# LOW AND HIGH DOSE PHOTON IRRADIATION OF QUARTZ

# CRYSTAL RESONATORS

**J. J. Suter and R. H. Maurer**
The Johns Hopkins University
Applied Physics Laboratory
Johns Hopkins Road
Laurel, MD 20707

## I. Introduction

The Johns Hopkins University Applied Physics Laboratory has been involved in studies concerning the radiation susceptibility of quartz crystal resonators for several years. In this paper we will report on results of a study of the sensitivity of AT cut quartz crystal resonators to low [4.0 Rads (Si)] and high [1000 Rads (Si)] accumulated doses which were generated by 1.25 MeV photons from two Cobalt 60 sources. In previous experiments, the same AT quartz crystal resonators were subjected to low level [4.0 Rads (Si)] photon irradiation and higher levels [12.0 Rads (Si] of proton radiation from the Harvard University Cyclotron (Ref. 1). It was concluded from those tests that the frequency susceptibility of AT quartz crystal resonators to proton radiation is approximately equal in magnitude to the frequency shifts induced in the resonator by photon irradation from Cobalt 60 sources. These experiments also showed, as was previously reported, that the frequency susceptibility of AT quartz crystal resonators to low level radiation [4.0 Rads (Si)] can <u>not</u> be extrapolated from high level [≥ 10 kRads (Si)] radiation tests (Refs. 2, 3, 4 and 5). This paper will discuss the results of radiation tests performed on AT quartz crystal resonators in which their frequency susceptibility as a function of accumulated photon radiation over an interval from 0 to 1000 Rads (Si) was measured. It is shown that the frequency shift ($\Delta f/f$) per rad is a function of total accumulated dose and that this slope ($\Delta$ f/f/ per rad) is the largest for low [4.0 Rads (Si)] accumulated doses. Furthermore, the paper will report on the recovery of AT quartz crystal resonators after radiation exposure and the magnitude of the oscillator's Allan Variances before, during and after the radiation events.

## II. Details Of Experiments

The test bed oscillators used for these radiation tests were all equipped with 5.0 MHz AT resonnators. As shown in Table 1, each of the oscillator test beds was first characterized by measuring its aging and 10 second Allen variance (refer to Figure 1). The RF output from the test bed oscillators was applied to one of two input ports of a frequency mixer which was part of a so-called $\Delta f/f$ data acquisition system (refer to Figure 2). The fractional frequency shift of the test bed oscillators before, during and after the radiation event was measured by beating their RF output versus the RF signal from a 5.0 MHz reference oscillator.

As shown in Figure 2 the beat frequency of 400 Hz between the reference and test bed oscillator supplied the input to an HP 5335 counter. An HP85 computer system, together with the required software, was used to make continuous measurements of the fractional frequency shift ($\Delta f/f$) of the test bed oscillator. The software, developed by APL, also provided an option of selecting sampling times from 1 to 100 seconds. Typically, data were collected in 10 second intervals before, after and in 1 second intervals during the event. The data were permanently stored on disks for later processing. Fractional frequency shift ($\Delta f/f$) data were also monitored by a strip chart recorder so that a continuous analog signal of crystal oscillator stability as a function of real clock time was available.

The three 5 MHz AT cut resonators (serial numbers 33820, 34632, and 34744) were made by Bliley Electric Company from Sawyer swept synthetic quartz bars. Samples 33820 and 34744 were from Sawyer bar number K13F. Quartz from this bar was analyzed as having a 0.79 ppm (parts per million) aluminum impurity content by Halliburton at Oklahoma State University (Ref. b). Resonator 34632 was fabricated from Sawyer bar K11K, which was analyzed as having an aluminum impurity content of 8.3 ppm. Each of the three AT cut quartz crystal resonators was installed and evaluated using APL test bed oscillators (refer to Table 1).

## III. Low Dose Gamma Ray Tests

Table 2 and Figures 3 - 5 show the results of the low dose [4 RADS (Si)] and low dose rate [0.068 RADS (Si) per minute = 4.08 RADS (Si) per hour] experiments carried out at APL during June 1985.

The results for AT cut resonators 33820 and 34632 are basically straight forward. Both resonators had pre-irradiation test drift rates that were considerably less than $5 \times 10^{-10}$ per day. Ten second Allan variances were less than $6 \times 10^{-12}$. The specific values are shown in Table 1. Each resonator had been preconditioned with a $Co^{60}$ gamma ray dose of 20,000 RADS (Si) and was tested as shown in Figure 6. Resonators 33820 and 34632 both show monotonically increasing positive shifts in frequency under irradiation. After the end of the radiation exposure, only modest recoveries or adjustments of $\Delta f/f$ are observed before a drift rate comparable to the pre-irradiation drift rate was resumed. The residual positive offsets for $\Delta f/f$ were $1.97 \times 10^{-10}$ or $4.92 \times 10^{-11}$ per RAD for SN33820 and $1.29 \times 10^{-10}$ or $3.22 \times 10^{-11}$ per RAD for SN34632. It should be noted that Figure 4 showing the data for SN34632 has an expanded time scale when compared with Figure 3 for SN33820.

AT cut resonator 34744 had a distinctly different response to the 4 RADS (Si) gamma ray irradiation (see Figure 5). A rapid positive excursion of $2.43 \times 10^{-10}$ was followed by a negative excursion of $-4.53 \times 10^{-10}$ at which point the irradiation ceased. A slight positive recovery left the residual offset of $\Delta f/f$ equal to $-1.78 \times 10^{-10}$ or $-4.45 \times 10^{-11}$ per RAD when referenced to the $\Delta f/f$ point at which the irradiation began. The unusual response of this resonator is probably due to competing physical mechanisms some of which are surface effects.

For these three AT cut resonators the dose sensitivity is several parts in $10^{11}$ for doses up to 4 Rads (Si).

## IV. High Dose Gamma Ray Tests

The test configuration shown in Figure 6 was used for a long term, low dose rate experiment during November 1985 in order to examine the response of an AT cut resonator when a total dose of about 1000 Rads (Si) was allowed to accumulate. By November the Cobalt 60 pill's source strength had decayed to 0.065 Rads (Si) per minute or 3.9 RADs (Si) per hour. The experiment duration was 259 hours; thus, 1010 Rads (Si) total dose was received by AT resonator 33820. Table 3 shows that the response of $\Delta f/f$ per Rad for 33820 was $4.92 \times 10^{-11}$ for the June 1985 4 Rads (Si) test. Figures 7 or 8 show that the response of $\Delta f/f$ per Rad for the same resonator was $4.75 \times 10^{-11}$ for the first 4 Rads (Si) of the November 1985 experiment. Thus, at the beginning of the experiment, SN 33820 repeated its response to the low dose rate $Co^{60}$ radiation within 4%.

The interesting results of this experiment are shown by the $\Delta f/f$ versus Rads (Si) plots of Figures 7, 8 and 9 as well as the best least squares fit slopes in Table 3. The response of 33820 to the gamma rays saturates as the dose accumulates. In other words, this AT cut resonator becomes less sensitive to $Co^{60}$ radiation as the dose increases.

Figure 7 shows the $\Delta f/f$ profile for the complete experiment [from 0 to 1000 Rads (Si)], Figure 8 shows the response for the first 130 Rads (Si); Figure 9 shows $\Delta f/f$ for the last 498 Rads (Si) - approximately the last half of the exposure. Table 3 shows the least squares fit for the slope, $\Delta f/f$ per rad, for the first 4 Rads (Si) (Figures 3 or 8), the first 40 Rads (Si) (Figure 8) and the last 498 Rads (Si) (Figure 9). We observe that from the first 4 Rads (Si) to the last 498 Rads (Si), the slope, $\Delta f/f$ per Rad, decreased from $4.92 \times 10^{-11}$ to $5.72 \times 10^{-13}$, a factor of 86. The net change for the total of 1010 RADs (Si) was $3.46 \times 10^{-12}$ per Rad (Si) or $3.49 \times 10^{-9}$ achieved at different response rates (Figure 7).

Following the completion of the radiation exposure we allowed resonator 33820 to recover as shown in Figure 10. After a negative $\Delta f/f$ recovery of several parts in $10^{11}$ the resonator resumed a positive aging rate within about 4.5 hours. A least squares fit of this aging rate yielded $2.32 \times 10^{-11}$ per day (November 1985). Prior to our 4 Rads (Si) radiation exposure in June 1985 (Figure 3) we had measured the aging rate of this resonator to be $2.17 \times 10^{-11}$ per day (Table 1).

It is also useful to compare the drift rates and Allan Variances during the long term, low dose rate radiation test with the post irradiation recovery rate (refer to Table 4).

We observe that at the end of the long term, low dose rate test, the drift rate was slightly greater than twice the post irradiation aging rate. The 10 and 100 second Allan Variances during irradiation were only about 13% and 6% greater respectively than the post irradiaton values for the same parameters.

## V. Conclusions

We have presented the results from the first of a series of experiments showing the saturation of the $\Delta f/f$ response of an AT cut quartz resonator made from swept synthetic quartz. The drift rates and short term stabilities (Allan Variances) between 500 and 1000 RADs (Si) total doses are not severely degraded from the same parameters measured without any radiation stimulus after the completion of exposure.

If confirmed in future experiments, these results have significant implications for the evaluation and qualification of quartz crystal resonators. More importantly, we may be gaining further insight into the piezoelectric and materials science properties of these resonators and their response to radiation.

## VI. References


1. J. R. Norton, J. M. Cloeren, and J. J. Suter, "Results from Gamma Ray and Proton Beam Radiation Testing of Quartz Resonators." IEEE, Transactions on Nuclear Science, NS-31, p 1230 (1984).

2. T. M. Flanagan "Hardness Assurance in Quartz Crystal Resonators," IEEE, Transactions on Nuclear Science, NS-21, p. 390 (1974)

3. P. Pellegrini, e.a., "Steady State and Transient Radiation Effects in Precision Quartz Oscillators," IEEE, Transactions on Nuclear Science, NS-25, p. 1267 (1978).

4. H. G. Lipson, e.a., "Radiation Effects in Swept Premium -$\phi$ Quartz Material Resonators and Oscillators," Proc. of 33rd Annual Frequency Control Symposium, p. 122 (1979).

5. F. Euler, e.a., "Radiation Effects in Quartz Oscillators, Resonators and Materials," Proc. of 34th Annual Frequency Control Symposium, p. 72 (1980).

6. L. E. Halliburton, "Report on Evaluation of Synthetic Quartz," Department of Physics, Oklahoma State University Reports of 31 March 1981 and 28 April 1982.

Table 1

Pre-Radiation AT Resonator Characteristics

| At Resonator* ID | Quartz Bar | Auto Clave | Aluminum Content (PPM) | 24 Hour Aging Rate | 10 Sec Allan Variance |
|---|---|---|---|---|---|
| 33820 | K13F | A6-26 | 0.79 | $2.17 \times 10^{-11}$ | $<4.0 \times 10^{-12}$ |
| 34632 | K11K | H39-24 | 8.3 | $2.05 \times 10^{-10}$ | $<6.0 \times 10^{-12}$ |
| 34744 | K13F | A6-26 | 0.79 | $2.24 \times 10^{-10}$ | $<1.5 \times 10^{-12}$ |

* Premium - Q, Swept, Cultured, Synthetic Quartz produced by Sawyer Research Products. Resonators were Manufactured by Bliley Electric Company. Resonators were Pre-Conditioned to 20 Krads (SI).

Table 2

AT Resonator Low Dose Radiation Test Results

| Resonator ID | Accumulated Dose At Resonator | $\Delta f/f$ | $\Delta f/f$ Per Rad (Si) |
|---|---|---|---|
| 33820 | 4 Rads (Si) | $1.97 \times 10^{-10}$ | $4.92 \times 10^{-11}$ |
| 34632 | 4 Rads (Si) | $1.29 \times 10^{-10}$ | $3.22 \times 10^{-11}$ |
| 34744 | 4 Rads (Si) | $1.78 \times 10^{-10}$ | $-4.45 \times 10^{-11}$* |

* Following Positive Excursion of $+2.43 \times 10^{-10}$

Table 3

$\Delta f/f$ Per Rad For
AT Resonator 33820*

| Total Accumulated Dose [Rads (Si)] Range | Dose Rate [Rads/Hour] | $\Delta f/f$ Per Rad Over Range |
|---|---|---|
| 0 → 4 | 3.9 | $4.92 \times 10^{-11}$ |
| 0 → 40 | 3.9 | $3.31 \times 10^{-11}$ |
| 512 → 1010 | 3.9 | $5.72 \times 10^{-13}$ |

* Accumulated Net Oscillator Response For A Total Accumulated Dose Of 1010 Rads (Si) is : $3.46 \times 10^{-12}$ Per Rad.

Table 4

| | DRIFT RATE | 10 Second Allan Variance | 100 Second Allan Variance |
|---|---|---|---|
| DURING IRRADIATION | $5.35 \times 10^{-11}$ Day* | $4\text{-}5 \times 10^{-12}$ | $1\text{-}1.5 \times 10^{-12}$ |
| POST IRRADIATION | $2.32 \times 10^{-11}$/Day | $2.5\text{-}3.7 \times 10^{-12}$ | $0.97\text{-}1.4 \times 10^{-12}$ |

* during the last 498 Rads (Si) of irradiation [3.9 Rads (Si) $\hat{=}$ 1 Hour of radiation exposure]

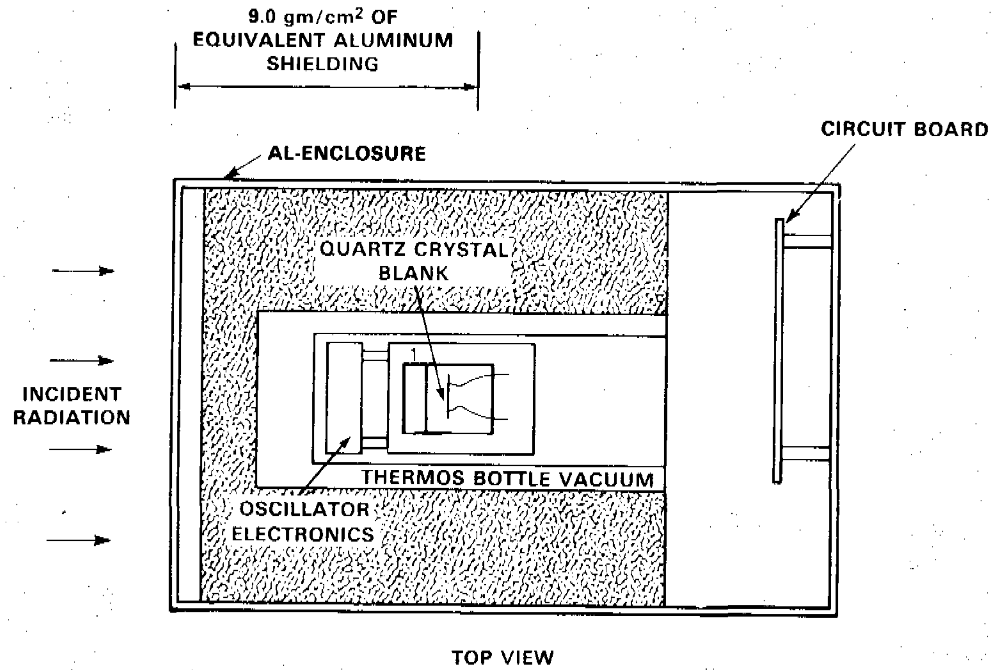


Fig. 1 Test bed oscillator.

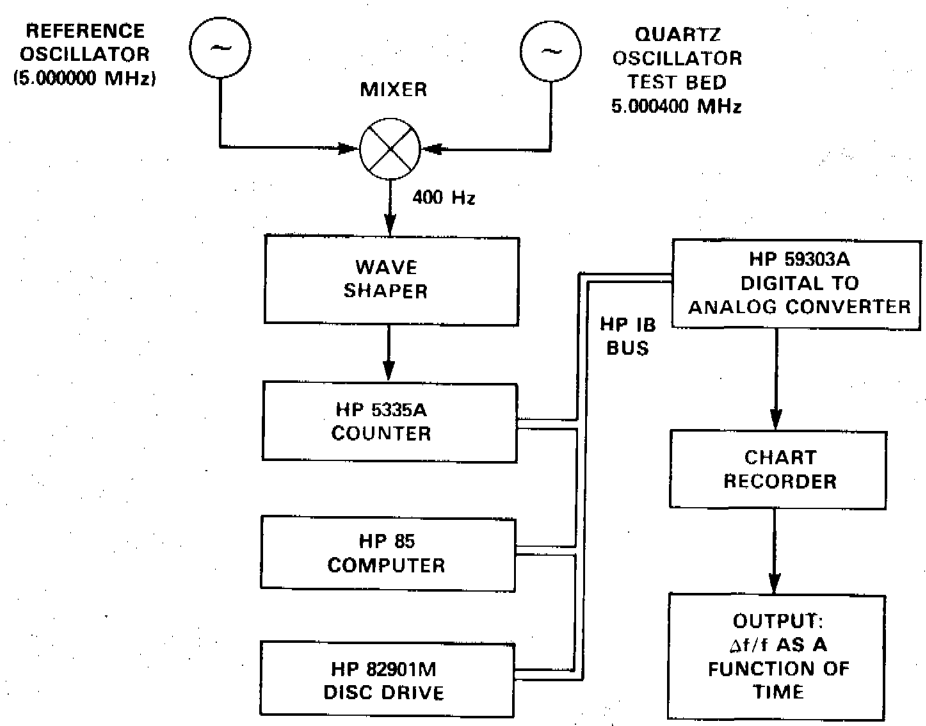


Fig. 2 Data acquisition system for radiation tests oscillator.

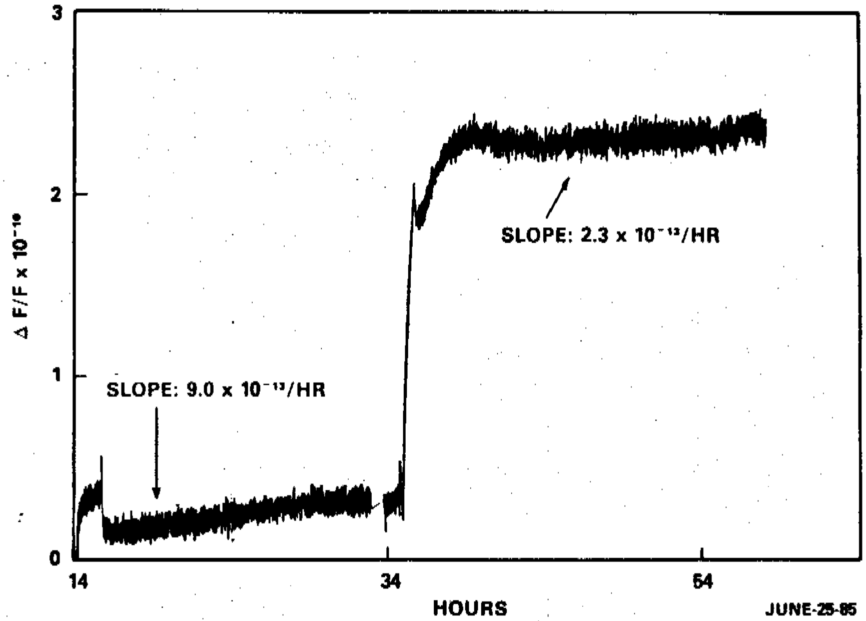


Fig. 3 Low-level radiation test on at resonator 33820.

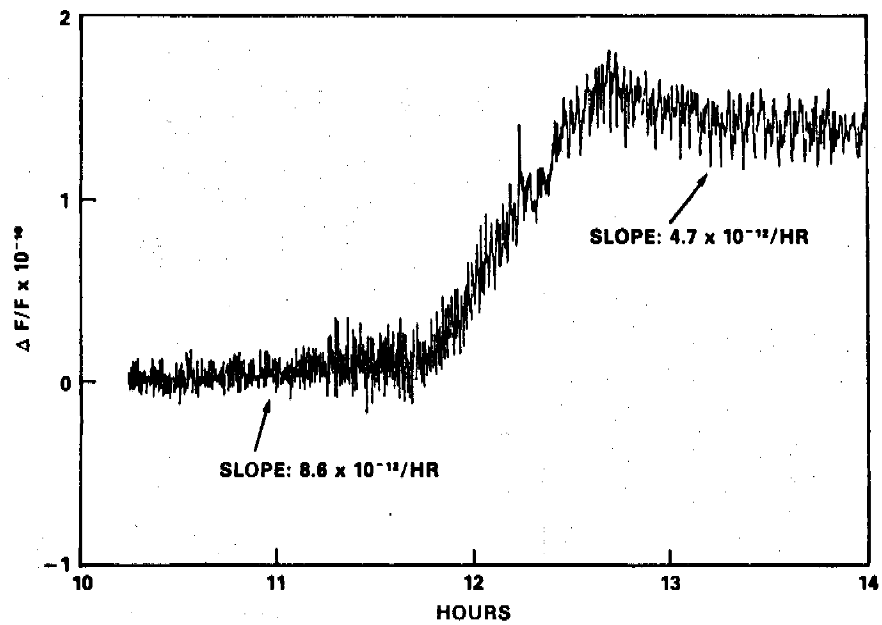


Fig. 4 Low-level radiation test on at resonator 34632.

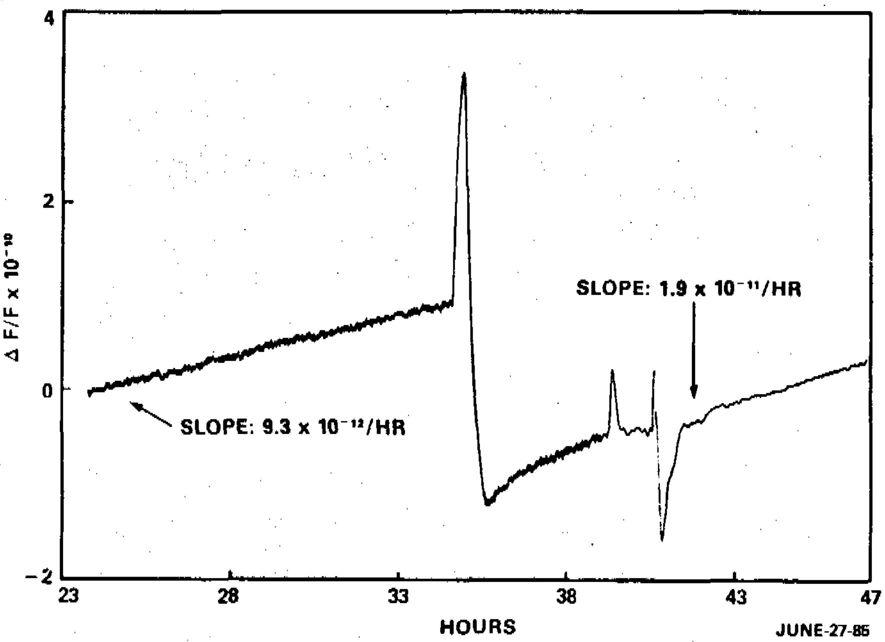


Fig. 5 Low-level radiation test on at resonator 34744.

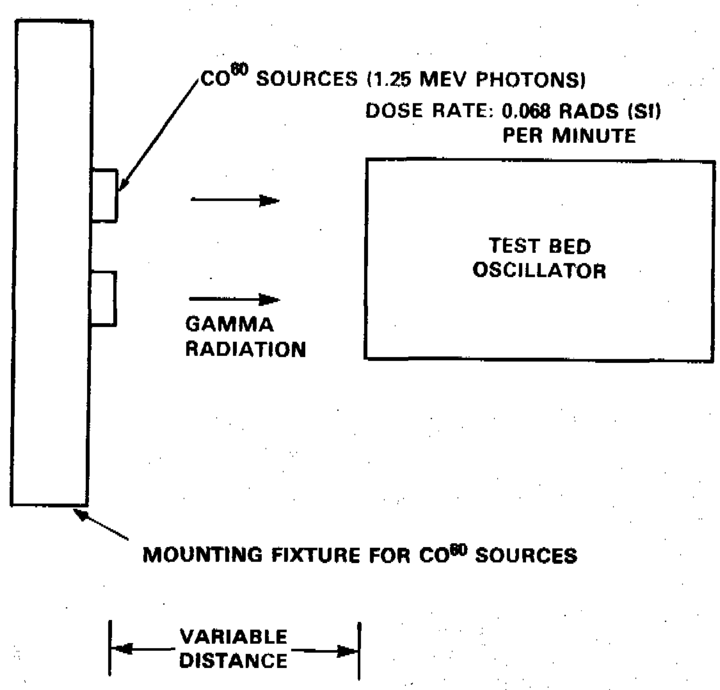


Fig. 6 Low-level radiation test configuration (top view).

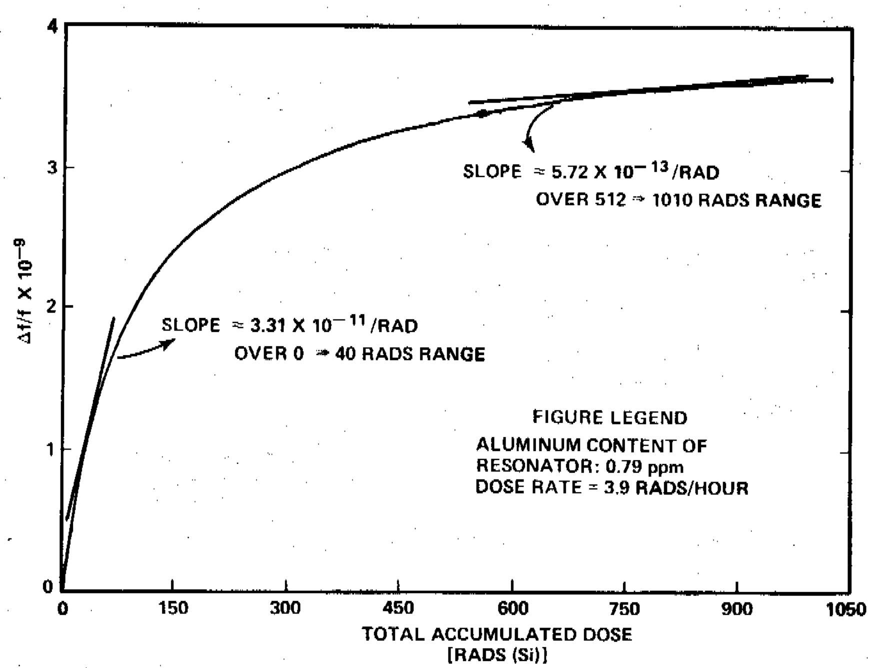


**Fig. 7** Long-term low-dose rate radiation test on 33820 resonator.

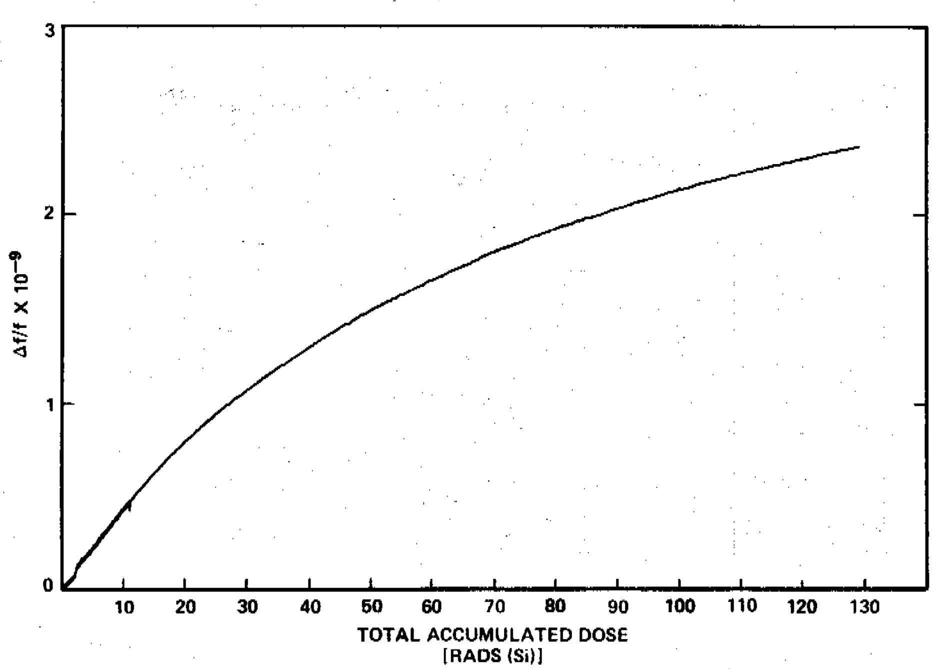


**Fig. 8** Long-term low-dose rate radiation test on 33820 resonator. (First 130 rads accumulated dose performance)

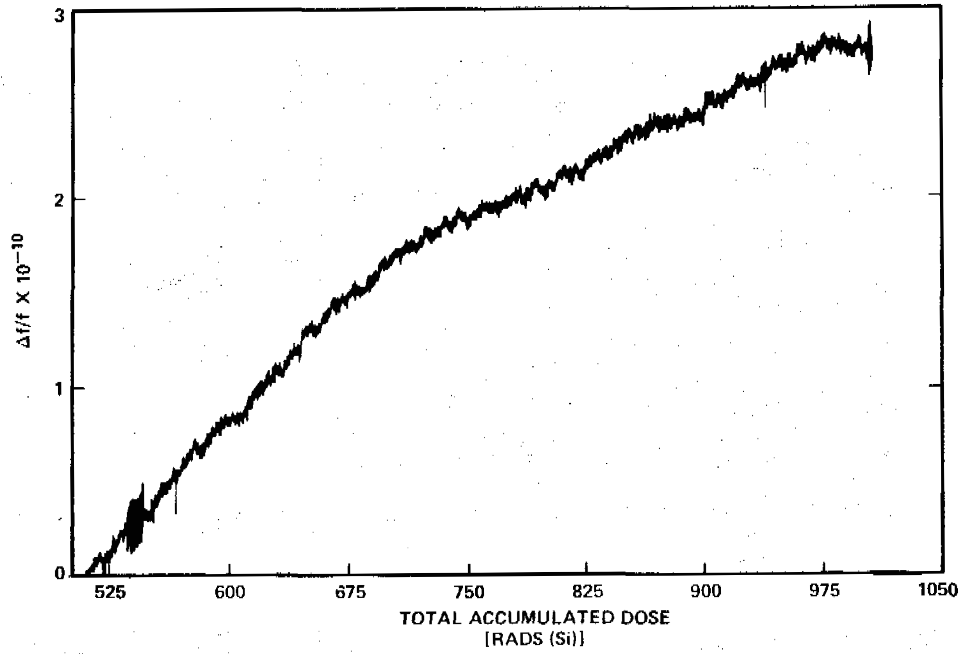


**Fig. 9** **Long-term low-dose rate radiation test on 33820 resonator. (Last 485 rads accumulated dose performance)**

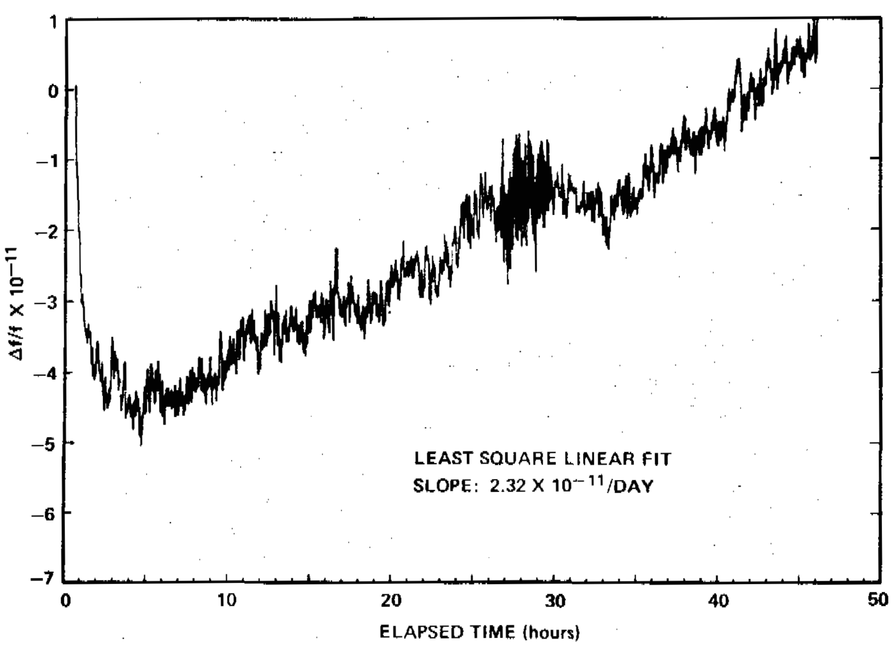


**Fig. 10** **Post radiation recovery and drift on 33820 resonator.**