NATIONAL BUREAU OF STANDARDS BOULDER LABORATORIES

A Frequency Standard at Low Temperature

by

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ABSTRACT

Investigation is under way at NBS Boulder Laboratories on the aging of precision quartz crystal units at temperatures below about -100 °C. Measurements at 4 °K over a period of several weeks have indicated a substantial improvement in stability. The measuring apparatus and low-temperature containers are described. The change in turnover temperature versus angle of cut is reported for the AT type plate as the angle was increased from about 35°. At -190 °C the angle was found to be very nearly 40°.

Purpose of a Frequency Standard at Low Temperature

Quartz clocks are used as transfer standards in the determination of Universal Time and in the generation and measurement of frequency. It is desirable that such clocks operate with practically zero rate over a long period, e.g., years, in order to: (1) determine with greater precision the new second as approved by the International Astronomical Union in September 1955(1); (2) precisely determine the microwave resonance frequencies of atoms and molecules: cesium, sodium, ammonia, etc., in terms of the ephemeris second. Also, greater frequency stability is needed to accurately transfer the standard of frequency for scientific and engineering applications, fixed and mobile, short and long intervals, e.g., in radio propagation research and in radio navigation facilities.

Quartz oscillators and resonators at the NBS Boulder Laboratories are operated at relatively constant temperatures in the range from about 12 to 40 °C. Aging amounts to 1 to 3 parts in 10^9 per month. Work at NBS and elsewhere(2) has indicated that a substantial reduction in aging results when the quartz resonator is operated at very low temperatures.

The measured fractional frequency variations in a precision quartz oscillator relative to another of similar design are about 1 part in 10^{10} in a few minutes. By operating at low temperature one may obtain higher Q, lower noise, lower aging and lower temperature coefficient. With such improvements it appears possible to obtain stabilities of 1 part in 10^{10} per month or better.

In the development of measurement methods and standards, which keep pace with advancements in the field of frequency control, it appears desirable to explore the possibilities at low temperature.

Sulzer(3) has shown that noise in the resonator and the associated circuit of a bridge type oscillator causes a small frequency amd amplitude modulation in the output.

$$\frac{\Delta f}{f} = \frac{2}{Q}\sqrt{\frac{KT}{P_R\, t}}$$ where K = Boltzmann's constant.

In an example given, $Q = 10^6$, $T = 320$ °K, power dissipated in the resonator $P_R = 2.5$ microwatts, and time of measurement $t = 0.06$ sec., the frequency deviation $\frac{\Delta f}{f}$ resulting from thermal noise in the bridge is approximately ± 3 parts in 10^{13}. Under equal conditions except at liquid helium temperature an order of magnitude improvement in short time stability would be obtained. Actually at 4 °K the Q has a higher value than at 320 °K.

Probably 3 parts in 10^{13} is a fair estimate, even for 4 °K, because it is usually desirable to operate with lower power to retain constant resonance frequency vs crystal current.

Description of Measurements

Measurements were made, at about 4 °K, on several GT type quartz crystal units and on two + 5° units all at approximately 100 kc, see Fig. 1. The GT units were sufficiently close to 100 kc for use of a GR 916 AL bridge and a W.E. type 0-76/U drive oscillator. A special container with liquid nitrogen jacket was assembled to accommodate the relatively large size GT units in liquid helium. An on-temperature run of about 24 hours was possible. The + 5° units had an overall diameter of about 1/2 inch and were placed under liquid helium in a commercial 25 liter container. Efficiency of the container, built in 1953, was such that an undisturbed on-temperature run of several months could be obtained if desired. A W.E. 0-76/U oscillator was used as shown upside down in Figure 2. Very little modification was necessary; it was possible to add resistors in parallel with the oscillator bridge arm and to operate at different crystal currents to as low as 50 microamperes and to add capacitors in series with the crystal unit in measuring Q.

It is interesting to note that, with the equipment used, the liquid helium boil off averaged about 200 cc of liquid per day. Boil off was measured with a commercial gas flow meter; to minimize the boil off the crystal unit was connected with constantan wire.

Outputs from the test oscillator and from a standard oscillator were each multiplied to 100 Mc with equipment like that shown in Figure 3. The beat frequency at 100 Mc was counted 10 seconds to give a sensitivity of ± 1 part in 10^9.

Gold-cobalt alloy to copper thermocouples were waxed to the glass bulbs of the 100 kc units. Temperature during cooling or warm-up was measured on a recording potentiometer.

Measurement Results

Without exception the precision GT units were found to have a turnover temperature at about -150°C. This was reported on two units, Nos. 38 and 40 of Figure 4, at the Frequency Control Symposium in 1953. The units had Q values in the range from 2 to 10 x 10^6. Uncertainty in temperature measurement still exists in the range 4 to 20 °K because of insensitive thermocouples and a relatively large time lag between temperature of the couple and that of the crystal. When covered with liquid helium the GT unit would become stable after about 1 1/2 hours. No aging trend while on-temperature at 4 °K was observed.

The + 5° crystal unit, whose temperature versus frequency deviation is also shown on Figure 4, has about 10 times the frequency change from 4 °K to 273 °K as the GT unit. The value shown at about 20 °K was obtained when immersed in liquid hydrogen.

Figure 5 shows interesting results during warmup (commencing at 5:45 hours) of a GT unit from 4 °K. The small increase in Q at about 7 °K was reproduced; it is believed to result from the gold electrode film having lower resistance at the higher temperature. This is mentioned for copper by Swenson and Emslie(4) as possibly resulting from impurities in the metal. Another interesting result, not shown on the curve, which was not precisely accounted for was a constant temperature for several minutes at about 7 °K during warmup from 4 °K. This is believed to have resulted from lead in the crystal unit and the large decrease in the specific heat of lead upon becoming super-conducting at about 7 °K.

The + 5° units having width to length ratios of 0.1 and 0.3 were built by E. M. Shidler for an experimental determination of the frequency at room temperature which would result in 100 kc at 4 °K. Although they were not intended as precision units they were carefully measured because there was no problem in maintaining them in liquid helium. Frequency of the thick bar was very sensitive to crystal current and mechanical shock; nevertheless, no definite aging trend was detected as may be seen from Figure 6; the striking reduction in aging in going from 12.1 °C to 4 °K is also shown. The higher temperature was established after 24 hours in a 50 foot underground well. Results for the thin bar are unconventional and probably because of relatively high crystal current.

AT Crystal Units

As mentioned last year it was planned to operate a modified AT unit at a regulated temperature near that of liquid nitrogen. Relaxations in quartz reported by Bommel, Mason and Warner(5) led to abandonment of this plan for highest constancy transfer standards. However, it is important to obtain information for design of units having turnover temperatures at any low temperature. Figure 7 gives experimental results to about -190 °C while Figure 8 gives both NBS experimental data and that published by others for the relatively wide range -200 to + 250 °C.

AT units over the range 4 °K and upward are shown in Figure 9. For application in the 4 °K region it may be more practical to use conventional AT cuts because of the high temperature coefficients encountered in adjustment of the units at ordinary temperatures.

Future Plans

An outline drawing of a large-neck liquid helium container is shown in the last illustration, Figure 10. One container was delivered on May 5 at NBS Boulder Laboratories. It was designed along with its heat-exchanger plug to have the same boil off as the small-neck containers. After careful heat measurements and satisfactory operation is determined, the unit will be supplied with GT units and put into long term operation.

Other work in FY'57 applicable to quartz crystal units is a planned paper study of small size, e.g., 1 cubic inch work space, temperature-regulated ovens operating in the range below -100 °C. Such ovens should have an overall volume of less than 1 cubic foot and should preferably operate from electrical energy.

Development work on high speed frequency measurement apparatus having sensitivities of 1 part in 10^{12} or better has been commenced. The error multiplier method in use at the Naval Research Laboratory and once used at NBS Will be further investigated. This method has the advantage of confining all frequency multiplication to relatively low ratios and frequencies; for example, a standard 100 kc is multiplied to 900 kc and an unknown 100 kc is multiplied to 1000 kc; the difference frequency has 10 times the error.

References

(1) IXth General Assembly of the International Astronomical Union, Commission 31, August 29 to September 5, 1955.

(2) "Ageing in quartz crystal vibrators", British Post Office Eng. Dept. Nature 174, 41, July 3, 1954.

(3) "High stability bridge balancing oscillator, P. G. Sulzer, Proc. IRE, 43, No. 6, 701 - 707 (1955).

(4) "Low temperature electronics", C. A. Swenson and A. G. Emslie, Proc. IRE, 42, No. 2, 408 - 413 (1954).

(5) "Experimental, evidence of dislocations in crystal quartz", H. E. Bommel, W. P. Mason, and A. W. Warner, Jr., Phys. Rev. 99, 1894 (1955).

(6) "Frequency multipliers and converters for measurement and control", J. M. Shaull, Tele Tech, 14 No 4, 86-89 (1955).

List of Illustrations

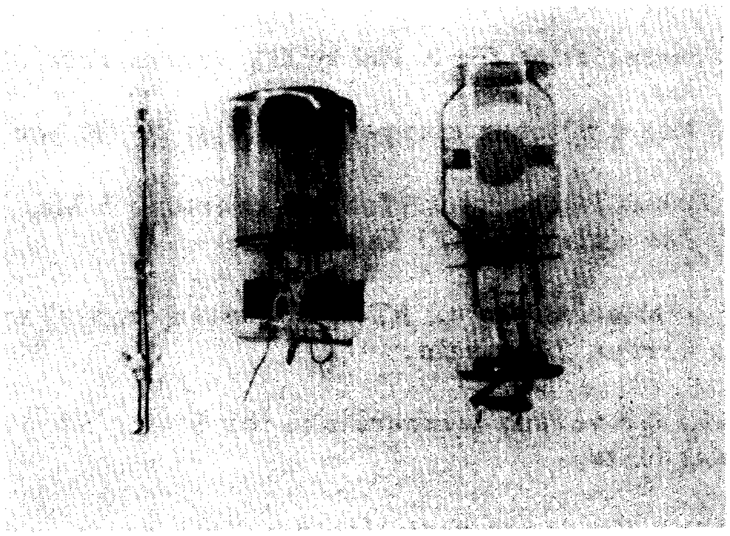


Fig. 1

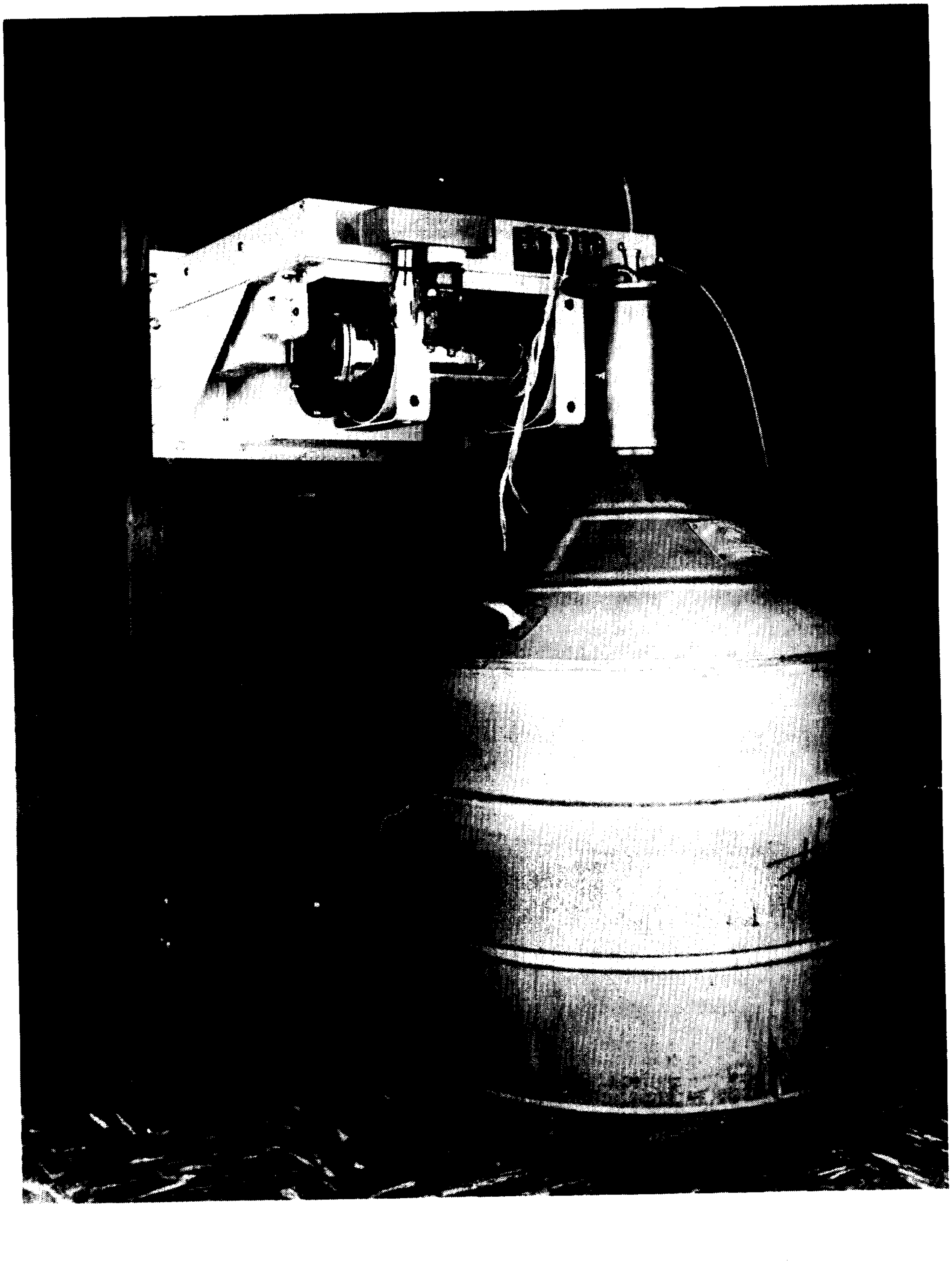


Fig. 2

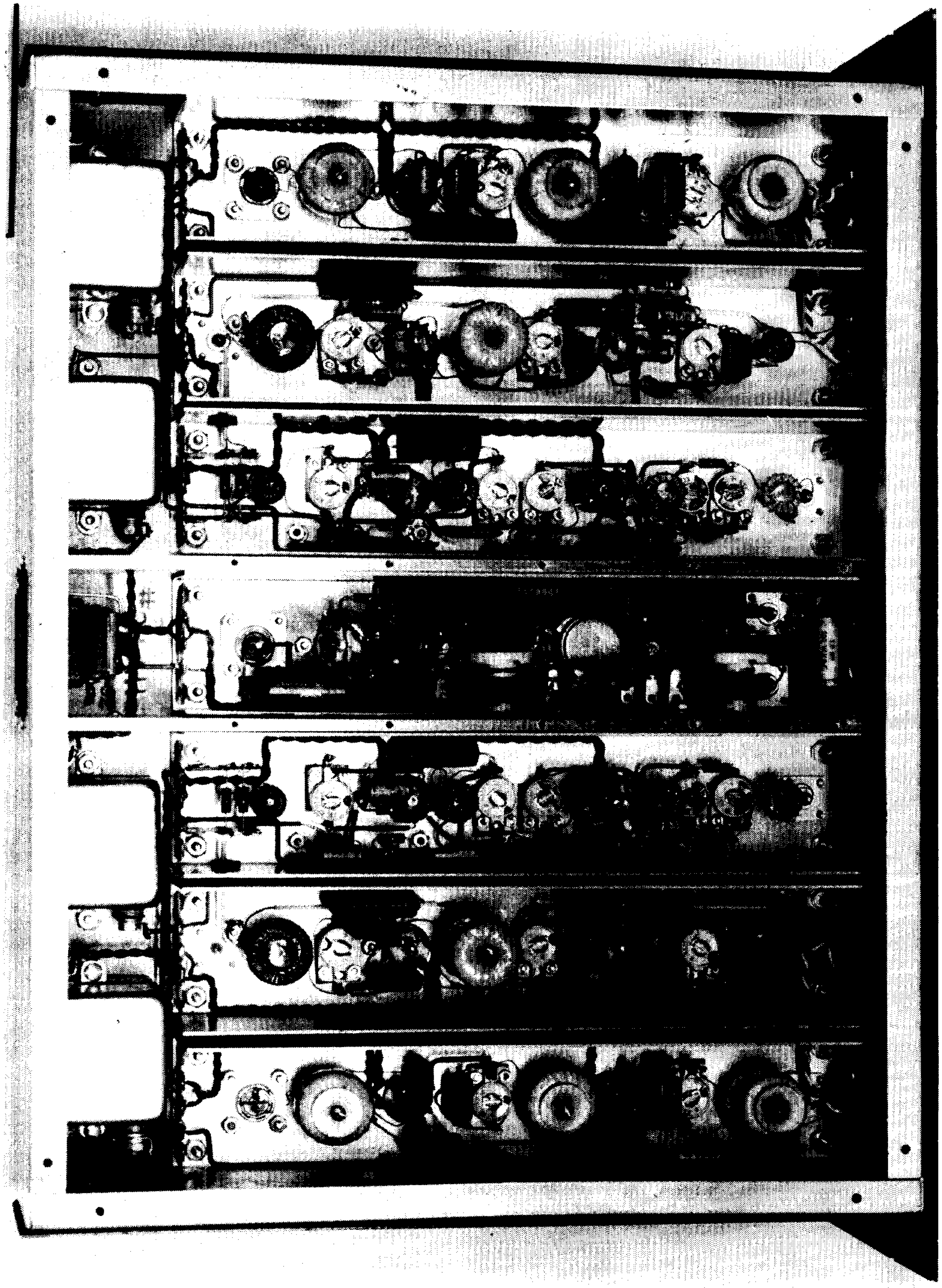


Figure 3

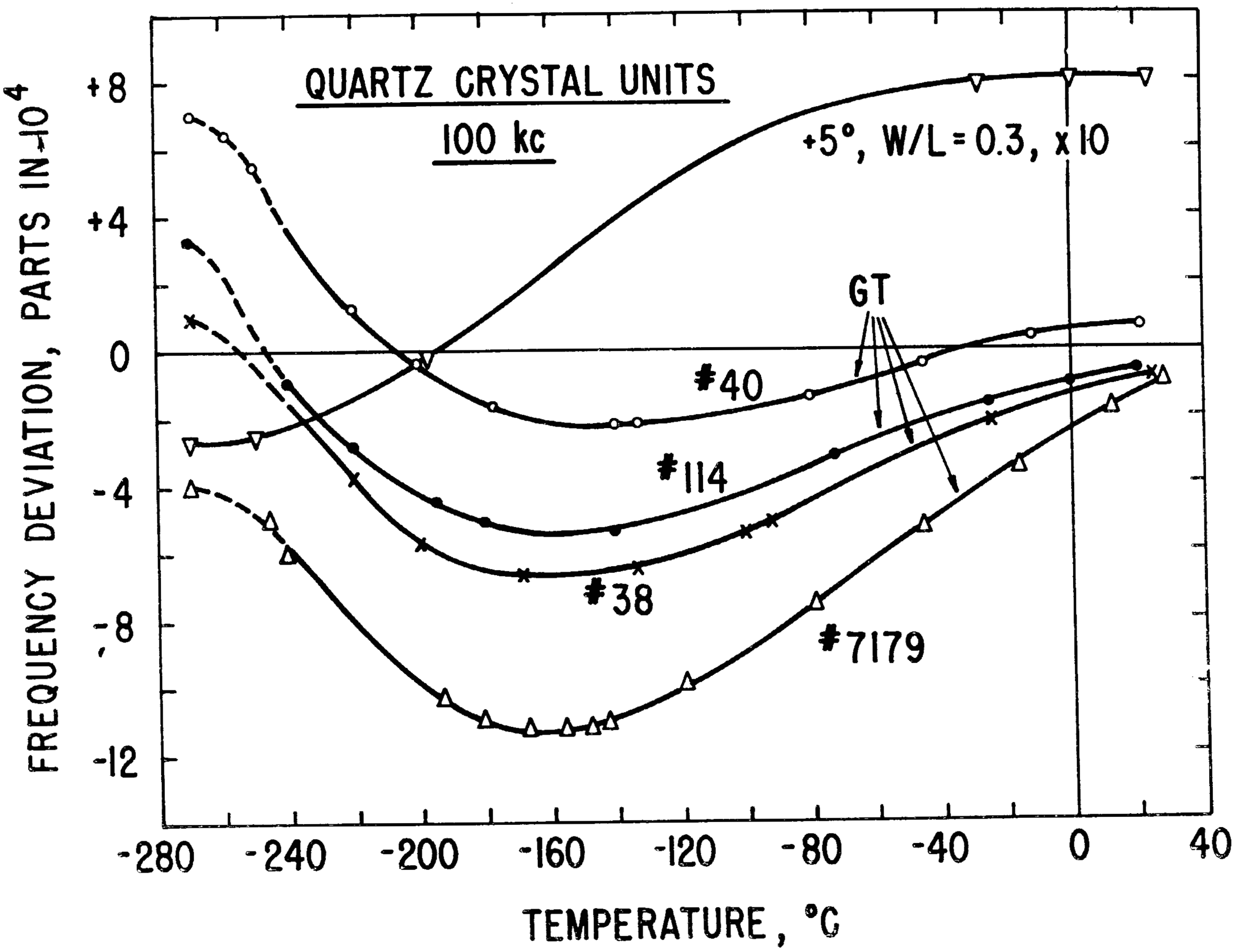

QUARTZ CRYSTAL UNITS
100 kc
+5°, W/L = 0.3, x 10
GT
#40
#114
#38
#7179
+8
+4
0
-4
-8
-12
FREQUENCY DEVIATION, PARTS IN 10^4
-280
-240
-200
-160
-120
-80
-40
0
40
TEMPERATURE, °C

Fig. 4

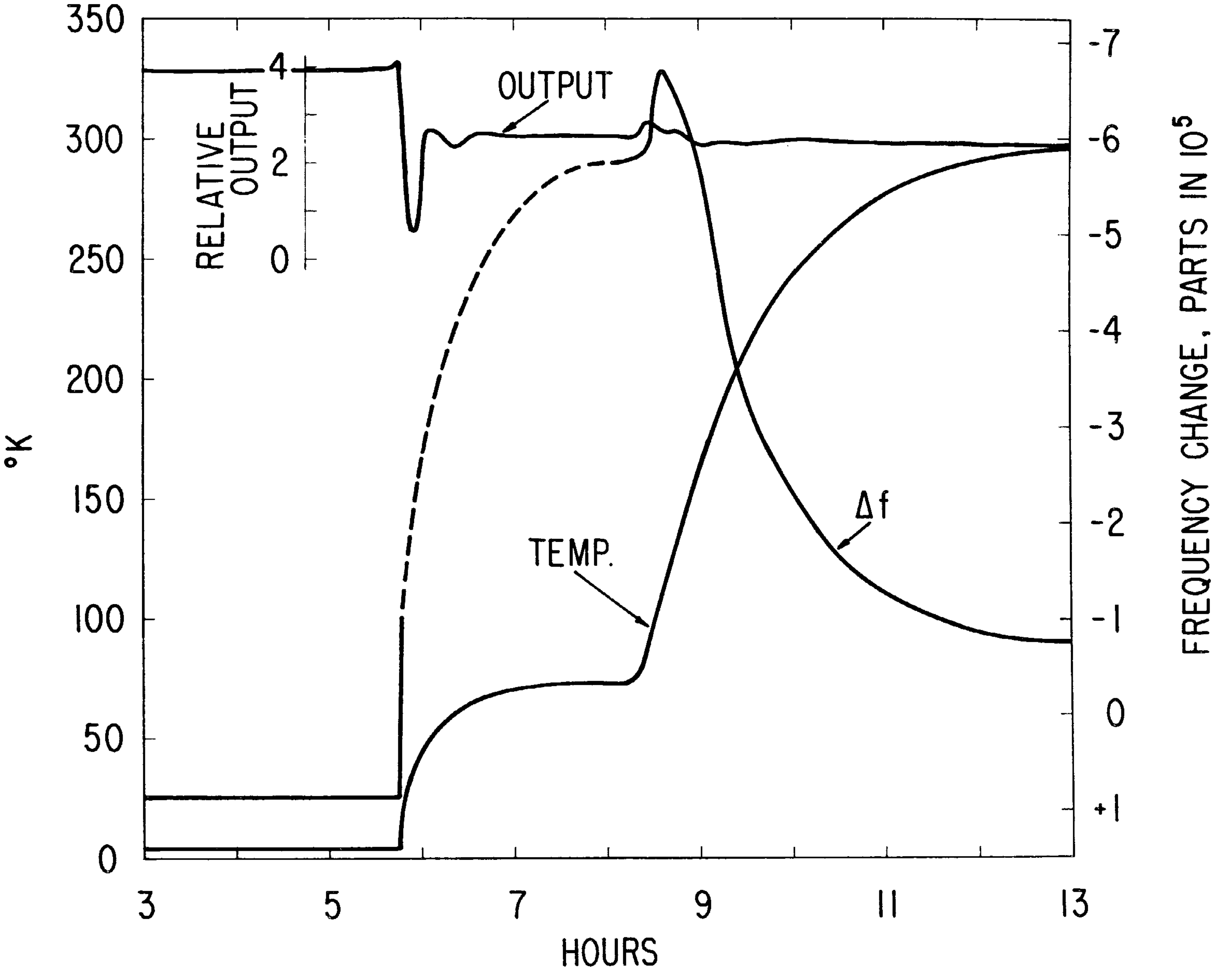

350
300
250
200
150
100
50
0
°K
RELATIVE OUTPUT
4
2
0
OUTPUT
TEMP.
Δf
3
5
7
9
11
13
HOURS
-7
-6
-5
-4
-3
-2
-1
0
+1
FREQUENCY CHANGE, PARTS IN 10^5

Fig. 5

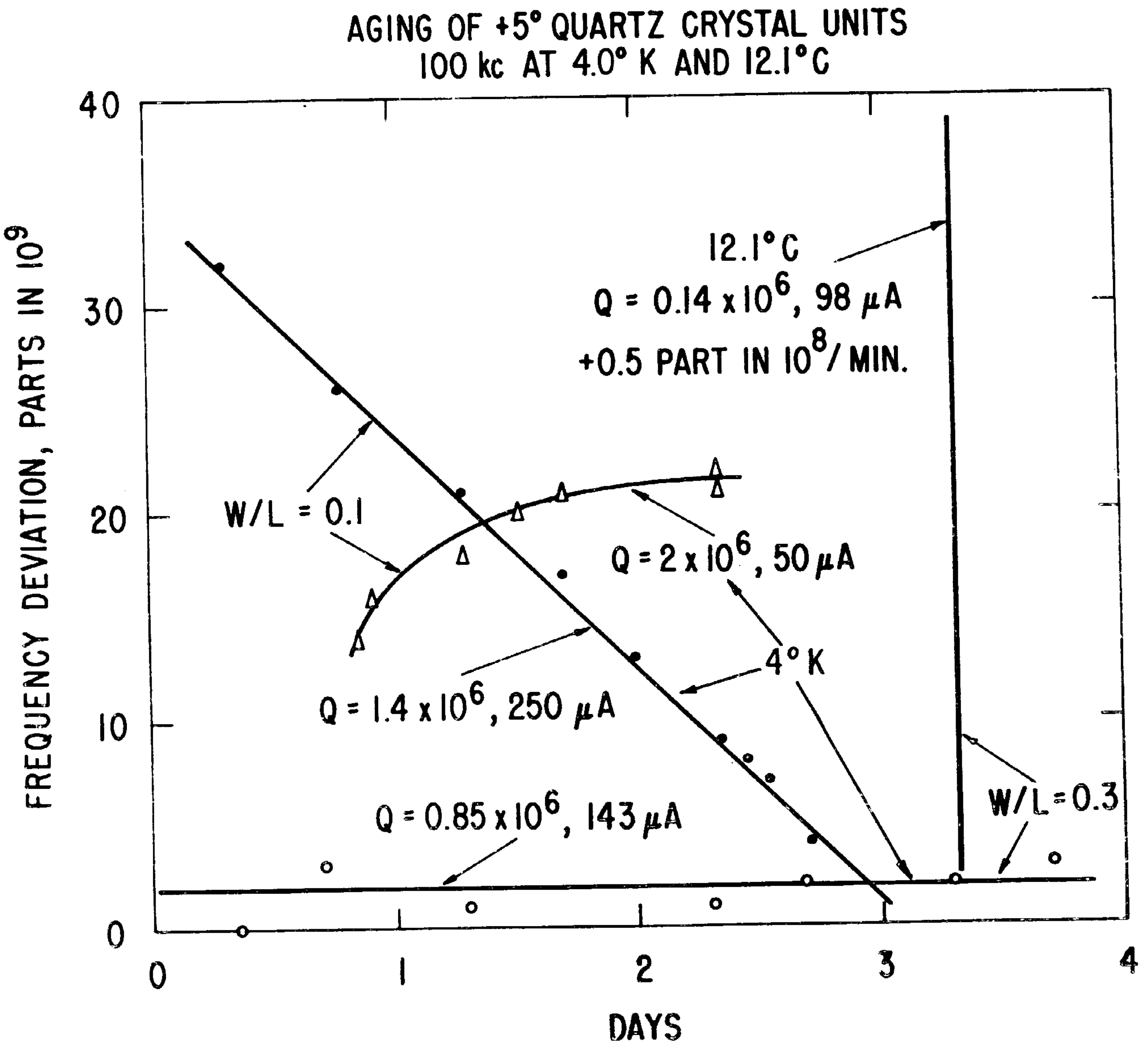
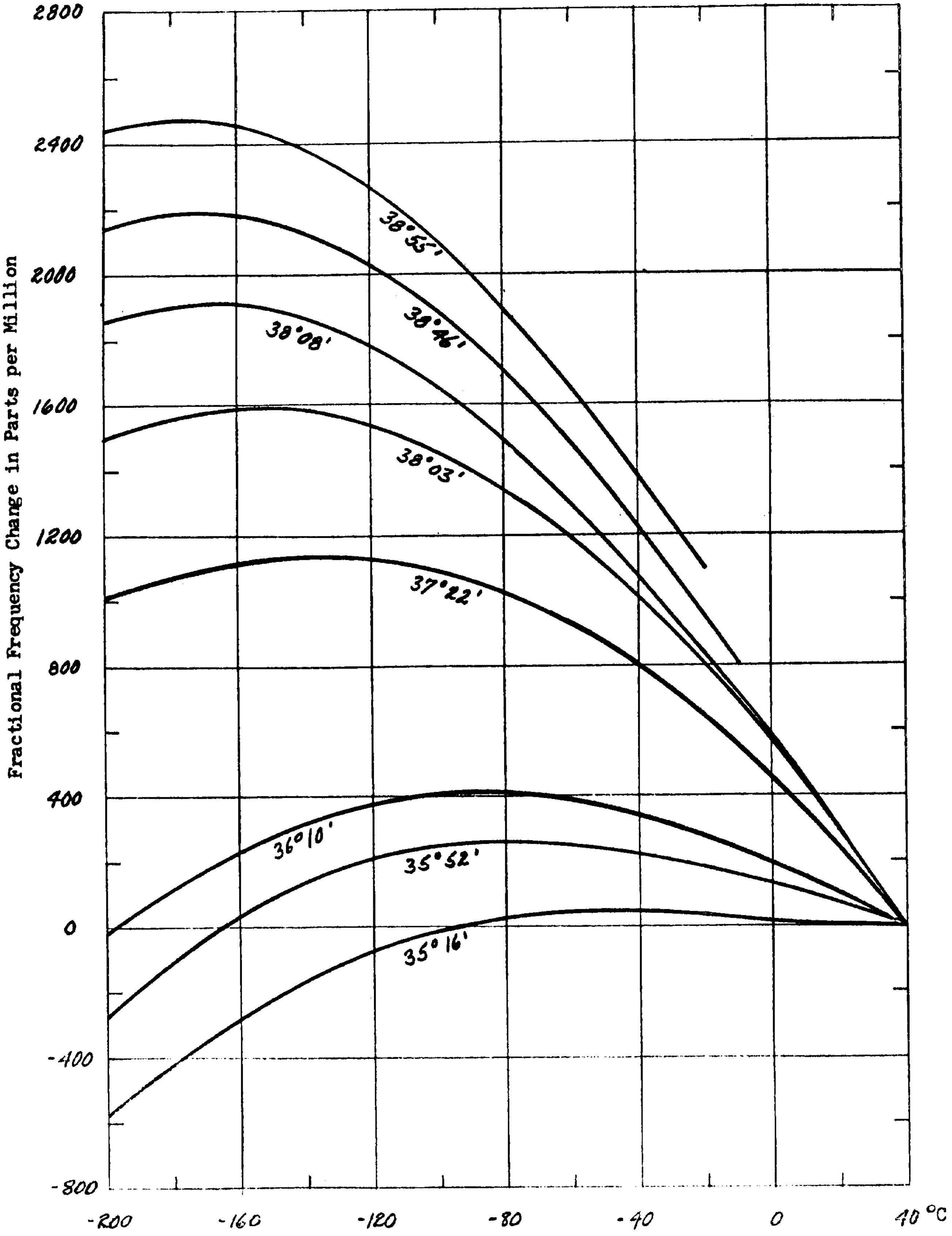

AGING OF +5° QUARTZ CRYSTAL UNITS
100 kc AT 4.0° K AND 12.1°C
FREQUENCY DEVIATION, PARTS IN 10^9
0
10
20
30
40
DAYS
0
1
2
3
4
12.1°C
Q = 0.14×10^6, 98 μA
+0.5 PART IN 10^8/ MIN.
W/L = 0.1
Q = 2×10^6, 50 μA
Q = 1.4×10^6, 250 μA
4° K
W/L = 0.3
Q = 0.85×10^6, 143 μA

Fig. 6



Fractional Frequency Change in Parts per Million
2800
2400
2000
1600
1200
800
400
0
-400
-800
-200
-160
-120
-80
-40
0
40 °C
38°55'
38°46'
38°08'
38°03'
37°22'
36°10'
35°52'
35°16'

Fig. 7

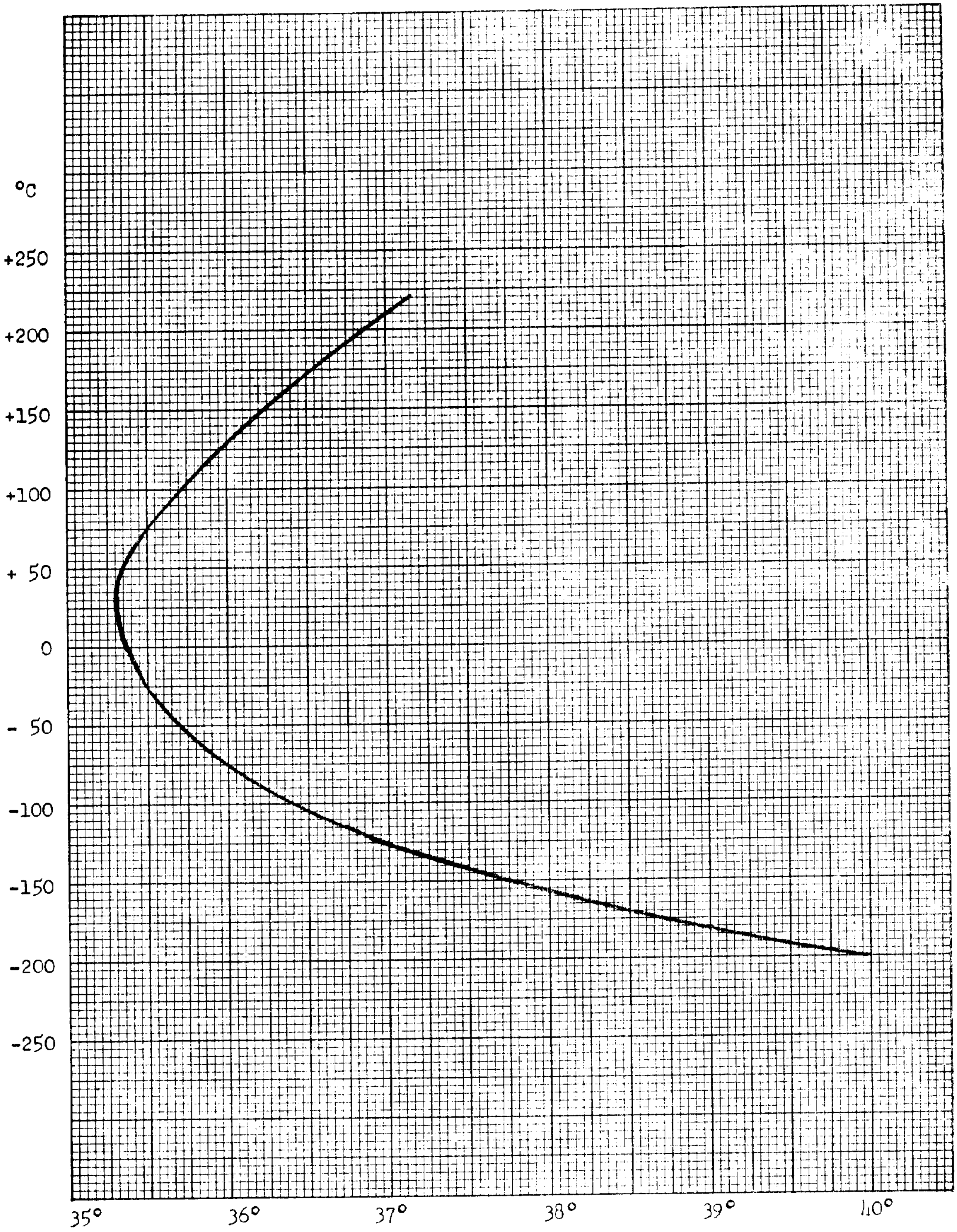


Fig. 8

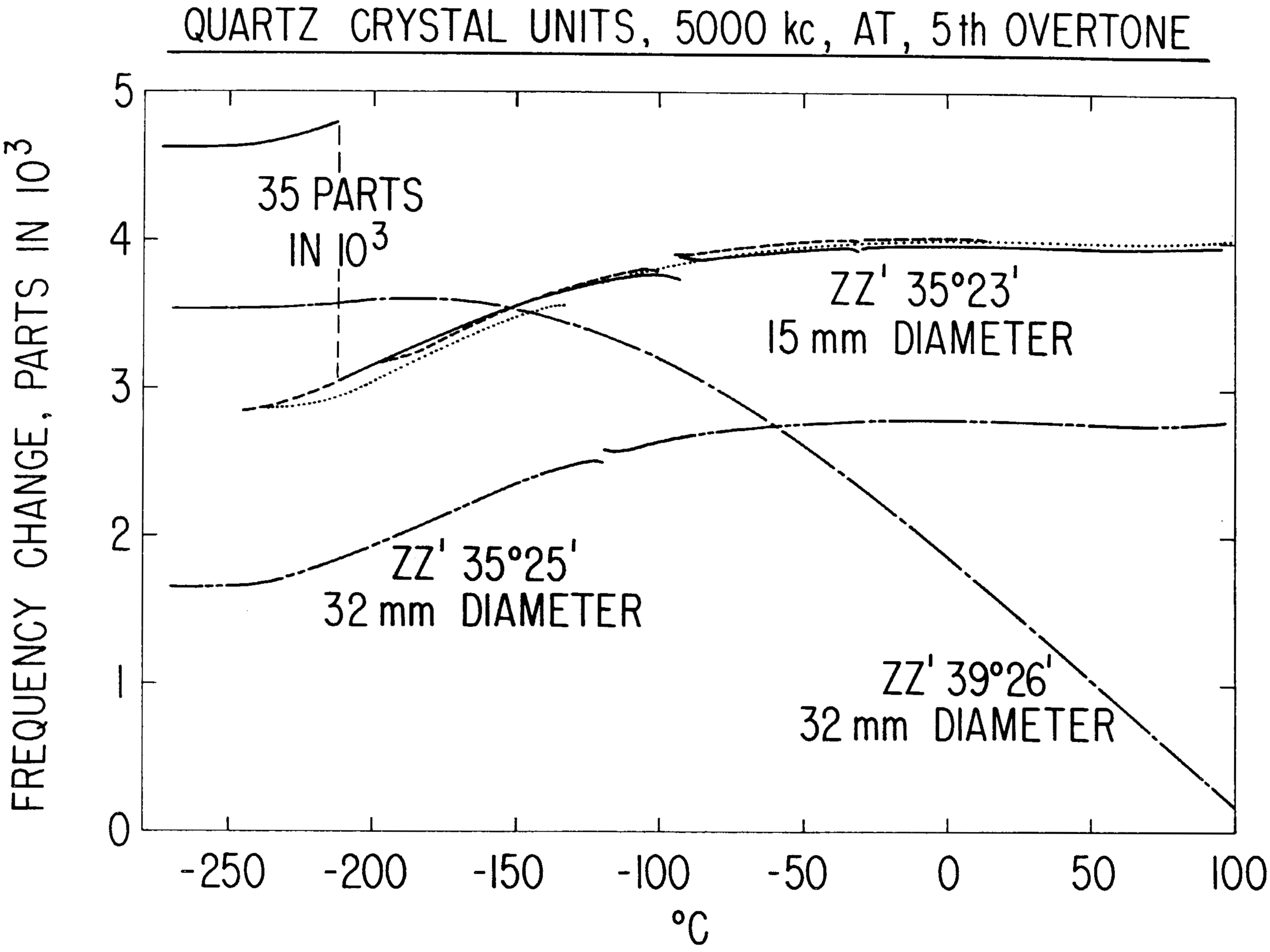

QUARTZ CRYSTAL UNITS, 5000 kc, AT, 5th OVERTONE
FREQUENCY CHANGE, PARTS IN 10^3
5
4
3
2
1
0
-250
-200
-150
-100
-50
0
50
100
°C
35 PARTS IN 10^3
ZZ' 35°23'
15 mm DIAMETER
ZZ' 35°25'
32 mm DIAMETER
ZZ' 39°26'
32 mm DIAMETER

Fig. 9

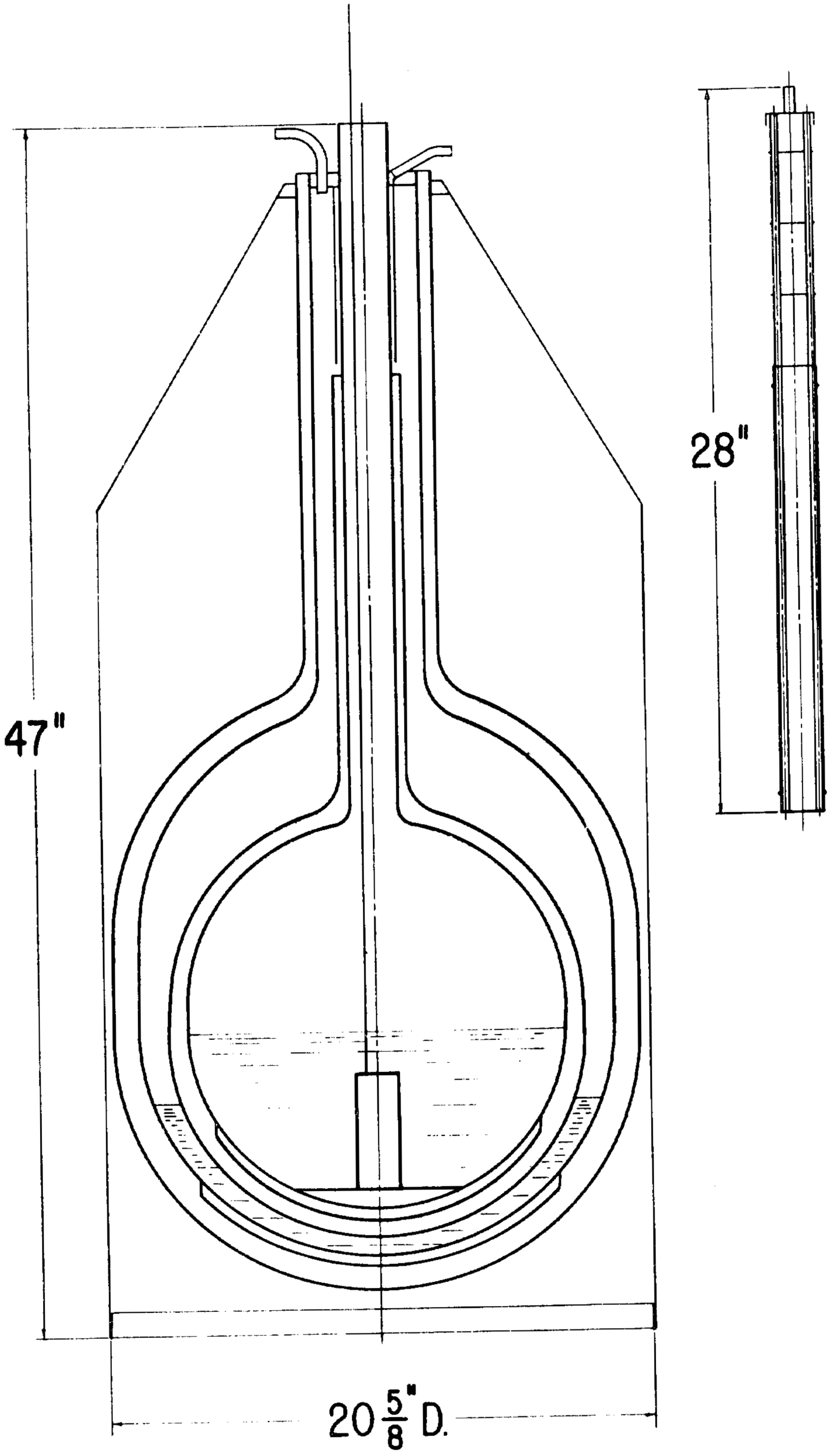


Fig. 10