ULTRASONIC ABSORPTION
IN CRYSTALLINE BENZENE
HOWARD W. ALEXANDER
and
JOHN E. ELMORE

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Lieutenant, United States Navy

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John E. Elmore

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Submitted in partial fulfillment of
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IN
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ABSTRACT

The dependence of the absorption of longitudinal ultrasonic waves in single crystals of benzene on the frequency and on the orientation of the crystal was investigated experimentally. Results were compared with the predictions of the resonance absorption theory.

Individual crystals were obtained by freezing a melt of highly purified reagent-grade benzene. The crystals were aligned to desired orientations by optical techniques.

Absorption coefficients were computed from direct measurements of the attenuation in longitudinal wave propagation at 5, 10, and 15 megacycles. Results were obtained at 255 $^\circ$K for absorption along each of the principal crystallographic axes and along three off-axial directions.

Absorption was found to be a function of crystal orientation, both in magnitude and frequency dependence. The frequency-squared dependence predicted from resonance absorption theory was observed for some, but not all orientations. Results indicate the presence of an additional absorption mechanism.

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1. Introduction.

The very high acoustic absorption observed experimentally in certain molecular crystals at ultrasonic frequencies is attributed to molecular vibrational resonance in the resonance absorption theory proposed by Liebermann (1). The basis of this absorption mechanism is an overlapping of the vibrational frequencies of the two modes of thermal oscillation of a molecule: internal vibrations of the atoms within the molecule and vibrations of the molecule as a whole about its lattice position. Such frequency coincidence results in a pseudo resonance, but with a consequent low rate of energy exchange between the two vibrational modes due to the weak intermolecular forces. This mechanism thus leads to a time dependent specific heat with a concomitant absorption for sound waves. In the case of benzene, such absorption has been observed to be four orders of magnitude greater than that in monocrystalline quartz.

Liebermann's experimental work (2) involved the use of benzene primarily because of the wide variety of thermodynamic data available for this compound which made possible a direct comparision of observed and calculated absorption. The writers of this thesis also utilized benzene in order to directly extend the results of prior experimentation, specifically in investigating the frequency dependence of absorption and the effects of crystal orientation.

Liebermann concluded from experimental results that the

acoustic absorption in benzene varied with the square of the frequency as predicted by the resonance absorption theory. He also found that the observed values of the absorption coefficients were approximately one-half of the theoretical values, the discrepancy being attributed to the uncertainty in the molecular constants used in calculating the coefficients. Apparently, no attempt was made to orient the specimens in his work; therefore, it is assumed that his conclusions were based on the observation of absorption in randomly oriented crystals. In the process of determining the elastic constants of crystalline benzene, Heseltine and Elliott (3) obtained incidental absorption data which strongly indicated that absorption was a function of crystal orientation. Their work involved measurements at a single frequency which precluded a direct comparison with Liebermann's results.

Rasmussen has amplified and refined the theory of resonance absorption (4) and has further investigated the validity of the theory by conducting ultrasonic absorption experiments with cyclohexane crystals. In brief, his results verified the square-law frequency dependence and again, as in the case of benzene, the observed absorption was only one-half the predicted value. Rasmussen's experiments were conducted on unoriented crystals; however, he did note that no effects of anisotropy were evident in his results. Dowling and Shultz, from similar work with cyclohexane (5), found a dependency of absorption on the two-thirds power of the frequency. Their values

of the absorption coefficient were approximately five times those determined by Rasmussen.

The objectives of the studies reported herein were those of investigating the validity of the frequency-square dependence of absorption for various crystal orientations and of determining the effects of orientation on the magnitude of absorption. Utilizing the procedures and techniques which will be described, the absorption coefficients for pure crystalline benzene at a temperature of $255^{\circ}K$ were determined at 5, 10, and 15 megacycles for longitudinal wave propagation along each of the principal crystallographic axes and along three directions, each of which was oriented at an angle of 45° with two of the principal axes and perpendicular to the third axis. These latter directions will henceforth be referred to as "oblique".

2. Sample preparation.

Crystals of the necessary size for subsequent alignment and mounting were obtained by freezing a melt of reagent grade benzene. Prior to freezing, the benzene was dehydrated and deaerated to minimize the possibility of entrapped impurities affecting absorption measurements. The drying agent, calcium chloride, was added directly to the benzene, and the mixture was stored for several days before further processing. After filtering-off the dehydrating agent, the benzene was boiled under a vacuum to remove all entrapped gases. The highly purified benzene was kept under vacuum in

a sealed container and placed in a refrigerated compartment maintained at a temperature several degrees below the freezing point of benzene (5.2°C). A period of from 10 to 14 days was required for a conglomerate mass of individual crystals to form. It was found unnecessary to seed the melt or to use a heat-leak to assist in crystal formation. The mass of irregularly-shaped crystals was separated by hand or by slight pressure with a sharp instrument along crystal boundaries. The crystals, thus prepared, could be preserved indefinitely at normal refrigerator temperatures, approximately -10°C.

Crystal alignment was accomplished by use of the optical techniques described by Mullens (6), as modified by Heseltine and Elliott (3). The techniques and equipment employed are described in detail in the latter; consequently, they will be only briefly summarized here.

Inasmuch as there is no preferred direction of growth for a benzene crystal, each sample had to be individually aligned. Benzene crystals belong to the orthorhombic crystal system and are characterized by a large positive birefringence. The center of the interference pattern produced around the two optic axes when a convergent beam of polarized light is passed through the specimen accurately defines the location of the b-crystallographic axis. The other principal axes can then be located from their known orientation with respect to the b-axis and the optic plane, as depicted in Fig. 1. The pictured orientations are based on the lengths of the three principal axes as determined by Cox (7) and the
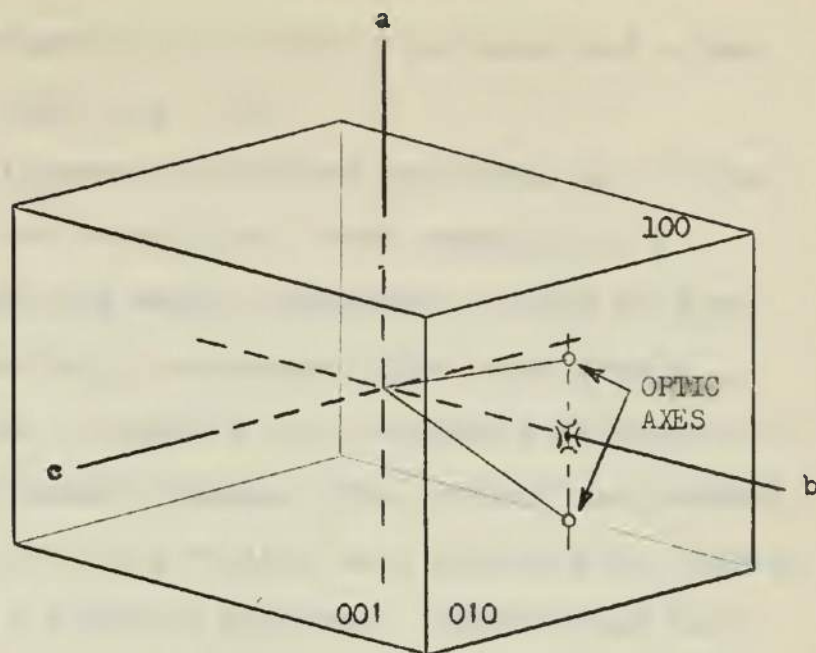
OPTIC
AXES

(a) PRINCIPAL AXES AND PRINCIPAL PLANES
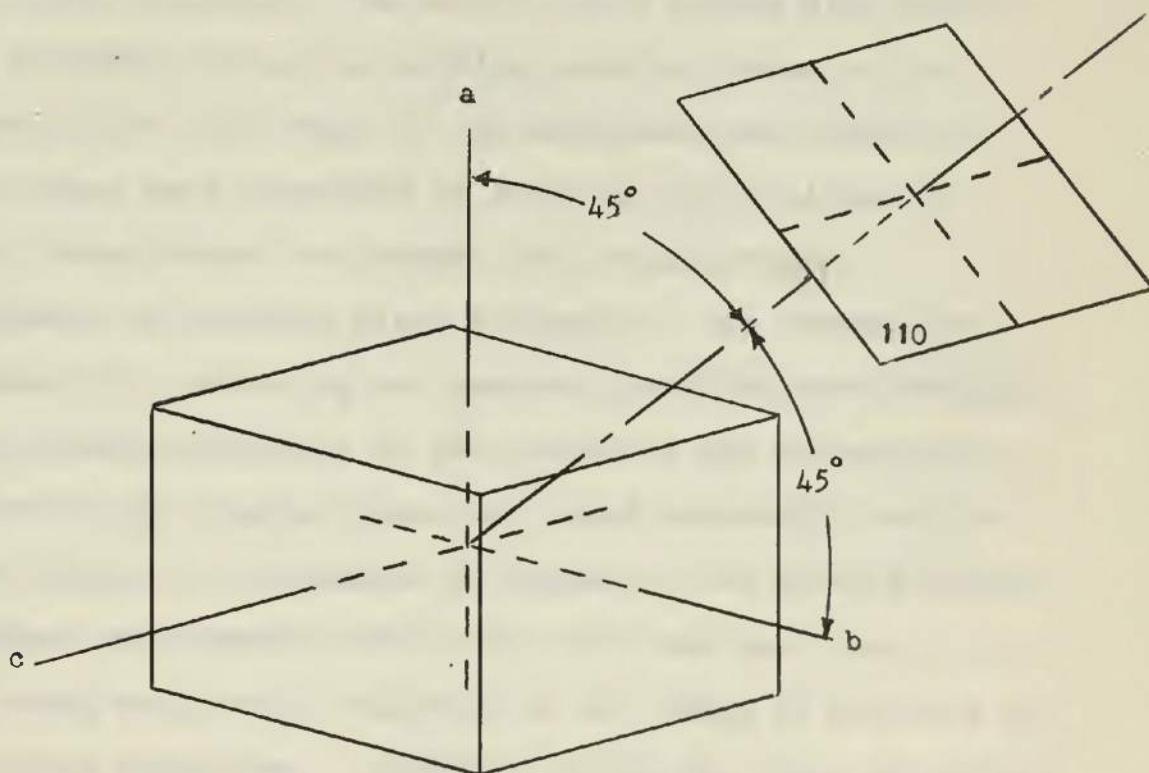
(b) 45°-110 ORIENTATION

FIGURE 1. CRYSTALLOGRAPHIC AXES AND PLANES OF BENZENE

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known optical properties of crystals as described in any crystallography text, e.g. (8).

The actual alignment procedures involved, as a first step, placing a raw crystal in a bath composed of a mixture of benzene and methyl salicylate. This bath was located in a polarized, convergent light beam from a mercury arc source. Figure 2 is a schematic illustration of the optical alignment system. The crystal was rotated until the optic axes were visible on a ground-glass plate positioned above a polaroid analyzer. Maintaining this approximate alignment, the crystal was transferred to a gimballed holder which permitted fine adjustments of the crystal until the center of the interference pattern was clearly visible. The base of this holder also served as a reference plane for melting parallel faces on the crystal after this stage of the alignment was completed. These faces were preserved by freezing glass slides in place, after which the crystal was transferred to a goniometer for precise final alignment. The corrections necessary for obtaining the desired precision were computed from measured movements of the center of the interference pattern as the viewing plate was moved vertically and the known indices of refraction of benzene. The final alignment procedure was repeated until the error was less than 0.5°, this being arbitrarily selected as the limit of accuracy of the facing operations. Alignment along the other principal axes or along the desired oblique axes was accomplished by rotating the crystal 90° or 45°, respectively, in the

GROUND
GLASS
SCREEN

ANALYZER

IMMERSION
TANK

SINGLE
CRYSTAL

GLASS
BOTTOM

LENS

POLARIZER
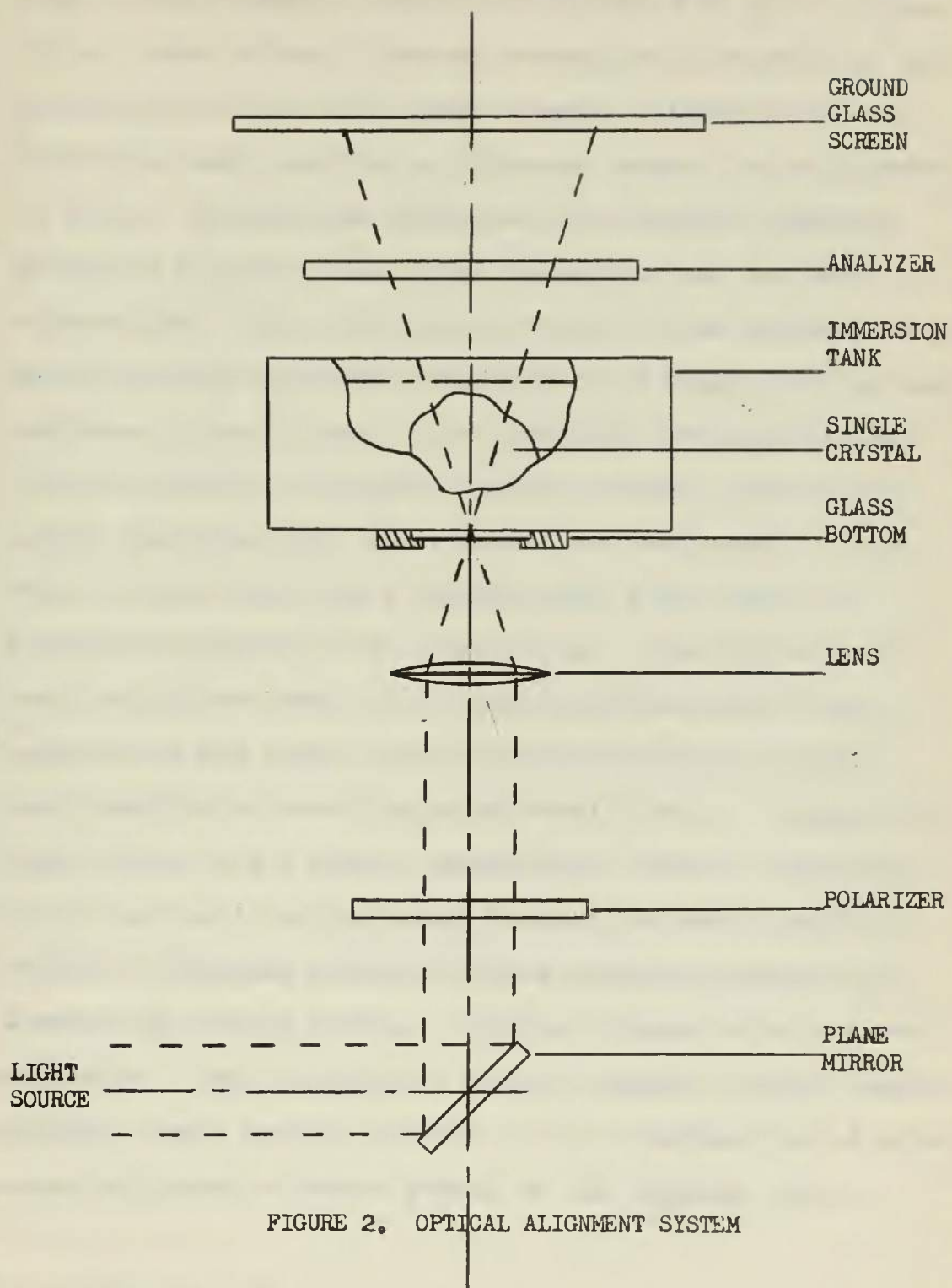
PLANE
MIRROR

LIGHT
SOURCE

FIGURE 2. OPTICAL ALIGNMENT SYSTEM

goniometer and melting new faces.

After alignment of the crystal to the desired orientation, it was necessary that it be reshaped to fit a cylindrical brass holder. This was accomplished by melting the periphery of the crystal with a metal cylinder until its dimensions were such as to allow the holder to be slipped in place. During this operation, the crystal remained frozen to a glass slide, thus preserving the desired orientation. With the crystal frozen in the holder, the end faces were finished until they were flush with the end surfaces of the holder. This finishing process involved initial melting, utilizing a metal surface, followed by smooth finishing with emery paper and bond paper. Both ends of the crystal were covered with $\frac{1}{4}$-mil mylar to protect the crystal from sublimation. Clear mylar was used on the free end, with aluminum-coated mylar being employed on the other end since this covering served a dual function of providing electrical contact between the metal holder and a quartz transducer. Several substances were tried as a bonding agent between the mylar and the crystal, including glycerol, Nonaq stopcock grease, and Dow-Corning vacuum grease. Glycerol proved to be the most effective. The transducers, $\frac{1}{2}$-inch diameter, X-cut quartz crystals, were bonded directly to the aluminum-coated mylar, employing Nonaq stopcock grease as the bonding agent.

3. Crystal pulsing.

Two arrangements for holding electrodes against the

transducers were used, the first of which was that used in prior work by Heseltine and Elliott. It featured a spring-loaded electrode, connected through a coaxial cable to a pulsed oscillator. With this assembly, the quartz transducers were bonded directly to the mylar seal covering the face of the crystal. The measurement of absorption at different frequencies required replacement of the transducers. The pressure between electrode and transducer could be varied by adjustment of holding nuts threaded onto the mounting rods of the assembly. The second assembly, illustrated in Figure 3, was devised and fabricated primarily to minimize any effects of non-parallelism between the electrode, transducer, and crystal. Its use also insured a uniform pressure between electrode and transducer, although there is no indication that any such pressure variations would effect acoustic absorption in the crystal. This holder utilized a fixed electrode upon which the transducer was directly mounted and covered with a mylar seal. Three such assemblies, each with a transducer of a different frequency, provided permanent pulsing assemblies upon which sample holders could be fitted. Investigations at different frequencies involved simply the transfer of a sample holder from one assembly to another.

A comparison of data taken on a single crystal using the different holders indicated that non-parallelism, if present due to assembly configuration, was of negligible magnitude and had no effect on measured absorption. Consequently, data
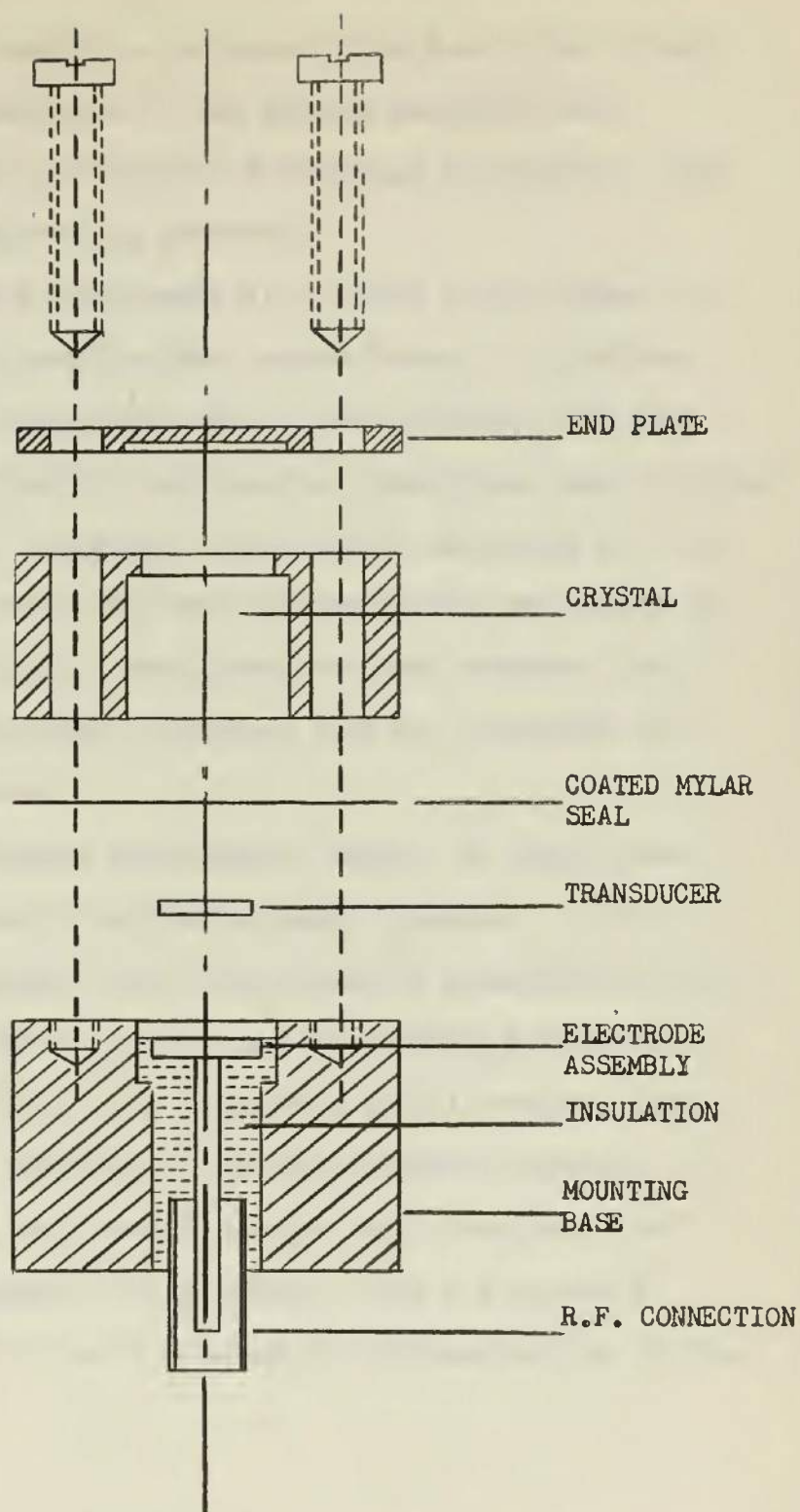
FIGURE 3. SAMPLE-MOUNTING ASSEMBLY

was combined for analysis purposes from test runs using either of the assemblies. The second assembly was preferred because it simplified mounting procedures and expedited the data-taking process.

The transducers used were all of the X-cut type for the generation of longitudinal sound waves. A limited number of Y-cut transducers were also employed for the purpose of investigating ultrasonic absorption due to shear wave propagation. However, the results obtained for the latter type wave were not very accurate due primarily to problems in achieving a satisfactory bond between the transducer and specimen, and they are not included in the results reported.

An Arenberg pulsed oscillator, Model PG 650-C, was used as the source of radio-frequency pulses. Return echoes from the sample were subjected to preamplification, after which they were fed through an Arenberg Wide-Band Amplifier, Model 600, to a Tektronic Oscilloscope, Type RM45A, for visual display. The dual channel display characteristic of this oscilloscope made possible the simultaneous display of the signal from a time-mark generator. Figure 4 is a schematic representation of the pulsing circuit.

4. Measurement of absorption.

The signal output from the time-mark generator was fed through an attenuator which allowed adjustments of its amplitude in increments of one decibel and was then displayed

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OSCILLO-SCOPE

WIDE-BAND AMPLIFIER

SAMPLE-MOUNTING ASSEMBLY

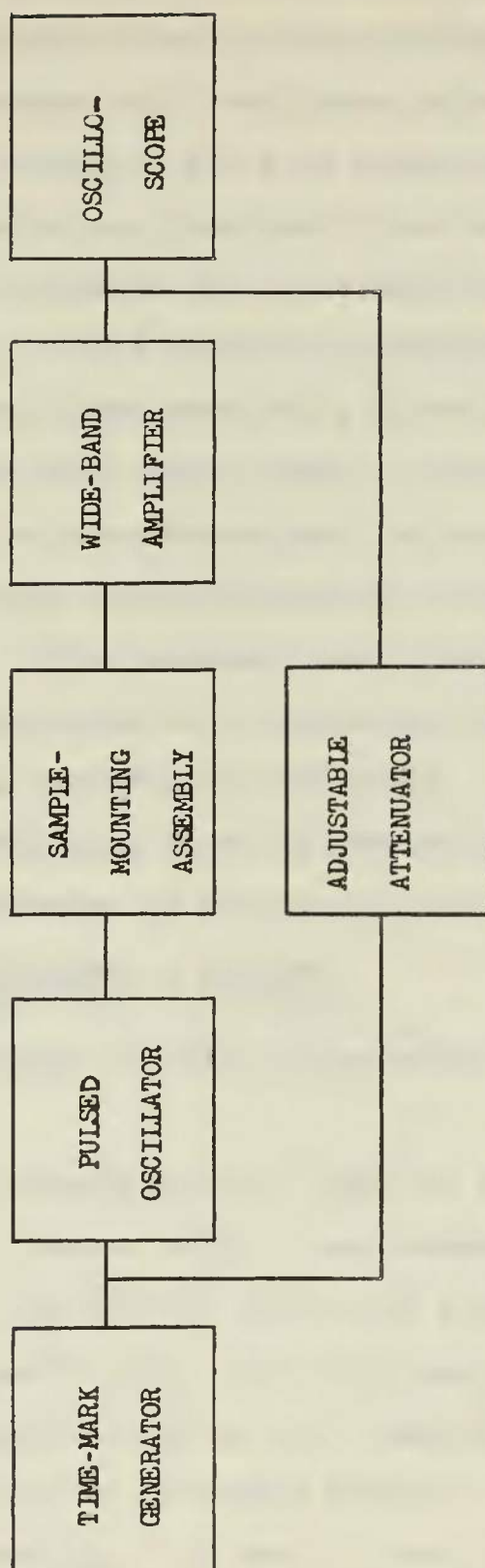ADJUSTABLE ATTENUATOR

PULSED OSCILLATOR

TIME-MARK GENERATOR

FIGURE 4. PULSING CIRCUIT BLOCK DIAGRAM

on the second channel of the oscilloscope. Measurements
were made by superimposing these signals on the echo
train from the crystal sample and adjusting their size
by attenuator changes until amplitude coincidence with the
first echo was attained. For this condition, the attenuator
setting was observed and recorded, after which the
attenuation was increased until the amplitude of the second
echo was matched. The difference in attenuator settings
for these two conditions provided a direct measure of the
absorption in one path length; that is, twice the length
of the sample. It was assumed that the train of sound
waves lost no energy during reflection at the end faces
of the specimen. This procedure was repeated for as many
pairs of echoes as possible - second and third, third and
fourth, etc. The absorption coefficient, α , was computed
in nepers per centimeter from the measured absorption in
decibels per centimeter by the relationship:

$$\alpha = \frac{attenuation(dbs)}{path\ length\ (cm)} \times \frac{1\ neper}{8.68\ dbs}$$

where the path length is twice the measured length of the
sample.

The number of readings which could be obtained on a
given sample was limited only by the attenuation itself
which determined the rate at which echo strength diminished
to immeasurably small size. Both the power of the transmitted
pulse and the amplification of the returned echoes were
adjustable and could be increased within the limits imposed
by amplifier saturation. As many as twenty-one echoes were
measured in a single sample at low frequency (5 mcs).

Several runs were made at each frequency for a given sample, and the average absorption for each such run computed. It was found that the average attenuation thus obtained could be duplicated to within 0.2 decibels per centimeter (approximately 0.02 nepers per centimeter). As time permitted, runs were repeated for additional crystals of like orientation.

The results reported herein are based on measurements made at the ambient refrigerator temperature of 255°K. Since all data were taken at the same temperature, no correction was necessary for thermal variations in sample length. The effects of temperature variation was investigated for one sample orientation at a single frequency. The results obtained in this case were not sufficient in quantity or in accuracy for a quantitative analysis; however, their nature was such as to warrant qualitative discussion.

5. Sources of error.

Several sources of error were present which were considered in the analysis of experimental results.

a. Errors in absorption measurements. Through use of the measurement techniques described, it was possible to measure the attenuation directly to the nearest decibel and, by visual logarithmic interpolation, to estimate it accurately to the nearest one-half decibel. For the sample lengths used - approximately three centimeters - such measurements corresponded to an accuracy of 0.17 decibels per centimeter, or 0.02 nepers per centimeter. Consequently,

the attenuation between two adjacent echoes could be determined to within this accuracy, and it was found that the measured absorption between the same two echoes in a particular crystal could be duplicated within these same limits. Absorption data for a given crystal orientation at a specific frequency were analyzed statistically to determine the "probable error" in an individual measurement based on root-mean-square deviations. This error was computed to vary from 21.5% for [110] crystals at 5 mcs to 4.0% for [011] crystals at 15 mcs. On this basis, the "probable error" in an individual measurement was relatively high at 5 mcs, averaging 19.8% for all orientations; but this error decreased significantly with increasing frequency, averaging 7.4% at 15 mcs. This variation between individual absorption measurements constituted the major source of experimental error.

b. Crystal orientation. Orientation of samples along the b-axis was subject to direct visual confirmation after alignment procedures had been completed; hence, the accuracy of orientation for such crystals could be verified. However, when subsequent rotation to other alignments was necessary, it was not possible to verify the final alignment. Any errors introduced were those incidental to the crystal facing operations, and these were considered to be less than 1° in magnitude. Alignment errors of this size would have a negligible effect on absorption determinations.

c. Length of samples. It was impossible to directly measure the crystal length after it had been mounted without the risk of defacing it. Consequently, the length of the sample was assumed to be approximately that of the holder, which could be determined to within ⌀0.0001 centimeter by direct micrometer measurement. Although the accuracy with which the face of the crystal could be brought into coincidence with the end of the holder could only be estimated, the error was certainly no greater than ⌀0.01 centimeter. This is less than one percent of the sample length and would thus introduce an insignificant error in the computed absorption.

d. Non-parallelism. Any non-parallelism between the crystal faces, or between the electrode, transducer, and crystal face would have introduced errors in observed absorption. However, such lack of alignment was always evidenced by poor quality in the observed echo train, and it was therefore immediately evident when such a condition existed. Corrections were made, when possible, by resurfacing the crystal or by adjustment of the sample-mounting assembly. When the condition could not be eliminated, it was necessary that the sample be discarded. This source of error thus did not contribute to the results obtained.

e. Dispersion and diffraction. The effects of dispersion were insignificant for samples of the lengths used in this work (5). However, diffraction effects were of possible consequence, particularly at the lower frequencies. Therefore, the absorption coefficients determined were

corrected for diffraction attenuation. The magnitudes of
such corrections will be noted in connection with the
presentation of quantitative results.

The results obtained are considered to be accurate
within the limits of the accuracy of the absorption
measurements themselves, all other errors being of
insignificant magnitude. In this respect, it is to be
noted that the "probable error" discussed is a statistical
error in an <u>individual measurement</u> and not an error in the
<u>mean</u> value of absorption for a particular crystal orientat-
ion. The mean constitutes the "best" value, and its computed
error is no greater than 6.0%, averaging 2.9% for all
orientations.

6. Results.

The results of absorption coefficient determinations are
presented in tabular and graphical form on pages 18 and
19. The tabulated data includes the number of different
crystals of each orientation used as samples and the number
of measurements taken at each frequency. The absorption
coefficients listed are mean values for the corresponding
number of measurements made. The computed statistical error
in the mean value is also tabulated.

The tabulated coefficients have been corrected for
diffraction effects by application of the approximate
relationship for diffraction attenuation of one decibel per
a^2/λ , where a is the transducer radius and λ is the wave-
length of the propagated sound (9). Since the velocity of

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ABSORPTION COEFFICIENTS
(nepers/centimeter)

Crystal orientation	a 100			b 010			c 001		
Frequency, megacycles	5	10	15	5	10	15	5	10	15
Samples tested	3	3	2	1	2	1	2	3	2
Measurements made	52	41	13	8	36	7	35	27	13
Absorption coefficient, cm^{-1}	.179	.341	.581	.067	.253	.518	.205	.446	.627
Error, cm^{-1}	.005 2.8%	.008 2.4%	.016 2.8%	.004 6.0%	.004 1.6%	.009 1.7%	.007 3.4%	.012 2.5%	.002 0.3%

Crystal orientation	110			101			011		
Frequency, megacycles	5	10	15	5	10	15	5	10	15
Samples tested	1	1	1	1	1	1	2	1	1
Measurements made	76	49	12	19	9	10	84	8	12
Absorption coefficient, cm^{-1}	.065	.384	.674	.114	.209	.320	.161	.227	.506
Error, cm^{-1}	.002 3.1%	.007 1.8%	.023 3.4%	.006 5.3%	.006 2.9%	.009 2.8%	.004 2.5%	.013 5.7%	.006 1.2%

ABSORPTION — FREQUENCY DEPENDENCE
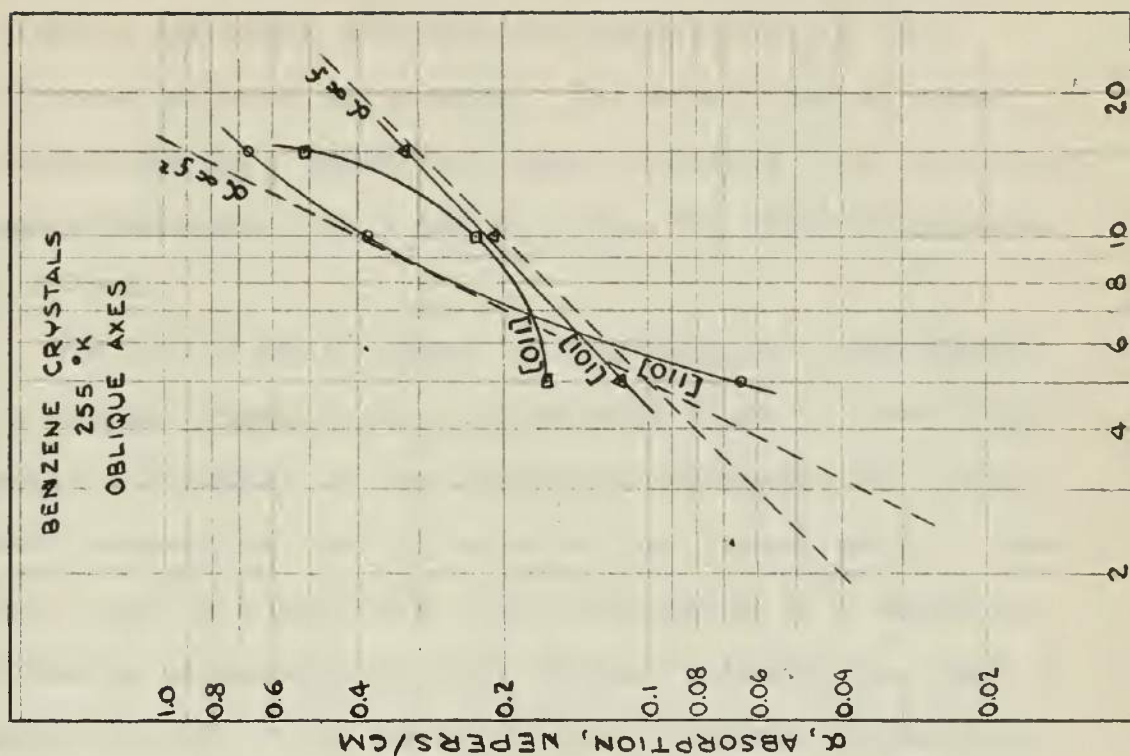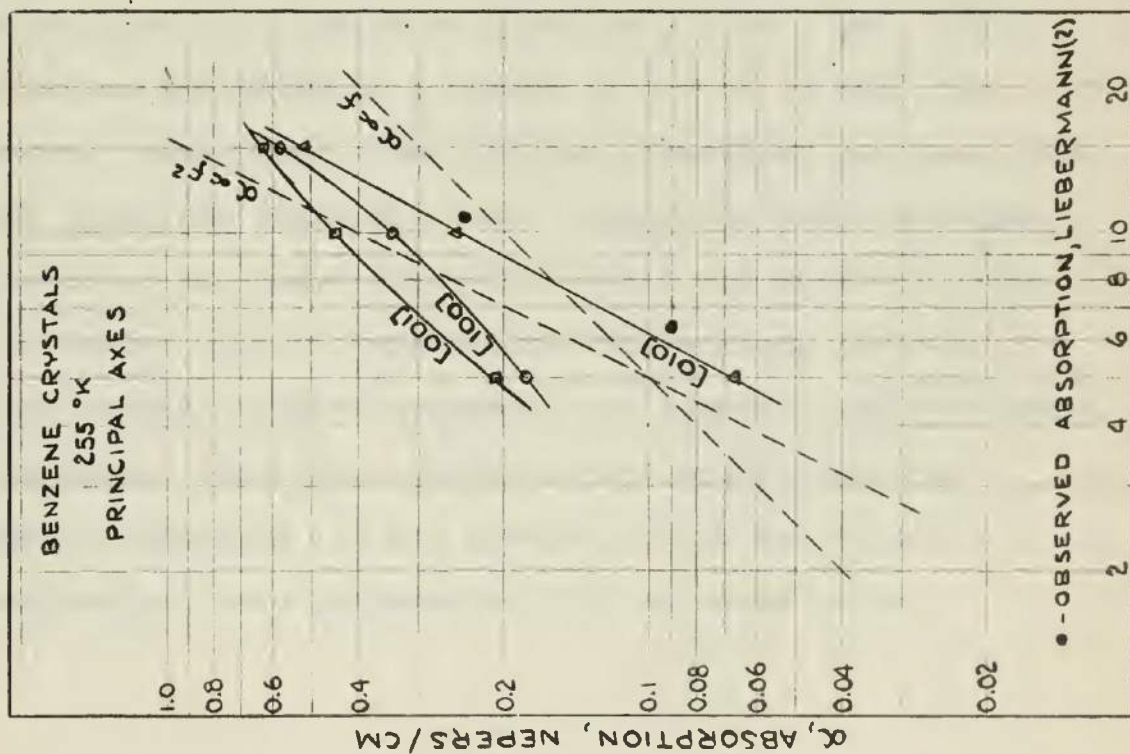
FIGURE 5

propagation varies slightly with crystal orientation, the wavelength exhibits a similar variation which results in slightly different diffraction corrections for the different crystal alignments. The magnitudes of these corrections were relatively small, varying from .013-.016 nepers/centimeter at 5 mcs to .004-.005 nepers/centimeter at 15 mcs.

Due to the small number of frequencies investigated, the curves presented may not be sufficient to specifically define the nature of the frequency dependence of ultra- sonic absorption for all orientations investigated. How- ever, they do constitute valid indication of a definite variation of absorption with crystal orientation, both in magnitude and in the nature of the frequency dependence. The magnitude of the absorption coefficients varied with orientation by a factor as great as 3.4 at 5 mcs. This variation decreased to a factor of 2.0 at 15 mcs. In general, absorption along oblique directions was less than that along the principal axes. Among the principal axes, absorption was least along the b-axis and greatest along the c-axis, with an intermediate value along the a-axis. These results compare favorably, as concerns their relative magnitudes, with known values of the coefficients of thermal expansion (7) and inversely with the velocity of longitudinal wave propagation (3), as shown below.
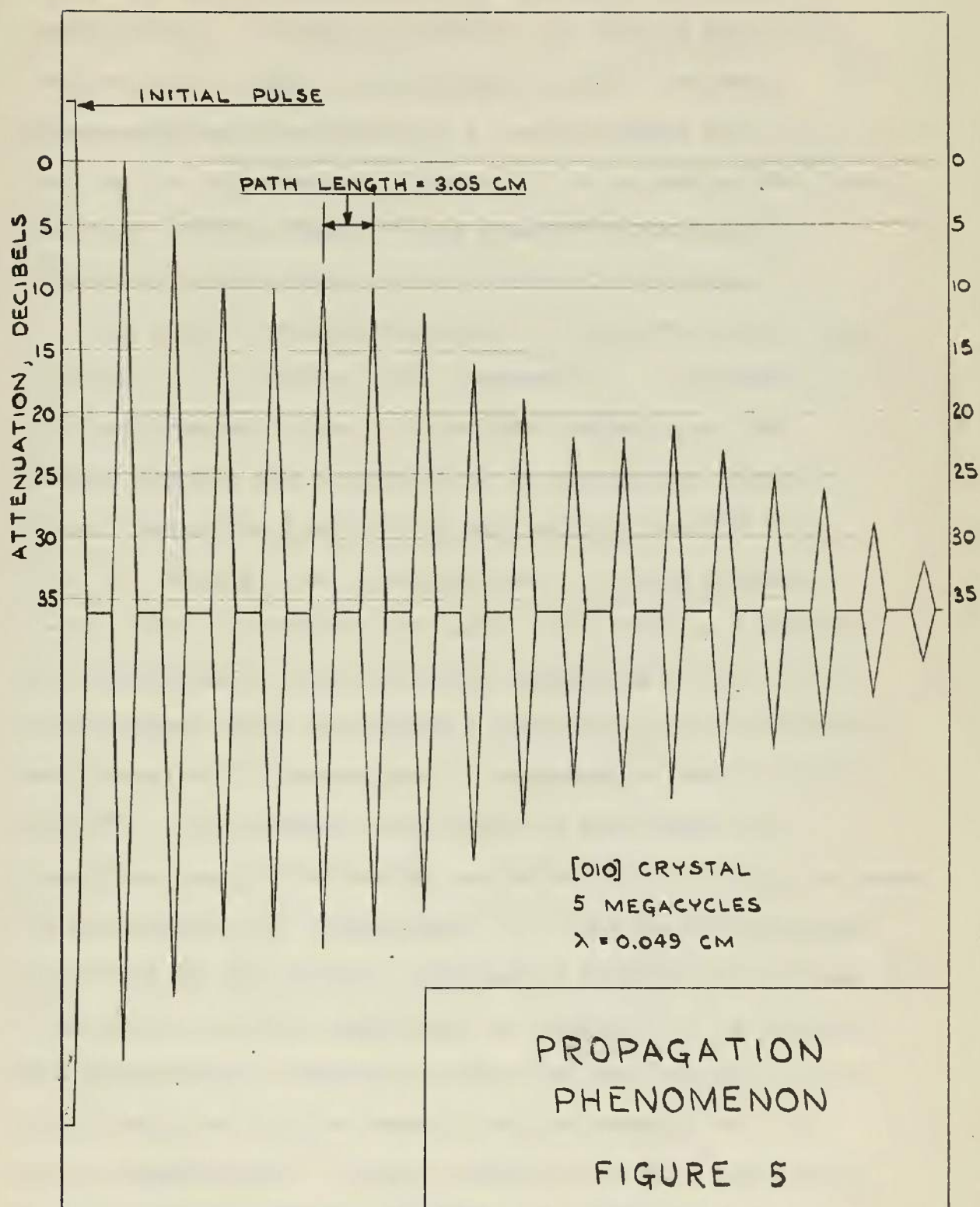
	Absorption coefficient. $(cm^{-1}; 10\ mcs)$	Coefficient of thermal expansion. $(\times 10^{-6}/^\circ C)$	Velocity of longitudinal wave propagation. (meters/second)
a	.348	119	2378
b	.260	106	2447
c	.453	221	2325

The increase of absorption was observed to vary accurately with the square of the frequency, as predicted by the resonance absorption theory, for only one crystal orientation, [010]. Along other directions, there were no simple, consistent variations with frequency. For one orientation, [101] , the frequency dependence was approximately linear. In other cases, the absorption varied in a manner which was intermediate between a linear and a frequency-squared dependence. For such orientations, the frequency dependence varied with the frequency itself.

In the course of obtaining data for the results tabulated and discussed, two unexplained phenomena were encountered. First, at low frequency (5 mcs), an unaccountable periodic variation in echo amplitude was observed. The attenuation was similar in appearance to that expected from diffraction effects. However, the magnitude of the effect and the characteristics of its periodic variations could not be accounted for by simple diffraction theory. Oscilloscope photographs of this propagation behavior were, unfortunately, not of suitable quality for reproduction; however, the observed absorption in a b-axis crystal at 5 mcs is indicated schematically in Fig. 5. The path

INITIAL PULSE

PATH LENGTH = 3.05 CM

ATTENUATION, DECIBELS

[010] CRYSTAL
5 MEGACYCLES
λ = 0.049 CM
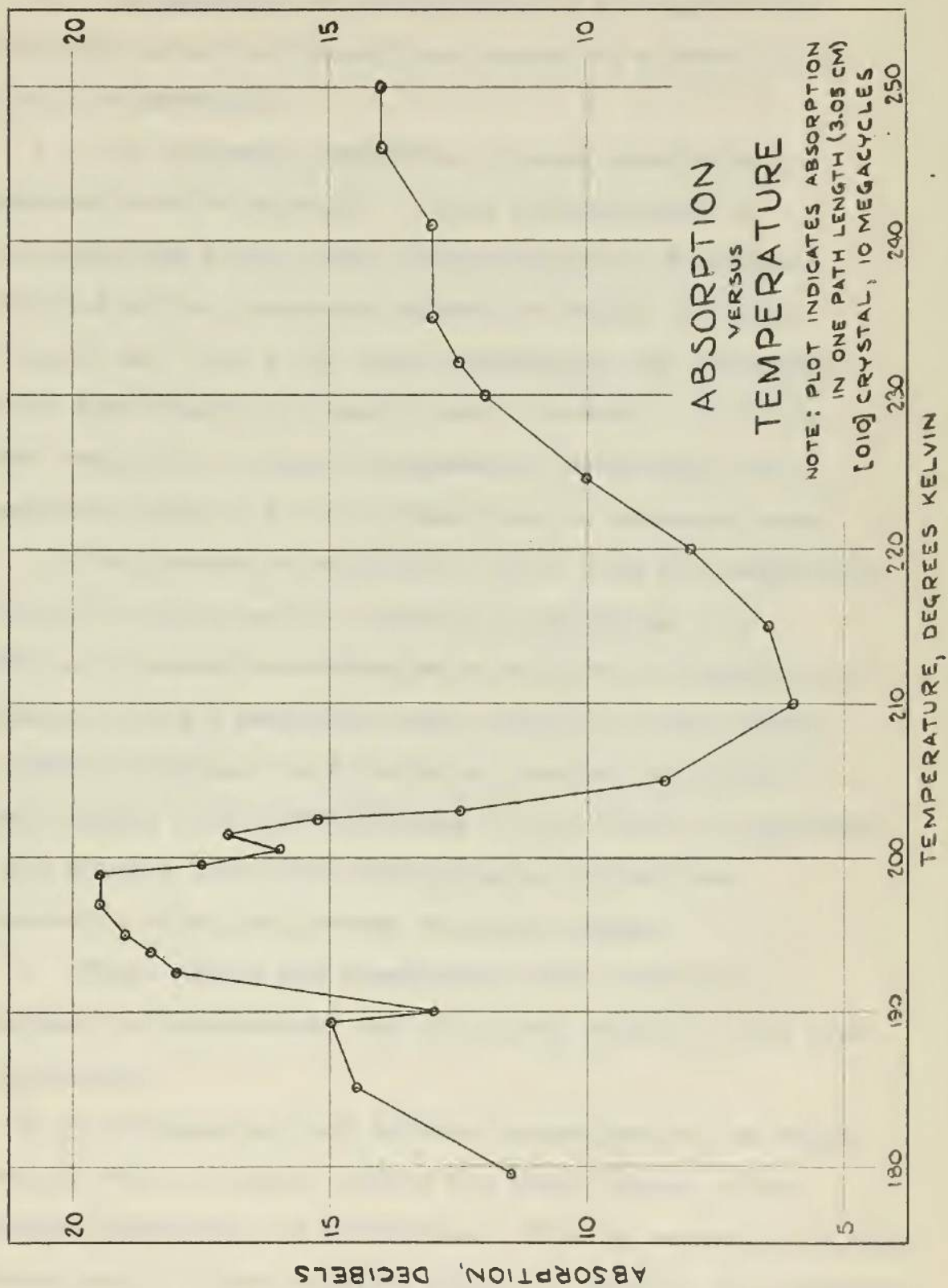
PROPAGATION
PHENOMENON

FIGURE 5

length in this crystal was 3.05 centimeters, and for this frequency and orientation the wavelength was 0.049 centimeters. A similar behavior, of varying magnitude, was noted for other orientations at this frequency. The phenomenon was observable to a lesser extent at 10 mcs, but was not apparent at 15 mcs. It is probable that this behavior was the cause of the greater variations in individual measurements at the lower frequencies.

The second unexplained aspect of observed absorption involved its variation with temperature. Incidental to the measurements taken at constant temperature, one b-axis crystal was subjected to an absorption study at 10 mcs while the temperature was varied from 250 ^{O}K to 180 ^{O}K. During this investigation, a $\frac{1}{4}$-inch diameter, X-cut quartz transducer was used. The results indicated an unaccountable variation with temperature which is in disagreement with Liebermann's conclusions that absorption was practically independent of temperature down to -58 ^{O}C (215 ^{O}K). The results also differed with those of Heseltine and Elliott which indicated only a slight decrease in absorption with temperature. The observed temperature variation of absorption is presented graphically on page 24, from which a marked dependency on temperature is apparent. The experimental results in this case are inconclusive at best, and time did not permit further pursuit of this type investigation. Further research on this particular aspect of the absorption behavior is warranted.

ABSORPTION
versus
TEMPERATURE

NOTE: PLOT INDICATES ABSORPTION
IN ONE PATH LENGTH (3.05 CM)
[010] CRYSTAL, 10 MEGACYCLES

TEMPERATURE, DEGREES KELVIN

ABSORPTION, DECIBELS

7. Conclusions.

From the results obtained, it is concluded that:

a. The magnitude of the absorption of longitudinal ultrasonic waves in crystalline benzene is a function of crystal orientation.

b. The frequency dependence of such absorption is dependent upon orientation. Along certain axes, in particular the b-axis, the frequency-squared dependence predicted by the resonance absorption theory is valid, or nearly so. For a few other alignments, the absorption varies approximately linearly with frequency. In still other cases, the frequency dependence varies with the frequency itself and lies between the two extremes noted.

c. The resonance absorption theory does not completely account for the observed ultrasonic absorption. An additional absorption mechanism is present and possibly is dominant. Such a mechanism must account for the effects of crystal alignment on absorption, and its definition might require a further knowledge of the relative magnitudes of the binding forces and corresponding vibrational frequencies along the various molecular planes.

d. There exists the possibility that absorption is dependent on temperature, and additional study in this area is warranted.

It is recommended that further investigations be undertaken in order to better define the exact nature of the frequency dependence of absorption. This is essential to the postulation of a more complete theory to explain the mechanism of absorption in a molecular crystal.
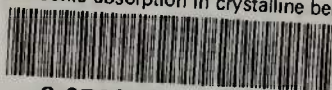
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