

SUPERSONIC VELOCITY IN GASES AND VAPOURS.

PART IV. MEASUREMENT OF THE ABSOLUTE FREQUENCY OF PIEZOELECTRIC QUARTZ OSCILLATORS.

By S. K. Kulkarni Jathkar.

INTRODUCTION.

In the course of the investigation on the dispersion of supersonic velocity in gases and vapours now in progress in our laboratory, it became necessary to know the absolute frequency of the quartz oscillators used, under the conditions of the experiment.

A good deal of the confusion in the literature about the anomalous dispersion in gases, is due to the fact that the quartz oscillators, which have been employed in most of the researches, possess spurious oscillating frequencies within as much as 1%. In the experiments conducted by the author it was found necessary, (as shown in Part I, *This Journal* 21A, 245-271), to make alterations in the valve oscillator circuit, such as the change in the anode coupling and variation of grid bias etc., in order to get a good wave form. Further, the quartz oscillator was heated to different temperatures during the experiments. It was found that whenever the crystal possessed nearby frequencies, one was stabilized at room temperature and the other was strengthened at higher temperature. The anode coupling, grid bias, etc., produced similar effects. It was also found necessary to plate the quartz oscillator, in order to solder a thin wire lead. This affected the frequency of the crystal appreciably. Most of the preliminary work of the author was done on the assumption that the effects produced by the various factors given above were negligible, and that the frequency of the crystal could be determined by measuring the wave lengths in air under known conditions. However, the latter was found to vary appreciably during the course of the numerous experiments and there was also the vexed question of tube dispersion. It was, therefore, found essential to have a frequency measuring apparatus which

would give accurate values for the absolute frequency of the crystal oscillators at the time of measuring the wave lengths.

The accurate determination of the frequency of an electrical oscillation is a measurement often required in the laboratory but one that is rarely undertaken without the use of elaborate apparatus. Frequency-meters of the Campbell type for audio frequencies, and carefully constructed wave-meters for radio frequencies, are of use in many cases, but their calibration is apt to vary, particularly during transport, and they cannot be relied upon for much greater accuracy than 1 part in 1000.

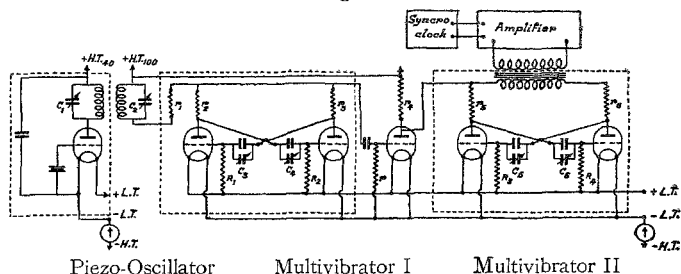
It is very necessary to employ a method of frequency measurement which is independent of outside calibration, and for this purpose the harmonic multivibrator as developed by the late Dr. Dye and others, has proved to be of the utmost value, and has been recently installed in the Standardising Laboratories of the Institute. The frequencies of all of the oscillators used in the present work, have been checked on this apparatus and were found to agree with the measurements of the author on his apparatus. While the purpose for which the apparatus was required, did not need the highest precision of the Dye multivibrator, because the other conditions of the experiment, such as purity of the substances, temperature etc., were not reproducible to that degree of accuracy, the accuracy of the harmonic multivibrator, however, could be obtained with a comparatively simple and inexpensive apparatus, the main advantage being that the absolute frequency could be checked on the spot.

EXPERIMENTAL.

The method consists in the use of a two-stage step-down, unsymmetrical multivibrator (Hull and Clapp, *Proc. Inst. Radio. Eng.*, 1929, p. 252) in conjunction with a phonic wheel, in the present case a "synchro-clock" by the General Radio Company of America kindly lent by the Department of Electrical Technology. The author has also constructed such a phonic wheel. This instrument is easily driven by a valve of the PX4 type and will run in synchronism with currents varying in frequency from 0.2 to 1.5 Khz. Timings can be recorded to 0.01 second but, for the series of experiments to be described, visual estimates of 0.1 second sufficed. All measurements were made against the 10

seconds signals of the laboratory electric clock, the timing of which was checked against radio signals.

Fig. 1



$$\begin{aligned}
 c_1 &= .005 \mu f, & c_2 &= .001 \mu f, & c_3 \text{ and } c_4 &= .0015 \mu f, & c_5 \text{ and } c_6 &= .0025 \mu f, \\
 R_1 \text{ and } R_2 &= .02 \text{ to } .04 & R_3 \text{ and } R_4 &= .5 \text{ megohm.} \\
 r_1, r_2, r_4, r_5 &= .01 & r_3 \text{ and } r_4 &= .25 \text{ megohm.} \\
 r &= 2 \text{ megohms.}
 \end{aligned}$$

The scheme of connections in Fig. 1, shows the multivibrator in detail. Instead of being used to generate harmonics of a known frequency, the multivibrator oscillates at its fundamental frequency, control being obtained by a drive (in this case the oscillator, the frequency of which is to be determined) oscillating at a harmonic. Hull and Clapp state that harmonics as high as the tenth may be used in each stage, giving a maximum step-down of one-hundredth, for the two. This is a good working rule, although we have found no great difficulty in controlling by means of the fiftieth harmonic, odd harmonics having been found to exercise better control. It will be noted that the multivibrators are unsymmetrical, the anode resistances being unequal; this appears to be essential for satisfactory working. The original instrument was provided with a buffer valve between the driving oscillator and the first multivibrator. This is desirable for work of the highest precision but in these experiments it was omitted. The variable condensers shown, were $0.0005 \mu f$ each, fitted with clips into which Loewe condensers of higher value could be inserted if necessary.

In order to use the instrument, the condensers C_3 , C_4 , C_5 and C_6 are set approximately to a convenient frequency, making use of the formula $N = 1/(R_1 C_3 + R_2 C_4)$, where N represents the frequency. The coupling between the driving circuit and the first multivibrator, which is fairly critical, is gradually increased until the multivibrator is found to be controlled. This is ascertained by the fact that small movements of the condensers have no effect on the output frequency, which is audible, while larger movements cause variations in jumps. The audio output is then adjusted so as to come within the range 0.7 to 1.5 KHz. and the amplifier is attached to the synchro-clock. The frequency is measured by comparison with the standard clock giving 10 second clicks at intervals of 100 seconds, for about 20 minutes. The condenser settings on the multivibrator are then changed to give a second output frequency, which is also measured, and so on. In this way a number of values are obtained which are harmonically related series, from which the fundamental can be calculated. In Table I an example is given of measurements with a 50 KHz. crystal oscillator.

TABLE I.

Interval No.	Standard time		Synchro clock		Interval for 100 secs.	Weight
	mins.	secs.	mins.	secs.		
0	1,	40	2,	43		
1	3,	20	4,	46,5	123,5	1 x 10
2	5,	0	6,	50,0	123,5	2 x 9
3	6,	40	8,	54,0	124,0	3 x 8
4	8,	20	10,	57,8	123,8	4 x 7
5	10,	0	12,	61,5	123,7	5 x 6
6	11,	40	14,	5,0	123,5	6 x 5
7	13,	20	16,	8,6	123,6	7 x 4
8	15,	0	18,	12,2	123,6	8 x 3
9	16,	40	20,	16,0	123,8	9 x 2
10	18,	20	22,	19,7	123,7	10 x 1
				Weighted mean =	123,67,	
				f =	1236,7,	

The reading of the synchro clock at 1 min., 40 sec. was 2, 43.0; at 3, 20, 4, 46.5; and so on. For 100 seconds of the standard clock, the weighted mean of the readings of the synchro clock was 123.67. As the synchro clock reads correct time at exactly 1000 cycles, the step-down frequency f is exactly 1236.7.

The observations were taken for thousand seconds, and as the error was of the order of 0.1 to 0.2 second, the error in measuring the frequency was less than 1 part in 5000 if importance was given to the first and the last observation. The actual error is considerably less than this, if a weighted mean is taken of all the ten or more observations. Although the average mean was sufficiently accurate for the purpose for which the crystals were used, the highest accuracy obtainable with a given number of observations was found to be 1 part in 100000, when weighted mean was taken by taking all combinations of the readings. The results given in Tables 3, 4, 5 and 6 have been treated in this way, which speak for themselves.

The following table shows the measurement of an audio frequency by a single stage multivibrator.

TABLE 2.

Multivibrator II						
c_3	c_6	R_3 megohm	R_4 megohm	Step-down n	f	$F =$ $n \times f, \text{ Hz}$
1000 $\mu\mu\text{f}$ +155°	1000 $\mu\mu\text{f}$ +170°	0.5	0.5	4	888	3552
1000 $\mu\mu\text{f}$ +10°	1000 $\mu\mu\text{f}$ +10°	0.5	0.5	3	1184	3552

The above frequency was emitted by the simultaneous oscillation of a crystal (B and H, 55 x 14 x 5 mms.) at two nearby frequencies 49.712 and 46.160.

Table 3 shows the results of the frequency measurements of a 24 Khz. crystal oscillator (115 x 20 x 5 mms.).

TABLE 3.

MULTIVIBRATOR I				MULTIVIBRATOR II				n ₁ x n ₂ = N	f	F = N x f Hz
C ₃	C ₄	R	n ₁		R	n ₂				
1000 $\mu\mu\text{f}$ +130°	1000 $\mu\mu\text{f}$ +150°	0.04	2	2000 $\mu\mu\text{f}$ +0°	2000 $\mu\mu\text{f}$ +3°	0.5	7	14	1684.6	235 83
—	—	"	3	—	—	"	5	15	1572.3	235 85
—	—	"	2	—	—	"	8	16	(1473.4)	(235 74)
+130°	+150°	"	2	+0°	+0°	"	9	18	1310.6	235 90
+110°	+130°	"	3	+180°	+180°	"	7	21	1123.0 ₈	235 85
+0°	+60°	"	2	+180°	+180°	"	11	22	1072.1	235 87
+50°	+60°	"	3	+180°	+180°	"	8	24	982.7 ₈	235 86
—	—	"	4	—	—	"	7	28	(842.0)	(235 76)
—	—	"	4	—	—	"	8	32	737.0	235 84
235 86										

The frequency of this crystal was checked on the Tuning Fork Standard Frequency Apparatus developed by Dye, which has been installed in the Electrical Technology laboratory of the Institute. The frequency was measured by interpolation from the readings of the standard condenser, corresponding to the various frequencies given by the multivibrator controlled by the 1 Khz. Standard Tuning Fork. The following readings were obtained:

$$\begin{aligned}
 22 \text{ Khz} &: 0.004 \mu\text{f} + (1150 + 90.0) \mu\mu\text{f} \\
 f_0 - 1 = f_x & \text{ ,, } : 0.0035 \text{ ,, } + (1450 + 69.0) \text{ ,,} \\
 23 & \text{ ,, } : \text{ ,, } + (1250 + 59.8) \text{ ,,} \\
 24 & \text{ ,, } : \text{ ,, } + (800 + 59.0) \text{ ,,} \\
 f_0 + 1 = f_x & \text{ ,, } : \text{ ,, } + (600 + 46.75) \text{ ,,} \\
 25 & \text{ ,, } : \text{ ,, } + (450 + 56.2) \text{ ,,}
 \end{aligned}$$

$f_0 + 1 = 24.585$ Khz. Therefore $f_0 = 23.585$, which is in exact agreement with the value of the step-down multivibrator. f_x was given by the expression $f_x = \frac{f_h}{\sqrt{1 + \frac{f_h^2 - f_1^2}{f_1^2} \left(\frac{C_x - C_h}{C_1 - C_h} \right)}}$ where

$f_x = f_c + 1$ Khz. f_h = frequency higher than f_x , f_l = frequency lower than f_x and c_x, c_h, c_l are capacities corresponding to f_x, f_h and f_l . When $f_h - f_l = 1$ Khz, the expression reduces to

$$f_x = \frac{f_h}{\sqrt{1 + \frac{f_h - f_l}{f_x^2} \left(\frac{c_x - c_h}{c_l - c_h} \right)}}$$

The following table summarises the results of the various step-down frequencies, which were measured from time to time spread over a period of three years, of the crystal (Sc. R) (53.5 x 21.5 x 2.6 mms).

TABLE 4.

	Step-down 1	Step-down 2	Total Step-down n	Output Frequency f	F = n x f Hz.
1.	2	17	34	1455.1	494 70
2.	2	19	38	1302.0	494 77
3.	2	20	40	1236.7 ₇	494 71
4.	2	21	42	1177.7 ₃	494 65
5.	2	21	42	1177.9	494 72
6.	4	11	44	1124.1 ₃	494 63
7.	5	9	45	1099.2 ₇	494 66
8.	3	16	48	1030.6 ₃	494 70
9.	7	7	49	1009.5 ₆	494 68
10.	5	10	50	989.3	494 65
11.	6	10	60	824.6 ₂	494 76
					494 70

These determinations were made on different occasions and the crystal temperatures varied from 23° to 28°. They show, however, that its frequency was very near to 49.470 KHz. Thus in the first example, the frequency of the first multivibrator was 24.735 KHz. and this was the 17th harmonic of the second multivibrator. It is, of course, not necessary to know the step-down in each case, the figures given being merely by way of illustration. In practice it is not easy to obtain every harmonic (as odd harmonics have preferential control), and this need not be done, there being little difficulty in determining the fundamental, if the difference between the reciprocals of measured frequencies are examined, or if the approximate frequency is known.

The frequency of this crystal was checked, more than five years after the above measurements were completed and the crystal was used under varying conditions of temperatures, on the Standard Frequency Apparatus. The following measurements were obtained.

48 KHz	:	0.003 μ f + 825.15 μ μ f
$f_x = f_c - 1$ KHz	:	„ + 746.2 „
49 „	:	„ + 661.1 „
50 „	:	„ + 507.3 „
$f'_x = f_c + 1$ „	:	„ + 437.7 „
51 „	:	„ + 363.35 „

$$f_c = f'_x - 1 = 49.474 \text{ KHz. } f_c = f_x + 1 = 49.471 \text{ KHz.}$$

Mean $f_c = 49.473$ KHz. which is in very good agreement with the value given above.

The frequency of another crystal (55 x 14 x 4 mms.) as determined by the step-down multivibrator was (1079.1 x 45 =) 48.559 KHz. and was checked by direct measurement of its beat frequency with 49.470 KHz. crystal on the Synchro-clock, which was 907 hz.

The frequency of the same crystal after re-plating was determined on the standard frequency apparatus by interpolation. The readings of the condenser for the different frequencies were as follows :—

50 KHz.	0.003 μ f + 497.0 μ μ f
$f_x + 1$ „	0.003 „ + 578.9 „
49 „	0.003 „ + 650.9 „
48 „	0.003 „ + 814.9 „
$f_c - 1$ KHz.	0.003 „ + 905.6 „
47 „	0.003 „ + 988.9 „

(B) (Sc. R) silvered and plated (26.5 x 15.5 x 2.1 mms.)

	$n_1 \times n_2$	N	f	$F = \frac{f}{N}$
1.	(12 x 5)	(60)	1578.9	959 40
2.	15 x 5	75	1279.2	959 40
3.	11 x 7	77	1245.8 ₅	959 30
4.	16 x 5	80	1199.1 ₅	959 32
5.	21 x 4	84	1142.0	959 28
6.	17 x 5	85	1128.5 ₅	959 27
7.	29 x 3	87	1102.6 ₅	959 31
8.	(11 x 8)	(88)	(1090.3)	(959 46 ⁵)
9.	12 x 8	96	999.5 ₅	959 52
10.	53 x 2	106	905.0	959 30
11.	12 x 9	108	888.3 ₅	959 45
12.	(11 x 11)	(121)	(793.1 ₅)	(959 65 ⁵)
				959 33

Table 6 (A) shows the results of the measurements of the step-down frequencies of a crystal (26.5 x 15.5 x 2.1 mms.) supplied by the Scientific Radio Company of America, in its holder without plating. The frequency of the crystal was 96.188 Khz. The higher accuracy of the measurements is due to the excellent oscillating property of this crystal, which was also very satisfactory for measurement of supersonic velocity.

On plating and silvering the crystal, the frequency changed to 95.933 Khz. as shown in Table 6 (B).

Table 7 shows the measurements of the crystal (E) 25.5 x 25.5 x 5 mms., which was kindly lent by the Electrical Technology Department. It gave three frequencies $F_1 = 94.483$ Khz., $F_2 = 127.408$ Khz. and $F_3 = 564.028$ Khz. The frequencies of this crystal were also measured when it was heated to 134° in a xylene bath, when the first two frequencies decreased to 93.889 and 126.264 Khz. respectively.

The higher frequency of the same crystal (E) was 127.408 KHz. at 24', as measured on the step-down multivibrator which was only 34 cycles higher than the value determined, on the standard frequency apparatus, the results of which are given below :

$f_c - 1 = f_x :$	$(0.01 + 0.0045) \mu f + 1001 \mu \mu f$	
$f_c + 1 = f'_x :$	„ „	+ 1512 „
126 KHz.	: „ „	+ 1095 „
127 „	: „ „	+ 846 „
128 „	: „ „	+ 602 „
129 „	: „ „	+ 364 „

$$f_x = 126.373 \text{ KHz.}$$

$$\text{Therefore } f_c = f_x + 1 = 127.373 \text{ KHz.}$$

$$f'_x = 128.374 \text{ KHz.}$$

$$\text{Therefore } f_c = f'_x - 1 = 127.375 \text{ KHz.}$$

$$\text{Mean } f_c = \mathbf{127.374} \text{ KHz.}$$

The third frequency F_3 of the same crystal was measured only on the Standard Frequency Apparatus. The author did not have any crystal, whose harmonic frequency could be compared with the above frequency. The following measurements of the condenser were used for interpolation of the frequency.

	563 KHz.	: 0.006 μf	+ 114.0 $\mu \mu f$	
$f_x = F_3 - 1$	„	: „ „	+ 113.35 „	
	564 „	: „ „	+ 90.2 „	
	565 „	: „ „	+ 66.3 „	
$f'_x = F_3 + 1$	„	: „ „	+ 65.6 „	
	566 „	: „ „	+ 42.55 „	
$F_3 = f_x + 1 =$	564.027 KHz.			$F_3 = f'_x - 1 = 564.029 \text{ KHz.}$
				Mean $f_c = \mathbf{564.028} \text{ KHz.}$

The Crystal (E) is very suitable for dispersion measurements.

Calibration of 396 K. C. crystal.

The frequency of this crystal was measured only on the Standard frequency apparatus, by the linear interpolation of the condenser readings corresponding to the various harmonics of the multivibrator.

Frequency Khz.	Condenser reading.
400	0.0124376
399	0.0125025
398	0.0125685
$f_x + 1000$ cycles	0.0126006
397	0.0126345
f_c	0.0126675
396	0.0127010
$f_x - 1000$ cycles	0.0127340
395	0.0127690

The frequency of the crystal is 396.525 Khz.

DISCUSSION.

Plating the crystal was found to lower the frequency of a crystal, although the weight and the size of a separate electrode did not appreciably affect the frequency. Thus, on being sputtered with a thin layer of silver, a crystal 55 x 14 x 4 mms. gave two frequencies 46.160 and 49.712 Khz. simultaneously, producing an audible frequency of 3552 hz., which was also measured on the single stage multivibrator. As it was difficult to solder a lead to the sputtered surface, the crystal was chemically silvered and electroplated with copper and silver, and a thin copper wire was soldered in the centre of the top electrode. The frequency of this crystal decreased to 48.560 Khz., as determined by the measurement of its beat note (0.907 Khz.) with another crystal of frequency 49.471 Khz., and the lower frequency disappeared. This crystal (48.56 Khz.) was used for measurements of volume resonance described in Part III. On replating this crystal, the frequency changed to 48.464 Khz., which was measured on the Standard Frequency Apparatus.

In the same way, a crystal whose frequency was 96.188 Khz. in the holder in which it was supplied, was found to have changed its frequency to 95.933 Khz. on being silvered and plated. The plating of the crystal spoiled its oscillating property somewhat, although some of the crystals, which would not oscillate in an ordinary holder, could be made to oscillate after plating. It was also found that plating only the top electrode was preferable and convenient, as it did not affect the oscillating property, nor changed the frequency, to the same extent as plating on both sides. The plating did not peel off the crystal, even on heating to 134°, as the surface was matt.

Work is in progress to find out the changes in the equivalent circuit of the quartz oscillator, which are produced by plating.

Crystal (E) gave three frequencies ($F_1 = 94.49$ Khz., $F_2 = 127.408$ Khz. and $F_3 = 564.028$ Khz. respectively) at 25° . The first two frequencies were checked on the standard frequency apparatus after a lapse of five years and were found to be correct to 1 part in 10,000 in absolute units. The third frequency was measured on the latter apparatus only.

The temperature coefficients of these crystals were found by determining the beat frequency between two suitable crystals, one at room temperature and the other heated to the desired temperature. Thus the 49.471 Khz. crystal decreased in frequency by only 50 Hz. on heating from 20° to 100° . It was also found possible to step-down the frequency of the hot crystal directly. Thus the first two frequencies of the crystal (E) at 134° as determined by the step-down multivibrator were 93.899 and 126.264 Khz. respectively.

For frequencies higher than 150 Khz., it was found possible to obtain a beat note of suitable frequency between the source to be measured and a harmonic of one of the crystal oscillators. This was amplified and measured on the multivibrator if it was higher than 1500 cycles, or measured directly on the synchro-clock if it was lower. For example, a crystal oscillator of a nominal frequency of 480 Khz. was found to give an audio note of 540 cycles with the fifth harmonic of 95.931 Khz. crystal. The frequency was therefore 479.10 Khz.

The author has given a detailed account of a convenient method of measurement of absolute frequencies very accurately. Although the purpose for which the apparatus was required, did not need the highest precision attainable, the accuracy of the Dye harmonic multivibrator could be obtained with comparatively simple apparatus. With the establishment of the standard frequency apparatus in the Institute, it has been found possible to check the measurements on the step-down multivibrator repeatedly, which gives the author confidence to recommend the inexpensive equipment used by him as a standard frequency apparatus, the calibration of which is directly measured against standard clock. The cost of many excellent precision frequency equipments, does not lie within the reach of institutions of modest means.

The frequency measurements recorded in this paper are accurate to better than 1 part in 5000 and the absolute frequencies of the various quartz oscillators have been checked after a lapse of more than five years on the standard frequency apparatus and were found to be correct within the accuracy of the original measurements, which could be easily increased by taking observations for intervals longer than 20 minutes, on a syphon recorder. It was, however, found that the absolute accuracy in measurement of supersonic velocity was more limited by the purity of the substances used and the constancy of temperature and pressure, which affected the reproducibility in the wave length measurements, the details of which will be discussed in the subsequent parts of this series.

The following table shows the comparison of the results of the measurements of the frequencies of the various piezoelectric quartz oscillators used in the supersonic interferometer in gases and vapours, by the step-down multivibrator, and by the Dye Standard Frequency Apparatus.

TABLE 8.

Crystal	Dimensions mm.	(1932.33) Step-down multivibrator Author Khz.	(1938) Dye step up multivibrator Khz.
Bernard and Halle (B and H)	115 x 20 x 5	23.586	23.585
Scientific Radio (Sc. R).	53.5 x 21.5 x 2.6	49.471	49.472
Bernard and Halle (B and H)	27.1 x 10 x 3	94.396	94.400
Scientific Radio (Sc. R)	26.5 x 15.5 x 2.1	95.933	95.926
E	25.5 x 25.5 x 5	96.188	
		F ₁ 94.483	94.467
		F ₂ 127.408	127.374
		F ₃ —	564.028

Thanks of the author are due to Dr. H. E. Watson for his keen interest and helpful guidance during the course of this work, and to Prof. K. Aston for affording facilities for checking the measurements of the frequencies on the standard frequency apparatus which has been installed in the communication Engineering Section.

SUMMARY.

A comparatively simple and inexpensive apparatus using a double, step-down, unsymmetrical multivibrator, has been described for determination of the absolute frequency of the piezoelectric quartz oscillators, used in the measurement of supersonic velocities in gases and vapours at different temperatures. The resulting low frequencies were measured by a synchro-clock, the observations on which were checked against the laboratory standard clock. Frequencies from 300 — 1500 Hz. could be measured directly on the synchro-clock and the multivibrator could give easily a step-down ratio as high as 120 : 1, without a high frequency stage, so that with the combination, frequencies up to 150 KHz. per second could be measured with a degree of accuracy limited only by the patience in observation. For higher frequencies, the beat between a harmonic of a standard crystal oscillator and the frequency to be determined could be applied to the system. There is apparently no difficulty in connecting one more multivibrator stage for stepping down the higher frequencies.

A single multivibrator could step-down frequencies in the ratio as high as 50 : 1. The odd multiples were found to exercise preferential control.

The mechanical and electrical stability of the piezoelectric quartz oscillators used in supersonic interferometers in gases and vapours at different temperatures, was greatly enhanced by coating the top electrode with chemical silvering followed by electroplating with copper and silver, to which a thin lead wire could be directly soldered. This process, however, decreased the oscillating frequency of the crystals appreciably.

The accuracy, in absolute units, of the measurements on the simplified step-down multivibrator has been established by careful checking on the Dye Tuning Fork Standard Frequency Apparatus.

*Department of General Chemistry,
Indian Institute of Science,
Bangalore (India).*

[Received, 28-11-1938]