QUARTZ CRYSTALS AT LOW TEMPERATURES

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Introduction

During the last ten years the Quartz Crystal Group of the National Bureau of Standards has been doing research on the performance of crystal units at temperatures below the normal ambient range. For the last three years this program has received a major part of its support from the U. S. Army Signal Research and Development Laboratories. Its primary goal has been an investigation of the aging of high precision crystal units as a function of temperature in the range 4°K to 250°K. Also studied during this period were related factors such as temperature control devices, frequency measuring systems, the development of high precision oscillators as well as the study of certain other crystal properties, like frequency versus temperature characteristics, susceptibility to mechanical shock, Q factor and the like. Some of this work has been reported on at previous Symposiums.

Frequency Measuring Systems

In order to determine the frequency stability of the various crystals under test, the measuring systems must have a resolution of 1 part in 10^9 or better. One of two basic approaches has been adopted for use.

The first employs the principle of multiplying the unknown frequency to the vicinity of 100 Mc and comparing it to a standard 100 Mc. Using a 10 second measuring period a precision of 1 part in 10^9 can be attained, while a 100 second measuring period results in a precision of a part in 10^{10}. One of two types of multipliers is used. The first is the dual-channel multiplier such as is used in the daily assessment of the USA Primary Standard of Frequency and Time Interval. A block diagram is shown in Figure 1. The standard signal is multiplied by the channels on one side while the unknown by those on the other side. The difference between the two frequencies is obtained from the mixer in the center. In one case mentioned later, i.e., the measurement of Crystal No. 8, the 100 Mc signals are fed through a crystal detector into a cavity tuned to the tenth harmonic (1000 Mc) and the difference frequency is obtained at that higher frequency. This results in a precision of 1×10^{-11} for a 100 second count.

The other type multiplier, shown in Figure 2, also has an output frequency around 100 Mc but is tunable over a much greater range than the dual-channel type. It will accept frequencies of from 4 to 6 Mc at the nominal 5 Mc input and deliver corresponding outputs of 80 to 120 Mc. This output can then be measured by some available method such as an electronic counter. Again the precision is a part in 10^9 or 10^{10}, depending on the measuring time.

The second approach to a method of precise frequency measurement employs some frequency multiplication, but its high precision is obtained by combining this with the period measurement available in electronic counters. The block diagram of the system is shown in Figure 3. The unknown and standard frequencies are multiplied to a nominal 10 Mc and then mixed. The difference frequency, which may be anywhere from one to one thousand cycles per second, is then sent to a counter where its period is precisely measured. Very high precisions can be obtained by measuring the length of, say, 100 or 1000 periods, but due to such practical limitations as noise and phase jitter only one or two parts in 10^{11} can be realized at present. This system has proved very useful in analyzing both the short term and long term characteristics of oscillators.

Crystals at 13°C (286°K)

Aging studies have been made on two crystals at a temperature of 13°C (286°K). Although slightly outside the range previously mentioned, it was used because a very convenient, stable environment at this temperature is readily available at the Boulder Laboratories.

Each crystal unit is located at the bottom of a 50-foot well underneath the Radio Building. The temperature of each well is measured by means of a platinum resistance thermometer and a Mueller bridge. It is approximately 13°C and is rising at a rate of slightly less than a millidegree per day.

The crystals are GT-cut plates at a frequency of 100 kc/s, and each is connected by means of shielded cable to a separate Western Electric Loran oscillator, as a replacement for the internal crystal supplied with the equipment. Suitable shunts were placed across the appropriate arms of the Meacham bridge to reduce the crystal current to less than 50 microamperes.

The results of two years' measurements are shown in the following three graphs. Figure 4 shows the first eight months' performance of the two crystals under this test. Here can be seen the start of the characteristic logarithmic aging curve of GT type crystals. The frequencies were measured by using the dual-channel multiplier and the 1000 Mc cavity and comparing directly against the USA Primary Standard. In fact, all measurements in this paper, unless otherwise stated, were referred to the USA Frequency Standard. The curve for No. 8 stops around August 16, because of a power supply failure, and it is resumed again on Figure 5 which shows the next eight months' data. Crystal No. 6 was stopped October 29 in order to place it in liquid helium for testing at 4°K. No. 8, after some erratic behavior following the power supply repairs, returned to its previous curve and soon appeared to cease aging.

Figure 6 shows the next eight months' data taken on No. 8. Notice the frequency has still remained constant to parts in 10^9. The performance of this crystal is probably due to a set of fortunate circumstances. Because the temperature of the well has increased by 0.3°C during the year that the frequency has remained unchanged, it does not seem likely that the crystal unit has ceased aging for this would imply a zero temperature coefficient of frequency, to parts in 10^9, over quite an extended temperature range. What is more probable is that the rate of frequency change caused by gradually increasing temperature is equal, but opposite in sign, to the aging rate of the crystal. However, it is planned to measure the temperature coefficient of frequency of this crystal at 13°C sometime in the future in order to fully determine all the factors that contributed in producing such remarkable results.

Crystals at Liquid Nitrogen Temperature (76°K)

Another temperature readily attainable at the Boulder Laboratories is that of boiling liquid nitrogen. This is because of the fine facilities for producing liquified gases at the NBS Cryogenics Laboratories, located not far from the Radio Building. The temperature of liquid nitrogen at Boulder is -197°C (76°K) because of the reduced atmospheric pressure of 625 mm.

The crystal units tested were 5 Mc plates operating on the fifth mechanical overtone. Figure 7 shows a group of such units fabricated at NBSBL, which are 32 mm in diameter and are cut at an angle somewhat removed from that of the normal AT plate. The orientation of the plates is around 39° 49' so as to give a temperature versus frequency turnover point slightly above the temperature of liquid nitrogen. Figure 8 gives the frequency versus temperature characteristic of one such crystal unit which has a turnover point at 640 mm pressure or a temperature of -197.2°C.

In order to attain better temperature stabilization the pressure of the vapor over the boiling liquid was controlled. The regulators controlled the pressure to better than 1 mm, so that the temperature was constant to approximately 0.01°C. Figure 9 shows the oven used to hold the crystal unit. The crystal was enclosed in a copper cylinder, along with the critical oscillator components, and the two leads brought out through thin wall stainless steel tubes which act as shields as well as mechanical supports. The oven was immersed in a standard narrow neck 4 liter glass dewar, which was, in turn, enclosed in a tank containing the pressurized gas. Figure 10 shows the equipment for conducting tests on four crystal units.

The results of aging measurements on three of the units are shown in Figure 11. These are smoothed curves and the scatter of the points from which they were obtained is labelled "Short Time Variations." Also shown is a straight line whose slope is the aging rate of crystal 5A, one of the better crystal units in USA Primary Standard.

At first glance it might appear that the crystal units have changed frequency. However, after considering the curves of CZM-8 and CZM-3, it can be seen that the average frequency change is approximately zero. Furthermore, the oscillators driving the crystals were somewhat sensitive to temperature. Due to this fact and the apparent periodicity of these curves, it is believed that these variations are due to a seasonal temperature change in the room and therefore the aging of the crystal units was quite small. As for CZM-1, it appears to have aged at the same rate as 5A, and there is also some indication of the above-mentioned seasonal variations in the two lower curves. The cause of this small aging is not known at present.

Crystals at Liquid Helium Temperature (4°K)

Liquid Helium is also available at the Boulder Laboratories. Operation at this temperature offers several advantages over operation at liquid nitrogen temperatures or higher. First, the Q factor of the crystal unit is increased by an appreciable amount; this makes the frequency control problem much easier. Then, too, temperature control is much easier because the temperature versus pressure coefficient of helium is 0.0016°C/mm, as opposed to 0.014°C/mm for nitrogen. The biggest disadvantage in using liquid helium is its cost, i.e., $10 per liter as compared to $.25 per liter for liquid nitrogen.

The first crystals put under test were a group of 100 kc GT-cut plates made for this project by Bell Telephone Laboratories. They were placed in a dewar flask and the refrigerant was left open to atmospheric pressure. The units were either measured on a bridge as resonators or were used as the controlling element of a Western Electric Loran oscillator. Some typical results are shown in Figure 12, which is a plot of the frequency of three crystal units in the same dewar for a period of eight months. Crystal No. 6 is the same unit that was first tested at 13°C in the well. The dewar ran out of liquid helium twice, on March 16 and September 10, and are easily seen in this slide.

The day-to-day scatter of the points, which is several parts in 10^9, may be attributed to two factors. First, because the pressure of the vapor over the liquid helium varies with the atmospheric pressure many of the changes are undoubtedly due to temperature changes of the liquid. Furthermore, some changes may be due to vibrations in the building, because we have found, as have other observers, that crystals at this temperature are quite sensitive to shock. The crystals were located on the fourth floor of the Radio Building but not near any main building supports. They have since been moved to a room on the ground floor. While there is a large daily scatter of points, it will be noted that the average frequency indicates little or no aging. In fact, this is true for a period of over a year and it is believed that the aging rate is considerably less than $1x10^{-11}$ per day at this temperature.

Four AT-cut plates for operation at $4^{o}K$ have also been furnished by Bell Telephone Laboratories for testing. Two are 5 Mc plates operating on the fifth mechanical overtone and two are 10 Mc plates operating on the seventh mechanical overtone.

The tests conducted on these crystals have benefited from the experience gained in testing the previously mentioned ones. It appeared from the tests on the GT plates in helium that the temperature versus pressure coefficient of helium could not be neglected, in spite of the small frequency versus temperature coefficient of AT plates at this temperature.

Accordingly, a pressurizing system, shown in Figure 13, was constructed to stabilize the temperature of the helium bath. The helium bottle was used to replace gas lost through leaks. The system has since been modified so as to handle four dewars, as shown in Figure 14. The leaks in this system have been sufficiently reduced so as to permit removing the helium bottle and replacing it with a relief valve to take care of the helium boil off from the dewars. Also, a quiet oil-free pump has replaced the noisy, oil type unit shown in the previous slide.

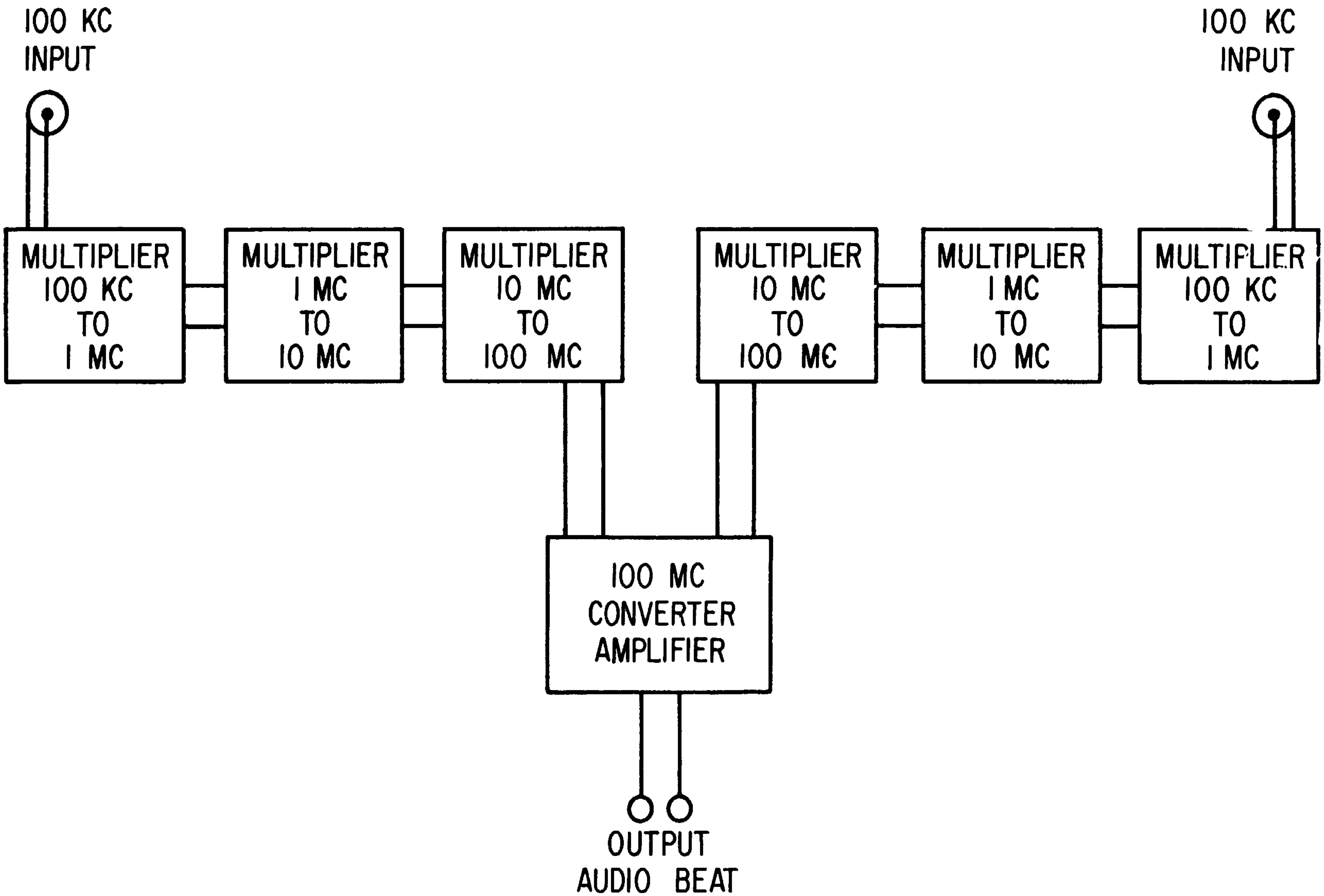
Figure 15 shows the latest model of the transmission line used to bring the crystal leads out of the dewar and up to the oscillator. It is the result of many small improvements on the first model that was used in liquid nitrogen. The outer walls are 0.010 inch stainless steel and the center conductor is No. 32 copper wire; the copper discs are heat reflectors to help conserve the helium. The tubes are closed on each end with hermetic seals and there is a provision to pump out the air space inside the shield in order to prevent any possible effect on the oscillator frequency when the nitrogen jacket of the dewar is filled.

The oscillator used is the one developed for the 5 Mc crystal plates in liquid nitrogen. Its schematic diagram is shown in Figure 16. One slight change was made in order to conserve the helium, and to facilitate adjustment of the components in the crystal network. This part of the crystal network was moved from the copper cylinder in the dewar and placed on the oscillator chassis. Circuit heaters were then added and the entire oscillator chassis temperature controlled with a thermostat with $0.02^{o}C$ sensitivity.

Figure 17 shows the performance of one 5 Mc unit for a period of twenty-five days. While there are several changes in frequency, each can be traced to some change in operating conditions; thus, the average aging rate appears to be less than a few parts in 10^{10} per day.

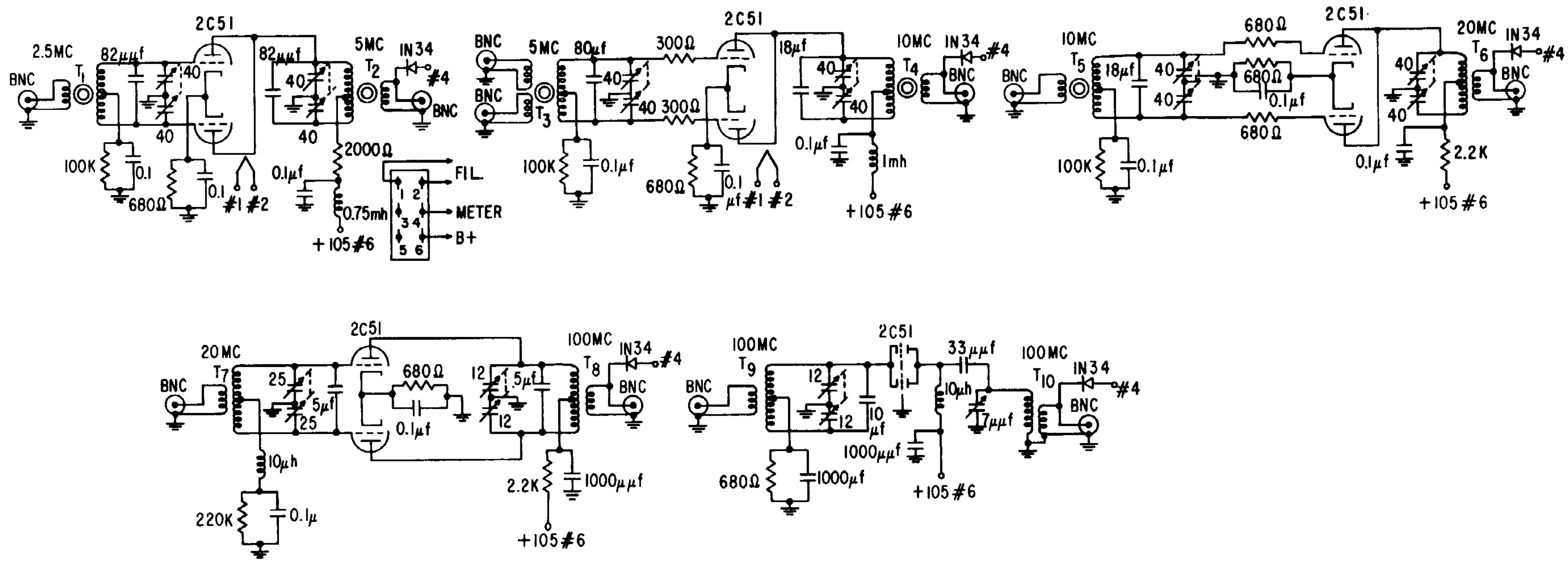
Figure 18 shows one-second stability of the same crystal as compared against an Ammonia Beam Maser. It appears as though the pressure regulating system is degrading the short-time stability from $\pm 4x10^{-11}$ to $\pm 1x10^{-10}$.

Figure 19 shows a part of the record of two 5 Mc crystals in helium when compared together. Chart A is a twelve-hour recording. Here the frequency has remained constant to better than $5x10^{-10}$ and the average drift is less than $1x10^{-10}$. Chart B shows the effect of filling the nitrogen jacket of a dewar containing a transmission line without the evacuated shields. The frequency is disturbed nearly $6x10^{-9}$ and does not return to normal for over three hours. Chart C shows the effect of the same operation when an evacuated transmission line is used. The frequency is changed by about $1.5x10^{-9}$ and in less than one hour the oscillator frequency has returned to normal.



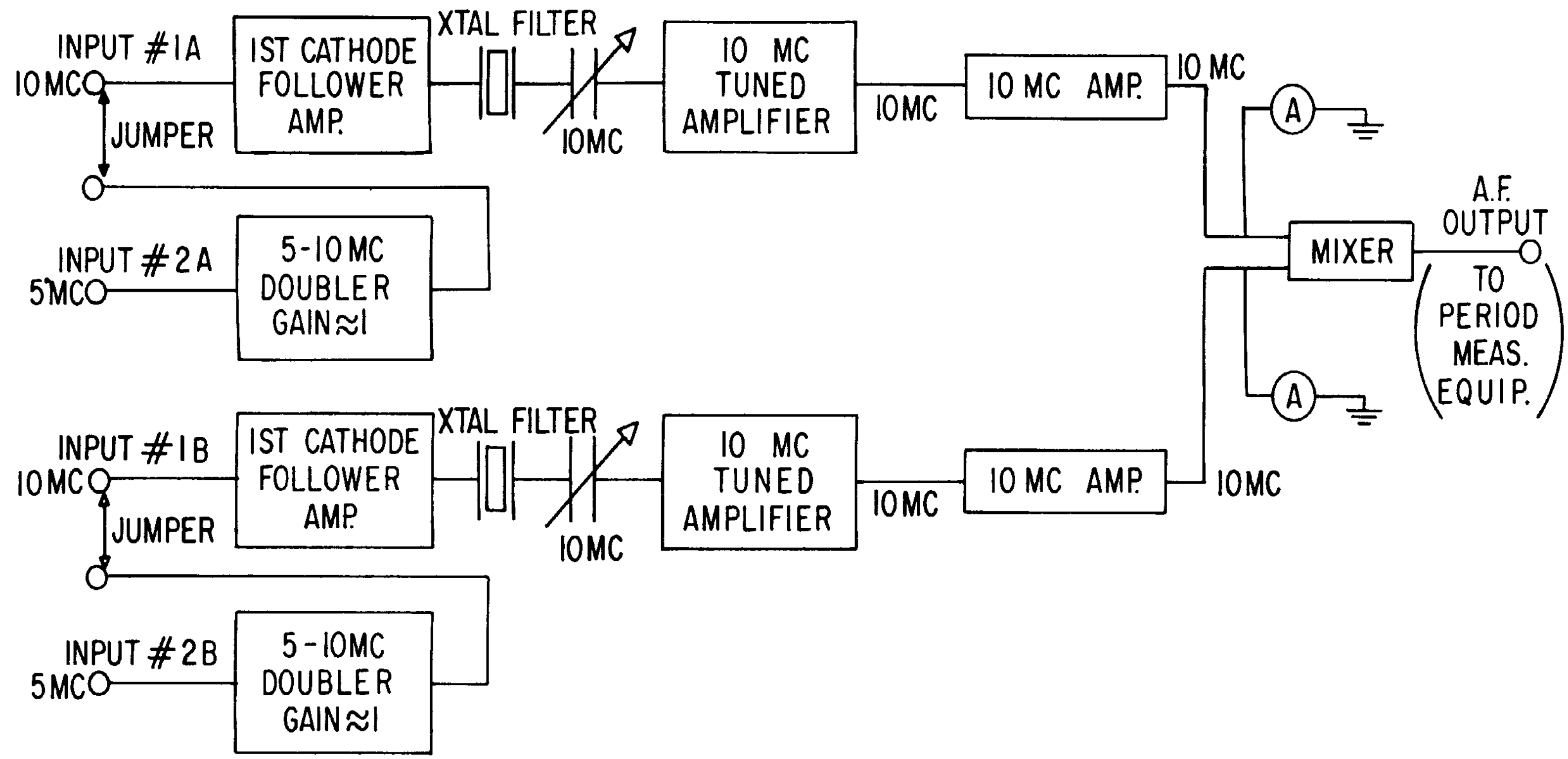
BLOCK DIAGRAM OF DUAL CHANNEL MULTIPLIER

Figure 1



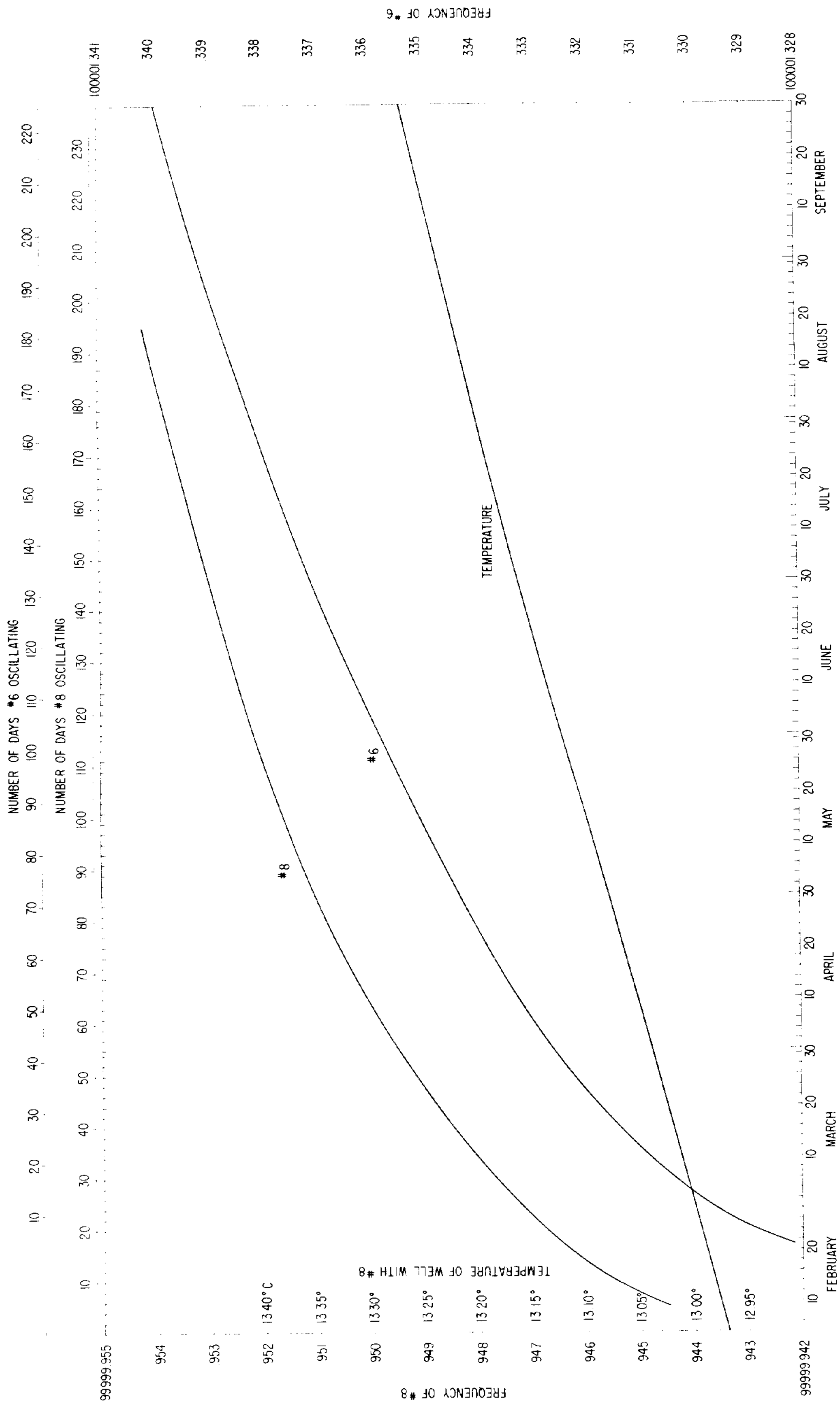
TUNEABLE FREQUENCY MULTIPLIER, 2.5 MC TO 100 MC

Figure 2



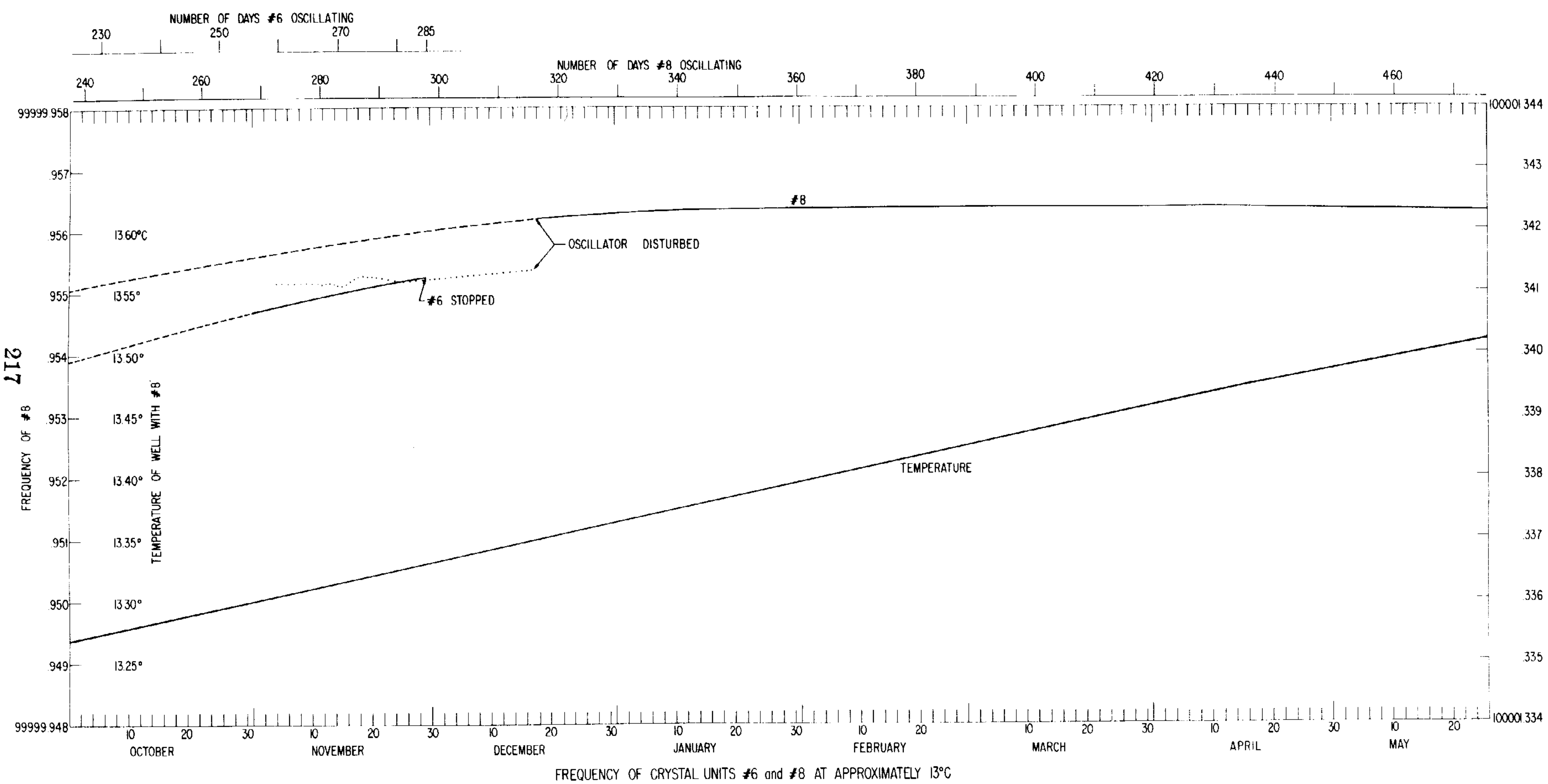
BLOCK DIAGRAM OF PRECISION FREQ. MEAS. APPARATUS

Figure 3



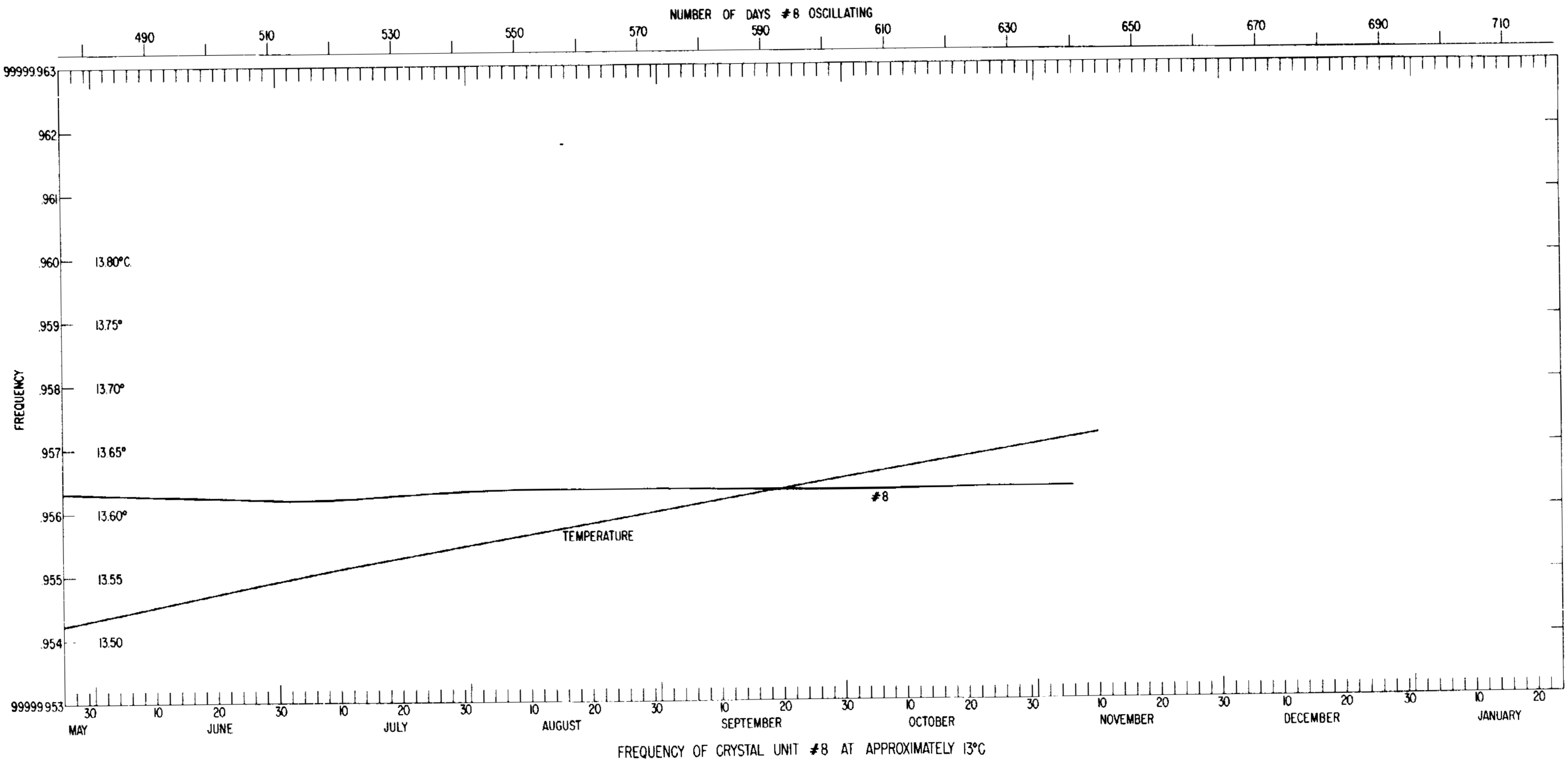
FREQUENCY OF CRYSTAL UNITS #6 and #8 AT APPROXIMATELY 13°C.

Figure 4



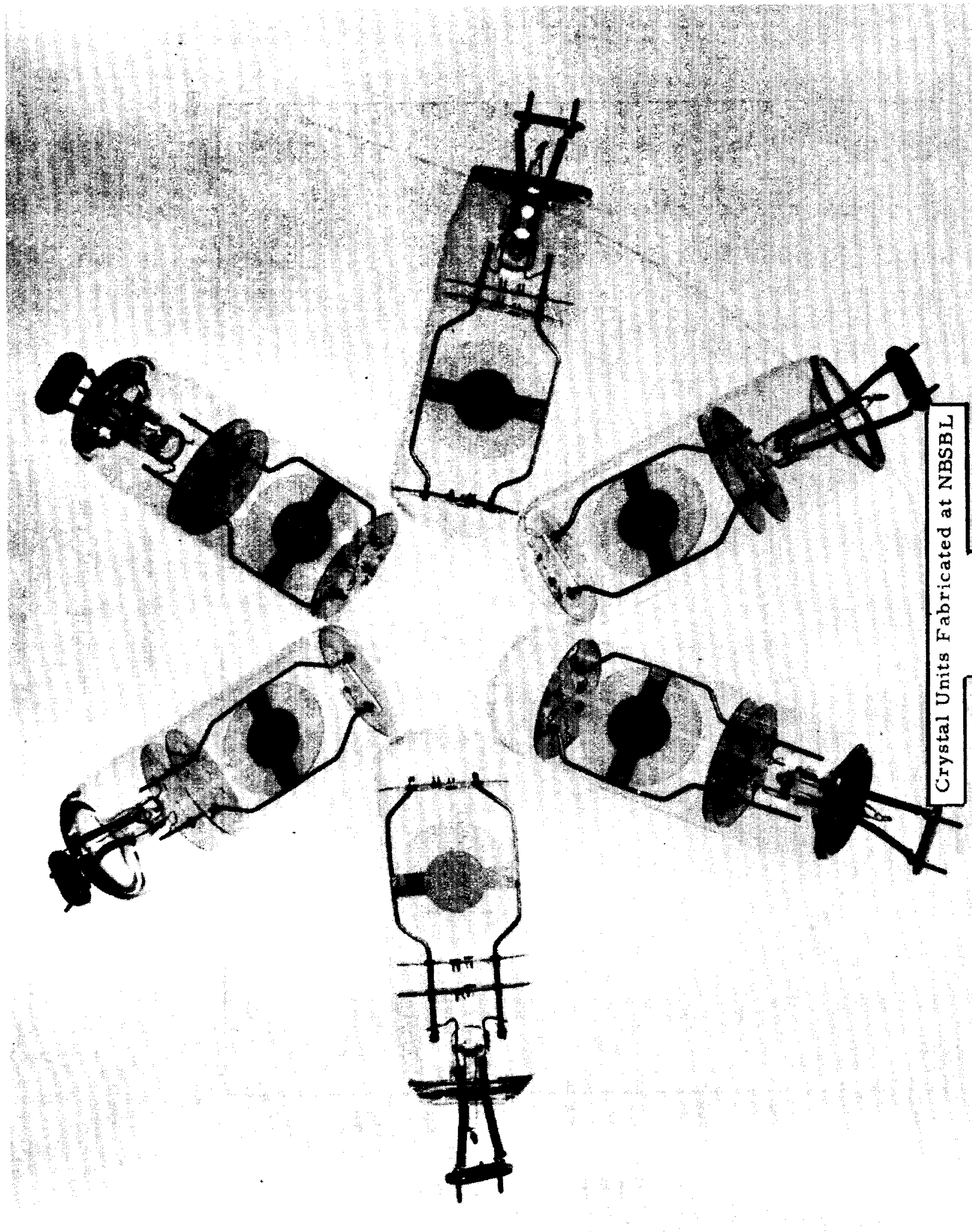
Frequency of Crystal Units #6 and #8 at Approximately 13°C

Figure 5



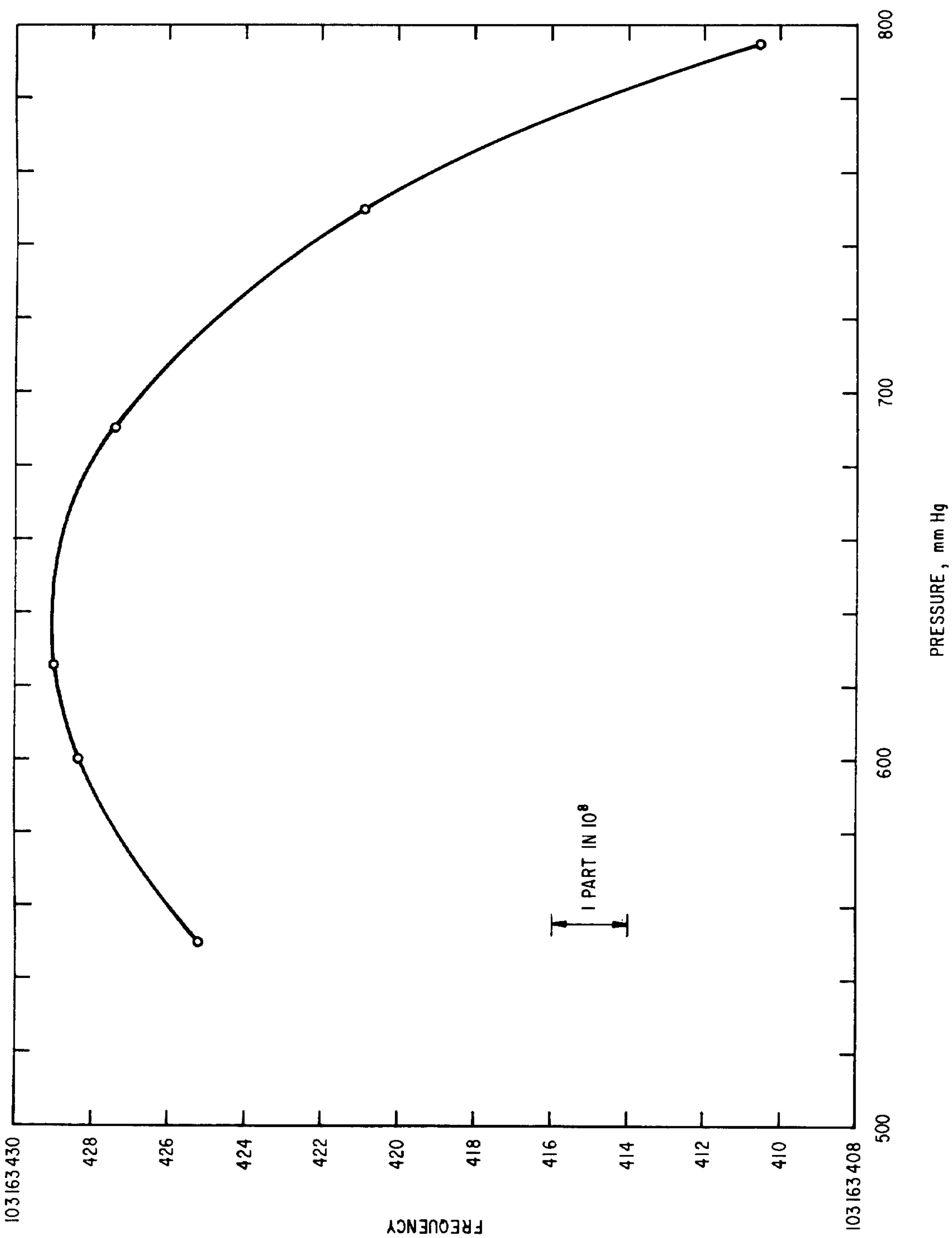
Frequency of Crystal Units #6 and #8 at Approximately 13°C

Figure 6



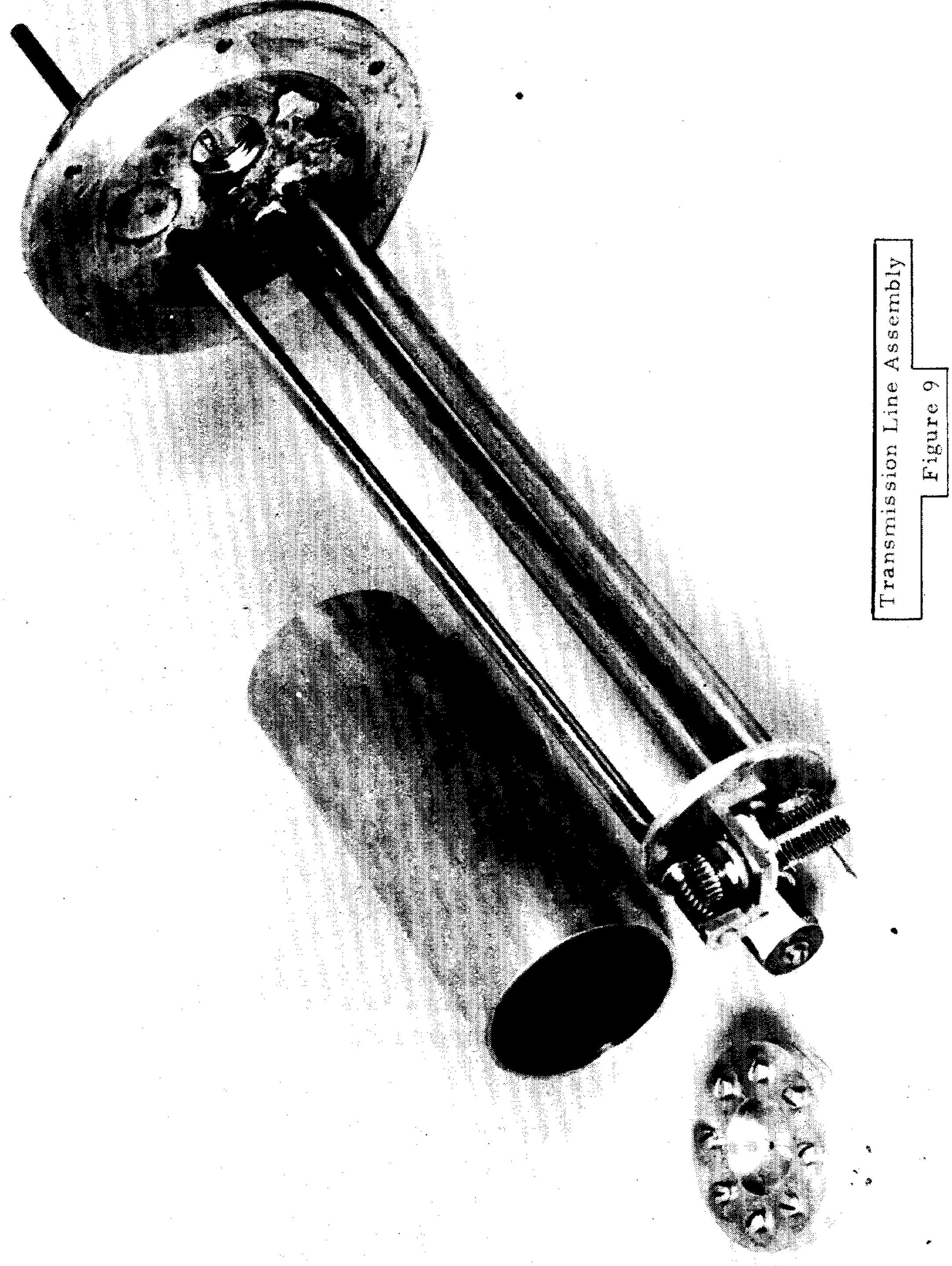
Crystal Units Fabricated at NBSBL

Figure 7



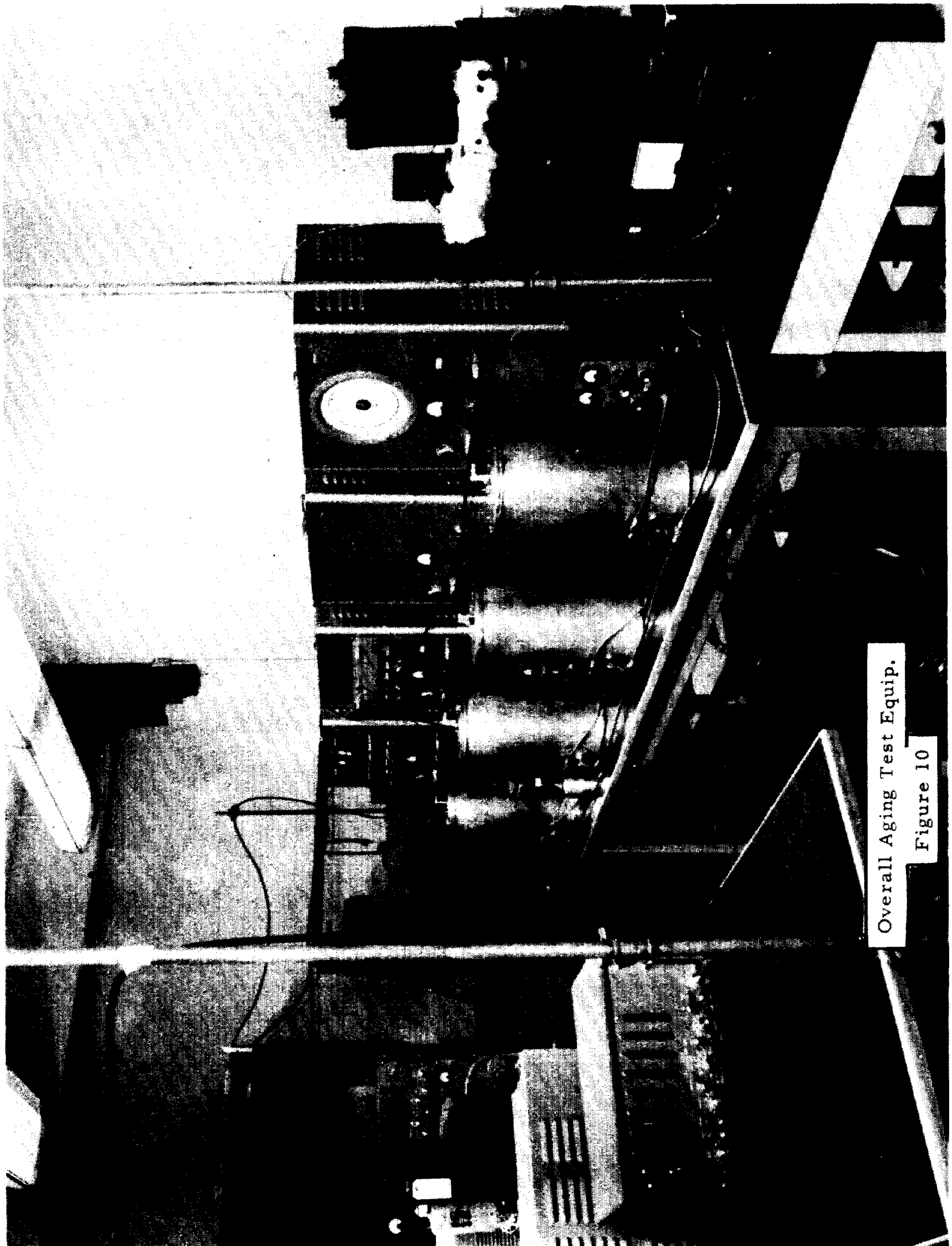
FREQUENCY VS. PRESSURE, CZM-8 IN LIQUID NITROGEN

Figure 8



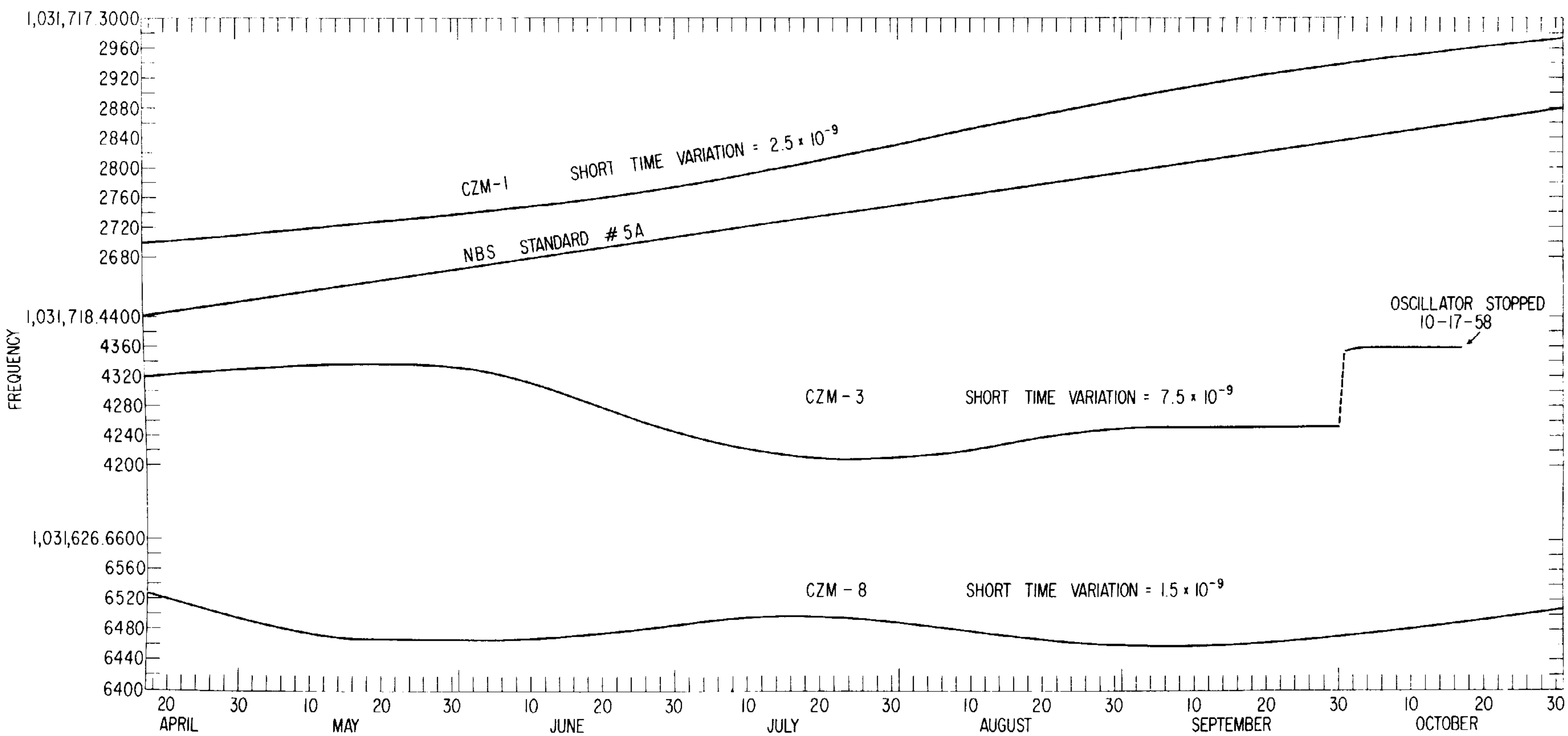
Transmission Line Assembly

Figure 9



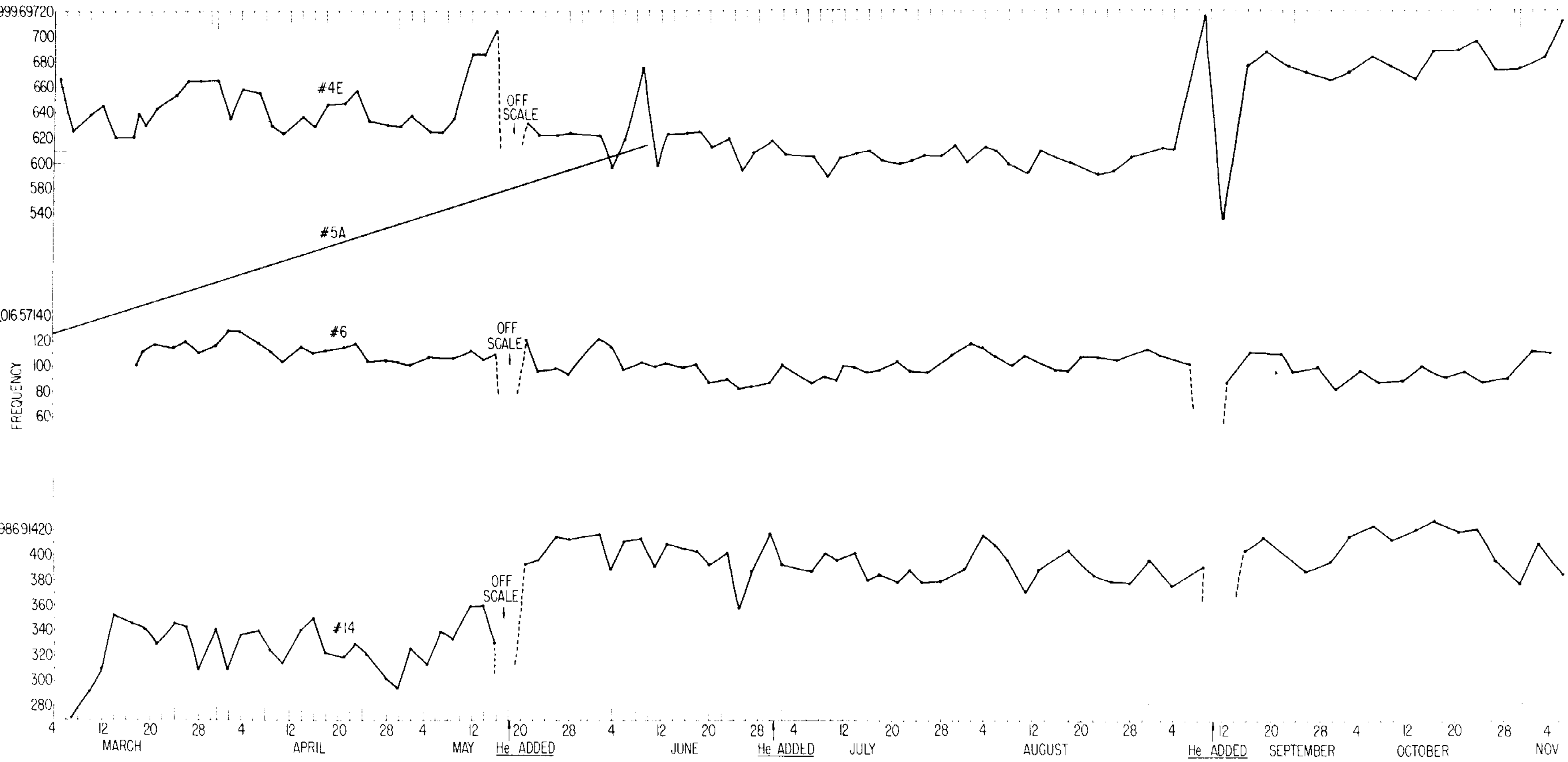
Overall Aging Test Equip.

Figure 10



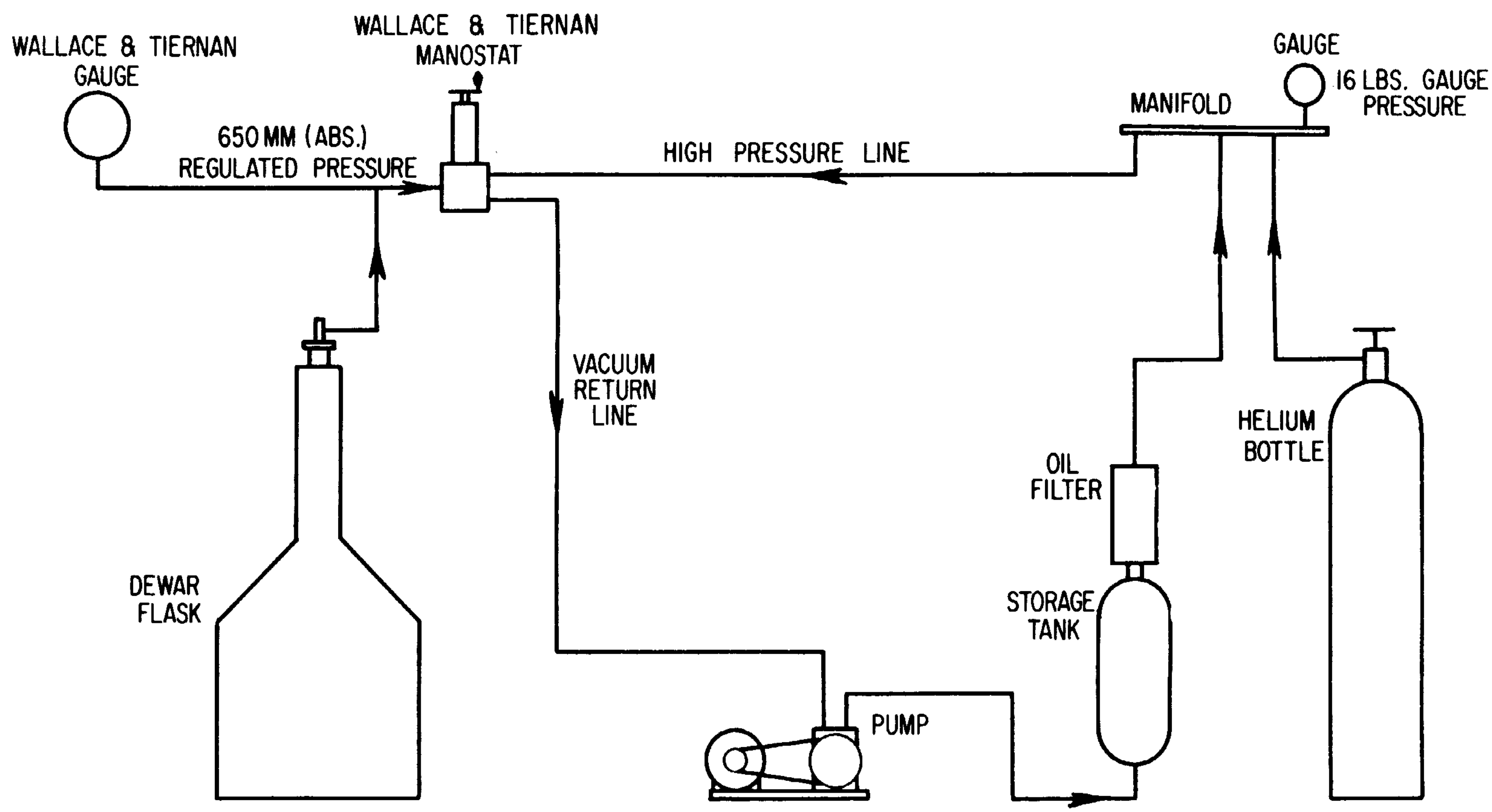
AGING OF CRYSTAL UNITS CZM 1,3, AND 8 AT 76° K

Figure 11



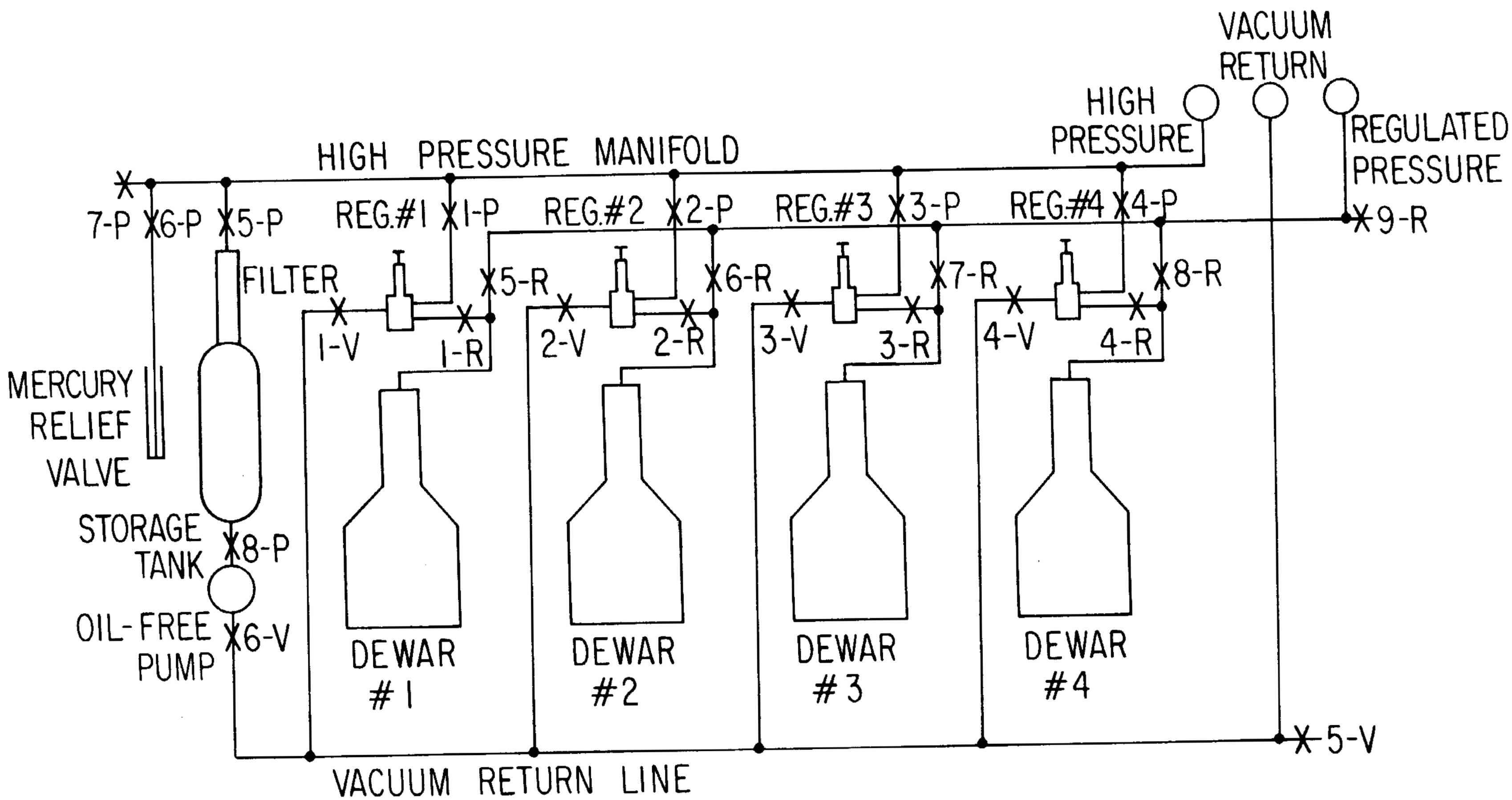
AGING OF 100KC CRYSTAL AT He TEMPERATURE

Figure 12



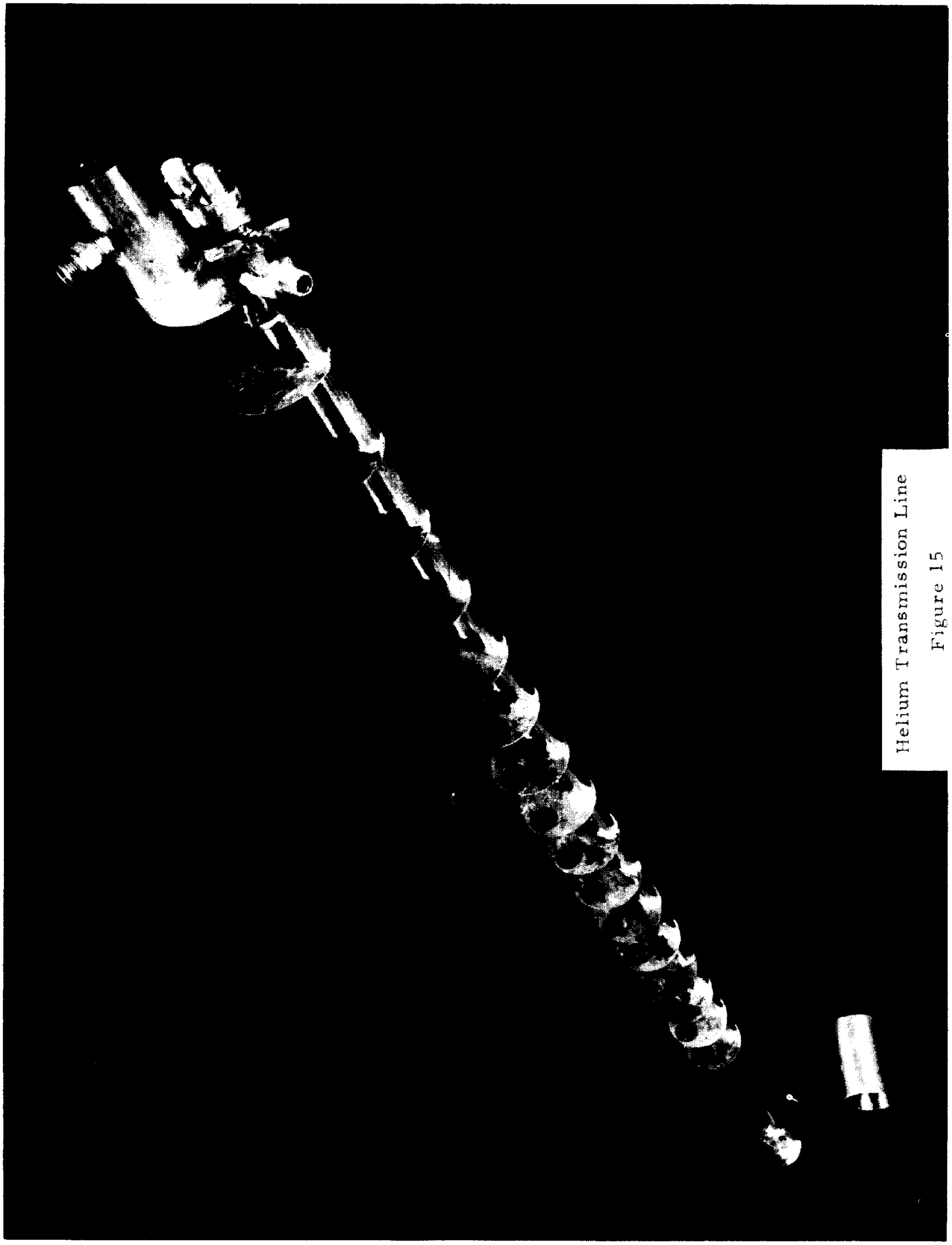
PRESSURIZING SYSTEM FOR 5 MC CRYSTAL IN LIQUID HELIUM

Figure 13



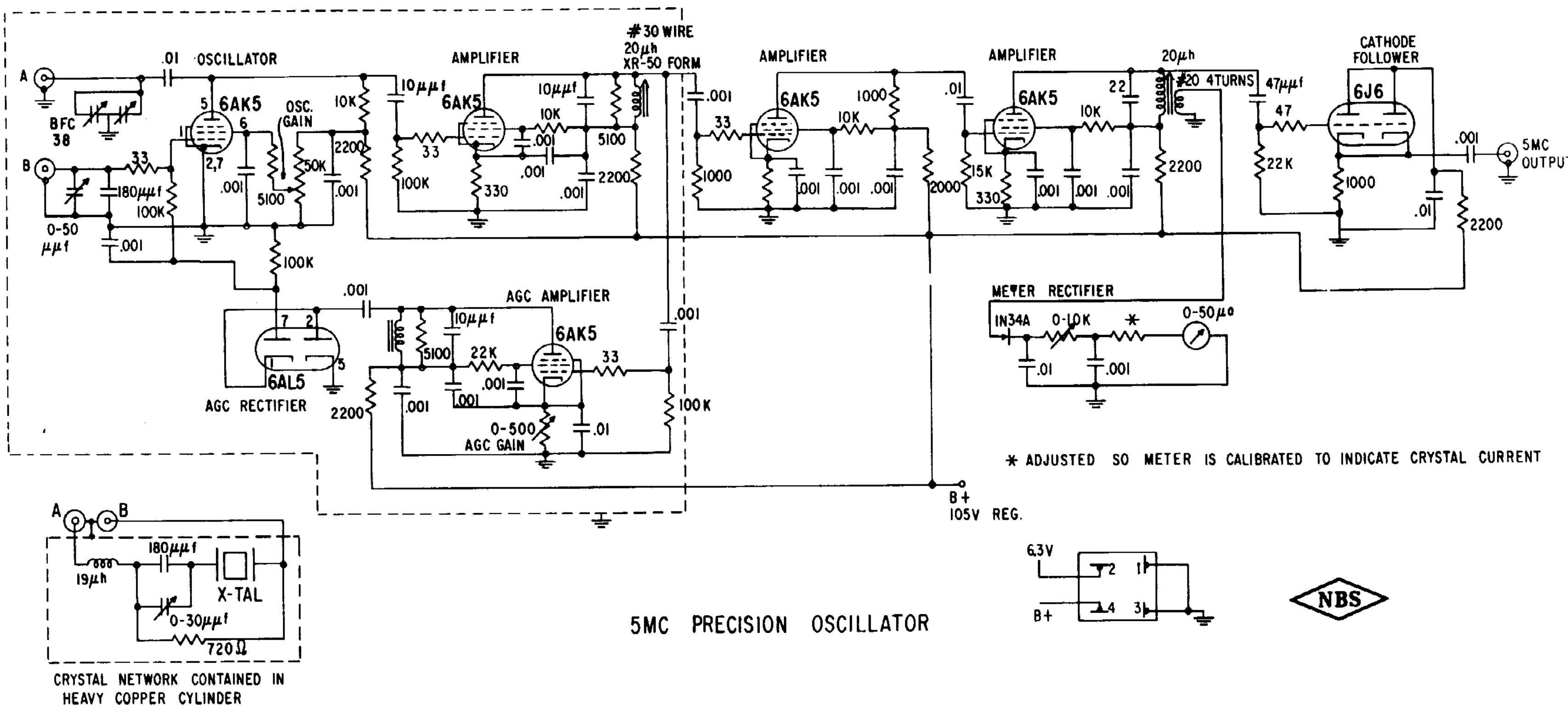
PRESSURIZING SYSTEM FOR FOUR HELIUM DEWARS

Figure 14



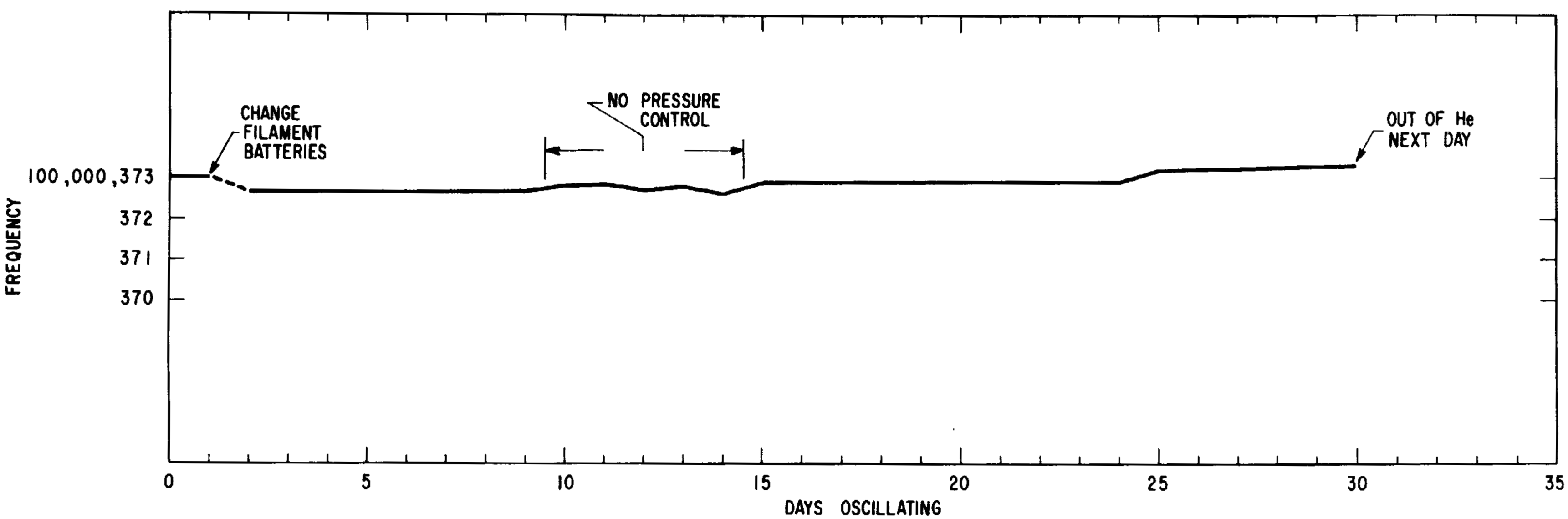
Helium Transmission Line

Figure 15



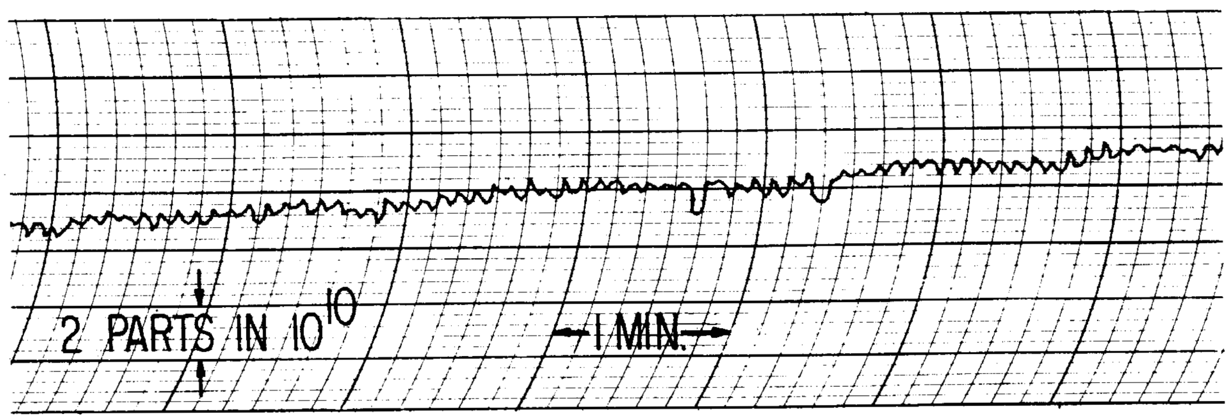
5MC PRECISION OSCILLATOR

Figure 16

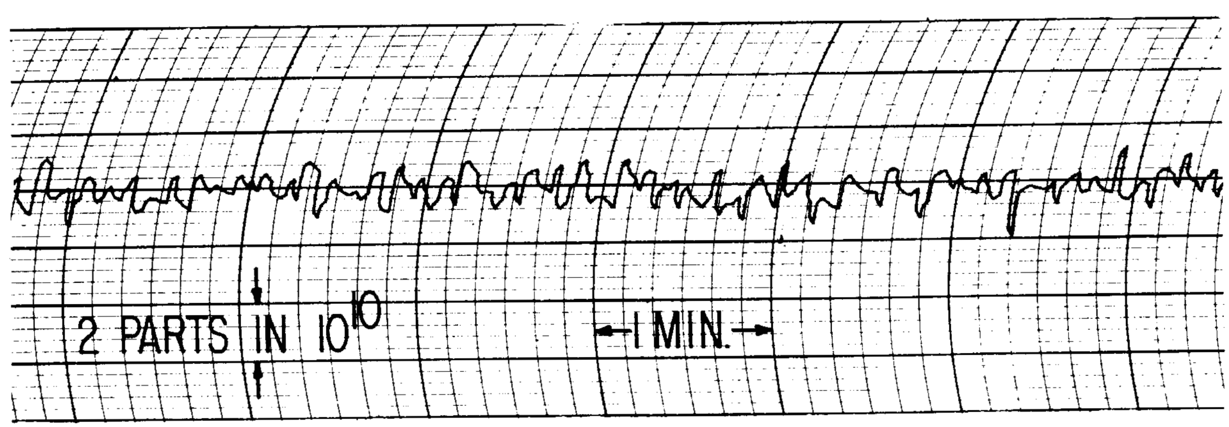


FREQUENCY OF CRYSTAL UNIT # 4
WITH CLOSED RE-CIRCULATING PRESSURIZING SYSTEM

Figure 17

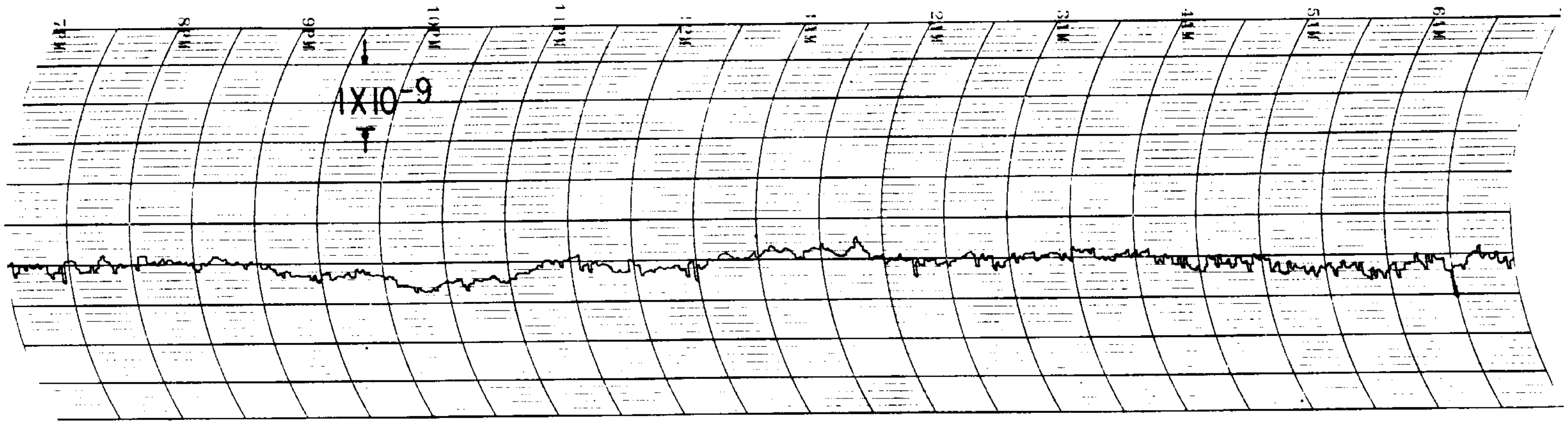


HELIUM OSCILLATOR VS MASER (NO PRESSURE CONTROL)

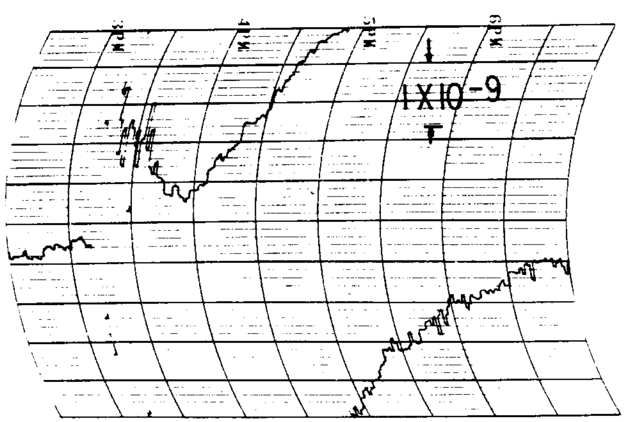


HELIUM OSCILLATOR VS MASER (PRESSURE CONTROL)

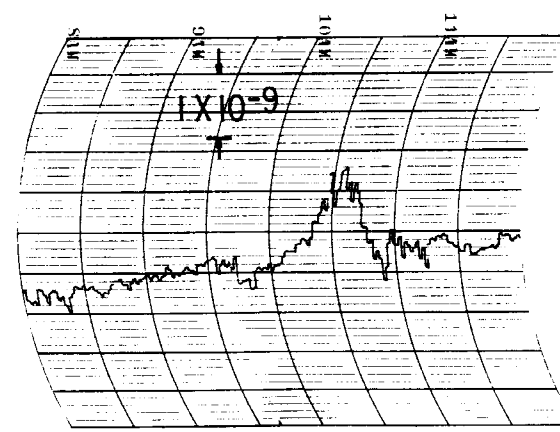
Figure 18



(a) FREQUENCY OF #1 VS #4 FOR 12 HOURS



(b) FREQUENCY CHANGE CAUSED BY FILLING NITROGEN JACKET OF DEWAR WITH UNEVACUATED TRANSMISSION LINE SHIELD



(c) FREQUENCY CHANGE CAUSED BY FILLING NITROGEN JACKET OF DEWAR WITH EVACUATED TRANSMISSION LINE SHIELD

SAMPLE RECORDS OF CRYSTAL #1 VS #4

Figure 19