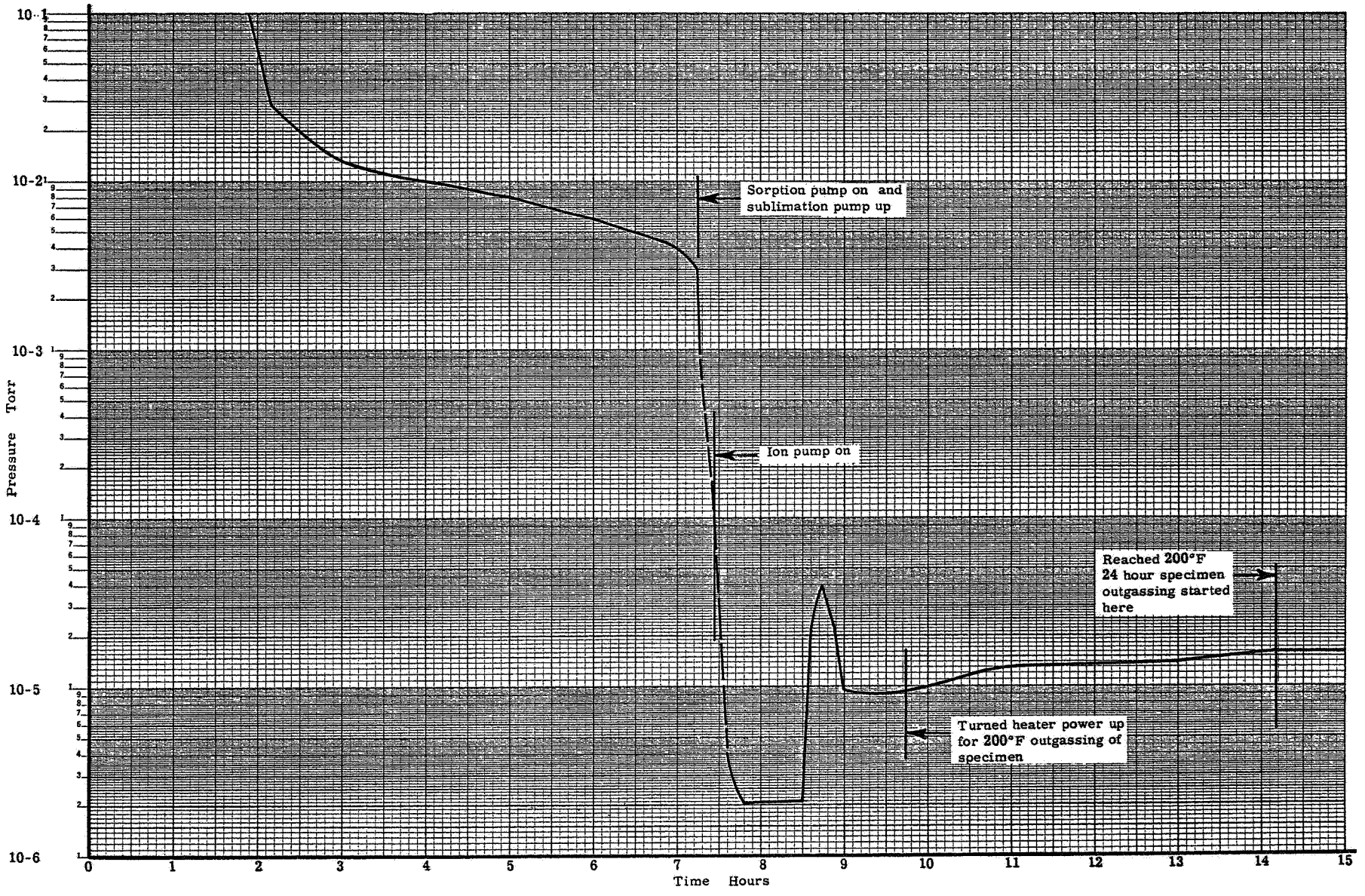


Figure 17. Plot of pressure vs time during portion vacuum exposure, Run 3

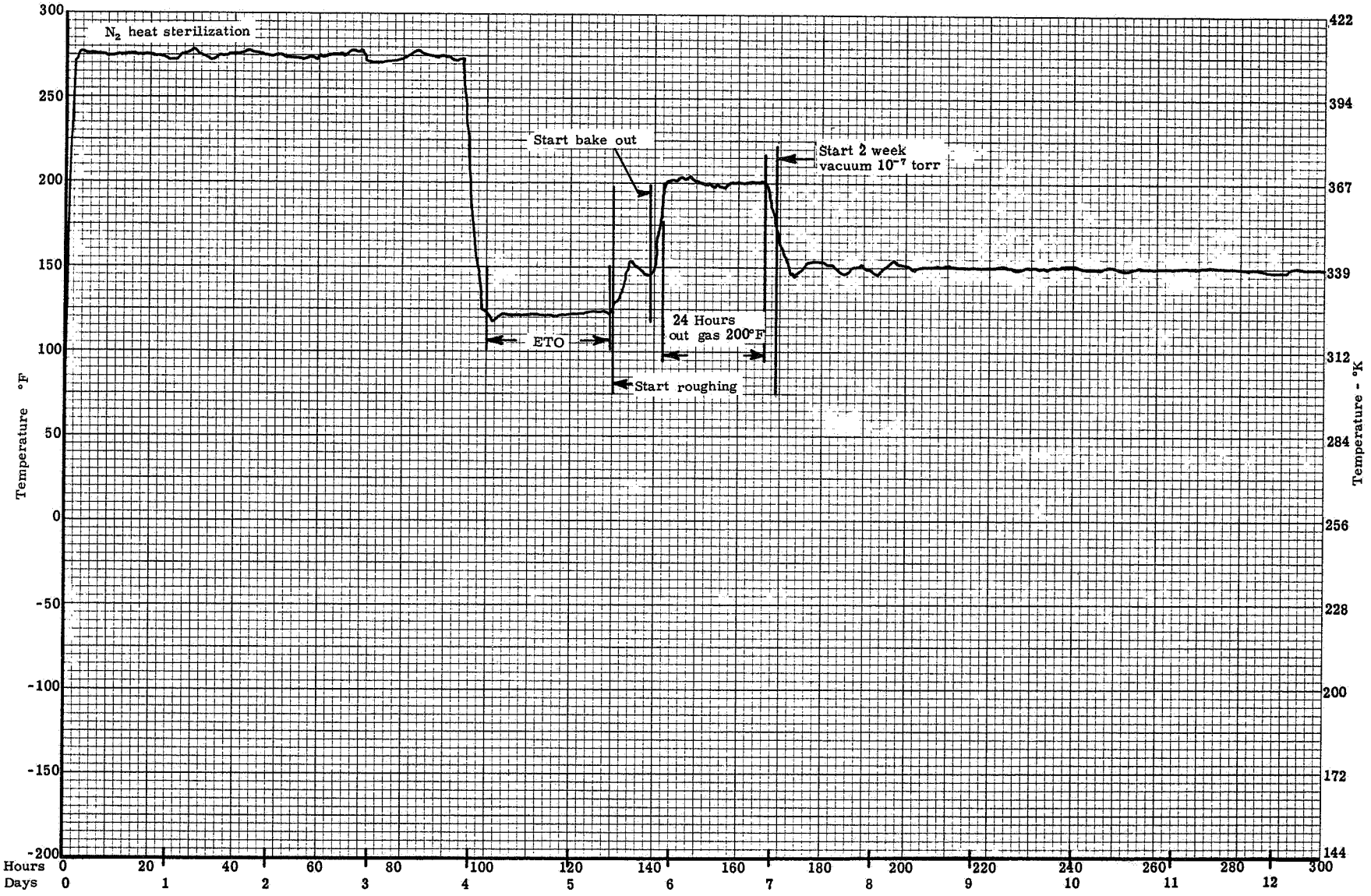


Figure 18. Average specimen temperature versus time-Sterilization-Vacuum Run 4

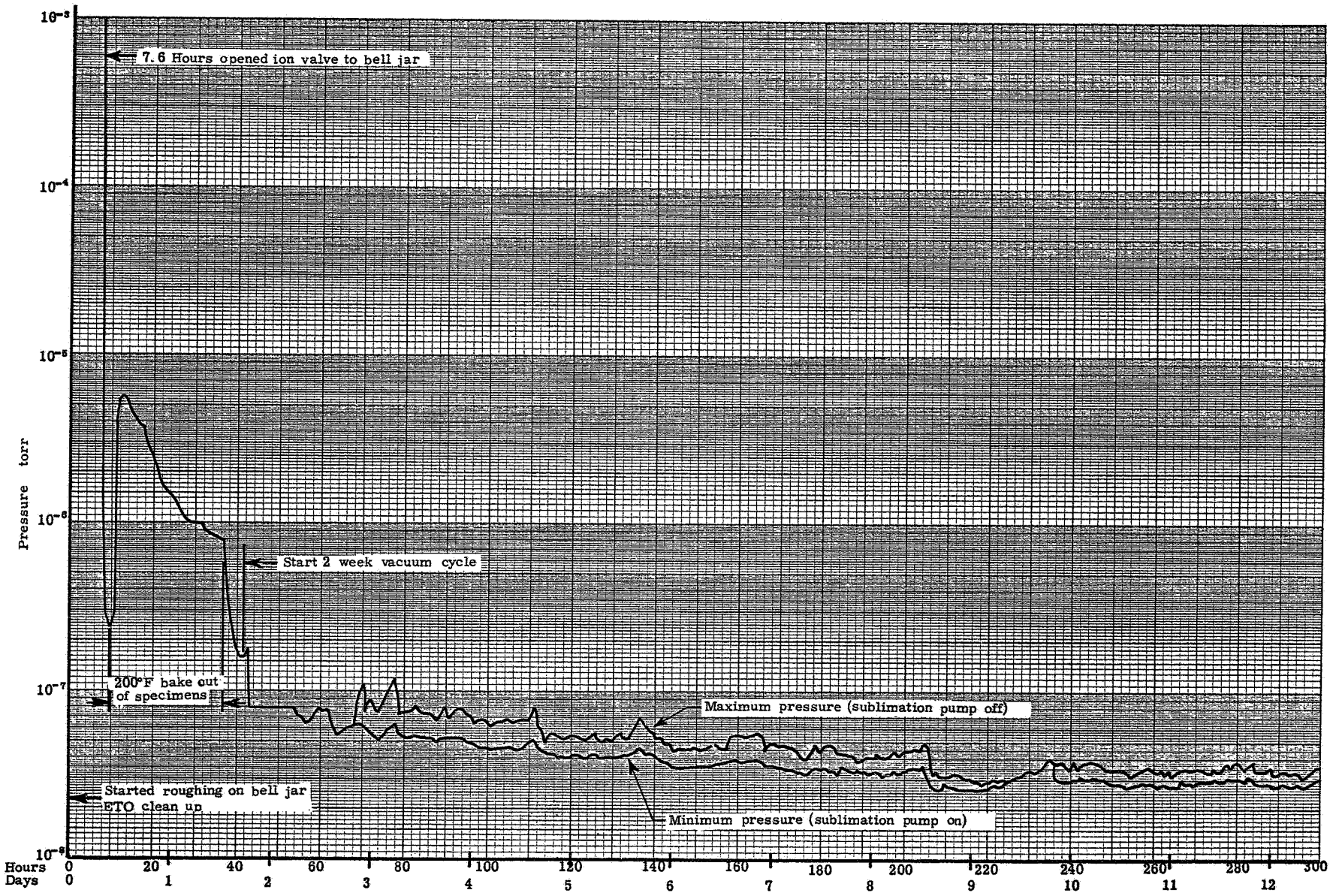


Figure 19. Pressure versus time during vacuum exposure portion of Run 4

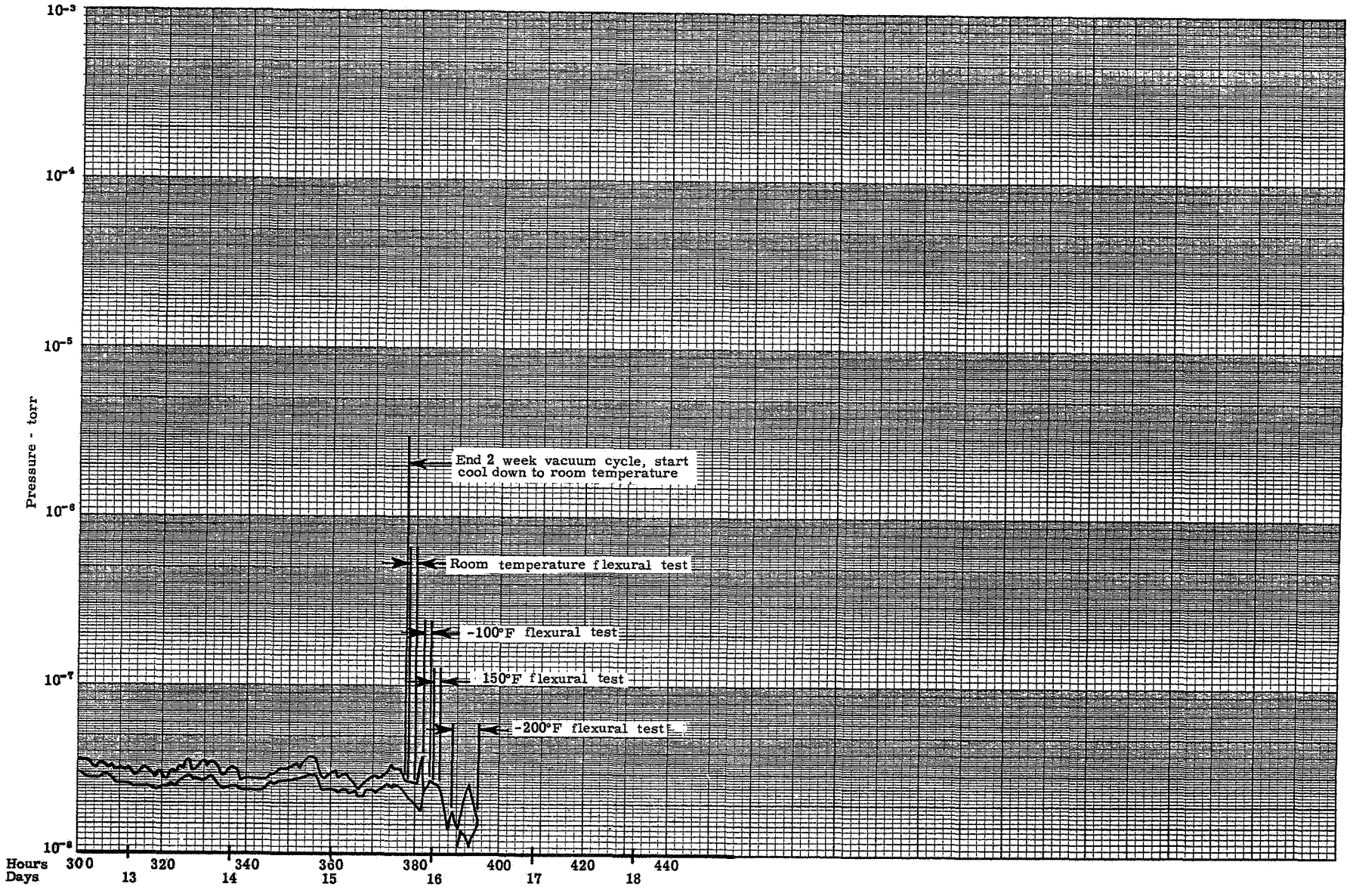


Figure 19 (continued). Pressure versus time during vacuum exposure portion of Run 4

APPENDIX A

DRAWING OF HEATING AND COOLING SECTIONS AND TYPICAL STRESS-STRAIN CURVE SHOWING CORRECTION FOR DEFLECTION IN LOAD TRAIN

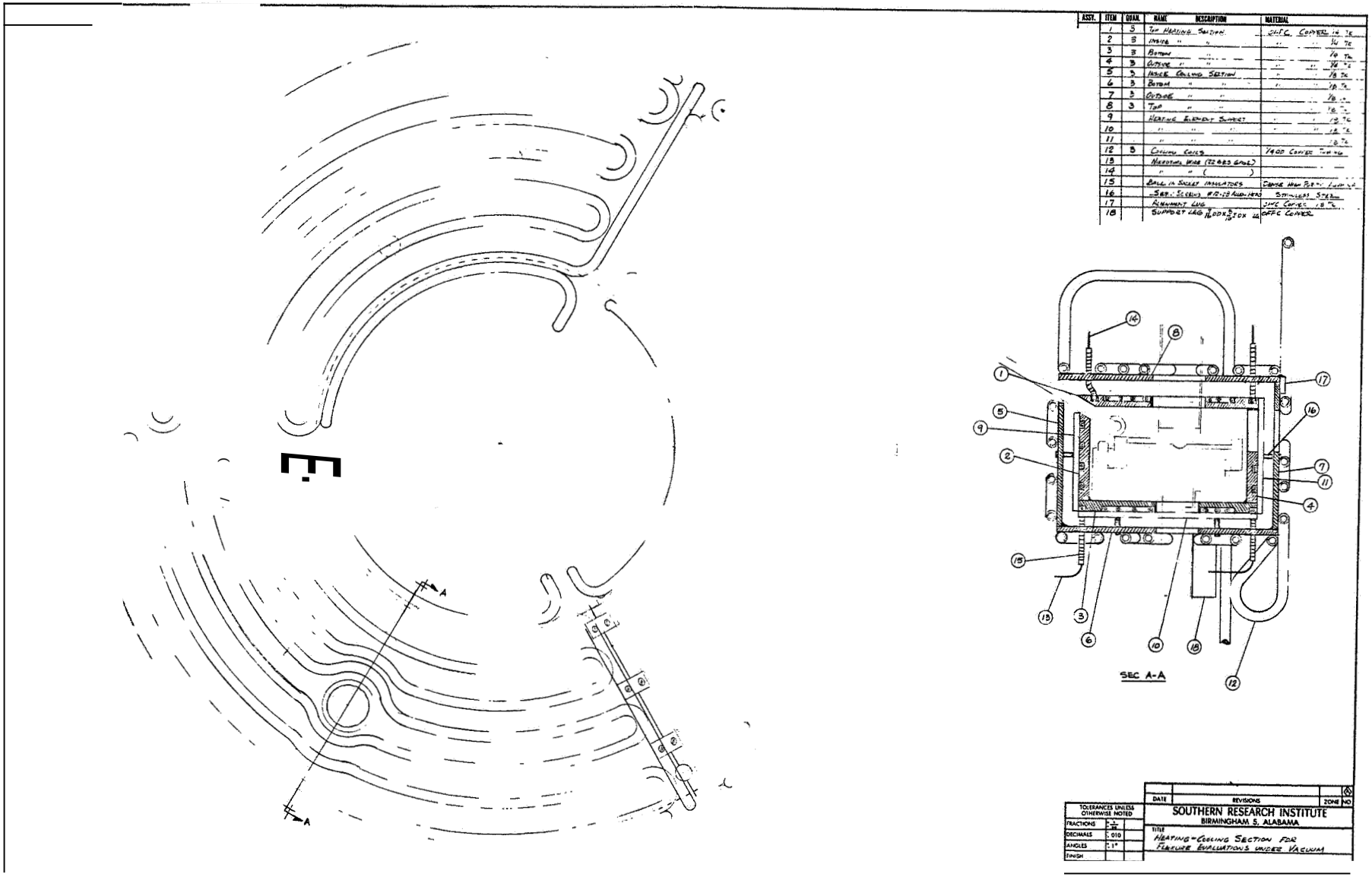


Figure A1. Heating and cooling sections used in environmental control system

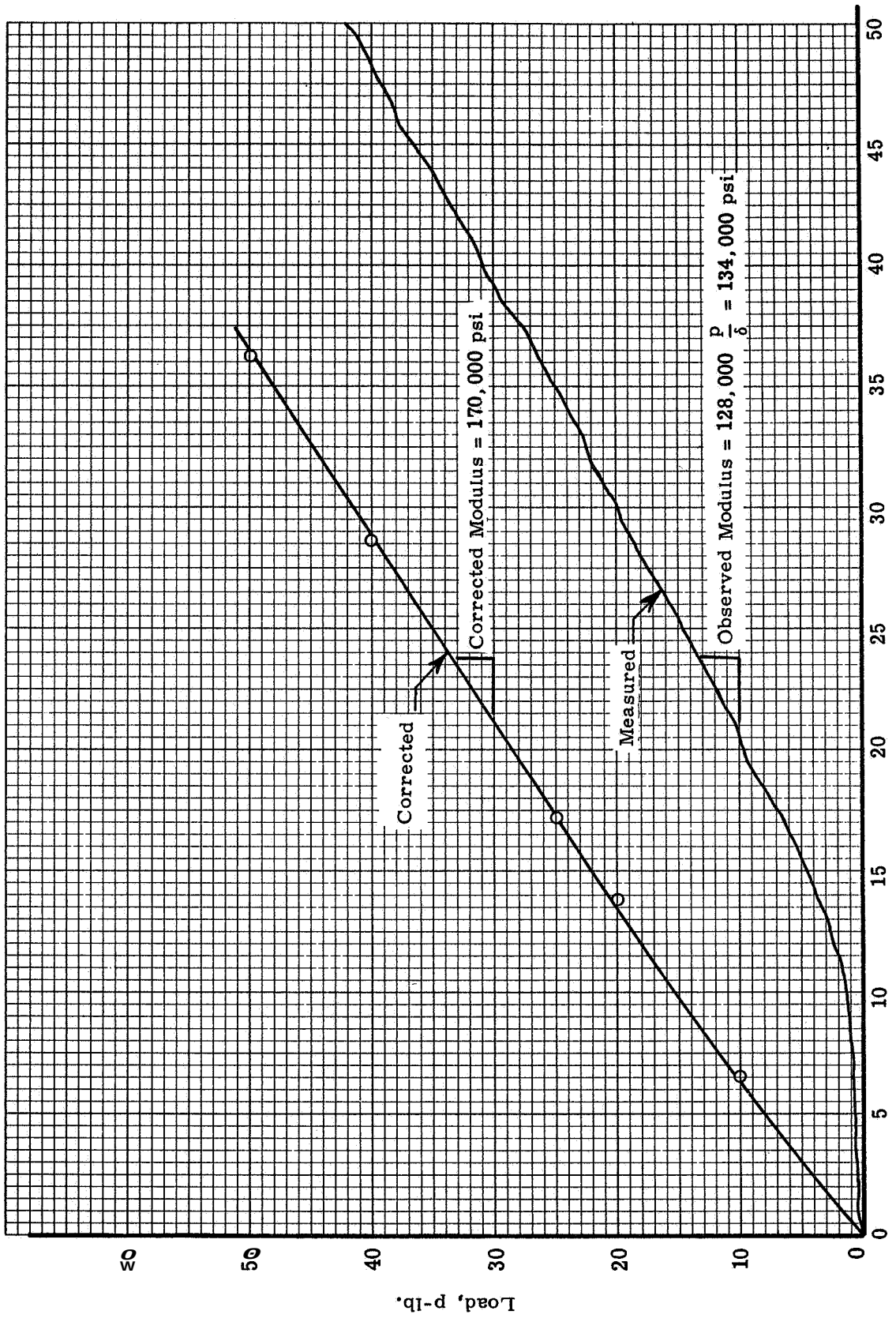


Figure A2.. Corrected stress-strain curve for Boeing Carborazole specimen 11 at - 200° F (Run 4)

APPENDIX B

PHOTOGRAPHSOFTGAAPPARATUSAND
TGA CURVES AND EXPOSED AND UNEXPOSED SPECIMENS

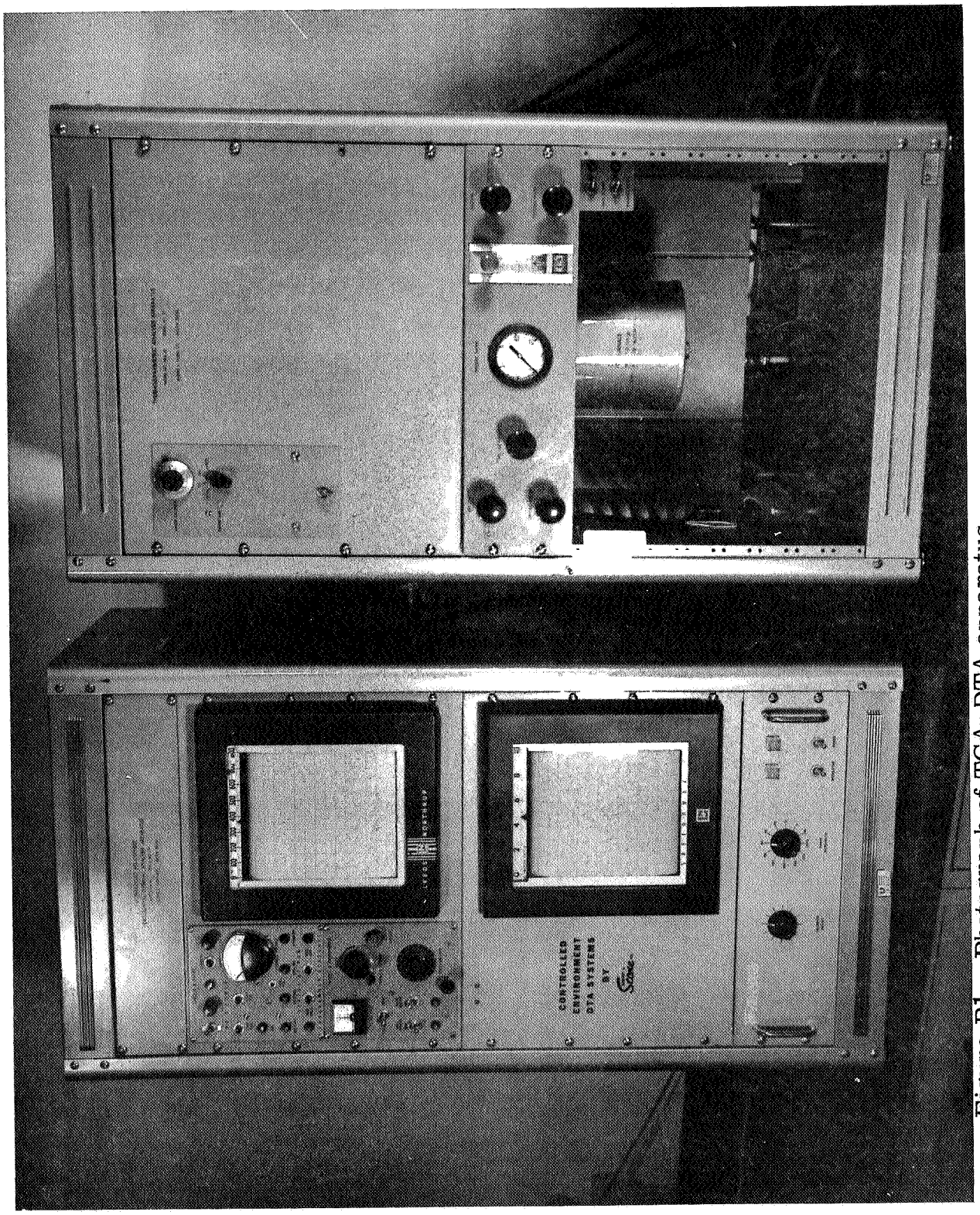
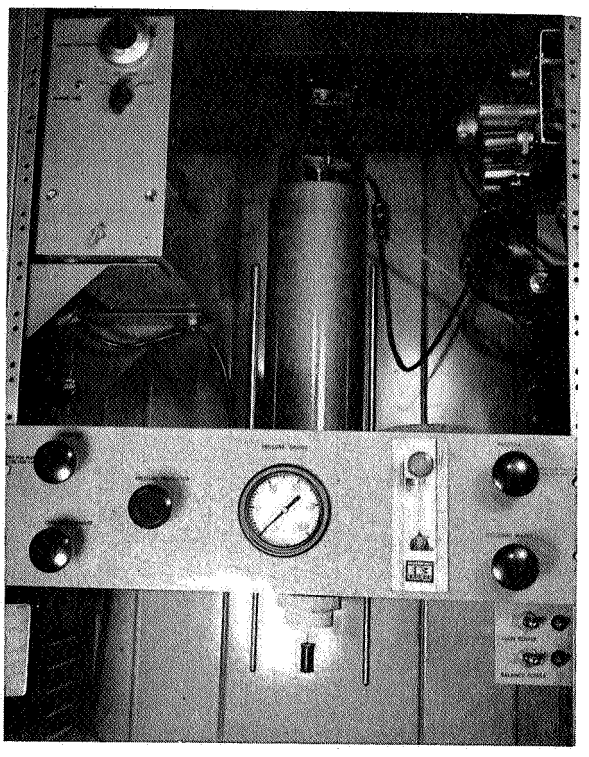


Figure B1. Photograph of TGA-DTA apparatus



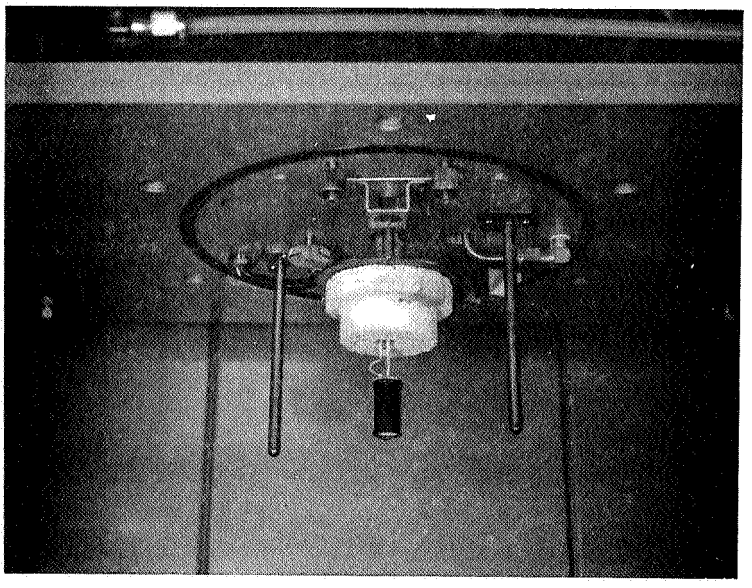


Figure B2. Photograph of TGA apparatus showing sample container suspended from weighing system (top) and in close-up (bottom)

Figure B3. TGA curves for GE1004 AP-20 lb/ft³

Figure R4. TGA curves for GE1004 AP-35 lb/ft³

Figure 28 TGA curves for MoDennell PAEPT

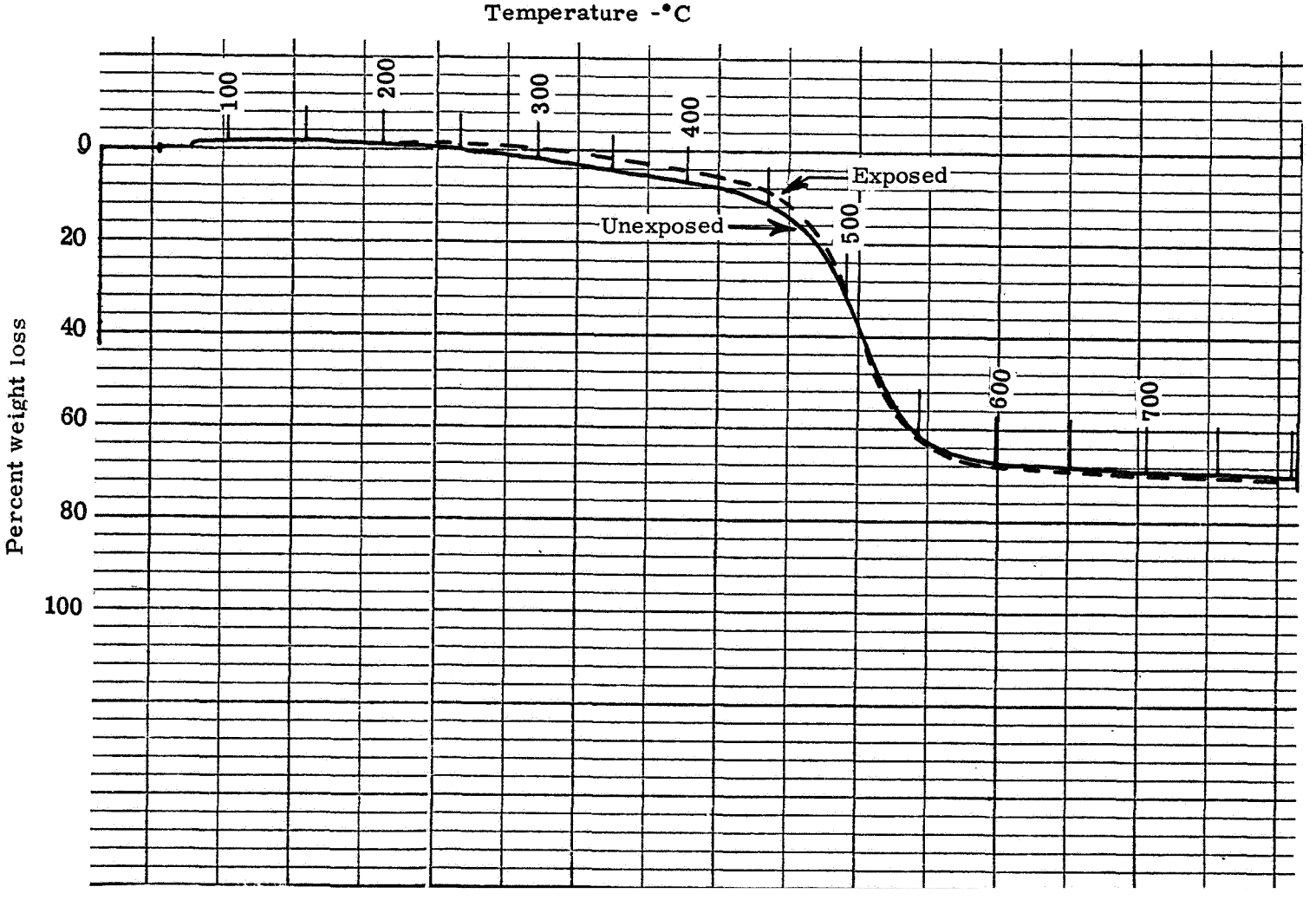


Figure B6. TGA curves for McDonnell B47RF

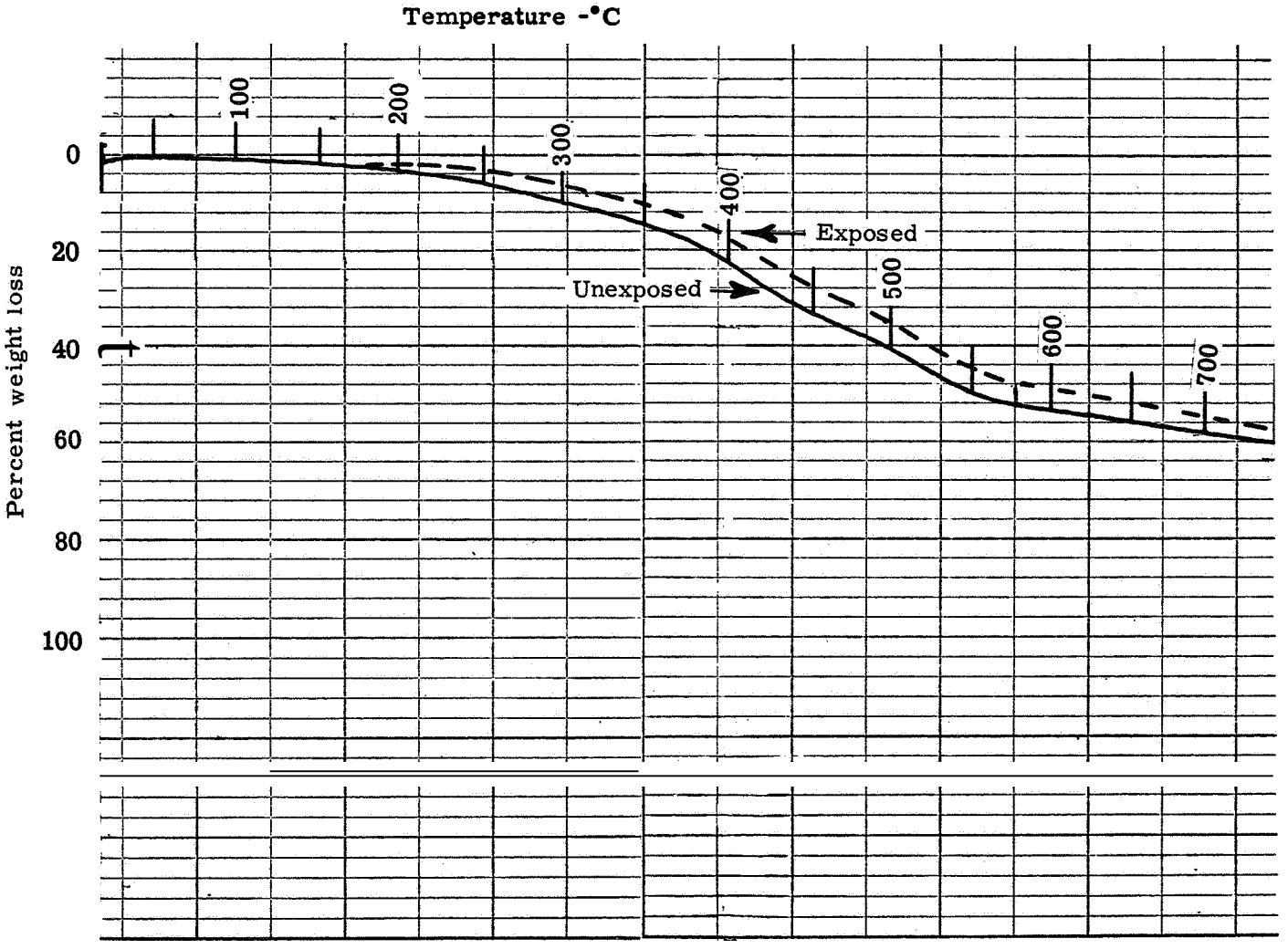


Figure B7. TGA curves for Martin SLA-561

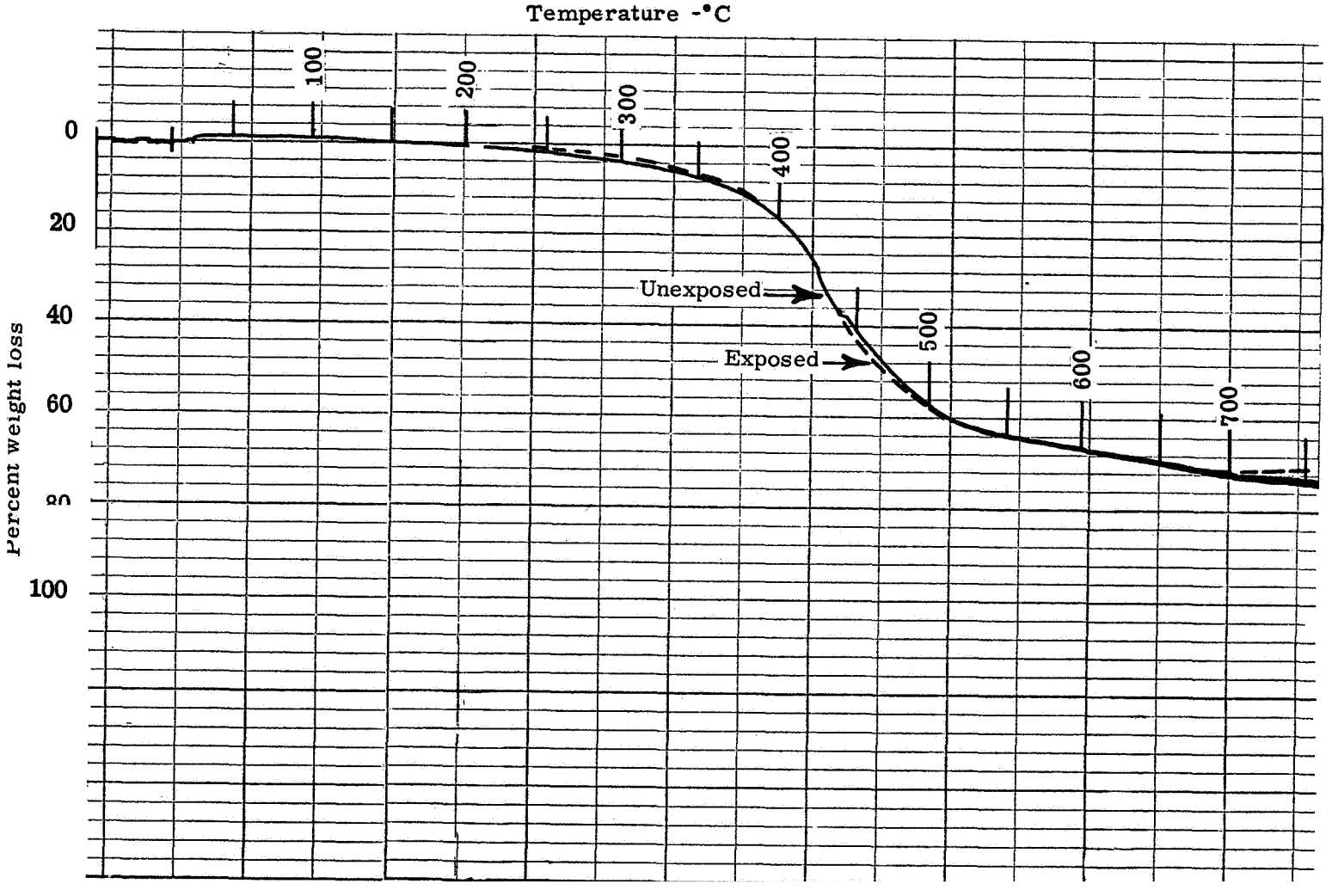


Figure B8. TGA curves for Avco Mod 5

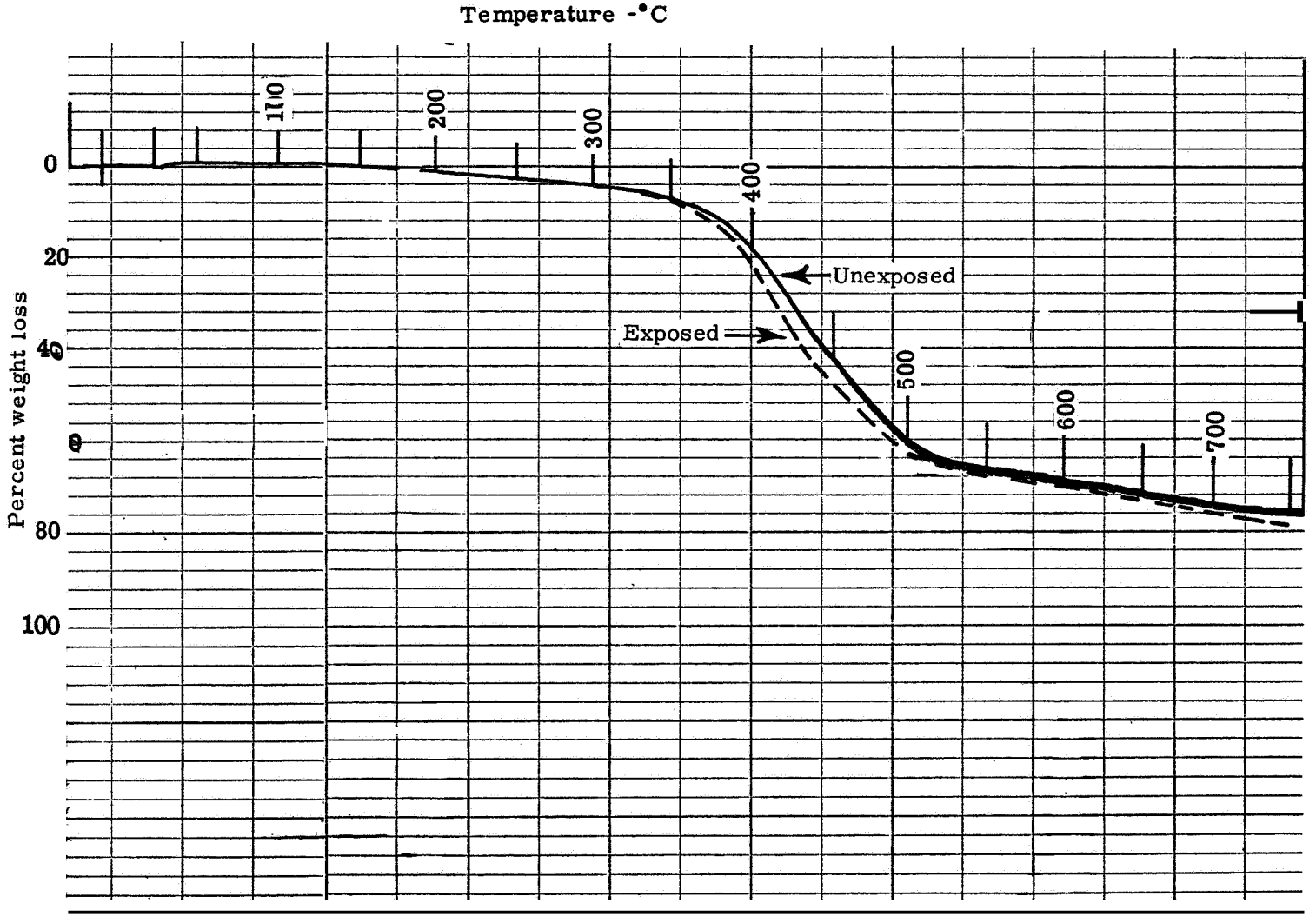


Figure B9. TGA curves for Avco Mod 7

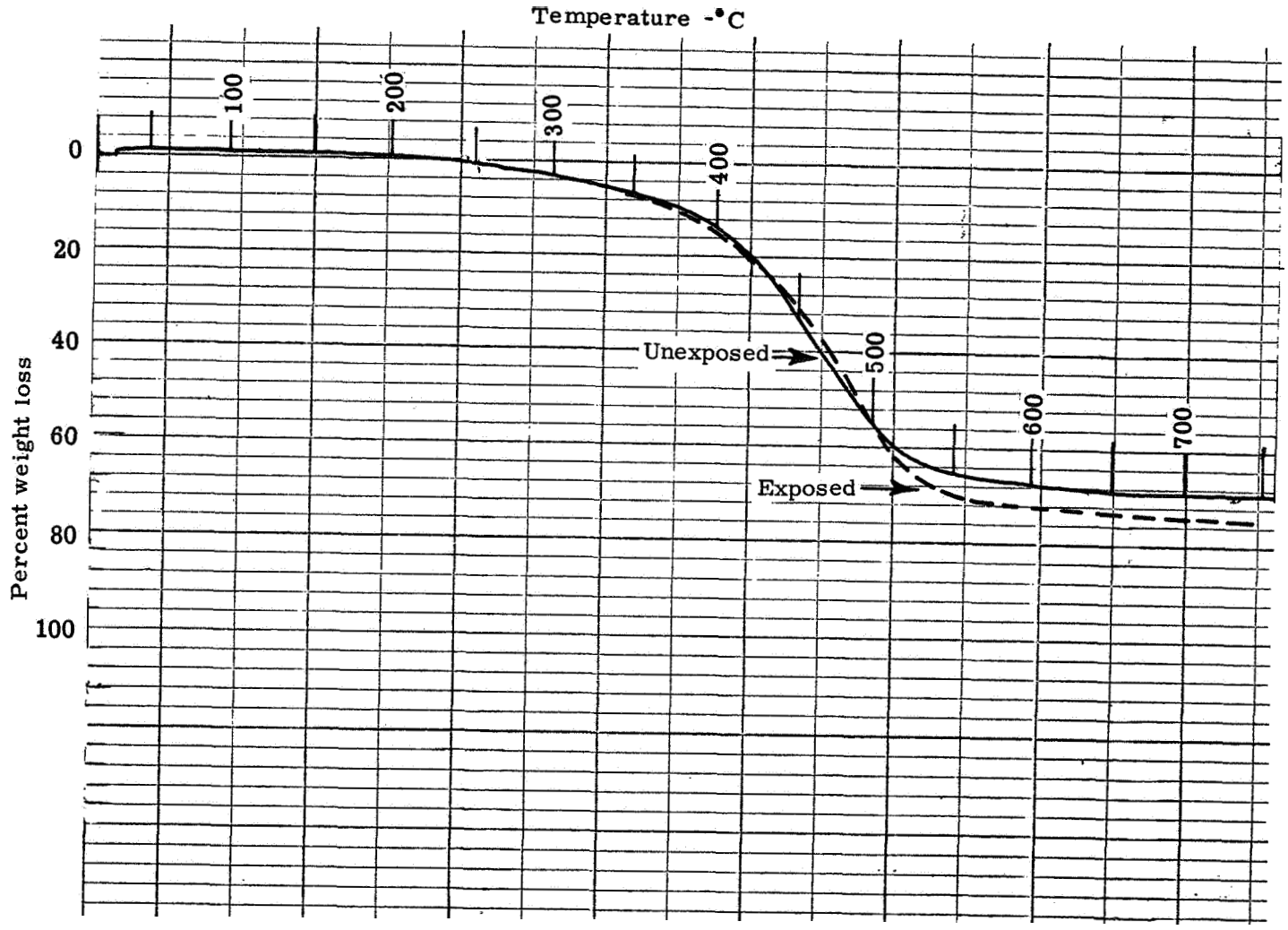


Figure B10. TGA curves for Avco Mod 20

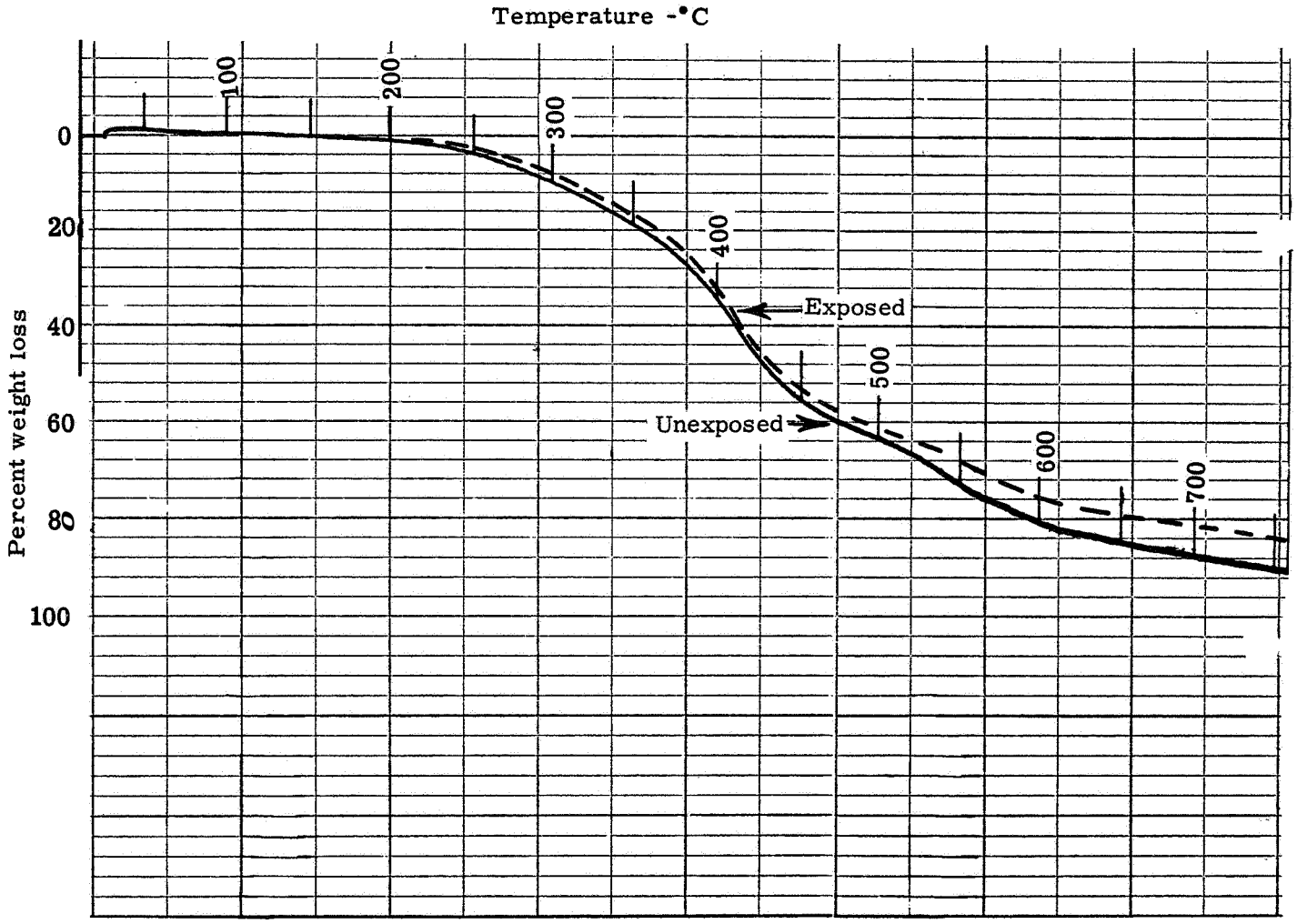


Figure B11. TGA curves for Avco 893-150

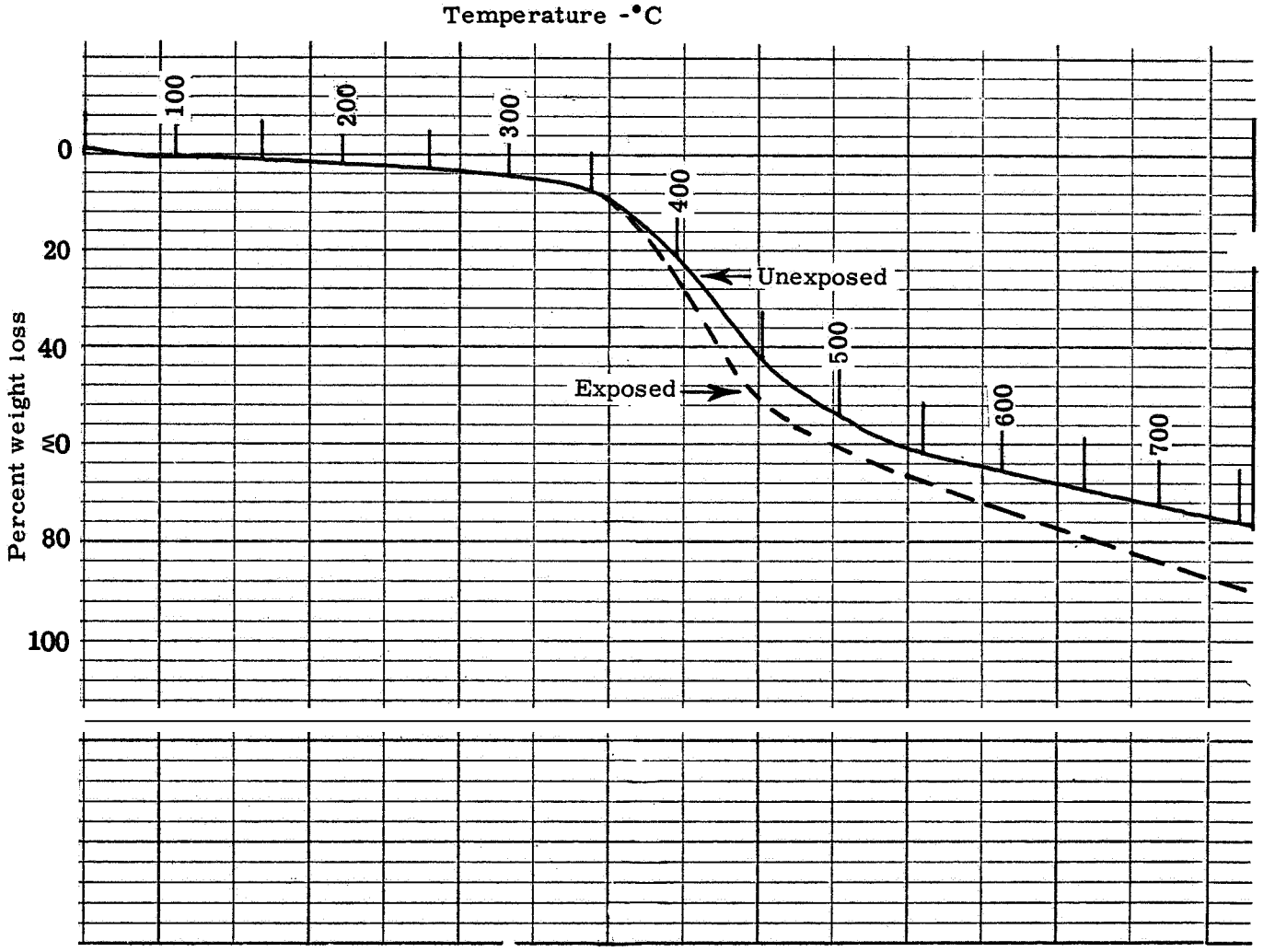


Figure B12. TGA curves for NASA E6A7 silicone-phenolic

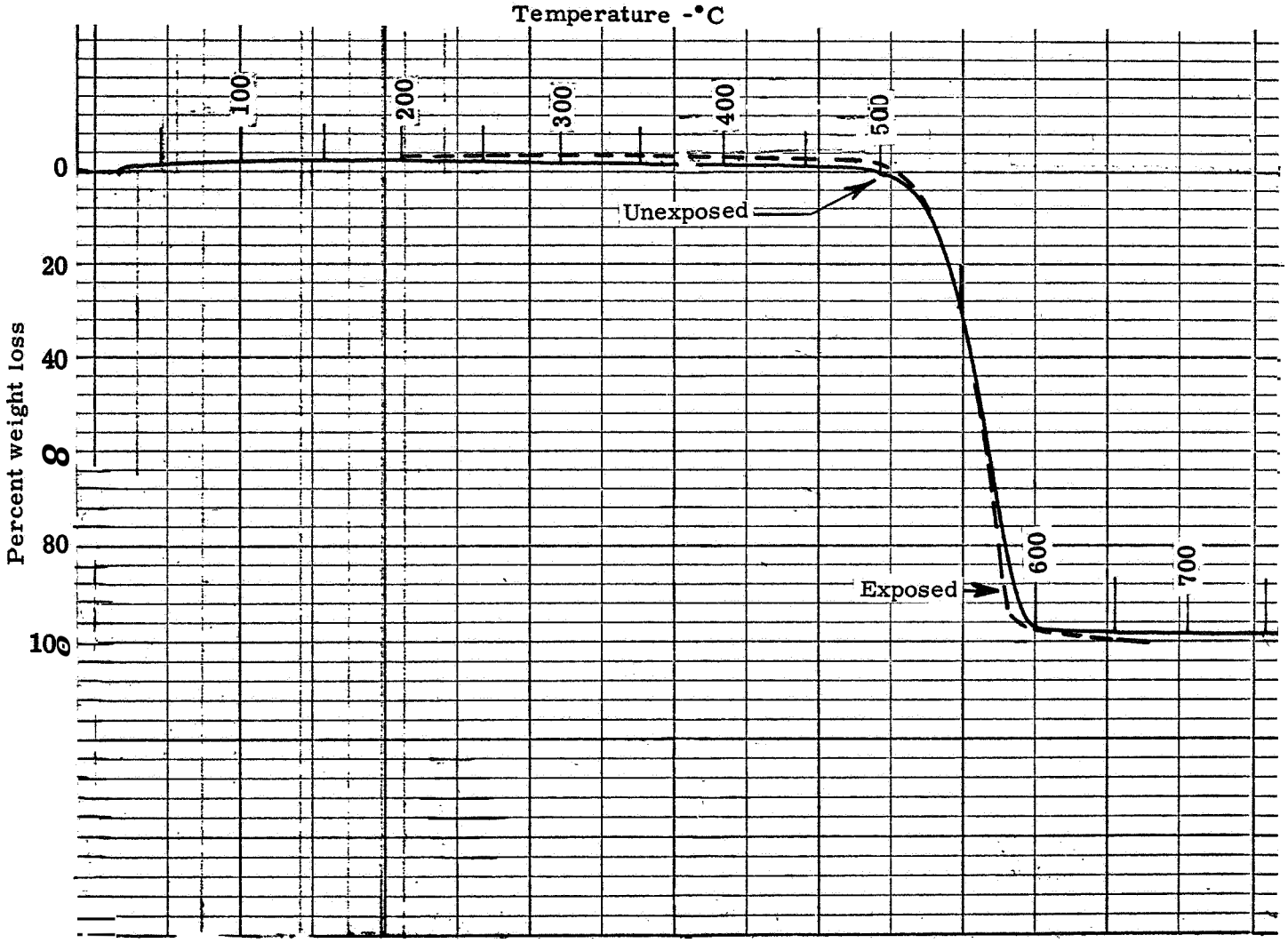


Figure B13. TGA curves for Avco Teflon 0.75 gm/cm^3

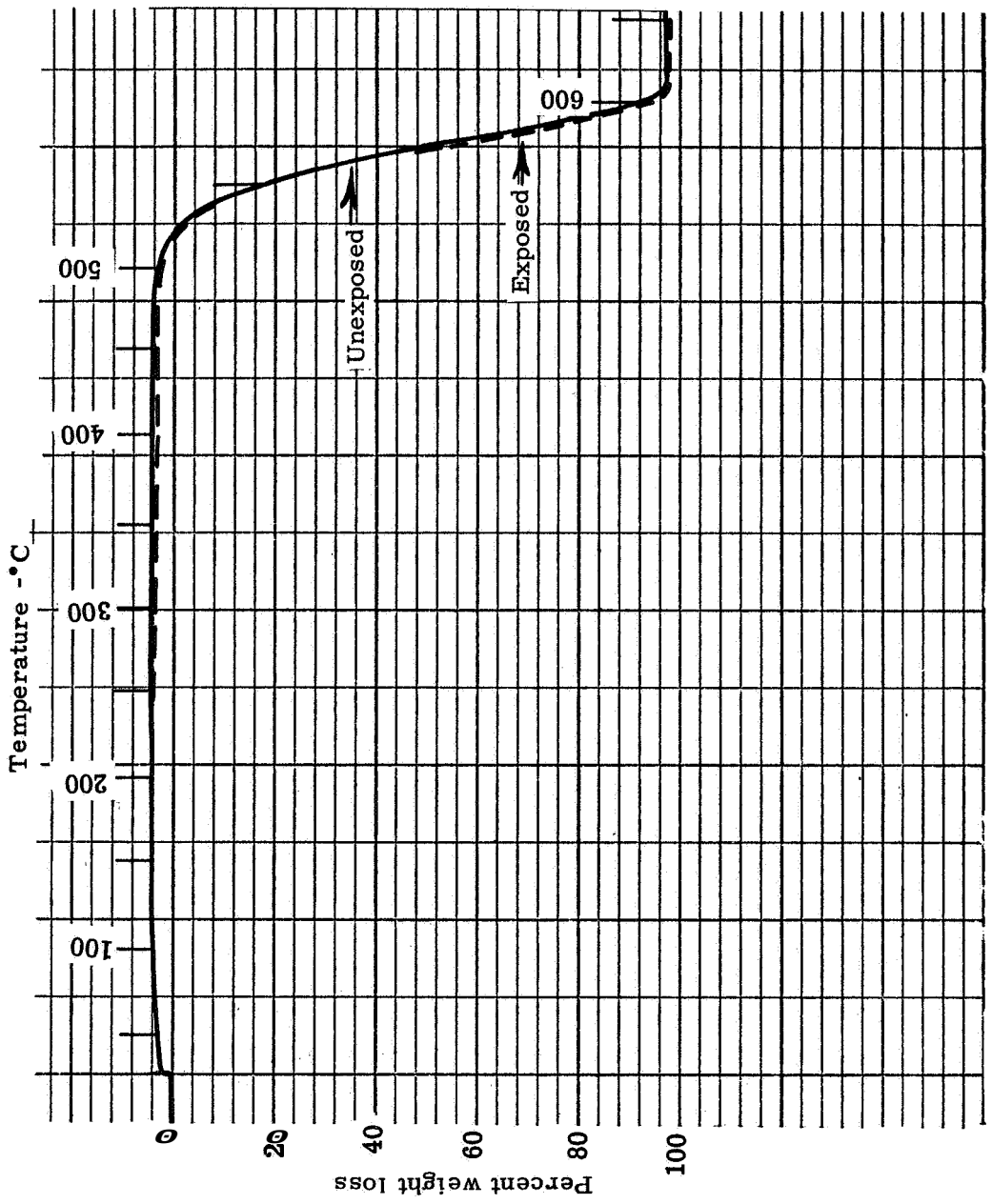


Figure B14. TGA curves for Avco Teflon 0.54 gm/cm³

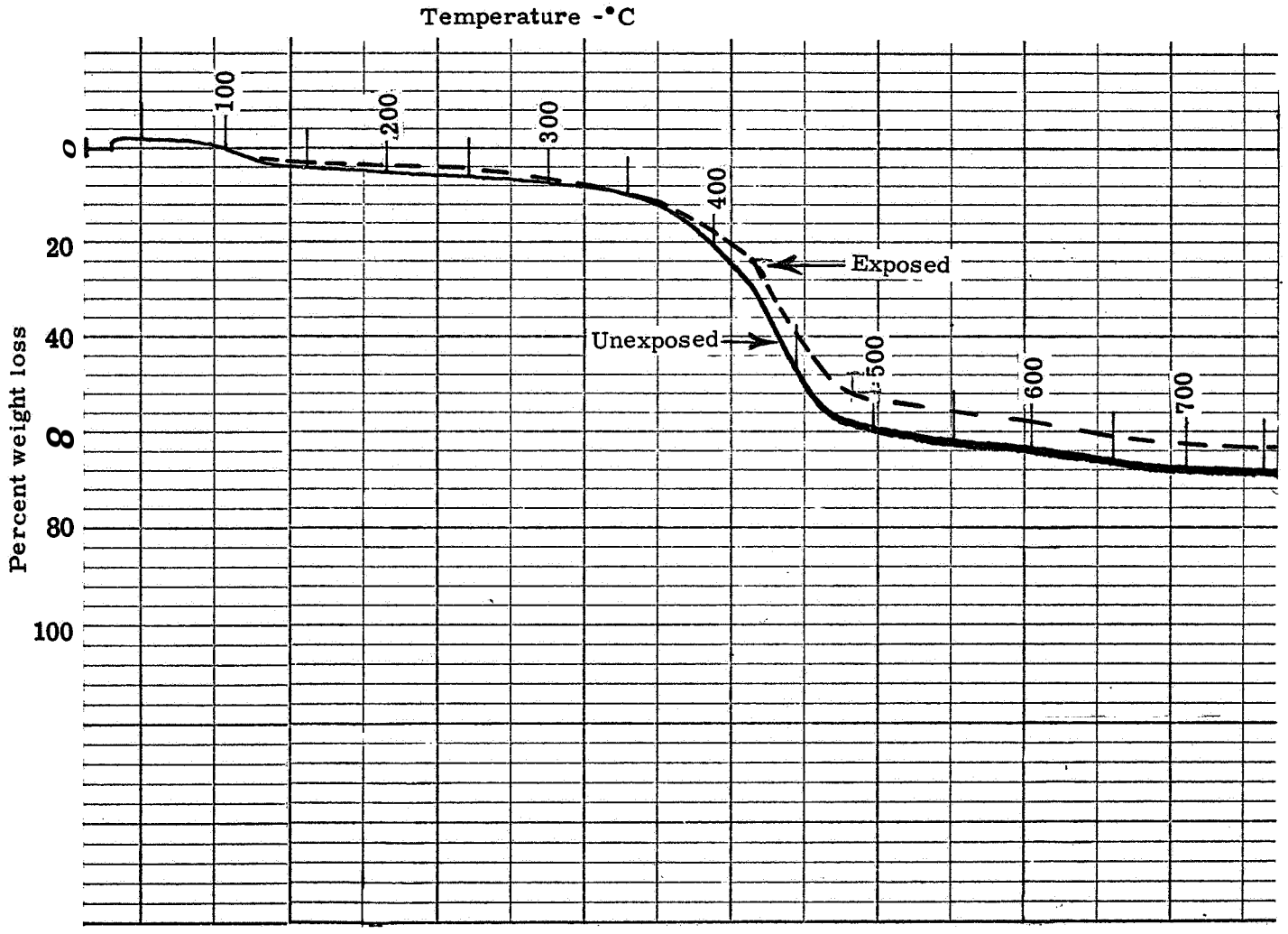


Figure B15 A curves for Boeing carborazole

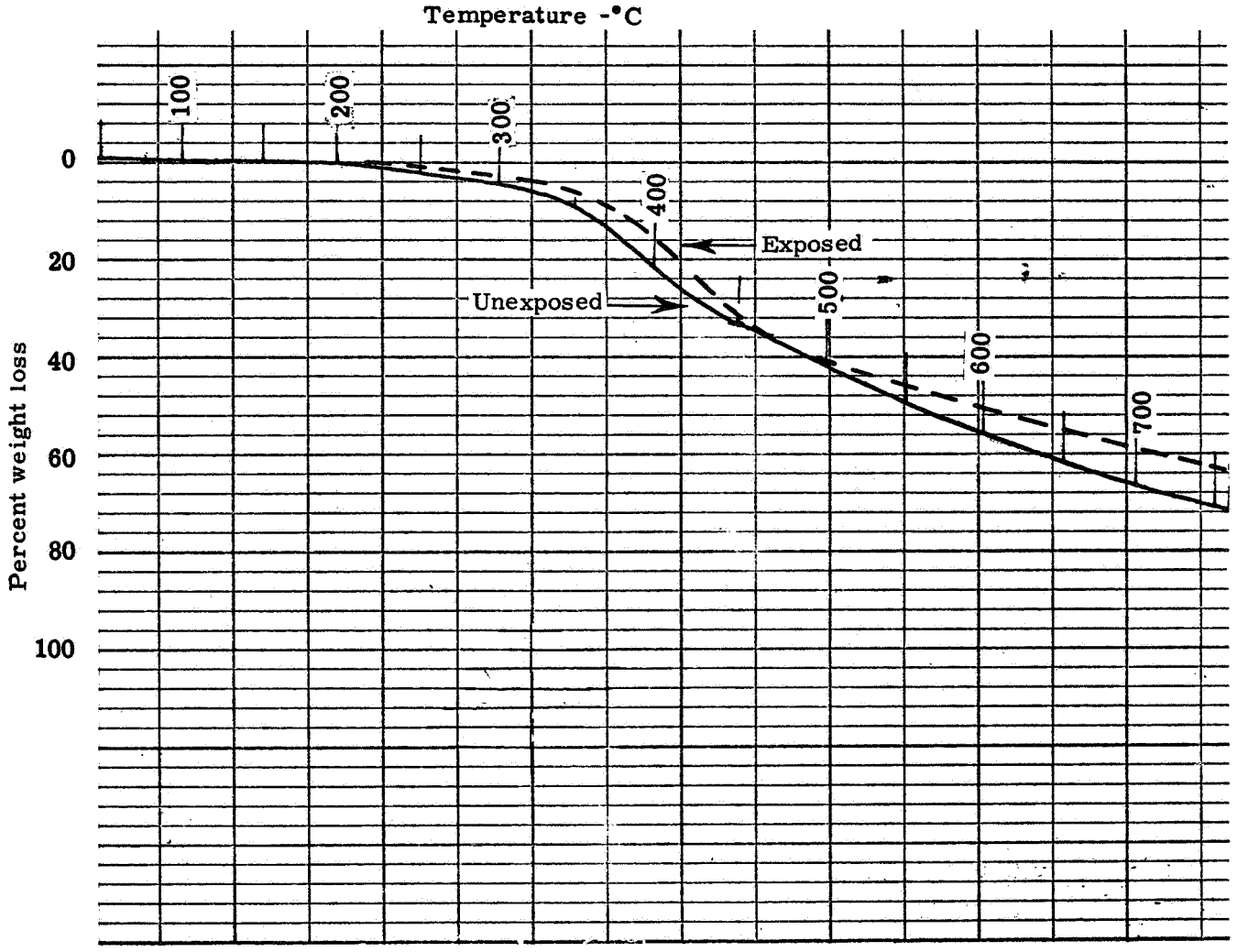


Figure B16. TGA curves for Avco 5026-39

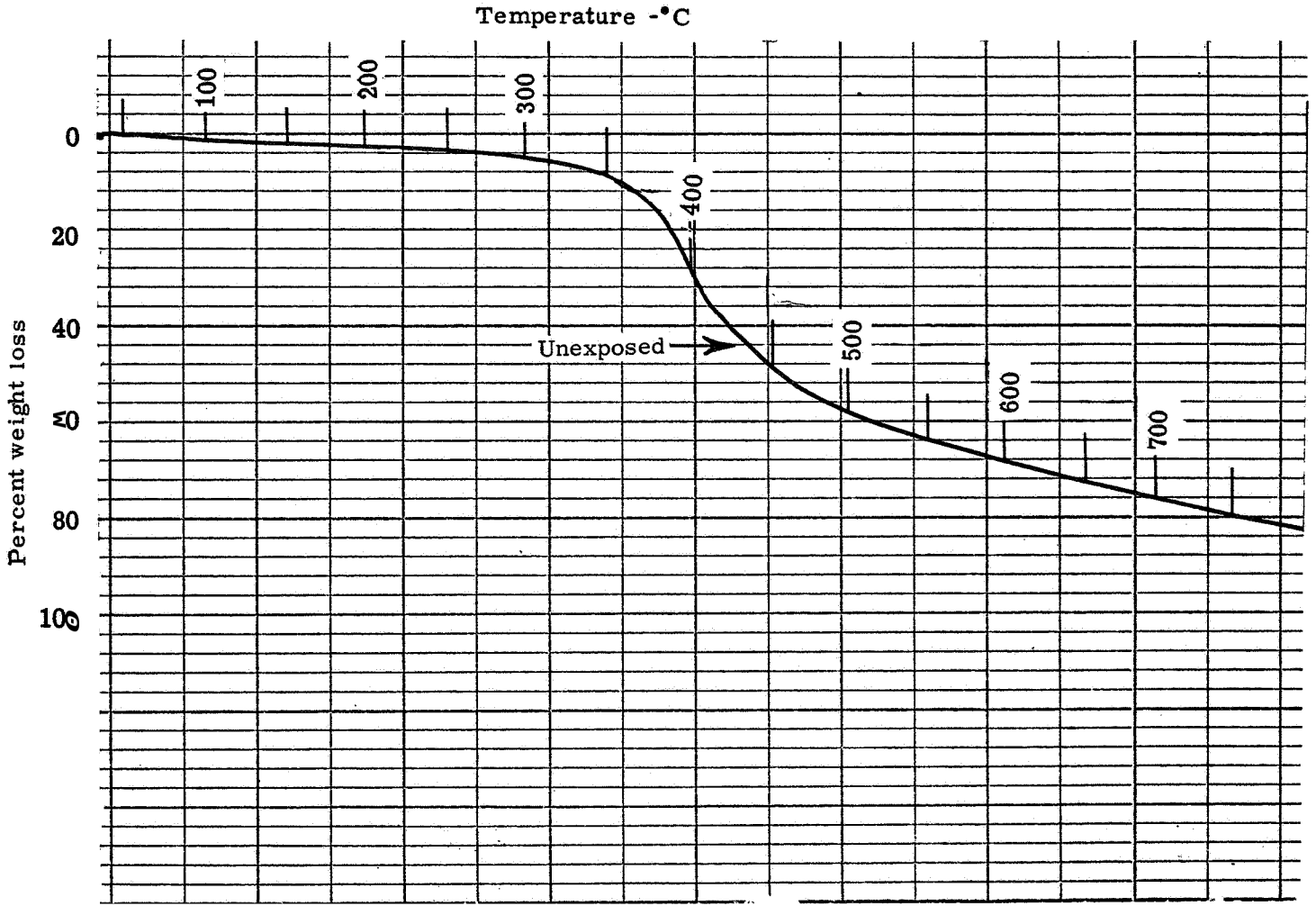


Figure B17. TGA curve for NASA 50-50 low-density phenolic-nylon

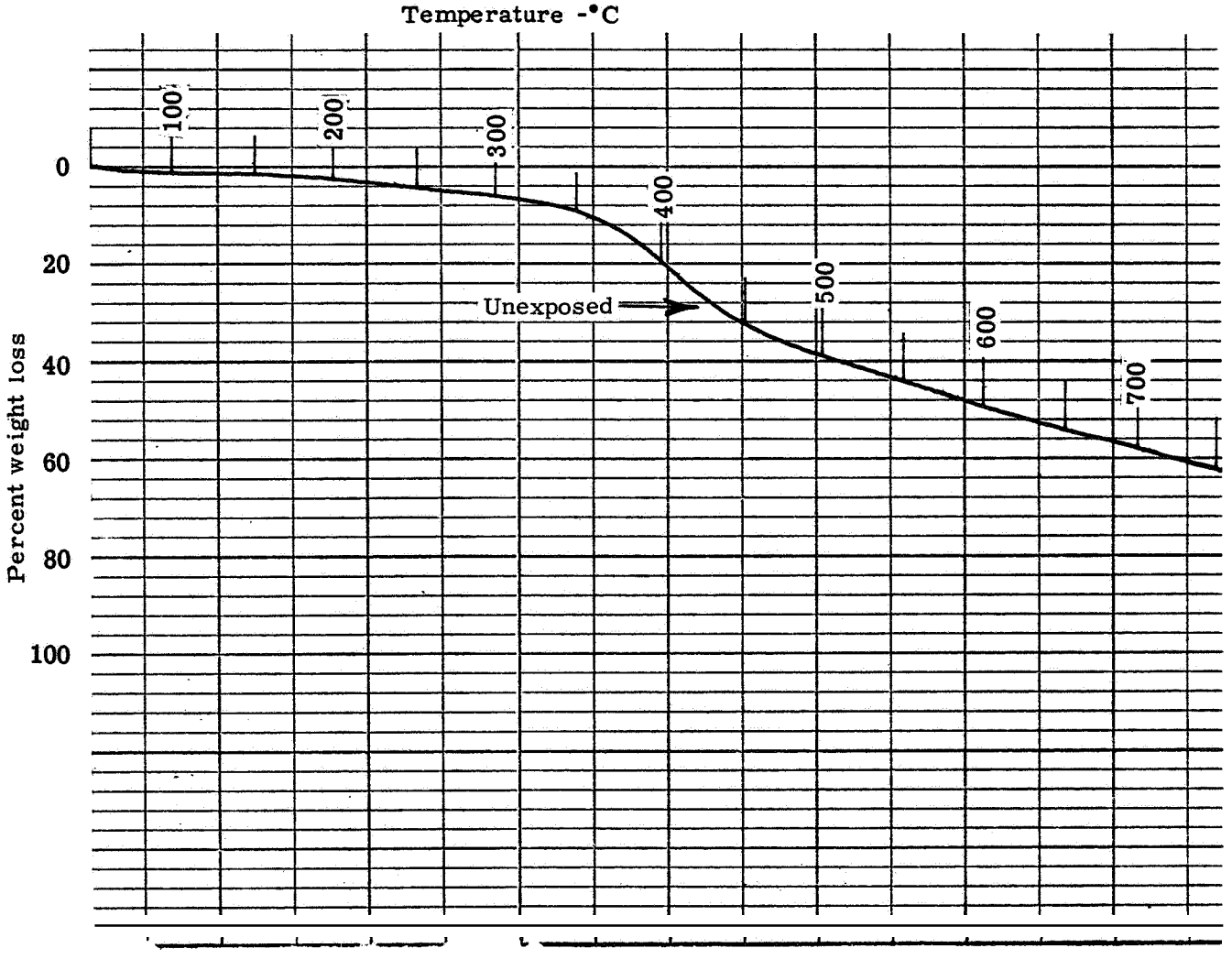


Figure B18. TGA curve for Avco 5026-99