Ultrasonic manometers for low and medium vacua under development at the National Bureau of Standards*

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The first part of this paper describes the use of ultrasonic interferometry for the measurement of the lengths of manometer columns. The major known sources of errors of manometers are then analyzed, and design criteria are developed which reduce these errors below set limits. Finally, two ultrasonic manometers now being developed at the National Bureau of Standards for measurements in the low and medium vacuum ranges are described. The ultrasonic mercury manometer, with a range of 13 kPa, has been operated with a resolution of 1.4 mPa.

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I. INTRODUCTION

A number of scientific and technical applications require increasingly accurate and reproducible vacuum measurements, particularly in the low and medium vacuum range from about 10^4 down to about 10^{-4} Pa (1 Torr = 133.322 Pa; 1 Pa = 1 N/m^2). One of the few instruments available for use as standards in this range is the micrometer point manometer, developed by Thomas and Cross[1] and used as the basis for calibration work at the National Bureau of Standards by Ruthberg[2] and at a number of other laboratories. Manometers, as pressure standards, offer such advantages as their ability to measure pressure independently of gas species, their construction and operation are relatively straightforward, and the results are converted to a measured pressure using only the most elementary theory. However, micrometer point manometers are limited to the high-pressure part of the vacuum range by the resolution of the length measurement of about 10^{-6} m for an oil manometer[2] and by the difficulty in maintaining the mechanical and thermal stability necessary for low-pressure manometry with a manually operated instrument. In addition, the difficulty in precisely locating the liquid surface with the micrometer points and holding the pressure stable while a skilled operator performs the measurement makes these instruments awkward to use.

The development of a sensitive pulsed ultrasonic interferometer[3] and its application to the measurement of liquid-column lengths has made possible vacuum manometry with greatly improved accuracy and resolution. The interferometer attains a length resolution of about 10^{-8} m, automatically makes a complete measurement in 2 s, and does not disturb the mechanical or thermal stability of the manometer. Ultrasonic mercury and oil manometers are under development at the National Bureau of Standards and may form the basis of a vacuum-gauge calibration service. This paper briefly reviews the ultrasonic interferometer, discusses the factors important for accurate low-pressure manometry, with special emphasis on the limiting factors at the lowest pressures, and describes the NBS ultrasonic manometers.

II. ULTRASONIC INTERFEROMETER

In an ultrasonic manometer the measurements of column lengths are made automatically, in terms of wavelengths of the ultrasonic signal, with the help of a fringe-counting interferometer.[4] This measurement, which requires the knowledge of the speed of sound in the particular medium, works as follows: A short (20 μs) pulse with a carrier frequency of 10 MHz is sent to the transducer mounted under the bottom closure of the liquid column (Fig. 1). The transducer transforms the electrical signal into the mechanical analog, and a short ultrasonic wavetrain is launched vertically upwards into the mercury. This signal is reflected at the surface of the liquid column and travels back to the transducer. Here a very small part of the ultrasonic energy is reconverted into an electrical signal. The rest is reflected back into the column. This process continues until all ultrasonic energy is lost from the signal by absorption and diffraction. In a mercury column of a few centimeters length, 20 or more echoes can usually be observed, whereas in oil the number is reduced by at least a factor of four. Figure 2 shows part of such a series of echoes. The echoes received by the transducer are amplified and then phase-sensitive detected in two detectors with a 90° phase shift between their reference signals. The resulting signals are shown in Fig. 3. Each of the pulses represents one echo received from the liquid column. The amplitude of the phase sensitive detected signals is proportional to the amplitude of the received signal and to the phase shift between signal and reference. When the liquid-column length changes, the phase of each received echoes changes also. Specifically, if the length changes monotonically through one-half wavelength of the ultrasonic signal, the amplitude of the first detected pulse will describe a full sine wave cycle as a function of length, since the total path length for the ultrasonic signal will have changed by one wavelength, causing a 2π phase change. Similarly the second pulse will go through two full cycles, the third through three, and so forth. The transducer is pulsed every 10 ms. One must therefore sample the instantaneous value of a chosen echo once every 10 ms and then hold it until

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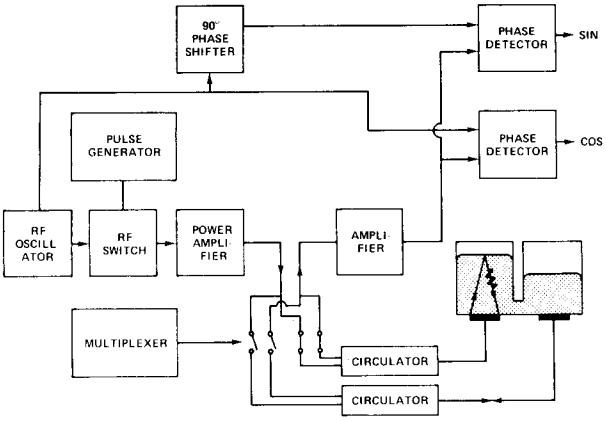

FIG. 1. Block diagram of the ultrasonic interferometer, showing the generation of the transmitted signal, multiplexing to two columns, and amplification and quadrature detection of the received echoes.

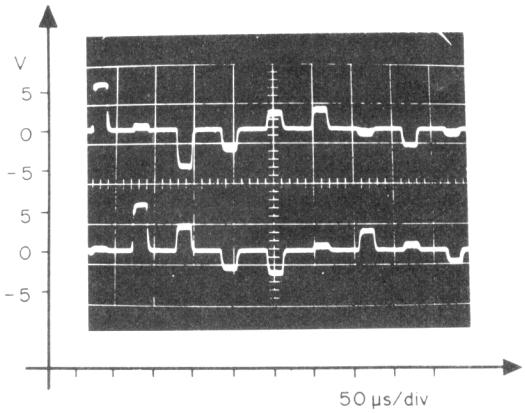

FIG. 3. Dual-channel phase-sensitive-detected echo sequence with 90° phase difference between reference channels.

it is updated 10 ms later. The sampled and held signals are then smoothed with a low-pass filter. The resulting quadrature signals are shown in Fig. 4 for a slowly changing mercury column. These signals are related to the column length L by

$$u_1 = A \cos kL$$
$$u_2 = A \sin kL, \qquad (1)$$

where A is an amplitude factor, $k = 2\pi f/c$, f is the carrier frequency, and c is the speed of sound in the liquid. These signals are connected to an up–down counter, which will count each zero crossing. The counter will also determine from the lead or lag of signal u_1 versus u_2 whether the column is rising or falling and will count accordingly. The change of column length ΔL per count is

$$\Delta L = \lambda/8q, \; q = 1, \cdots, n, \qquad (2)$$

where λ is the wavelength of the ultrasonic signal ($\simeq 150\ \mu$m) and q is the order number of the echo. Using, for example, the fourth echo, a resolution of less than 5 μm per count can be achieved in mercury. In order to further improve the sensitivity, the instantaneous voltages u_1 and u_2 can be measured and the fractional fringe ∂L (see Fig. 4) can be determined from

$$\partial L = (\lambda/4\pi q) \operatorname{arccot} (u_1/u_2). \qquad (3)$$

The lengths of several columns can be measured by multiplexing the interferometer. Multiplexing, operation of the interferometer, and data collection can conveniently be done under the control of a microprocessor.

Liquids generally used for vacuum manometry are mercury and diffusion-pump oils. Diffusion-pump oils offer the dual advantage of reduced vapor pressure and lower density. Among the oils investigated, only di-2-ethyl hexyl phthalate and di-2-ethyl hexyl sebacate(di-octyl sebacate), hereafter referred to as DEHS, have an ultrasonic attenuation sufficiently low for use with an ultrasonic interferometer. The speed of sound in mercury is 1449.57 ± 0.1 m/s as determined in this laboratory in an unpublished measurement; another, more accurate, determination is under way. Rather than measuring the speed of sound and density of diffusion-pump oil, the oil manometer will be calibrated against the mercury manometer.

It should be noted that the speed of sound in liquids is temperature dependent, typical values for $\Delta c/c$ being $-3 \times 10^{-4}\ \mathrm{K}^{-1}$ for mercury and $(-2 \text{ to } -4) \times 10^{-3}\ \mathrm{K}^{-1}$ for oils. This must be taken into account in evaluating the temperature effects discussed later in this paper.

The pulsed ultrasonic interferometer will only work if the pulses are short enough so that the returning echoes do not overlap one another. Furthermore, accurate measurements require that the phase of the returned echoes be measured in a steady-state portion of the pulse, i.e., away from the leading or trailing edge of the pulse. This requires a pulse with fast rise

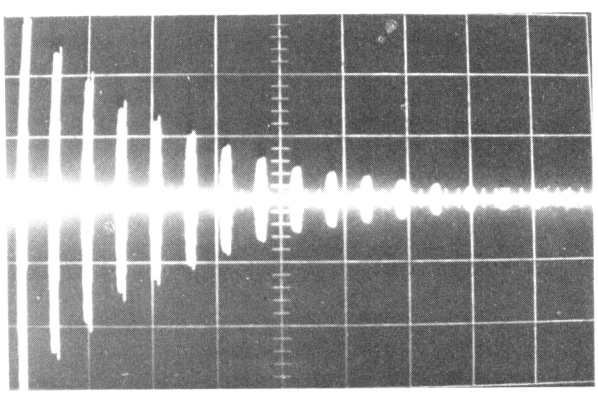

FIG. 2. A series of echoes from the surface of a mercury column.

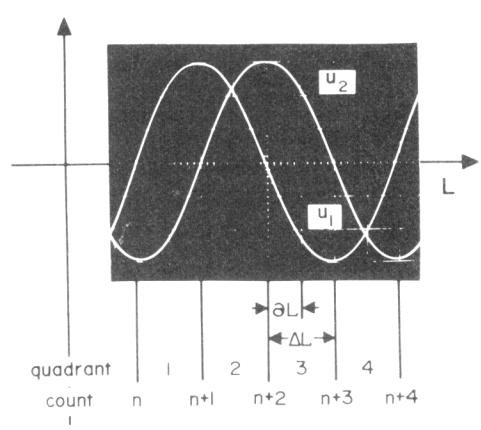

FIG. 4. Quadrature signals as a function of column length.

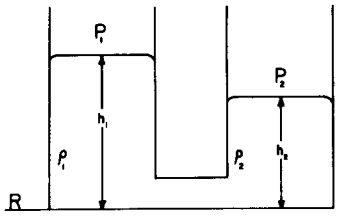

FIG. 5. Idealized U-tube manometer. With pressures P_1 and P_2 applied to the tubes, a height difference $h_1 - h_2$ results. Temperature gradients will cause the average densities in the columns to differ.

and fall times, which can be achieved only if the ultrasonic signal is well coupled into the liquid. Insulating liquids, such as oils, present no problems since the transducer can be mounted in direct contact with the liquid. However, mercury would short out the transducer, which therefore must be mounted on a buffer material. Coupling between the buffer material and the mercury is determined by the power-reflection coefficient at the interface,

$$R = \left[\frac{\rho_1 c_1 - \rho_2 c_2}{\rho_1 c_1 + \rho_2 c_2}\right]^2, \tag{4}$$

where ρ_1, ρ_2 and c_1, c_2 are the density and speed of sound in media 1 and 2, respectively. Two construction materials that are chemically compatible with mercury and have a good acoustic impedance match are beryllium ($R = 0.007$) and titanium ($R = 0.024$). We have not been able to obtain the theoretical coupling coefficient for beryllium. However, we have obtained good results with titanium when it is carefully cleaned and prepared.

III. LIQUID MANOMETERS

Many design considerations incorporated in the mercury and oil manometers to be described in Sec. IV were prompted by the attempt to minimize errors. In the following general analysis of manometer errors, the specific sources of errors that were considered are discussed and the measures taken to reduce these errors in a specific type of manometer are described. Figure 5 illustrates an idealized U-tube liquid manometer. Pressures P_1 and P_2 are applied to the liquid surfaces and the differential pressure is expressed by

$$P_2 - P_1 = g\rho(h_1 - h_2), \tag{5}$$

where ρ is the average liquid density, g is the acceleration of gravity at the location of the measurement, and h_1 and h_2 are the column heights measured from a reference plane R.

In order to properly discuss the sources of error in low-pressure manometry, it is necessary to refine this picture somewhat and express the differential pressure as

$$P_2 - P_1 = g(\rho_1 h_1 - \rho_2 h_2), \tag{6}$$

where ρ_1 and ρ_2 are now the average liquid densities in the respective columns. Contributions of these quantities to the uncertainty of the measured differential pressure will be discussed, along with the contribution of the reference pressure P_1 to the uncertainty of the absolute pressure P_2. These uncertainties will be divided into those proportional to the measured differential pressure, contributed by the uncertainties in the values of gravity, density, and height, and those independent of the pressure, contributed by the uncertainties in the values of density, height, and reference pressure. The magnitudes of the proportional errors are linearly dependent on pressure, and consequently lead to constant relative errors.

A. Errors proportional to pressure

1. Gravity

The value of the local acceleration of gravity and its error assessment have to be made at the site of the measurment. The relative uncertainty in g contributes the same relative uncertainty to the differential pressure measurement.

2. Density of manometric fluid

The density of mercury has been determined with an uncertainty of 1 ppm, and probably varies by only a few parts per million between different lots of virgin material.[5] Since it is an easily purified element and does not dissolve gases, the density is quite reproducible. The coefficient of thermal expansion is well known ($181 \pm 0.05 \times 10^{-6}$ K^{-1} at 20° C)[6] as a function of temperature, so it is possible to accurately determine the density of mercury for a given temperature. The density of diffusion-pump oils is generally not very well known, may vary significantly from lot to lot, and depends in an unknown way on absorbed gases. However, the density and thermal expansion of a given sample can be determined by conventional weighting techniques,[7] density float measurements,[8] or intercomparing mercury and oil manometers. The last technique will be applied to the fluids used in the ultrasonic mercury and oil manometers at NBS to obtain a value and temperature coefficient for the density and speed of sound product for DEHS and to study the effect of absorbed gases. The low-pressure compressibility of mercury is 4×10^{-11} Pa^{-1}, and for oils it is of the order of 10^{-9} Pa^{-1}. The pressure dependence of the density can therefore be safely neglected for vacuum manometry.

3. Determination of height

The uncertainty proportional to pressure introduced by the height determination depends on the proportional uncertainty of the length measurement and the orientation of the measurement axis with respect to the vertical. The ultrasonic interferometer measures column length in terms of the ultrasonic wavelength $\lambda = c/f$. Since the frequency f can be measured with negligible error, only the speed of sound c will contribute to the uncertainty in the wavelength. We are presently determining the speed of sound in mercury at 10 MHz by simultaneously measuring the change in transit time through a mercury column with an ultrasonic interferometer[3] and the change of path length within the same column with a frequency-stabilized CO_2 laser interferometer.[9] The product of density and speed of sound of diffusion-pump oil as a function of temperature will be determined by calibrating an oil manometer against a mercury manometer.

An additional proportional uncertainty can be introduced by elastic mechanical distortion of the length-measuring apparatus caused by the applied pressure. For example, in the

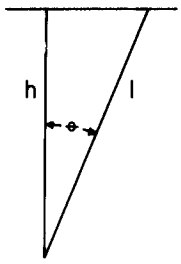

FIG. 6. Illustration of the cosine error; l is the measured length, h is the height.

ultrasonic mercury manometer the baseplate will bow with the applied pressure, causing a measured proportional error of 0.2 ppm. Where these effects can be identified and calculated or measured they can be corrected, as long as the distortion is elastic. Where possible, the manometer should be designed to be so rigid and stable as to effectively eliminate these effects.

If the measurement axis is displaced from the vertical by an angle θ, as in Fig. 6, the measured length l differs from the actual height h by the factor $\cos\theta$, that is

$$h = l \cos\theta.$$

If θ is small, $h \approx l(1 - \theta^2/2)$, and the error is proportional to θ^2. This cosine or verticality error can be made negligibly small by careful alignment of the manometer. Initial alignment of the ultrasonic manometer is achieved with a sensitive spirit level mounted on a surface designed to be parallel to the surfaces of the ultrasonic transducers. Final alignment is achieved by leveling the manometer for maximum strength of the ultrasonic signal reflected from the horizontal liquid surfaces. With mercury columns this latter technique has a sensitivity of about 10^{-4} rad.

B. Errors independent of pressure

Pressure-independent errors in the differential pressure include random errors caused by thermal, mechanical, and electronic instabilities, and systematic errors caused by nonlinearities and misalignment of the phase-sensitive detectors. The value of the reference pressure adds an additional uncertainty to a measurement of an absolute pressure. The accuracy of vacuum-manometry measurements is largely limited by the pressure-independent errors. A number of these errors can be evaluated by monitoring the stability and repeatability of the indicated zero differential pressure.

1. Thermal instabilities

In the ultrasonic manometer, the heights can be considered as the product of the ultrasonic transit time and the speed of sound in the liquid column. Equation (6) can be rewritten as

$$P_2 = g(\rho_1 t_1 c_1 - \rho_2 t_2 c_2) + P_1 \tag{7}$$

where c_1 and c_2 are the temperature-dependent speeds of sound in columns 1 and 2 and t_1 and t_2 are the measured transit times for the two columns. If α_d and α_c are the temperature coefficients for the density and speed of sound, we can write

$$\begin{aligned} P_2 &= g[\rho_1 t_1 c_1 - \rho_1(1 + \alpha_d \Delta T) t_2 c_1 (1 + \alpha_c \Delta T)] + P_1 \\ &\simeq g\rho_1 c_1 \{t_1 - t_2[1 + (\alpha_d + \alpha_c)\Delta T]\} + P_1, \end{aligned} \tag{8}$$

where ΔT is the temperature difference between columns 1 and 2. For an ultrasonic mercury manometer with $\alpha_d + \alpha_c = 5 \times 10^{-4}\ \mathrm{K}^{-1}$ and a column length of 100 mm, an undetected change in the temperature difference of 2 mK will cause an error in the indicated differential pressure of about 10^{-2} Pa. The quantity $\alpha_d + \alpha_c$ is about $4 \times 10^{-3}\ \mathrm{K}^{-1}$ for an ultrasonic manometer using DEHS. Therefore, for a column length of 10 mm, a change in the temperature gradient of 0.2 mK will cause an indicated change in the differential pressure of 10^{-4} Pa. Manometers must be designed to achieve this stability over the period of hours typically required to perform a calibration. This is most easily accomplished by designing the manometer to minimize the temperature gradient. If ΔT can be made so small that $(\alpha_d + \alpha_c)\Delta T$ is less than the allowed proportional uncertainty, Eq. (8) can be reduced to

$$\begin{aligned} P_2 &= g\rho h + P_1 \\ &= g\rho c(t_1 - t_2) + P_1, \end{aligned} \tag{9}$$

where g and ρ are again average values for the entire manometer. The effectiveness of the precautions designed to prevent temperature gradients can be assessed, for if ΔT does vary it will appear as changes in the indicated zero differential pressure.

2. Mechanical instabilities

(*a*) *Noise in the determination of length.* Random noise in the length measurement will contribute an uncertainty independent of the pressure. In the ultrasonic manometer this will be of electrical and mechanical origin. An example of the latter is the disturbances on the mercury surface.

The random-length noise contributions cannot be separately assessed but since they contribute to the instability of the indicated zero differential pressure, their upper bound can be experimentally assessed.

(*b*) *Tilt.* We have previously discussed the proportional

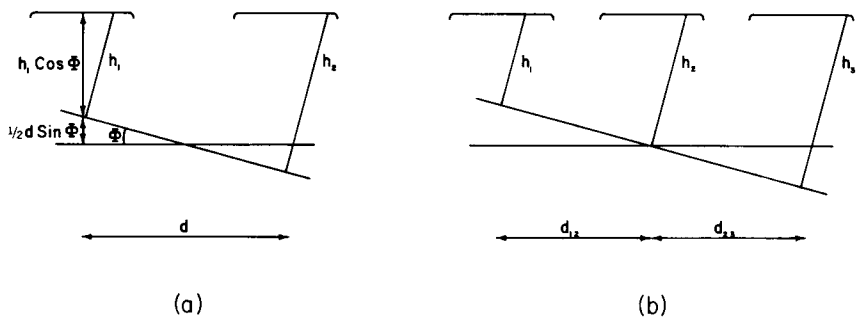

FIG. 7 (a). Illustration of tilt error. Tilting the manometer by an angle ϕ causes an indicated height difference, at zero pressure difference, of $h_1 - h_2 = d \sin\phi$. (b). A three-tube manometer compensates for the tilt, to first order.

uncertainty caused by misalignment of the measurement axis to the vertical. After obtaining a zero pressure reading, small changes in this angle during the measurement caused by tilt of the manometer about a horizontal axis perpendicular to the lines between the measuring points will contribute an additional error independent of pressure. In Fig. 7(a) the error in the measured height difference for a tilt ϕ is

$$\epsilon(h_1 - h_2) = h_1 \cos\phi + \tfrac{1}{2}d \sin\phi - h_1 - [h_2 \cos\phi - \tfrac{1}{2}d \sin\phi - h_2] = (h_2 - h_1)(1 - \cos\phi) + d \sin\phi.$$

For vacuum manometry, the distance between the measuring points d is of the same order or greater than the column heights, and the tilt angles are small, so that $1 - \cos\phi \approx 0$ and the tilt or sine error is

$$\epsilon(h_1 - h_2) \simeq d \sin\phi \simeq d\phi.$$

The illustration is for $P_1 = P_2$ but the resultant error will be the same, independent of the pressure at which the tilt occurs. It should be noted that, in general, the tilt is so small that it causes a negligible change in the verticality or cosine error, which depends on the second power of the angle. However, the tilt is large enough so that the associated error, which is linear with the tilt, cannot be neglected in vacuum manometry. The separation between measuring points is typically of the order of 10 cm, and the tilt varies upwards from a level of about a microradian. Thus, the resultant changes in indicated height difference will be much greater than the required resolution.

The effects of tilt can be greatly reduced with a three-tube manometer as illustrated in Fig. 7(b). The same pressure is applied to the outer two columns and, if the measuring points are equally spaced about the center column ($d_{12} = d_{23}$), a tilt about the horizontal axis perpendicular to the line between the outer columns will cause equal and opposite changes in the indicated heights of the outer columns, so that their average height is unaffected by the tilt. The differential height is then $\Delta h = \tfrac{1}{2}(h_1 + h_3) - h_2$. If $d_{12} \neq d_{23}$, the error ϵ in the measured differential height will be

$$\epsilon \simeq (d_{12} - d_{23}) \sin\phi.$$

However, the change in the differential length of the outer columns, $\delta(h_1 - h_3)$, can be used as a tilt meter,

$$\sin\phi \simeq \frac{\delta(h_1 - h_3)}{d_{12} + d_{23}}$$

so that

$$\epsilon \simeq \left(\frac{d_{12} - d_{23}}{d_{12} + d_{23}}\right) \delta(h_1 - h_3).$$

The constant $k = (d_{12} - d_{23})/(d_{12} + d_{23})$ can be evaluated by intentionally tilting the manometer, with zero differential pressure applied, through an angle larger than the maximum expected tilt and measuring h_1', h_2', h_3'. Then

$$k = \frac{\delta'[\tfrac{1}{2}(h_1' + h_3') - h_2']}{\delta'(h_1' - h_3')}$$

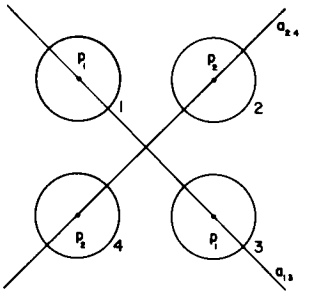

FIG. 8. Layout of a four-tube manometer, showing the diagonal positions of the pressure tubes to which P_1 and P_2 are applied, and the axes about which the manometer is tilted, at zero pressure, to obtain the correction constants k_{13}, k_{24}.

where δ' indicates measured changes in the bracketed quantities caused by the intentional tilt. All subsequent measurements of the indicated height difference are then corrected by $k[\delta(h_1 - h_3)]$, i.e.,

$$\Delta h = \tfrac{1}{2}(h_1 + h_3) - h_2 - k[\delta(h_1 - h_3)].$$

However, if the measuring points for the three columns are not collinear i.e., if the center column is displaced from the line between the outer columns, a tilt about the axis between the outer columns will also cause an indicated differential height change. Since it is not possible in practice to arrange the measuring points to be perfectly collinear, a three-tube manometer cannot eliminate all effects of tilt. This can be done using a four-tube manometer, as illustrated in Fig. 8. The two pairs of tubes can be used as two tilt meters about two different horizontal axes. Any tilt of the manometer can be resolved into tilts about these two axes and can be corrected for in a manner analogous to that discussed for the three-tube manometer. If the manometer is tilted about axis a_{13} connecting columns 1 and 3, and heights $h_1' \cdots h_4'$ are measured, we can determine

$$k_{24} = \frac{\delta'[\tfrac{1}{2}(h_1' + h_3') - \tfrac{1}{2}(h_2' + h_4')}{\delta'(h_2' - h_4')}.$$

Similarly, by tilting about axis a_{24} between columns 2 and 4 and measuring $h_1'', \cdots, h_4''$, we can determine

$$k_{13} = \frac{\delta'[\tfrac{1}{2}(h_1'' + h_3'') - \tfrac{1}{2}(h_2'' + h_4'')]}{\delta'(h_1'' - h_3'')}.$$

The corrected differential heights will then be

$$\Delta h = \tfrac{1}{2}(h_1 + h_3) - \tfrac{1}{2}(h_2 + h_4) - k_{13}\delta(h_1 - h_3) - k_{24}\delta(h_2 - h_4).$$

(*c*) *Mechanical deformation*. Application of pressure may strain parts of the measuring apparatus in a manner that is not completely elastic. As an example, in the ultrasonic mercury manometer, the applied pressure will bow the titanium disks used to couple the ultrasonic signal into the mercury. The elastic deflection will affect all columns equally. Any inelastic behavior will appear as a hysteresis in the measured zero of the manometer. The mechanical structure of the ultrasonic oil manometer is designed so that the possible elastic deflections are less than the desired length resolution. This eliminates the possibility of significant inelastic distortion.

(*d*) *Capillary effects*. The surface of a liquid confined in a vessel is distorted from the horizontal plane it would assume in an infinite pool. This distortion depends on the contact

angle between the liquid and the vessel walls, and on the surface tension of the liquid–gas interface. Since mercury does not wet most substances, this generally results in a depression of the mercury surface; since oil wets most materials the oil meniscus is generally elevated. However, capillary effects are notoriously irreproducible, particularly for mercury. It is not unusual to see inverted as well as normal mercury menisci in either scrupulously clean or dirty manometer tubes. Indeed, the same meniscus may have normal and inverted contact angles at different parts of the liquid–wall–gas contact line. The only solution to this problem is to make the diameter of the manometer tube large enough so that the change in the measured surface position will be less than the desired resolution if the contact angle changes from normal to inverted. Fortunately, the elevation or depression and shape of the center of large diameter menisci are well known[10] and it is not difficult to calculate the maximum height uncertainty, given the tube diameter and area of the meniscus over which the measurement is performed. Oil menisci are much better behaved than mercury menisci but it is prudent to apply the same criterion to the design of an oil manometer.

In the assembled instrument an experimental estimate of an upper limit on capillary effects can be obtained by checking for reproducibility of the zero reading. This will not be definitive, however, as capillary behavior will vary with position in the tubes.

(*e*) *Demodulation error.* In practice, signals u_1 and u_2 in Eq. (3) are not exactly 90° out of phase, which causes a periodic nonlinearity of the measured lengths. If the lengths of the columns change, this nonlinearity will cause an apparent change in the measured differential height. This will contribute to instability of the measured zero differential pressure. Column-length changes can be caused by changes of the average temperature, air diffusing into the liquid columns, and changes in the meniscus volume because of changing contact angles. It may be possible to measure this nonlinearity and correct for it, thus reducing the demodulation error.

3. Reference pressure

Manometers measure only differential pressures. In order to measure an absolute pressure applied to one side of a manometer, the reference pressure on the other side must be known. The reference pressure can never be lower than the vapor pressure of the manometric fluid. In an oil manometer, the vapor pressure may be low enough that the reference pressure can be reduced well below the allowed level of uncertainty. This pressure can be measured with a device such as an ionization gauge. The relatively high vapor pressure of mercury and consequent large temperature dependence require that precautions be taken to avoid cold spots that will reduce the equilibrium vapor pressure and introduce pressure gradients in the vacuum lines.

IV. ULTRASONIC MERCURY MANOMETER

The mercury manometer is shown schematically in Fig. 9. It was designed to have a differential range of about 10^4 Pa. This manometer has been in operation for several months and has achieved a resolution 1.4×10^{-3} Pa. A typical set of zero readings indicating the resolution is shown in Table I.

In order to reduce the tilt error, the manometer is of the three-tube type with the reference pressure applied to the center column and the pressure applied to the two outer columns. The pressure lines to the two outer tubes are externally connected together. The tubes are 7.5-cm-i.d. Pyrex, and coated on the inside with a conductive layer of chromium and nickel, vacuum evaporated from a nichrome wire. The conductive coating is designed to eliminate electrostatic forces between the glass and the mercury surface. This, along with the large tube diameter, minimizes height variations of the center of the mercury surfaces and keeps the capillary depression below the resolution of the instrument. The tubes, mounted 10 cm apart on an 18-mm-thick stainless-steel baseplate, are polished on the ends and sealed to the baseplate and the top flanges with polytetrafluoroethylene or polytrichlorofluoroethylene gaskets. The ultrasonic signal is generated and received by 18-mm-diam, 10-MHz ceramic transducers. These are bonded with a thin layer of epoxy to 6-mm-thick titanium disks, whose upper and lower surfaces are ground plane and parallel, and polished. The disks are securely clamped against the lower side of the baseplate. The baseplate is likewise ground so that the upper and lower surfaces are plane and parallel. The plane and parallel surfaces and rigid mechanical structure are designed to insure that all three transducers lie in the same plane. Initial alignment of the transducers in the horizontal plane is achieved using a spirit level mounted on the top surface of the baseplate. Final adjustment is obtained by maximizing the signal strength of the reflected ultrasonic signal. In order to ensure that the measuring points for the three tubes are nearly equally spaced and collinear, the transducers are mounted on the center of the titanium disks using a tight fitting jig. The three titanium disks are positioned by another jig. The assembly jigs were designed to position the geometric centers of the three transducers within 0.1 mm. In order to obtain maximum coupling of the ultrasonic signal from the titanium to the mercury, the titanium had to be thoroughly cleaned, degreased, lightly acid etched, rinsed, and carefully dried. Acid-washed and triply distilled mercury is distilled directly into the manometer as soon as possible after the titanium disks have been prepared. The mercury in the three tubes com-

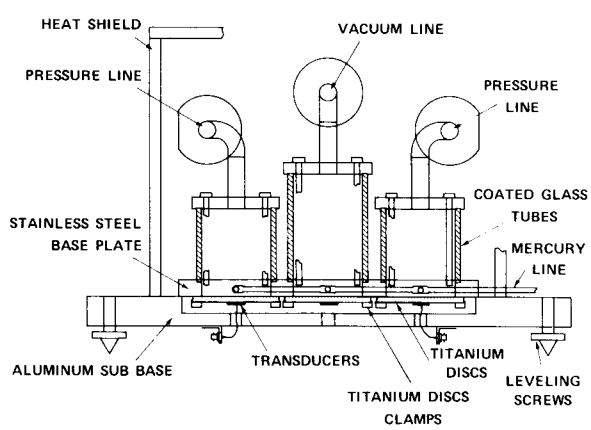

FIG. 9. Schematic of the US Nat. Bur. Stand. ultrasonic mercury manometer.

TABLE I. Pressure resolution of the ultrasonic mercury manometer. Manometer readings at zero applied pressure of over a 5-h period. Test II-047. Standard deviation of the residuals of the indicated pressure: 0.0014 Pa.

Time of day	Temperature of manometer	Difference of column lengths	Indicated pressure
(h:m)	(°C)	(μm)	(Pa)
18:00	22.335	0.00	0.0000
18:30	22.334	0.02	0.0027
19:00	22.334	0.00	0.0000
19:30	22.334	−0.02	−0.0027
20:00	22.334	0.00	0.0000
20:30	22.335	−0.01	−0.0013
21:00	22.336	0.00	0.0000
21:30	22.336	0.00	0.0000
22:00	22.338	0.01	0.0013
22:30	22.340	0.00	0.0000

municates by a horizontal, 4.5-mm-i.d. stainless-steel tube welded to the baseplate.

The assembled manometer is mounted on a 25-mm-thick aluminum plate with attached leveling screws. In order to minimize temperature gradients and average temperature changes, the entire manometer is surrounded by a 12-mm-thick aluminum box bolted to the aluminum baseplate. This in turn is surrounded by 5 cm of polystyrene foam. Only the vacuum and pressure lines, electrical leads for the signal to the transducers, and thermometer leads pass through the thermal enclosure. Temperatures in the manometer are measured either with a quartz crystal or platinum-resistance thermometer mounted on the stainless-steel baseplate. The behavior of measured vertical temperature gradients in a similar manometer indicates that the average temperature is within 10 mK of the thermometer temperature. Measured horizontal temperature differences between tubes typically change by less than 1 mK/day (Table II). A problem with the present design is the diffusion of air through seals, particularly the fluorocarbon (vinylidene fluoride–hexafluoropropylene copolymer) O rings sealing the titanium disks to the baseplate. The diffusion rate of about 3×10^{-9} m^3Pa/s is enough to cause a detectable and continuous length change of the columns until a bubble breaks off and rises to the surface of the mercury two or three times a day. This disruption causes the fringe counting interferometer to lose count. In the future, either metallic seals or a guard vacuum will be used to prevent this.

The performance of the mercury manometer is summarized in Table III. Some of these figures are based on prenary measurements or estimates. The speed of sound value currently used is based on an unpublished value obtained in this laboratory. Measurements currently under way should reduce its uncertainty by an order of magnitude. The values for the independent uncertainties other than capillarity are based on experimental observations. The capillarity uncertainty is an upper bound calculated for this manometer using the equations in Ref. 10 and assuming that the contact angle may arbitrarily change from normal to inverted. Temperature-gradient changes, mechanical and electrical noise, and tilt contribute to long-term instability of the measured zero reading when the manometer is left at zero. Over a period of 5 h the temperature of the manometer, and consequently also the column lengths, were held constant so that the demodulation error did not affect the measurements. During this

TABLE II. Temperature difference changes in the ultrasonic mercury manometer. Temperatures at the manometer base and temperature differences between the bases of the outer columns as function of time. Test II-058. This table is abbreviated; the original data were taken every hour.

Time of day	Temperature	Temperature gradient
(h)	(°C)	(°C)
6	23.005	0.0143
8	23.004	0.0142
10	23.002	0.0141
12	23.001	0.0140
14	23.002	0.0140
16	23.002	0.0141
18	23.000	0.0143
20	22.994	0.0144
22	22.992	0.0143
24	22.990	0.0143
2	22.988	0.0145
4	22.986	0.0143
6	22.988	0.0142

TABLE III. Uncertainties affecting pressure measurements with ultrasonic interferometer manometers. The values given represent present measurements or estimates, some of which will be substantially reduced in the future.

Source	Mercury	Oil
(a) Uncertainties proportional to pressure		
	systematic uncertainty[a] in ppm	systematic uncertainty[a] in ppm
Density	2	100
Acceleration of gravity	1	1
Temperature	5	10
Speed of sound	70	no est. avail.
Carrier frequency	1	1
Flexure of mounting plate	<1	<1
Verticality	<1	<1
Sum total	81 ppm	
(b) Uncertainties independent of pressure:		
	random(1σ) systematic uncertainty in mPa	random systematic uncertainty in mPa
Temperature gradient	3	0.01
Mech. and elect. noise	1	0.1
Demodulation	10	0.7
Capillarity	0.1	<0.01
Tilt	0.1	0.01
3 × RSS of random	9.6	0.10
Sum of systematic uncertainty	10.1	0.7
Sum total	19.7	0.80

[a] Random errors are estimated to be less than 1 ppm.

TABLE IV. Repeatability of the ultrasonic mercury manometer. Repeatability of zero applied differential pressure reading after pressure excursions. Test II-051. Maximum deviation: 0.02 Pa. Standard deviation of the residuals of the indicated pressure: 0.01 Pa.

Applied Pressure (Pa)	Indicated Pressure (Pa)
0	0.00327
130	
0	−0.00473
−130	
0	−0.01539
130	
0	0.02060
−260	
0	−0.01139
260	
0	−0.00873
−520	
0	0.01400
520	
0	−0.01000
−1300	
0	0.00467
1300	
0	0.00600
−1300	
0	0.00200
1300	
0	0.00067

period the zero readings had a standard deviation of 1.4×10^{-3} Pa (Table I). This compares with 3.2×10^{-3} Pa for the square-root of the sum of the squares (RSS) of the random errors in Table III. Repeated measurements of zero after pressure cycling will include the above errors plus those due to capillarity and demodulation. Such measurements had a standard deviation of 1.0×10^{-2} Pa (Table IV), and a maximum deviation of 2×10^{-2} Pa. The latter value compares with 2×10^{-2} Pa for the sum of all of the estimated independent errors shown in Table III.

V. ULTRASONIC OIL MANOMETER

The lowest absolute pressure that can be measured with a mercury manometer is limited by the mercury vapor pressure, on the order of 0.13 Pa at room temperature. This limitation can be overcome by using an oil, such as DEHS(di-2-ethylhexyl sebacate), whose vapor pressure is on the order of 10^{-6} Pa at room temperature, as the manometric fluid. If the length resolution obtainable with the mercury manometer can be maintained, the pressure resolution can be improved by an order of magnitude, due to the lower oil density. The ultrasonic oil manometer illustrated in Fig. 10 was designed with particular attention paid to the factors which contribute to the pressure-independent errors, in order to optimize pressure resolution.

The manometer is constructed from two blocks of stress-relieved aluminum, each 10 cm thick and 38 cm square. This massive construction is sufficient to prevent mechanical deflections of the transducer-mounting surface greater than the desired length resolution of 10^{-5} mm. The size of the blocks also provides a large thermal mass with small temperature gradients and high thermal conductivity. The manometer has four oil wells so that corrections for tilt in any direction can be determined. The four tubes are machined into the lower surface of the top block. Each tube is 2.5 cm deep, 10 cm in diameter, and centered on a corner of a square 11 cm on a side. Vacuum lines machined into the block connect tubes on diagonal corners together in two pairs, one pair for the reference, the other pair for the pressure side.

The faces of both upper and lower blocks were ground flat and parallel. The 18-mm-diam, 10-MHz ceramic transducers are located on the upper surface of the lower block. A jig was used to locate the transducers at the centers of the tubes. They are bonded to the block with a thin layer of low-vapor-pressure epoxy with good outgassing properties. The transducer leads pass through the lower block via metal–ceramic feedthroughs.

The acoustic signals enter both the lower aluminum block and the oil. Returning echoes from the bottom side of the lower block can interfere with the echoes from the oil. The echoes from the aluminum are attenuated by two or three orders of magnitude by scattering the downward-traveling pulse from a shallow cone machined into the bottom surface of the block under each transducer, and by coating the lower surface of the block with a lead-filled epoxy which absorbs acoustic energy.

The upper and lower blocks are sealed together with two concentric fluorocarbon O rings, with a guard vacuum in between. The transducer signal feedthroughs pass into an extension of this guard vacuum. These measures are designed to prevent the leakage of air into the oil.

The manometer has been designed for isolation from room-temperature changes and gradients. The manometer blocks are mounted on three thin-walled stainless-steel legs, standing on a 25-mm-thick aluminum plate. This plate in turn is similarly supported above a second 25-mm-thick aluminum plate with attached leveling screws. The two aluminum plates serve as bases for nesting 12-mm-thick aluminum boxes, which act as heat shields. A 5-cm-thick layer of polyurethane foam is placed on the inside of each box. The vacuum lines and electrical leads are thermally anchored where they pass through the heat shields. Temperatures are measured using platinum-resistance thermometers, placed in wells in the top block. Horizontal temperature differences between the tubes

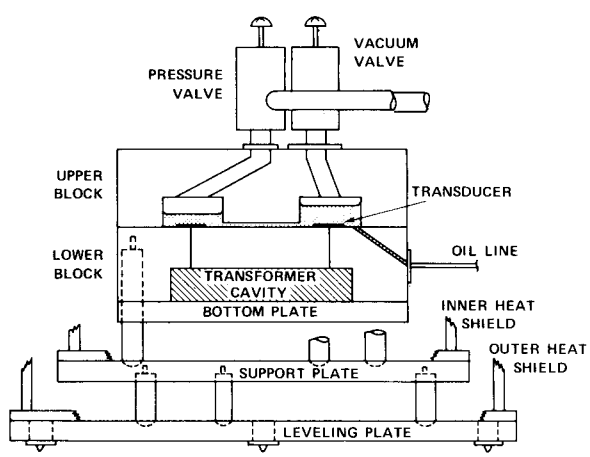

FIG. 10. Schematic of the US Nat. Bur. Stand. ultrasonic oil manometer.

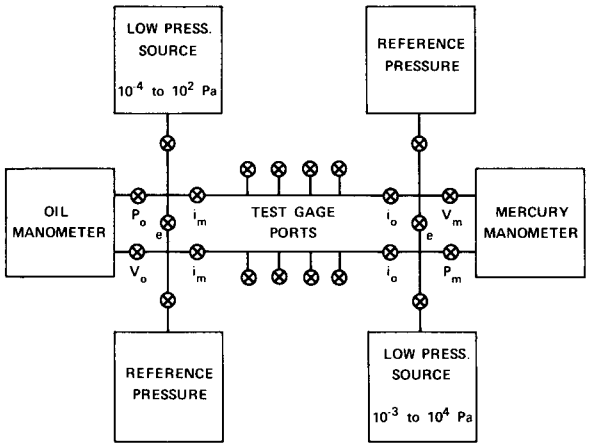

FIG. 11. Block diagram of the test stand.

have been monitored with a resolution of better than ten microdegrees. The differences are less than 0.1 mK and change by less than 25 μK over a 24-h period in a laboratory where the temperature variation is less than 1° C. A temperature gradient intentionally applied across the outer thermal shield was attenuated by a factor of 10^4 in the manometer block. The thermal time constant of the manometer for external temperature changes is about half a day.

Neither the speed of sound nor its temperature coefficients has yet been determined for DEHS. However, using a value of $\alpha_c + \alpha_d = -4.5 \times 10^{-3}\ \mathrm{K}^{-1}$, typical of other oils, and assuming a column length of 10 mm, a temperature-difference change of 25 μK will cause an indicated pressure change of 1×10^{-5} Pa.

Estimates of error contributed from the various sources are listed in Table III.

VI. TEST STAND

The proposed test for calibration of vacuum gauges using the ultrasonic manometers is shown schematically in Fig. 11. The mercury and oil manometers will be connected to the two ends of a manifold constructed of 36-mm-i.d. stainless-steel tubing. All joints will be either welded or will have crushed nickel seals. The lines into the oil manometer will be 24-mm i.d.; those into the mercury manometer will be 16-mm i.d.

The reference pressure for the mercury manometer will be maintained by a small untrapped mercury diffusion pump, while that for the oil manometer will be provided by a mercury diffusion pump with a cold baffle and liquid-nitrogen trap. A mercury rather than oil pump was chosen for the oil manometer to eliminate the possibility of diffusion-pump oil or its decomposition products migrating to the manometer and contaminating the manometric fluid.

Appropriate valving will be provided for roughing, isolation, and venting. The reference pressure of the oil manometer will be measured with a hot-filament ionization gauge calibrated on a conductance-limited expander.

The mercury manometer and a simple test manifold have been successfully used for a number of calibrations of capacitance diaphragm gauges. A static test pressure was generated by bleeding gas into or out of the system through two needle valves connected to a compressed gas bottle and a vacuum pump. The pressure was then trimmed with a variable volume. The same system will be used to generate the high pressures in the new system. Lower pressures will be generated by a system[2] that uses a servo-controlled leak valve to bleed gas into a volume pumped by an orifice-limited diffusion pump. It will be possible to connect or isolate either or both manometers from the manifolds, and bypass valves will be provided for both manometers to establish zero differential pressure. Parallel connection of the manometers will allow use of the mercury manometer for determining the density speed of sound product of the oil. Series connection of the manometers will allow the oil manometer to measure the reference pressure of the mercury manometer.

Thermistor, ionization, and capacitance diaphragm gauges will be placed at various points in the system to monitor the operation. A gas analyzer will be used to monitor gas composition during low pressure calibrations. Blank ports will be provided for the connection of differential or absolute gauges to be calibrated.

VII. SUMMARY

The operating range of manometric vacuum standards can be extended by the use of pulsed ultrasonic interferometer manometers. With mercury, the ultrasonic interferometer achieves a length resolution of 10^{-5} mm, and a similar resolution is expected with oil. The automatic operation permits a degree of isolation from thermal and mechanical disturbances not attained with manually operated instruments. The known sources of error in low-pressure manometry have been discussed, and were taken into consideration in the design of two ultrasonic manometers, one using mercury and the other using vacuum-pump oil as the manometric fluids. The manometers are intended to provide the basis for vacuum gauge calibration service for both absolute and differential pressures to 10^4 Pa.

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