RESEARCH AT NBS BOULDER LABORATORIES ON QUARTZ CRYSTAL RESONATORS AND OSCILLATORS AT LOW TEMPERATURES

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NATIONAL BUREAU OF STANDARDS

Measurements performed at NBS Boulder Laboratories on the short term and long term frequency stability of crystal controlled oscillators and quartz resonators, temperature stabilized by cryogenic fluid, are described. Included are some details on the cryogenic temperature ovens used, some of the troubles encountered with them, as well as their long term and short term performance characteristics. Effects of vibration, filling the cryogenic ovens, temperature inversion occurring in the liquid, and ambient temperature changes on the resonators and oscillators are discussed. Some information on the frequency measuring systems will also be given.

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NOTES

Research at NBS Boulder Laboratories on Quartz Crystal Resonators and Oscillators at Low Temperatures

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Last year at this symposium it was reported that in view of the increasing need for higher precision in the available frequency standards, and in measurement techniques and equipment, since these go hand in hand with Standards, work with this end in view was under way at the Boulder Laboratories of the Bureau of Standards along several different avenues of approach, including development of atomic and molecular frequency standards as well as improvement in the performance of Quartz Crystals. This work has been continued and in addition a commercial atomic frequency standard ("Atomichron") has been purchased and put into service along with the group of quartz resonators and oscillators which heretofore have comprised the frequency standard.

At this point, although it has no direct reference to the subject of the present talk, may I just mention, for the benefit of some who may not know about it, that the Boulder Laboratory is now operating a cw experimental standard frequency broadcast on 60 kc. The frequency transmitted is accurate to 1 part in 10^8 and is stable to about 2 parts in 10^{10}. It is believed to be usable for specialized measurements, anywhere in the country, to a precision of 1 part in 10^9. The bureau will appreciate any reports concerning it.

As to means for comparing frequencies with the standard, studies have been made along several different lines of approach, most of which have been outlined in our progress reports. Of these only two will be discussed upon here. The first is shown in block diagram in Slide O, upper part only. It parallels quite closely the system used for monitoring the Bureau's Standard Frequency Broadcasts station WWV.

The signal under test is fed into one channel of an NBS type dual channel multiplier while a standard frequency is fed into the other. Both are multiplied to 100 mc, fed to a mixer stage and the output difference frequency amplified, filtered and fed to an electronic counter. The multiplier will accept inputs of 100 kc, 1 mc, and 10 mc. When dealing with a 5 mc crystal the crystal frequency is doubled and fed into the 10 mc stage. Since the normal resolution of the counter is 1 cycle/sec a ten second count gives a precision of $\pm$0.1 c/s. At 100 mc this is $\pm$1 part in 10^9. We are now using a counter with a 100 second count resulting in a precision of $\pm$1 part in 10^{10}. Electronic counters normally are equipped with an internal frequency source to operate the gate. However, this is not sufficiently stable for the purpose in hand and in every case we supply the gate with a frequency through a distribution amplifier from the NBS Primary Frequency Standard. This dual channel multiplier is a very narrow-band device and the frequency to be measured must not be off by more than a few cycles at most from the center of the band. To obviate this difficulty a single channel multiplier has been designed and built very similar to the above multiplier except that all the stages are tunable, covering a range at each stage equivalent to about 500 kc at 5 mc. This equipment has been in use for quite some time and is performing very satisfactorily. I may mention however that we have had much trouble with the electronic counter not staying in adjustment, and many of our crystal aging runs have been interrupted because of this. The ambient in the room where the counter is located ranges from 28°C to 31°C and this appears to be just too hot for this particular counter. At any rate the troubles appear to be overcome by supplying more ventilation by adding an extra blower to the counter. This system is quite satisfactory where measurement of short-time stability is not important. However, in order to utilize the full precision afforded by atomic frequency standards, quartz crystal oscillators with better short-time stabilities are required.

It has been difficult in the past to observe the behavior of oscillators over very short-time intervals with any degree of precision. In fact very little work has been done on this problem, because it has only recently become of major significance. Most of the research to date has been on the improvement of the aging characteristics of quartz resonators over periods of days and months.

However, both the long-term and the short-term stability of oscillators is becoming more important. In some ways these qualities appear to be incompatible. For instance, some of our studies have indicated that rather high crystal current is required to give good short-time stability to the oscillator, whereas for best long-term performance rather low crystal current must exist. This is reasonable in view of the fact that good short-term stability is dependent on a high signal-to-noise ratio to achieve low phase jitter, whereas low crystal current permits better long-term oscillator performance because of less dependence of frequency on the amplitude of oscillations.

As an aid in the study of precision oscillators, a new frequency measuring instrument has been developed, under the direction of the co-author, Mr. Morgan, which has several advantages not enjoyed by previous units. It is based on the principle of precise period measurement developed in 1947 by Law (1), and others (2). A similar unit has been developed by the Bell Telephone Labs, also. It has some basic limitations that make it unsuitable, in its present form, for general use over a very wide range of frequencies, but for special types of measurements, wherein only a very limited range of frequencies is used, it is unexcelled at present.

The instrument utilizes a combination of two simple techniques, frequency multiplication and precision period measurements, made possible by modern electronic counters.

It may easily be explained by reference to the block diagram (Fig. 1). A reference oscillator (f_s) is adjusted so that the beat frequency obtained between it and the unit (fx) under study, after frequency multiplication of both to 10 mc, is within the range of from one cycle per second to 1000 cycles per second; the beat frequency chosen by the user will depend on the sensitivity desired and the time required for a single measurement. Using an electronic counter, adjusted for period measurements, the beat frequency is then measured; and by using a digital to analog converter, the frequency measurements may be continuously recorded.

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(1) H.B. Law, "An Instrument for Short-Period Frequency Comparisons of Great Accuracy," J IEE, Part 3, p. 38, Jan. 1947.

(2) R. Trainer, "A Recording Precision Frequency Comparator," Research Lab. Report No. 4170, dated 31 Jan 1956, Research Labs, Eng. Div., Postmaster General's Dept., Melbourne, Australia.

To indicate the wide range of sensitivities and measuring times of this instrument, the chart in Fig. 2 was devised. This chart is also useful, when making measurements, for determining the exact beat frequency (at 10 mc) from the count indication, and to establish which readout column on the counter corresponds to a specified precision, say 1 part in 10^9.

Measuring sensitivities shown on the chart beyond 10^{-11} have no practical significance since jitter or background noise of about this amount is present.

As mentioned before, Fig. 1 is the block diagram and Fig. 3, 4 and 5 are the circuit diagrams of the first model of the NBS frequency measuring instrument. It consists of two 10 mc amplifiers, with a simple crystal filter in the input of each, a mixer amplifier with an output filter and audio frequency amplifier, and an additional stage of audio amplification and filtering. This system has performed very well and the data shown in Fig. 6 was taken with it.

An improved model, as in the block diagram shown in Fig. 7, was developed after an exchange of ideas with the Bell Telephone Laboratories. In this unit, provision was made for using it with either 5 mc or 10 mc inputs by including a frequency doubler (5 to 10 mc) as shown in Fig. 8. A cathode follower circuit (Fig. 9) cascaded to this provides a low impedance driving source for the crystal filter in the first 10 mc amplifier, Fig. 10. This is followed by a second 10 mc amplifier stage, Fig. 11, which feeds the mixer shown in Fig. 12. In this system, all the filtering is done at 10 mc, in contrast to the first model where most of it was done at the audio frequency output.

As mentioned above, some tests of three NBS oscillators, with their crystals at about liquid nitrogen temperature, were made with the first model of this instrument, and measurements are plotted in Fig. 6. The one second and ten second stabilities of these oscillators are as shown in the graphs.

A few sample measurements on the short-time stability of one NBS oscillator, not at liquid nitrogen temperature, is given to illustrate the use of the new instrument and the frequency stability over various time intervals. Fig. 13 is the 100-second stability of one NBS oscillator versus an atomic frequency oscillator. The following three recordings taken subsequently used the same beat frequency, but with a measuring time of 10, 1 and 0.1 sec., respectively. Thus, Fig. 14 is the 10-second stability, Fig. 15, the one-second stability and Fig. 16 the 0.1-second stability of this oscillator versus an atomic standards. As mentioned before, the measuring times are determined by the number of periods of the beat frequency that are measured. In the above, 10 seconds was achieved by measuring 100 periods of the 10 c/s beat, 1 second by 10 periods and 0.1 second by one period.

A study of these recordings indicate the following: 100 second short-time stability was about ±1/2 part in 10^{10}; the 10 second about the same; but, the 1 second short-time stability was about ±1 part in 10^{10}, and the 0.1 second stability was approximately ±4 parts in 10^{10}. Further study of this type, including the causes of the very short-time instabilities, is being planned. Some theories have been advanced but are not complete enough yet to include here.

One or the other of these two systems have been used for crystal measurements given below.

Aging of Crystal No. 8

Fig. 17 shows aging rate of a 100 kc GT crystal located in a 50 ft. well. After four years the temperature of the well is still rising at somewhat less than a millidegree per day or about 1/3°C per year. The temperature is about 13°C. This curve is interesting in that aging appears to have nearly ceased (less than 5 parts in 10^{10} in three months).

It may be that the aging has reached a point where the frequency change due to this cause is just equal to but opposite in sign to that produced by the temperature rise of one millidegree per day.

Aging of CZM- 1, 3 and 8 and 1-2 at N_2 temperature

Fig. 18 shows the frequency versus time curves for crystals at liquid nitrogen temperature. Within the limits of error we cannot for certain say that there has been any change due to aging. The line marked NBS Standard shows the drift of one of the best of the NBS Group of Standard crystals. Certainly any aging of CZM-8 at N_2 temperature is less than this. The drift here was about 1 in 10^9 in a little over 100 days.

The sloping line shows how the curve would have gone if there had been a drift of 1 part in 10^9 per day. Fig. 19 shows a comparison of crystal 1-2 vs. NBS Standard R-1. The sensitivity in charts a, b and the first part of c is 1 part in 10^8 for full scale. Chart b is continuation of a and d is continuation of c. C and d comprise a four-day section from an aging test run. Chart c shows the drift after opening and refilling the cryostat with N_2 during which time the pressure was reduced from 1270 mm to 625 mm, causing large temperature changes. After the first twelve-hour break on the chart the sensitivity was reduced one order so that full scale is now 1 in 10^7 and the smallest division is 2 in 10^9.

It will be noted that in all this group there are both very short time and also relatively long time changes of the order of a few parts in 10^9. Some of the changes of the type shown here have been reduced considerably by stabilizing voltages, and by cleaning up some of the circuitry so that the beat frequency fed to the counter has a good sine wave form. Voltage stabilization is important notwithstanding the fact that the drive oscillator has provision for keeping the drive level constant. These crystals are very sensitive to drive level even for very low drive as will be seen from Fig. No. 19A. This slide shows the turnover point of one of the CZM series, (5.158 mc 5th overtone) crystals, the frequency having been multiplied to 103 mc before counting. It was operated in a low drive oscillator with about 2 millivolts or less RF across the crystal. The crystal current was roughly estimated to cover the range of about 5 to about 50 microamperes. This crystal was cut to have a zero temperature coefficient slightly above the boiling point of liquid nitrogen at the altitude of Boulder, Colorado, as determined from the curve in Slide 19b which gives the temperature at which the frequency temperature coefficient is zero versus the angle of cut (zz' angle). The ZZ' angle was 39°49' and the temperature of the turnover point was that of liquid nitrogen at 680 mm pressure. It will be seen that the frequency may change by as much as 1 part in 10^7 even at the low drive level used. Very precise regulation of drive level is indicated if a high order of frequency stability is to be attained.

Shock

The crystal units appear to be very sensitive to mechanical shock as evidenced by sudden jumps in frequency sometimes amounting to several parts in 10^9. Even the small shock incident to filling the cryostat with liquid nitrogen apparently causes changes of this order of magnitude. It is now suspected that the changes are due to slight change in position of the crystal in its copper case which is at ground potential, thus changing the capacity of the crystal to ground. We have not had time to investigate this theory fully but the clue leading to it is the fact that when a crystal was put into the cryostat without its copper shield it definitely was not shock sensitive.

There may be some interest in the type of cryostat we are using. Slide 19c is a photograph of the assembly. Inside the large brass cylinder is a 4l-glass dewar to hold liquid nitrogen. The crystal in its glass envelope is contained in a copper cylinder which is immersed in the liquid nitrogen. Leads are brought out through a transmission line. The brass vessel is closed gas tight and the pressure is regulated and controlled by means of a commercial manostat which controls over long periods to about 1/2 mm of mercury. The pressure is read on a highly precise gage which reads absolute pressure. This constitutes a rather accurate gas thermometer. Also an alumel-chromel thermocouple is in thermal contact with the copper cylinder and can be read to ± 0.1 microvolt on a type k-3 potentiometer.

the temperature remains constant to 0.01° until the nitrogen has to be replenished which is about every five days. At present it has to be opened to the atmosphere while adding nitrogen which causes greater or less temperature changes depending upon the operating pressure. Provision is being made to inject the liquid nitrogen under pressure to avoid this interruption.

Aging tests at Liquid Helium Temperature are shown in slide 20. Although the measurements on all the crystals in helium were made with a precision of a few parts in 10^{10} and repeat measurements made immediately gave values agreeing to this degree of precision, yet as can be seen, measurements made at one or two day intervals show differences amounting, sometimes, to several parts in 10^9. Whether these variations are due to interfering pickup on the transmission line between the crystal and the measuring equipment or whether they are due to shock and vibration in the building has not yet been determined. The latter seems the more probable since crystals at this low temperature seem to be highly susceptible to mechanical shock. Similar variations at Bell Telephone Laboratories have been noted and attributed to shock(3).

Until these random variations have been eliminated it will be difficult to properly assess the aging characteristics. However, I think in the case of No. 19E we can say with certainty that it has not aged more than 1 part in 10^9 in somewhat more than six months.

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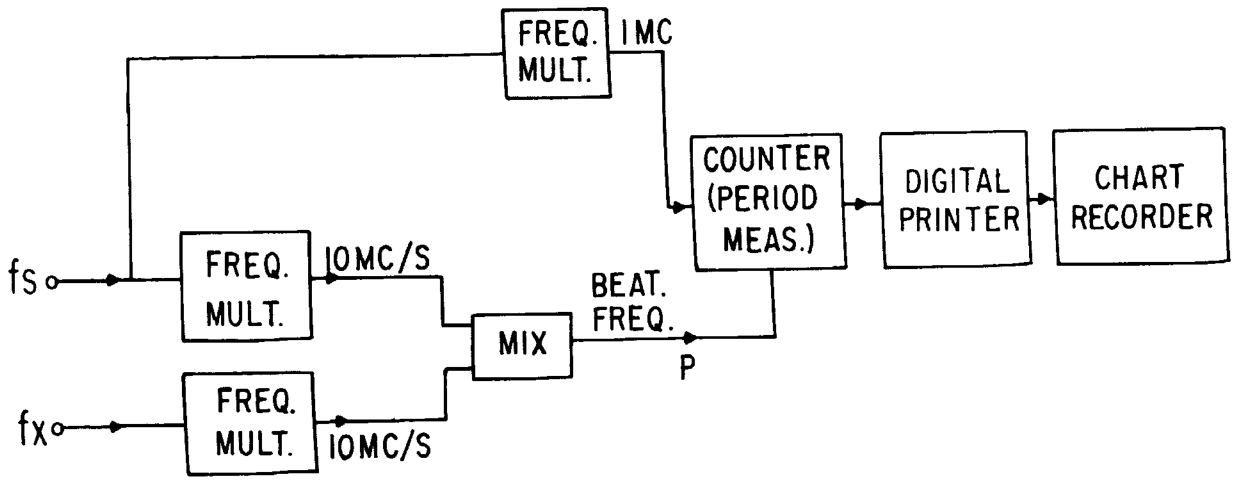
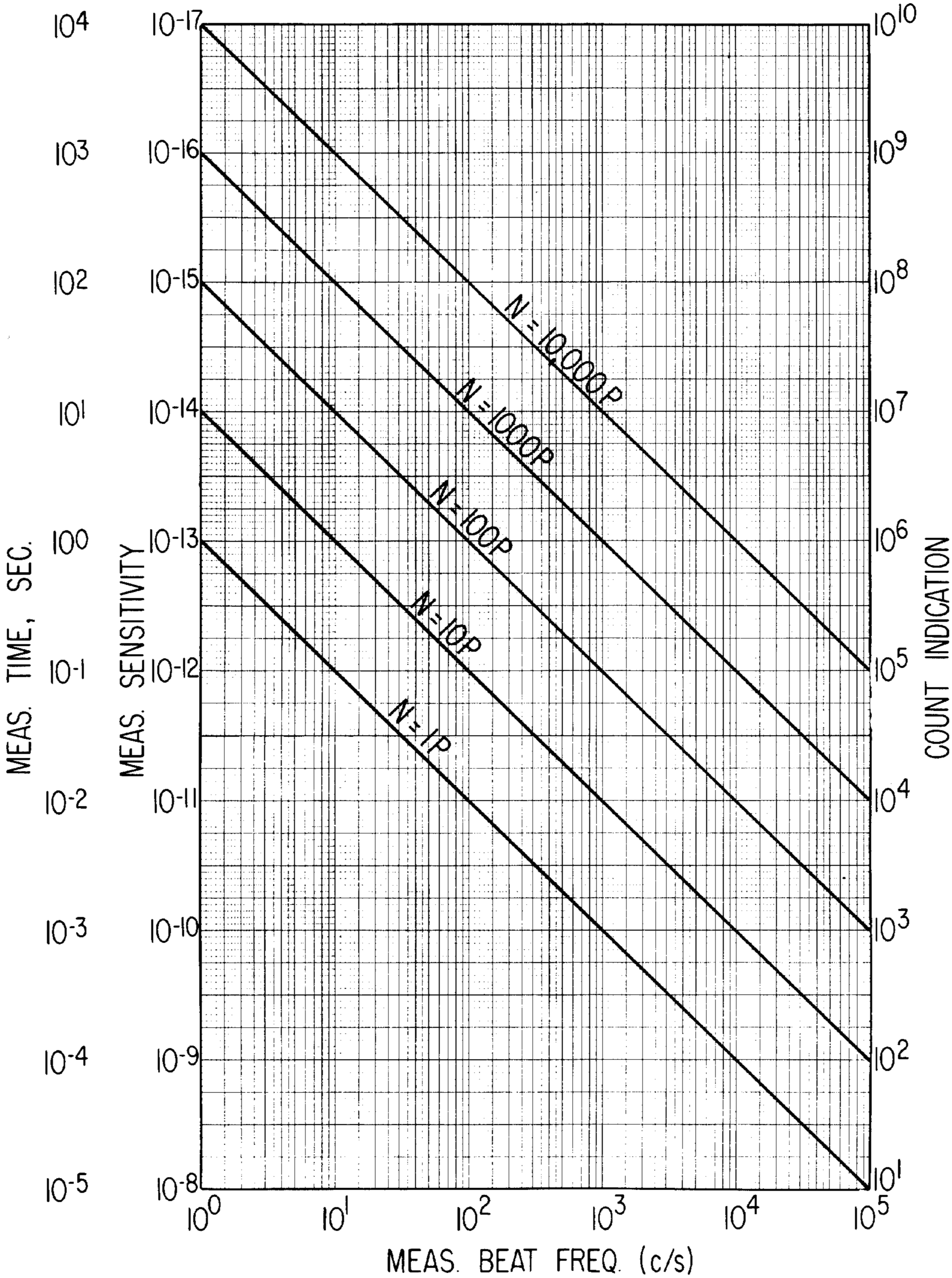
(3) USASEL Contract DA 36-039 sc 73078 "An Ultra Precise Standard of Frequency," Fifth Interim Report, Page 9.



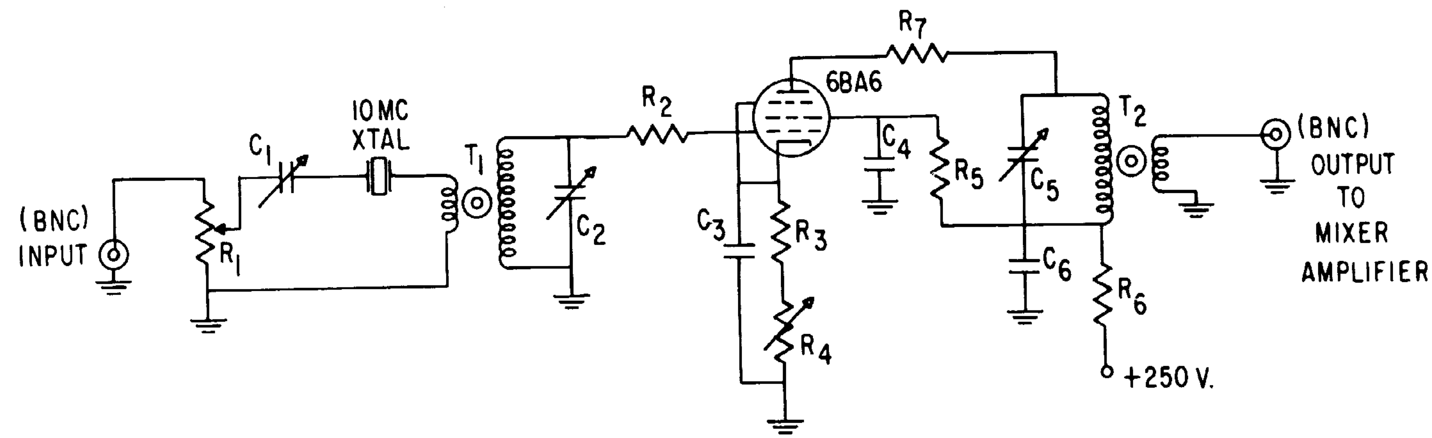
BLOCK DIAGRAM OF FIRST MODEL OF FREQUENCY MEASURING SYSTEM

FIGURE 1



CHART

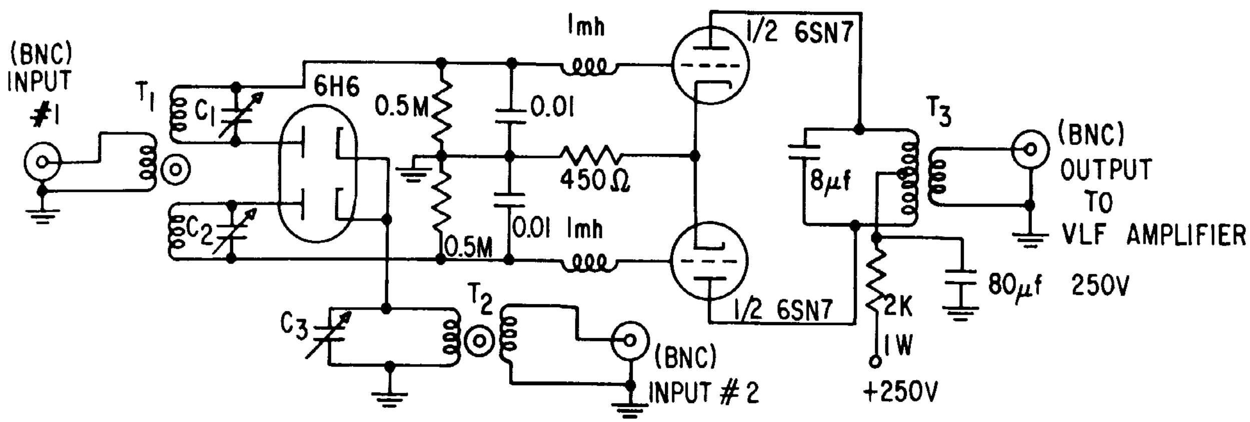
FIGURE 2



R_1 = 500Ω 2W POT.	C_1 = 100pf VAR. CAP.	T_1, T_2 = 20t #24e., 11.9μ CORE, Q=170, C = 47pf
R_2 = 270Ω 1/2W RES.	C_2 = 7-47pf CER. VAR. CAP.	PRI. 3t #24e.
R_3 = 1KΩ 1/2W RES.	C_3 = 0.01 DISC. CAP.	**POWER CONNECTIONS**
R_4 = 10KΩ 2W POT.	C_4 = 0.01 DISC. CAP.	1, 2, = FIL.
R_5 = 39KΩ 1/2W RES.	C_5 = 7-47pf CER. VAR. CAP.	3 = B+
R_6 = 1KΩ 1/2W RES.	C_6 = 0.01 DISC. CAP.	4 = GND.
R_7 = 270Ω 1/2W RES.		

10 MC CRYSTAL FILTER AND AMPLIFIER (SECOND UNIT IS A DUPLICATE)

FIGURE 3



$C_{1,2,3}$ = 7 − 45pf CER VAR WITH 20pf PADDER

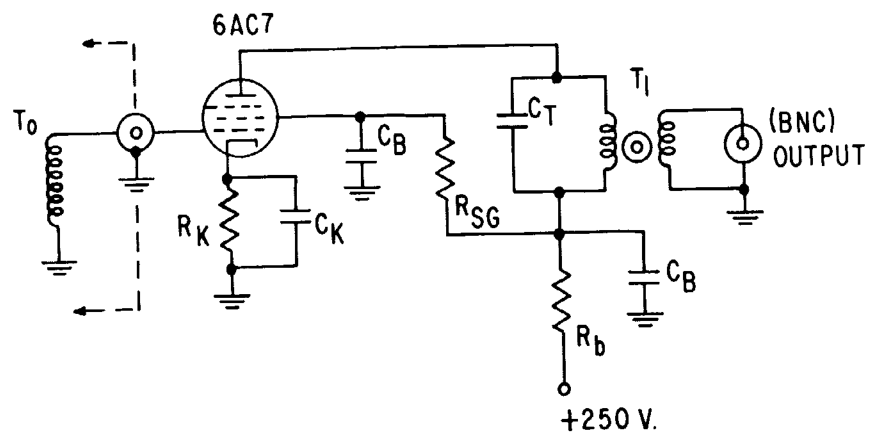
T_1 = PRI, 3t#24e., SECS. 16t(ea)#24e., Q=150, C_T = 50pf μ = 9.3

T_2 = PRI, 3t#24e., SECS. 16t(ea)#24e., Q=180, C_T = 60pf

T_3 = DE COURSEY S-4821, PRI, 12h MAIN WINDINGS
SECS. 252t, 4 EXTRA WINDINGS

MIXER AMPLIFIER

FIGURE 4

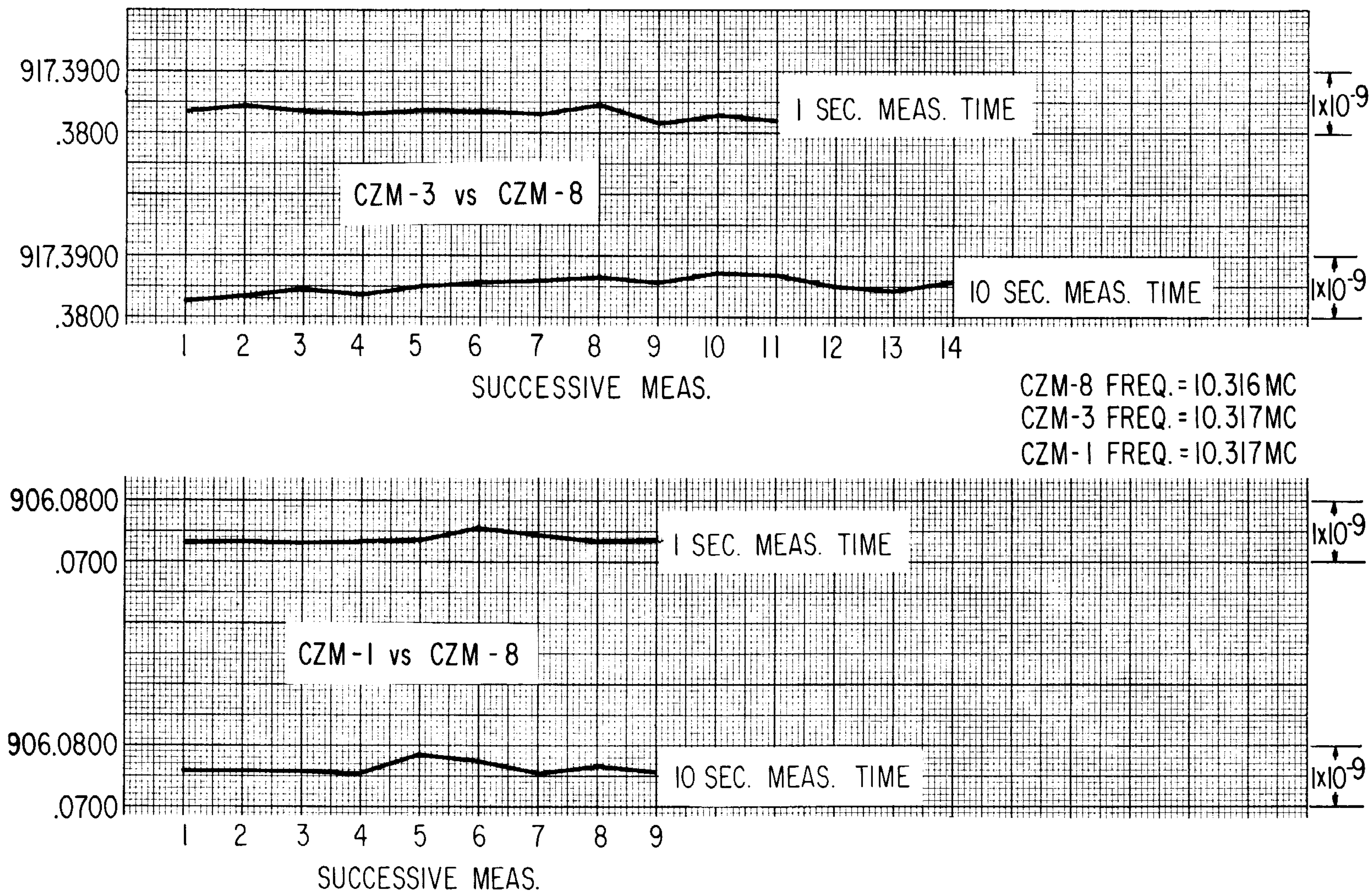


T_0 = OUTPUT TRANS. OF 10CPS MIXER AMPLIFIER
T_1 = DE COURSEY 10h TOROID RATIO 1:1 (S-4821)
C_T = 72μf 600V. PAPER CAPS
C_K = 1000μf 12VDC ELEC.
C_B = 80μf 450VDC ELEC.
R_K = 200Ω 2W POT.
R_{SG} = 58KΩ 1/2W CARBON
R_b = 2000Ω 1/2W CARBON

POWER CONNECTIONS
1,2 = FIL.
3 = B+
4 = GND.

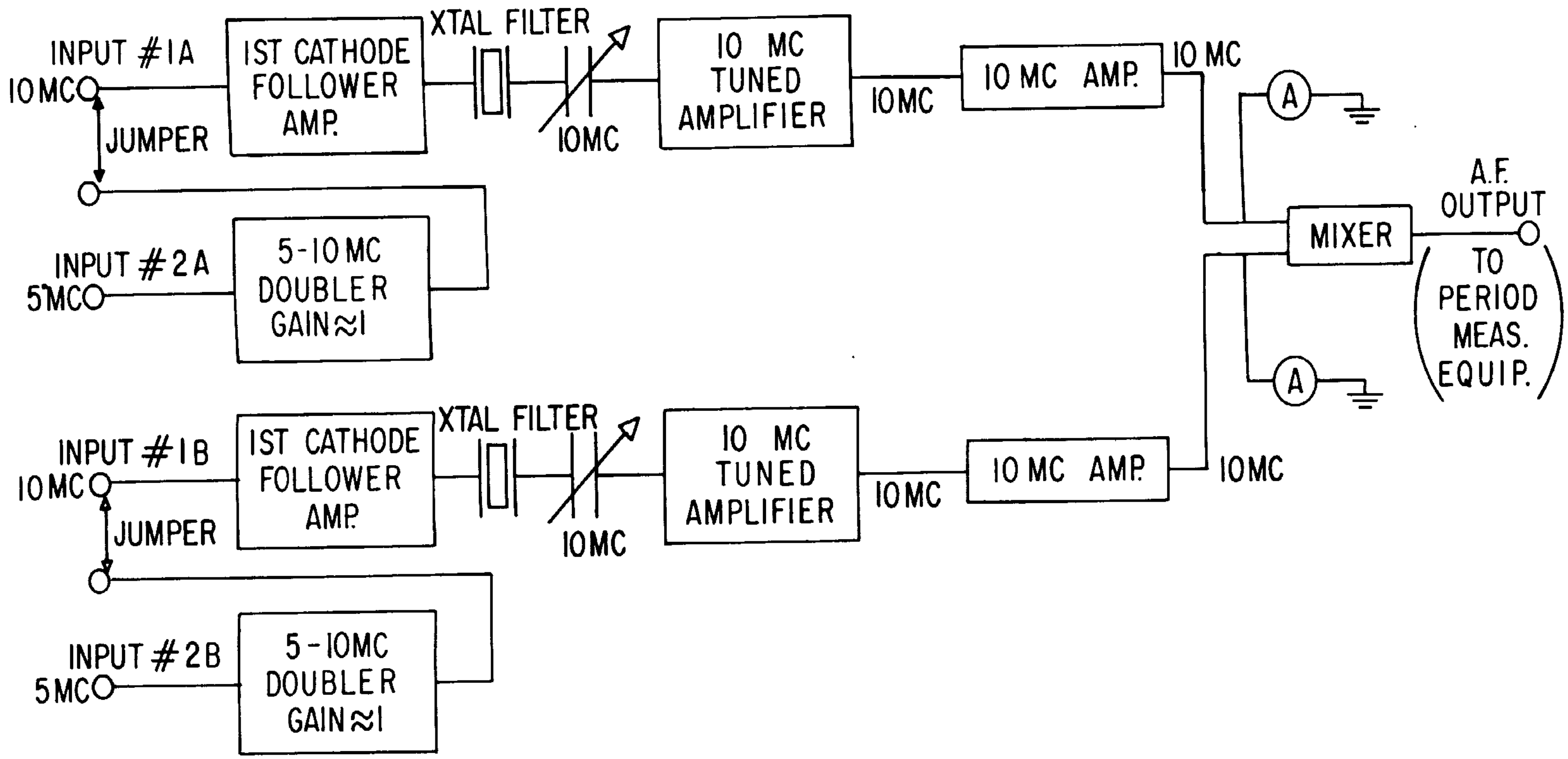
VLF AMPLIFIER (10 CPS TO 1000 CPS)

FIGURE 5



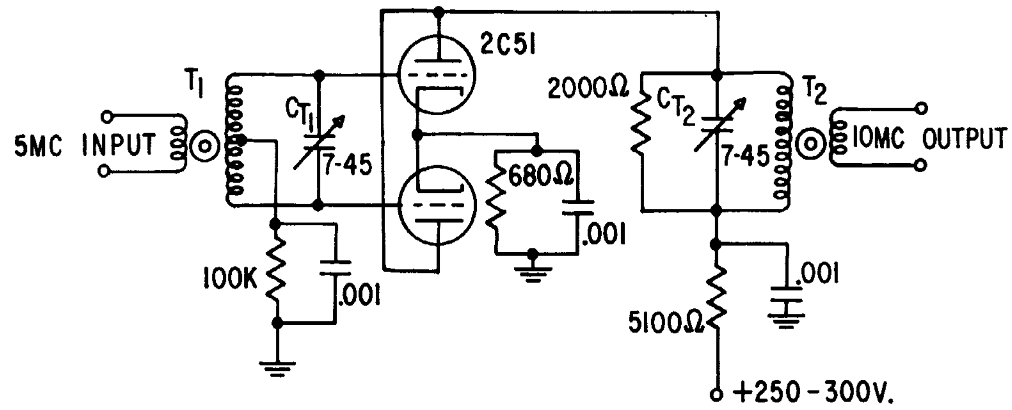
COMPARISON OF NBS OSCILLATORS: CZM-8, CZM-3, CZM-1

FIGURE 6



BLOCK DIAGRAM OF PRECISION FREQ. MEAS. APPARATUS

FIGURE 7



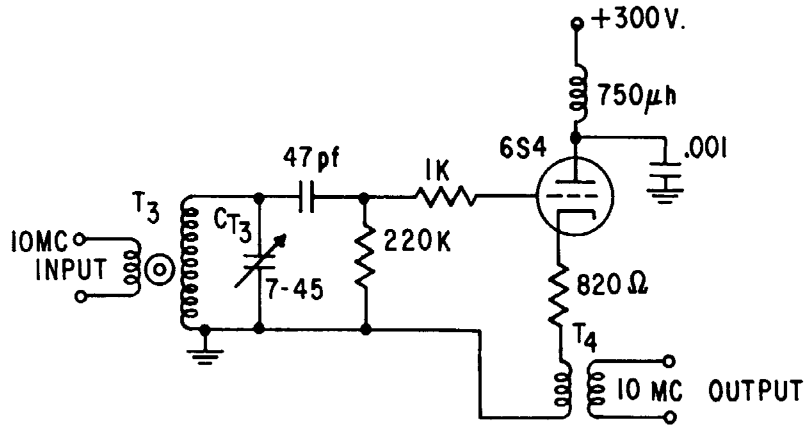
T_1 — PRI. 2t. #28, SEC. 70t. ct. #28. C_{T_1} = 18pf Q = 100

CORE: POWDERED IRON μ = 7.5, 0.75 O.D., 0.5 I.D., 5/10 h.

T_2 — PRI. 50t. #28, SEC. 6t. #28. C_{T_2} = 35pf Q = 100

5-10 MC DOUBLER (2 REQ'D.)

FIGURE 8

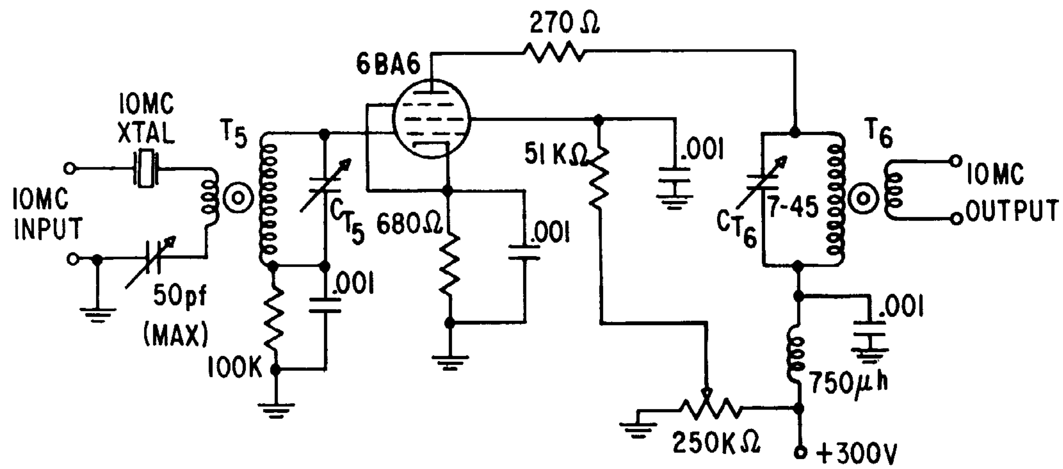


T_3 — PRI. 6t. #28, SEC. 50t. #28 C_{T_3} = 35pf Q = 140

T_4 — PRI. 8μh SOLONOID CHOKE, SEC. 2t. #28 WOUND ON CHOKE

CATHODE FOLLOWER (2 REQ'D.)

FIGURE 9

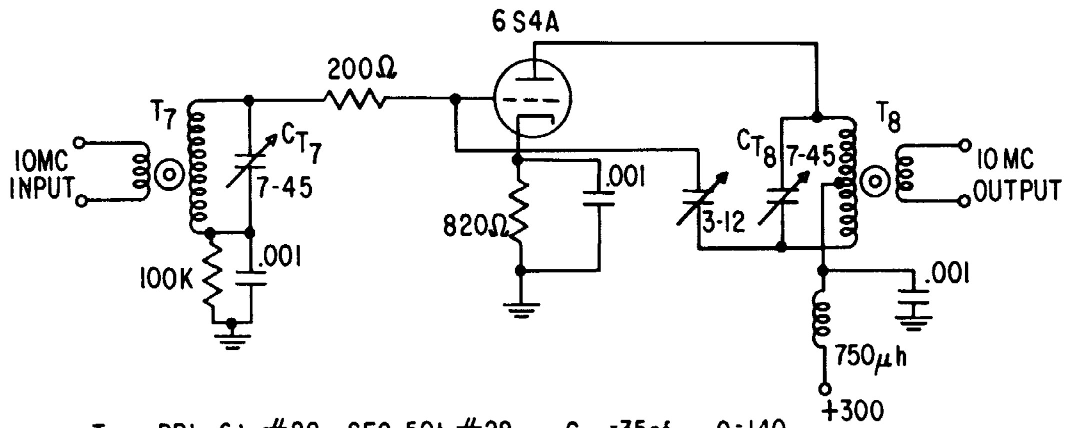


T_5 — PRI. 5t. #28, SEC. 50t. #28 C_{T_5} = 35pf Q = 140

T_6 — PRI. 50t. #28, SEC. 6t. #28 C_{T_6} = 35pf Q = 140

1ST 10MC AMPLIFIER (2 REQ'D.)

FIGURE 10

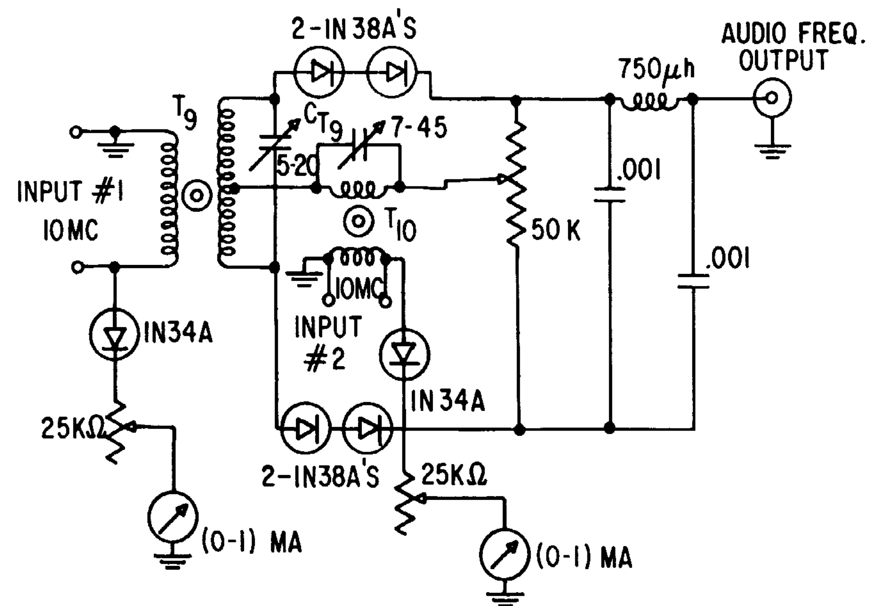


T_7 — PRI. 6t. #28, SEC. 50t. #28. C_{T_7} =35pf Q=140

T_8 —PRI. 50t. #28ct. SEC. 6t. #28. C_{T_8} =35pf Q=140

2ND 10MC AMPLIFIER (2 REQ'D)

FIGURE 11



T_9 — PRI. 11t. #28, SEC. 88t. #28ct. C_{T_9} = 18pf Q=100

T_{10} — PRI. 4t. #28, SEC. 50t. #28. $C_{T_{10}}$ = 35pf Q=140

10 MC - FREQUENCY MIXER (1 REQ'D.)

FIGURE 12

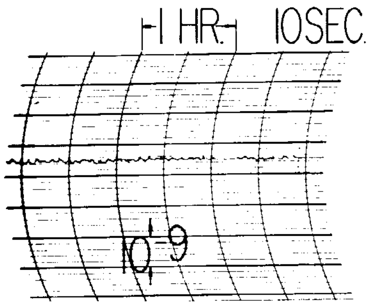


FIG. 14

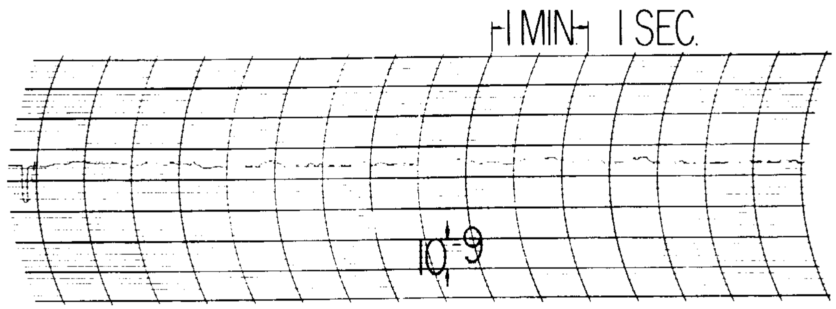


FIG. 15

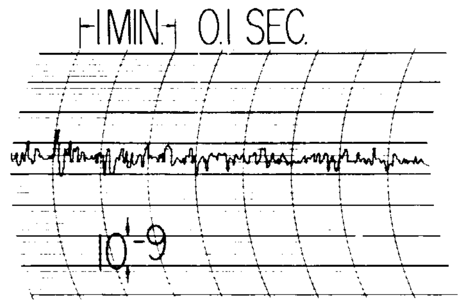


FIG. 16

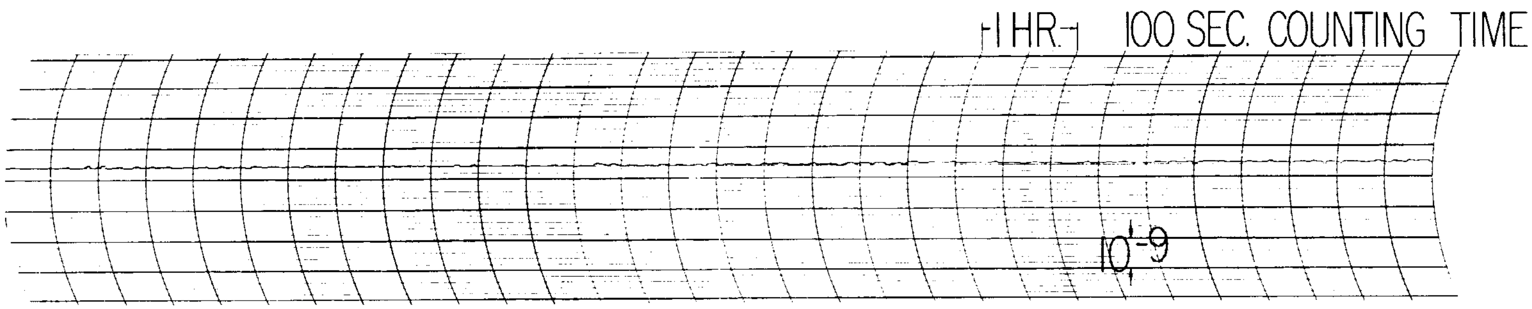
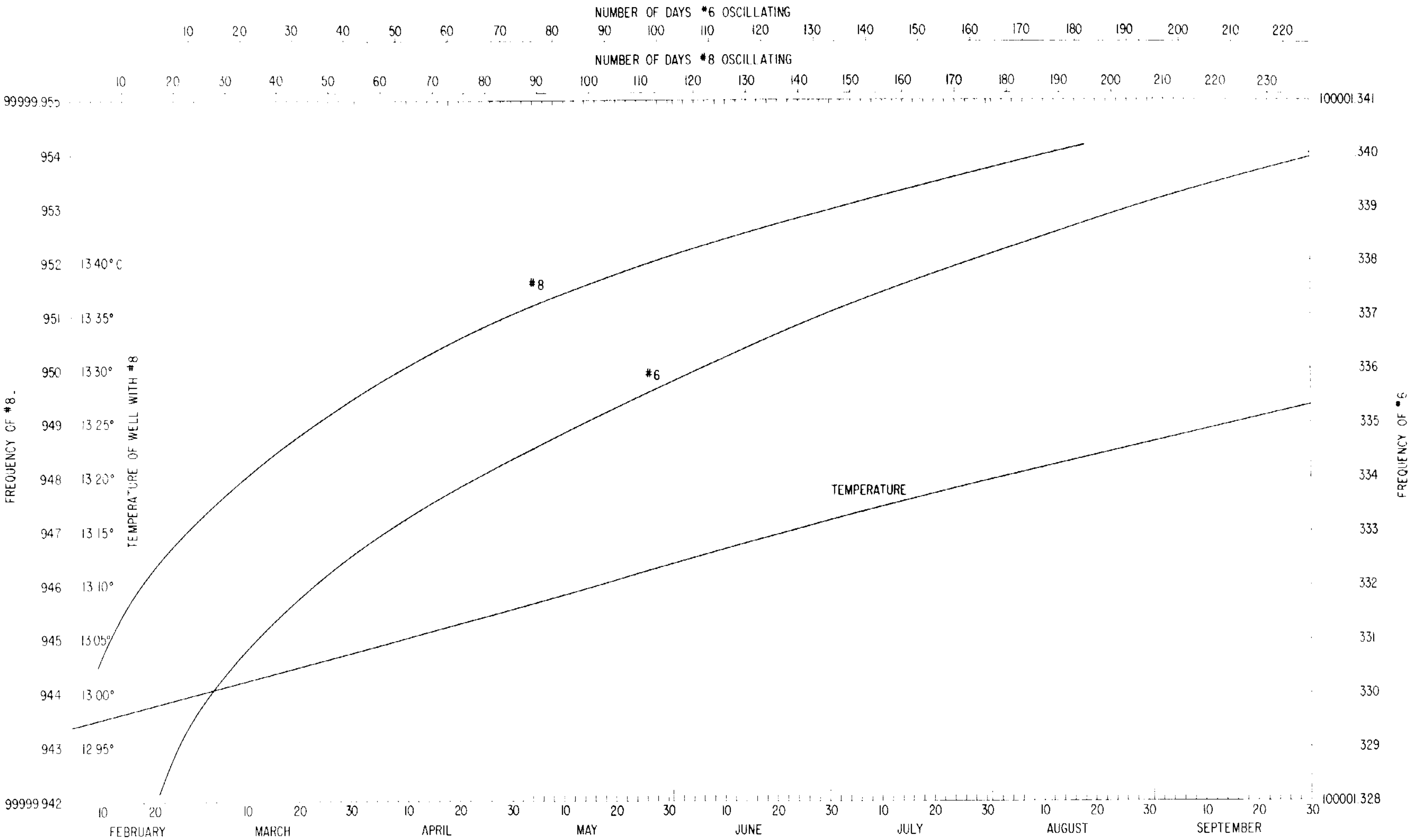


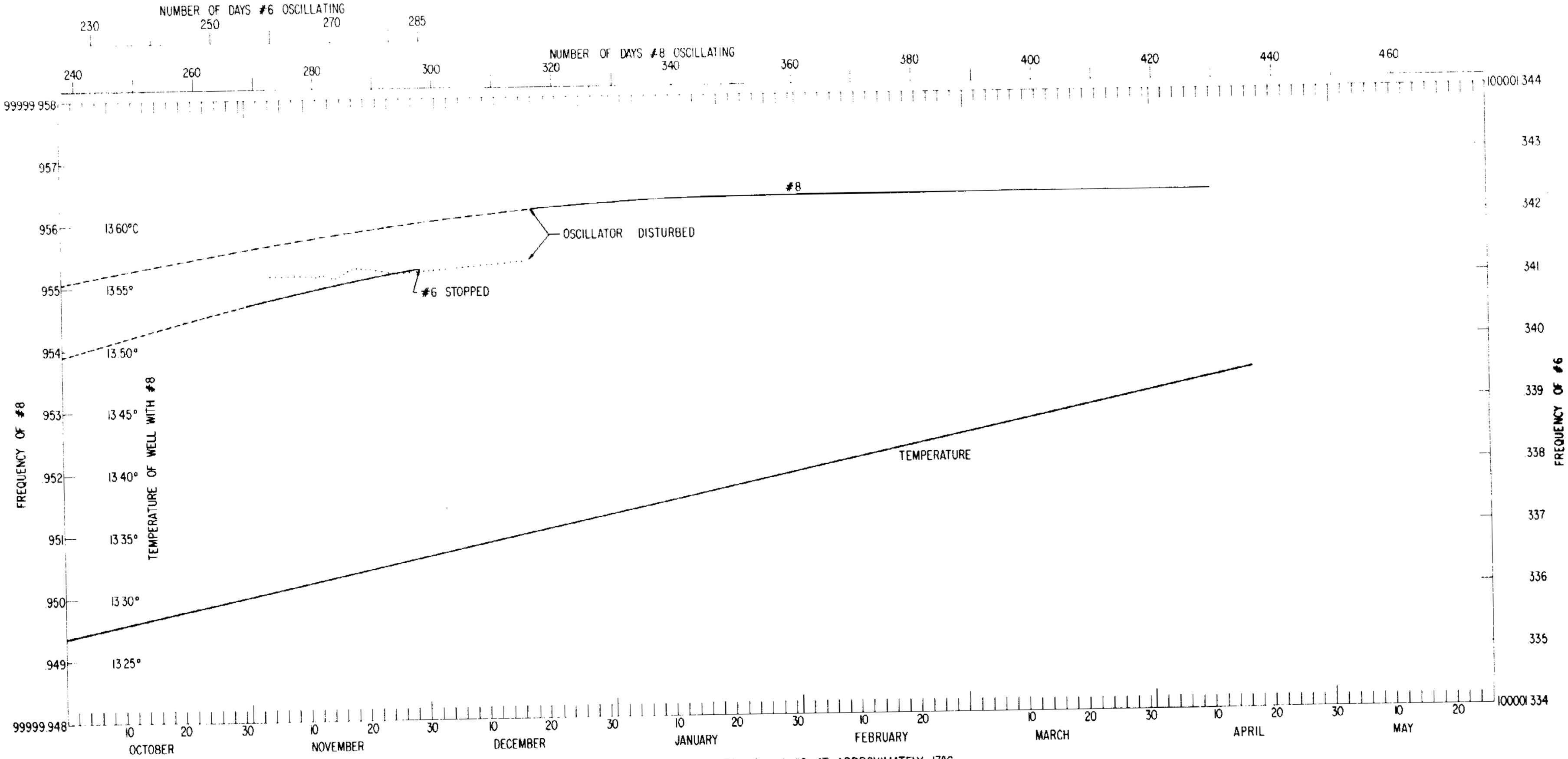
FIG. 13

NBS OSC. R-1 vs. ATOMIC FREQ. STD.



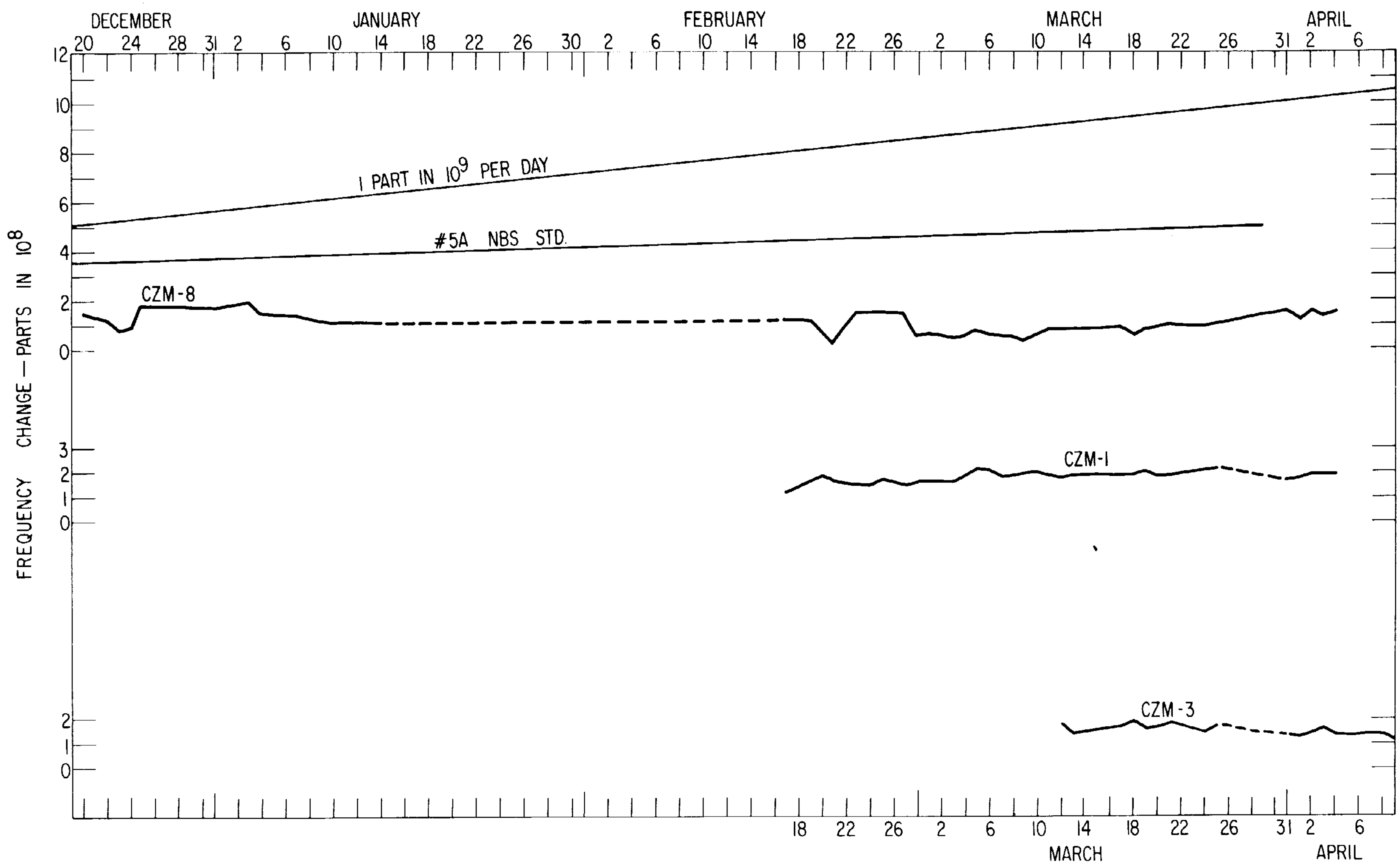
[redacted] FREQUENCY OF CRYSTAL UNITS #6 and #8 AT APPROXIMATELY 13°C

FIGURE 17 (SEE NEXT PAGE ALSO)



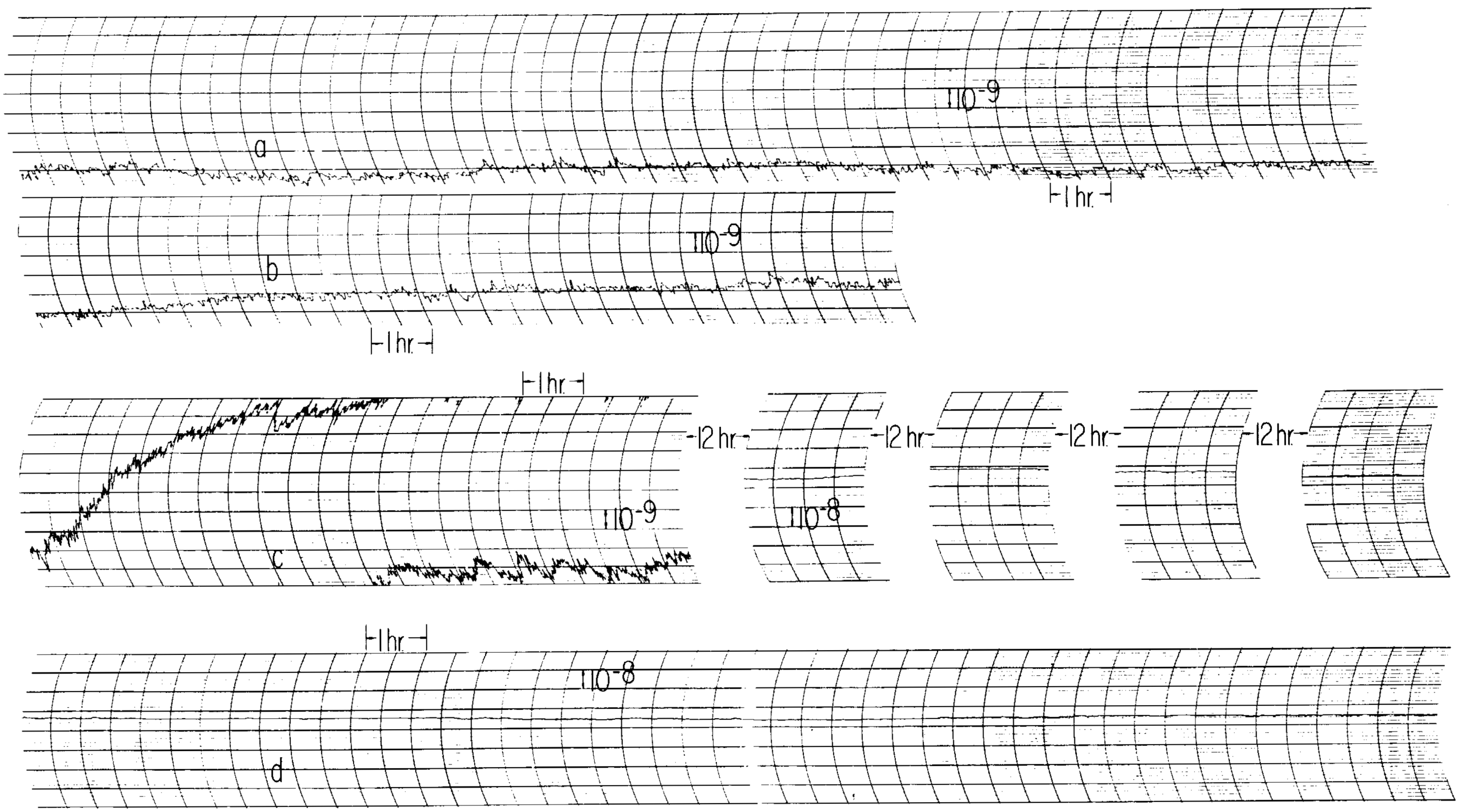
FREQUENCY OF CRYSTAL UNITS #6 and #8 AT APPROXIMATELY 13°C

FIGURE 17 (CONTINUED)



AGING OF CZM-1, 3, and 8

FIGURE 18



FREQ. OF CRYSTAL I-2 IN N_2 vs NBS STD. #R-I

FIGURE 19

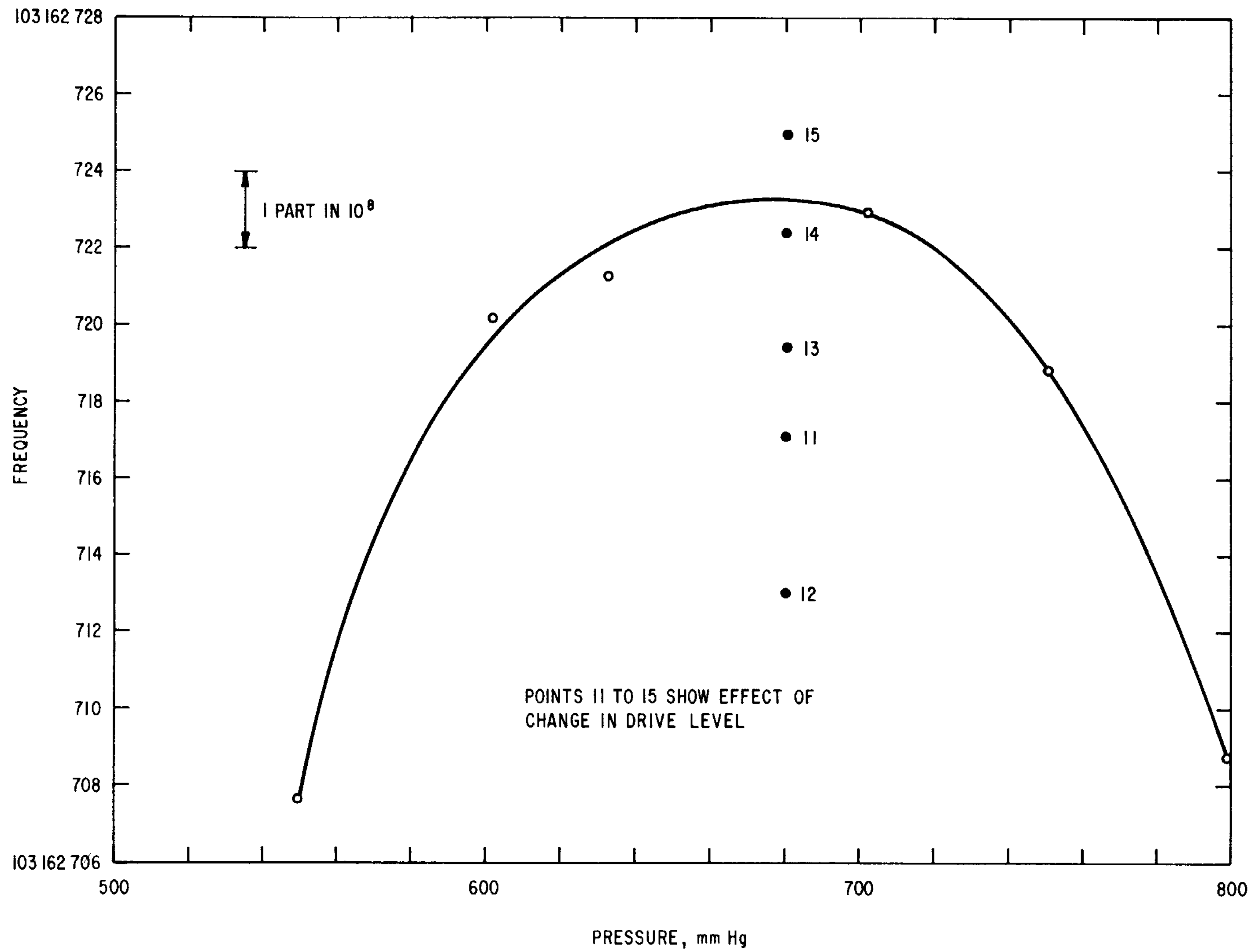
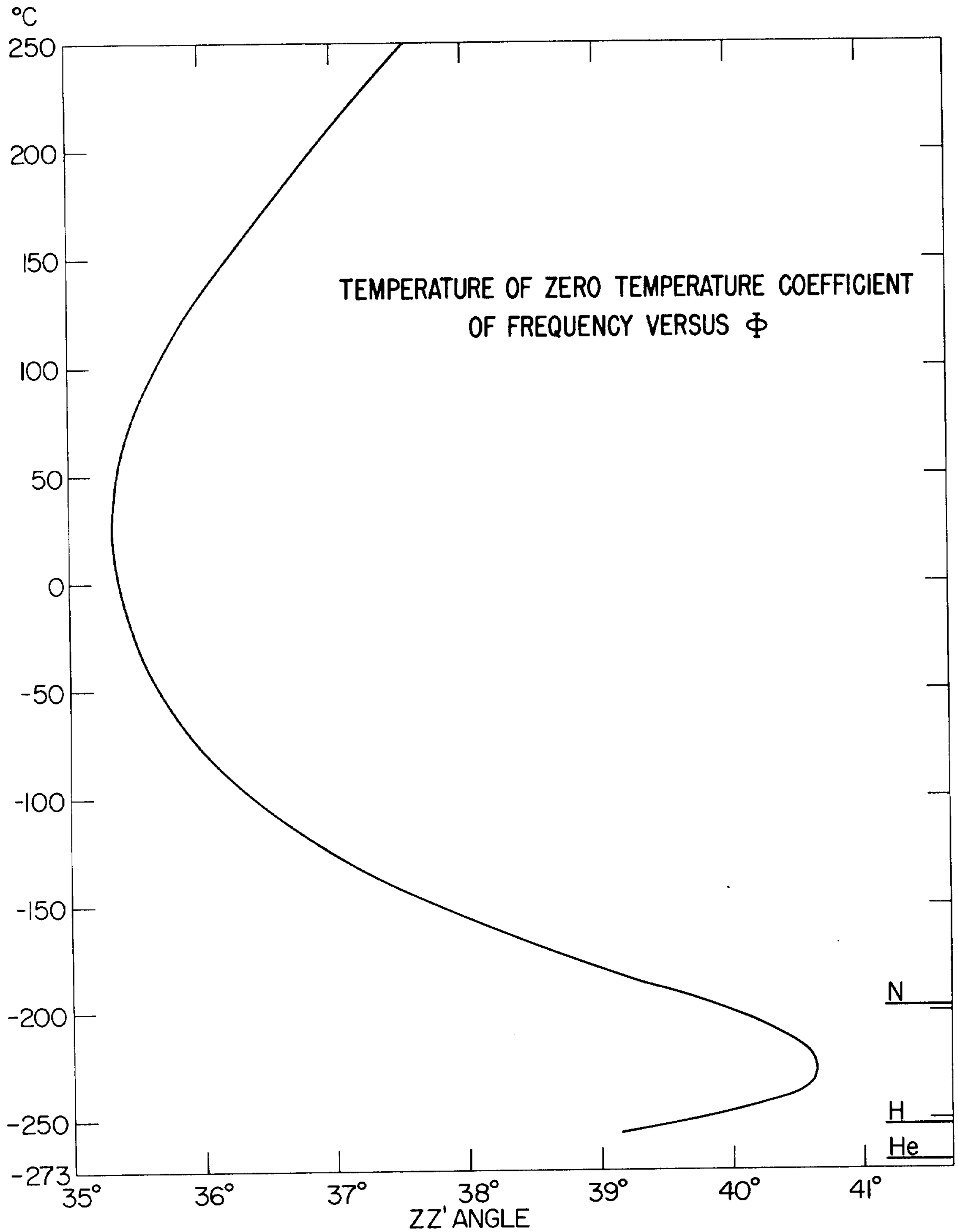
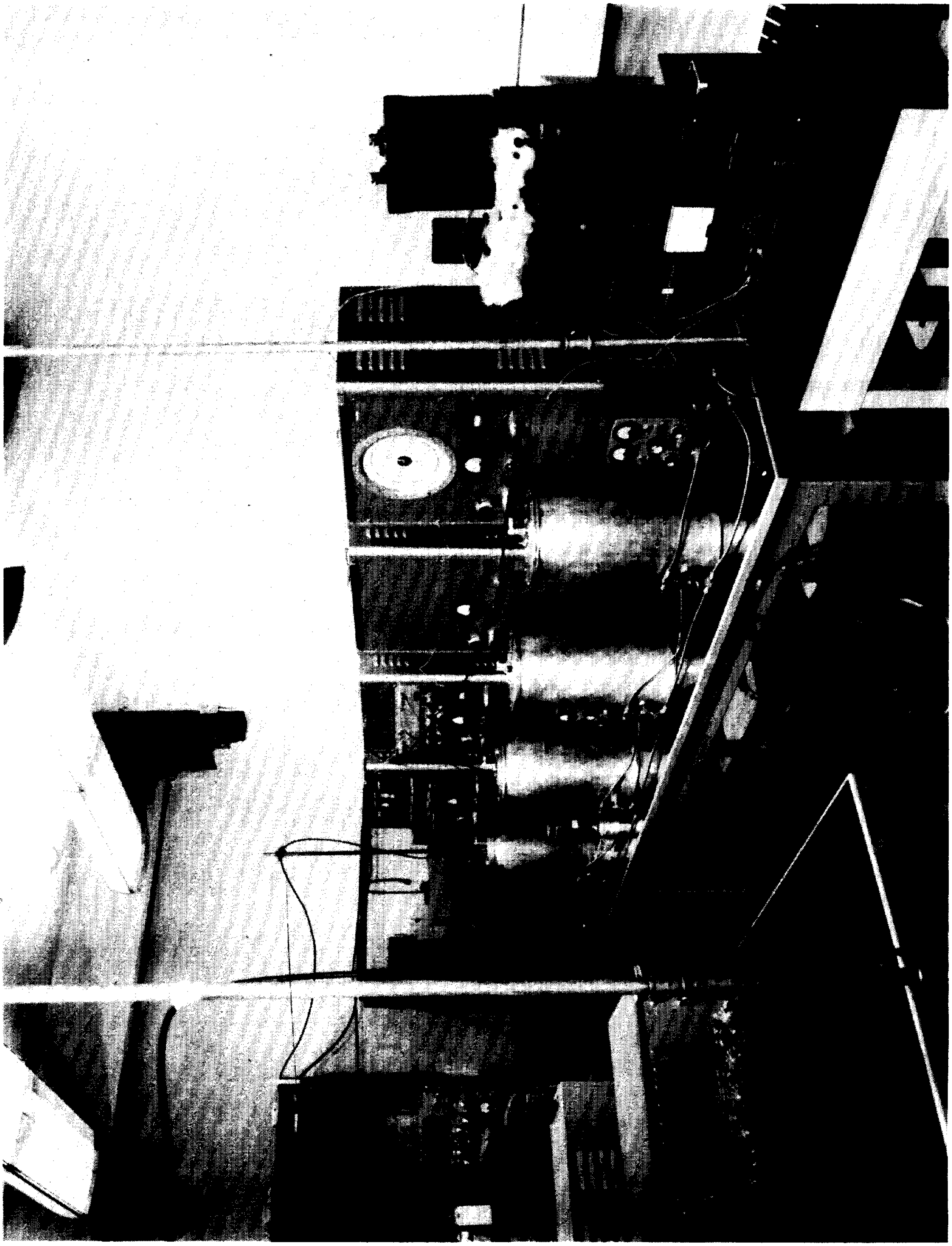


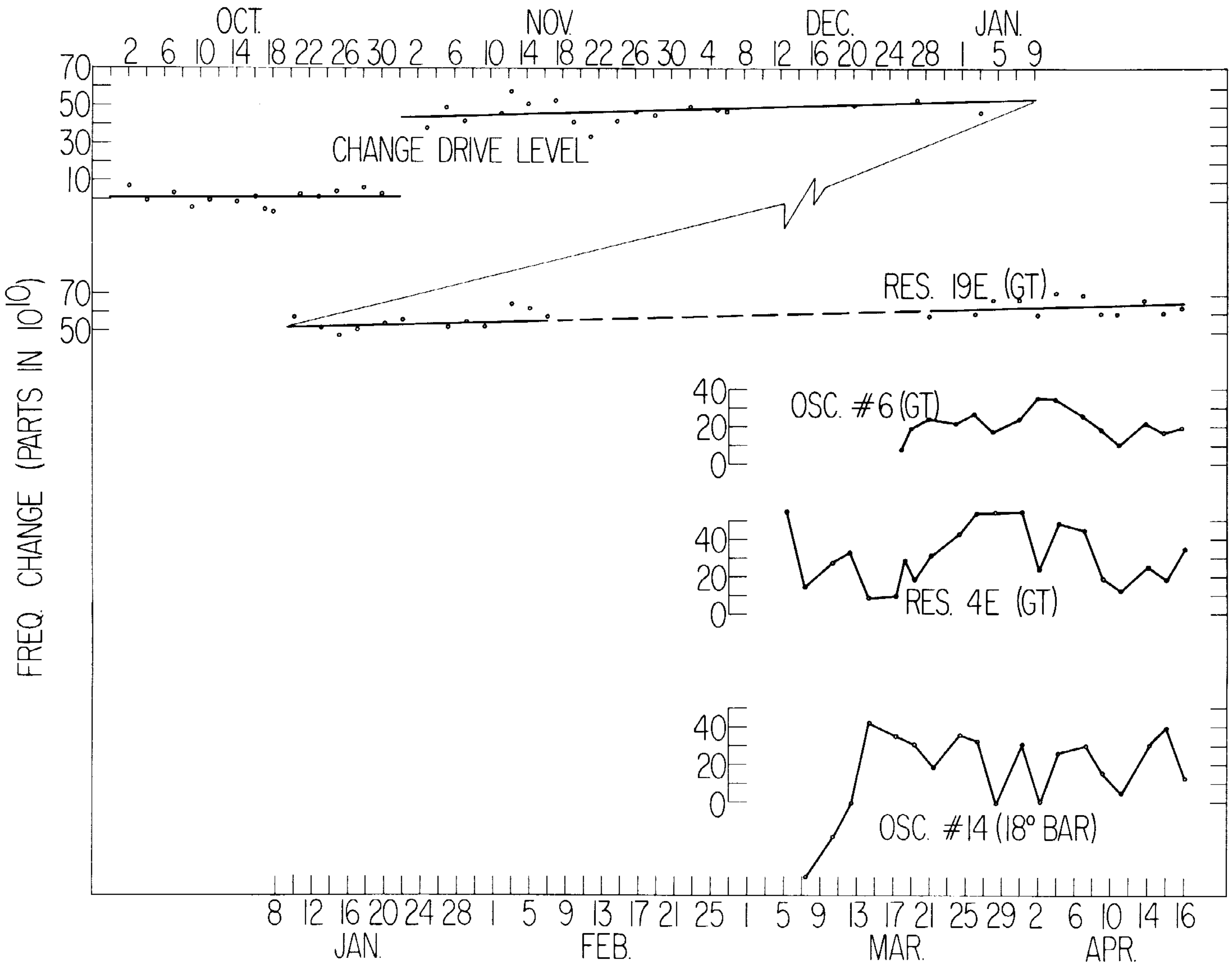
FIG. 19A FREQUENCY VS. PRESSURE, CZM - 2 IN LIQUID NITROGEN


°C
250
200
150
100
50
0
-50
-100
-150
-200
-250
-273
TEMPERATURE OF ZERO TEMPERATURE COEFFICIENT OF FREQUENCY VERSUS Φ
N
H
He
35°
36°
37°
38°
39°
40°
41°
ZZ' ANGLE

Slide 19B



Slice 19c



AGING OF 100KC CRYSTALS IN He
FIGURE 20