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Static and Dynamic Calibrations of Pressure-Measuring Instruments at the National Bureau of Standards

E. C. LLOYD and D. P. JOHNSON

Introduction

An important part of the pressure standards programme of the National Bureau of Standards is a continuous effort directed towards the development of improved pressure measurement methods and equipment. The programme is concerned with measurement of both static and transient pressures, and covers the range from very high pressures to pressures in the vacuum range. The work of the National Bureau of Standards in this field supports both industrial and scientific activities in the United States, which have ever-increasing needs for improved pressure measurement methods.

In this paper we shall briefly discuss the present state of the art from the standpoint of accuracy of pressure standards, and will describe the design and performance of some of the equipment developed or used at the National Bureau of Standards for pressure measurement and calibration. Some plans for future work aimed at improving the measurement of high pressures will be indicated.

Accuracy of Pressure Standards

A general view of the state of development of equipment for static pressure calibration may be obtained from a graphical presentation of measurement accuracy *versus* pressure. *Figure* 1 illustrates the accuracies now obtainable from a number of measurement. The solid lines represent instruments now in use; the dashed lines show moderately optimistic hopes for the near future. The standard mercury barometer referred to in *Figure* 1 measures absolute pressures, while the mercury manometers can be used as either absolute or differential instruments. The remaining instruments measure differential pressure with one side at or near 1 atm. The precision manometer of Stimson[1] was developed in the Temperature Physics Section of the National Bureau of Standards for use in measuring absolute pressure in gas thermometry.

The accuracies indicated here do not take into account uncertainties in the values of the acceleration of gravity at Washington, D.C. or in the density of mercury. At pressures above 6,000–7,000 atm the measurements depend at present on calibration points largely determined by P. W. Bridgman; accuracies are estimated to be within about 1 per cent at 15,000 atm and about 10 per cent at 100,000 atm. The shape of the curves of *Figure* 1 merits some comment. In general any pressure-measuring instrument may have errors which are: (*a*) independent of the pressure being measured (e.g. errors due to zero instability, least count of reading means, capillary depression), (*b*) proportional to the pressure (e.g. proportional error in measurement of the height of a mercury column, or diameter of a piston), and (*c*) errors relatively more important at high pressures such as the compressibility of mercury or the

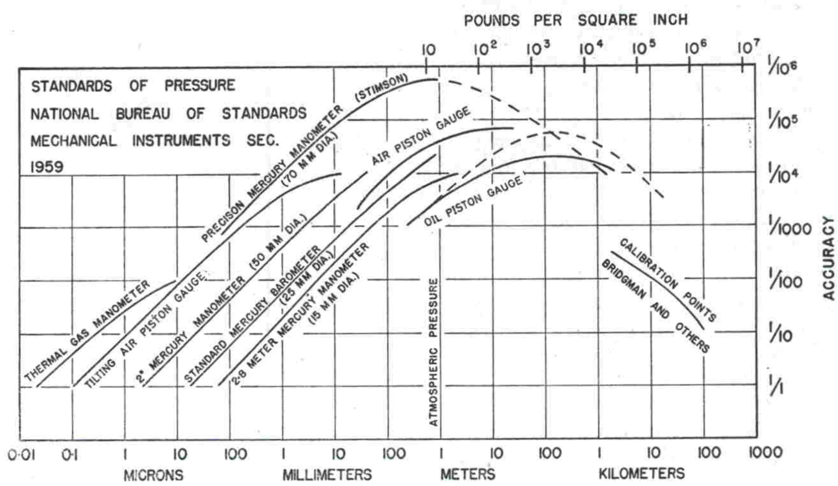

Figure 1. *Absolute accuracies of pressure standards*

fluid-column and dead-weight instruments used for static calibration over the pressure range from a few microns of mercury to 5,000 atm. Here the abscissa is the pressure expressed on a logarithmic scale, and the ordinate is a logarithmic scale of accuracy expressed as estimated uncertainty in the elastic distortion of a piston and cylinder for which the error is proportional to the square of the pressures.

At low pressures group (*a*) dominates; the intercept at accuracy 1/1 is drawn at the pressure equal to the estimated magnitude of these errors, and the curves start off with a 45°

slope. At intermediate pressures the proportional errors (*b*) dominate and the curves are horizontal. (This is usually the region of greatest accuracy.) At high pressure, the third group of errors (*c*) becomes important and the accuracy falls off.

These considerations apply not only to individual instruments but also to the state of the art as a whole. Thus at pressures of a metre of mercury or a little less, by a great effort, it is possible to approach an accuracy of a part in a million with a mercury manometer.

There is a definite loss in accuracy in measuring lengths greater than one metre, and it becomes very difficult to maintain and to know the temperature of a mercury column more than a few metres in height. The compressibility of mercury is about four parts in a thousand at 1,000 atm, and is known to about 1 per cent. The accuracy of pressure measurement by use of a mercury column is therefore at present limited to about $1/10^4$ at 2,000 atm. At the other end of the scale, the vibration level encountered in several of the National Laboratories makes it doubtful whether the level of a mercury surface can be found to an accuracy much better than 0·1 μ.

The attainment of these accuracies is at the cost of considerable time spent in making each measurement; thus, making a measurement with the 'precision mercury manometer' involving a change in pressure requires more than 24 hours. A working instrument must strike a practical compromise between speed in making a measurement and accuracy. Thus the 'standard mercury barometer', which has an accuracy of one part in 25,000 at one atmosphere, has been set and read in one minute, and a pressure change can be made in 5–10 minutes.

Design and Performance of Equipment for Generation of Static Pressures

Dead-weight piston gauges

The N.B.S. piston gauge standards are of the controlled-clearance design developed by Johnson and Newhall[2]. *Figure* 2 is a general view of this device, shown connected to a small dead-weight gauge to be calibrated. A weight load of nine 100-pound discs is suspended by a yoke below the piston-cylinder assembly, and additional graduated weights provide a total load of 1,000 lb. A pressure-supported ball-and-cup bearing is provided between the top of the piston and the yoke to permit rotation of the piston without rotation of the weights. A series of piston-cylinder assemblies are provided, the smallest of which has a piston diameter of approximately 2 mm, corresponding to a maximum pressure of 12,000 atm. *Figure* 3 is a sectional view of the piston-cylinder assembly showing the piston and the rotating device, the cylinder jacket, pressure connection, and the manner in which the load is transmitted to the piston.

Elastic distortion is the important source of error in high pressure piston gauges. The configurations we use are such that the elastic distortion error can be computed and correction applied to bring the uncertainty due to elastic distortion within several parts in 10,000 at 10,000 atm. In the middle range the important uncertainties are those associated with the measurement of the diameter of the piston and with the variations of the piston from an ideal cylinder. These are of the order of 1 or 2 tenths μ and for our largest pistons lead to an uncertainty of 1 or 2 parts in 100,000. The clearance can be estimated by means of the leak rate and the torque required to turn the piston, both of which are functions of the average clearance. Since the average clearance can be varied at will in these gauges the extrapolation may be made to zero clearance with an accuracy of the same order as the surface irregularity. At low pressures, the uncertainty in oil buoyancy on a non-uniform piston, in surface tension effects, and in accounting for the head of oil, limit the accuracy to a few tenths mm of mercury.

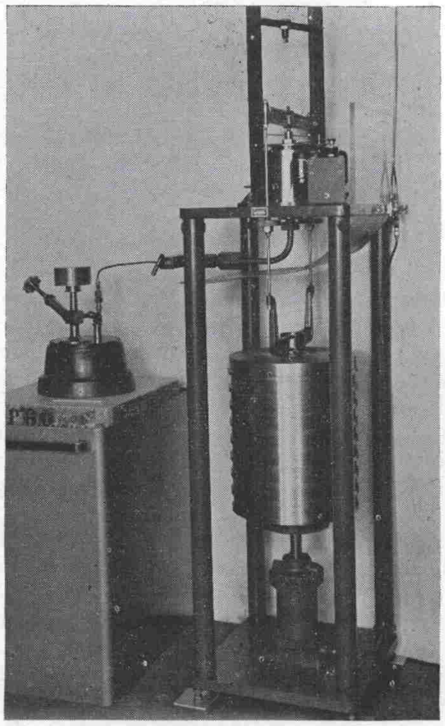

Figure 2. *N.B.S. controlled-clearance piston gauge designed for use as a primary standard up to* 12,000 atm

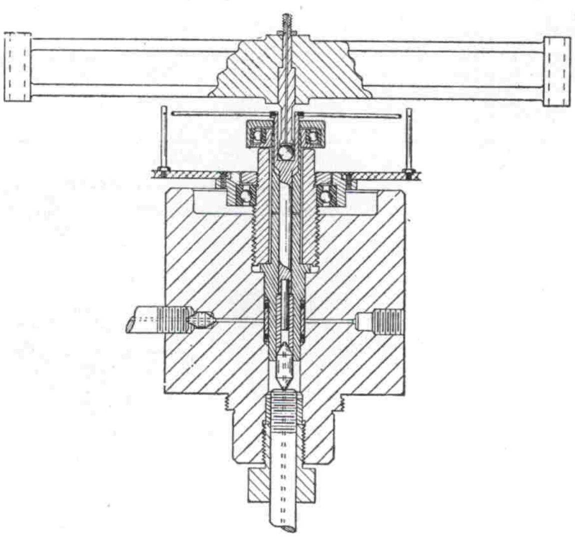

Figure 3. *Sectional view of N.B.S. controlled-clearance piston gauge showing jacket for application of hydraulic pressure to control clearance, and arrangement for application of weight load to the piston*

The greatest accuracy with an oil piston gauge is a few parts in a hundred thousand at about 100 atm, 1 part in 10,000 at a 1,000 atm, and possibly 1 part in 1,000 or a little better at 10,000 atm.

For pressures below 35 atm the air-lubricated piston gauge is coming into use as a pressure standard in the U.S.A. *Figure* 4 is a sectional view of such an instrument having a 1 in. piston diameter, constructed at the National Bureau of Standards for pressures between 1/2 and 35 atm. This device also utilizes the controlled clearance principle. Although the internal pressure causes no significant distortion of the piston or cylinder, the control of the cylinder bore makes it possible to determine the effective area more accurately by direct measurement of the piston diameter only than could be done in an instrument requiring measurement of both piston diameter and cylinder bore. The errors of oil buoyancy and fluid head are reduced, and surface tension effects eliminated. The sources of errors involved are otherwise the same as with the oil-lubricated gauge. An absolute accuracy within one part in 50,000 is obtainable over most of the range of this instrument, limited by accuracy of the determination of the effective area of the piston. A sensitivity of one part in a million has been obtained at pressures of a few atm.

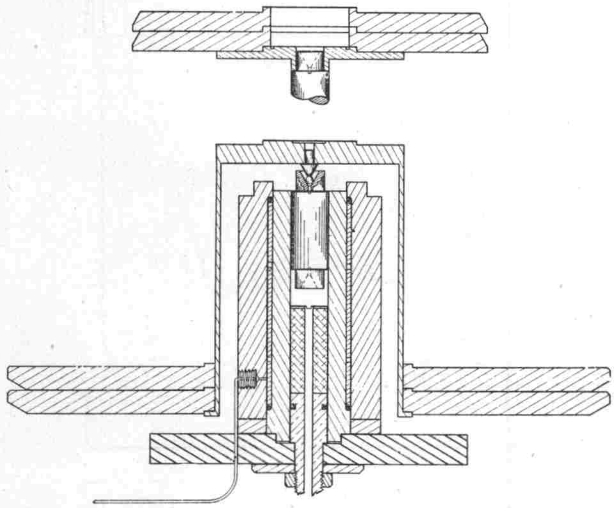

Figure 4. Sectional view of N.B.S. controlled-clearance air-lubricated piston gauge used as a pressure standard from 1/2 *to* 35 atm

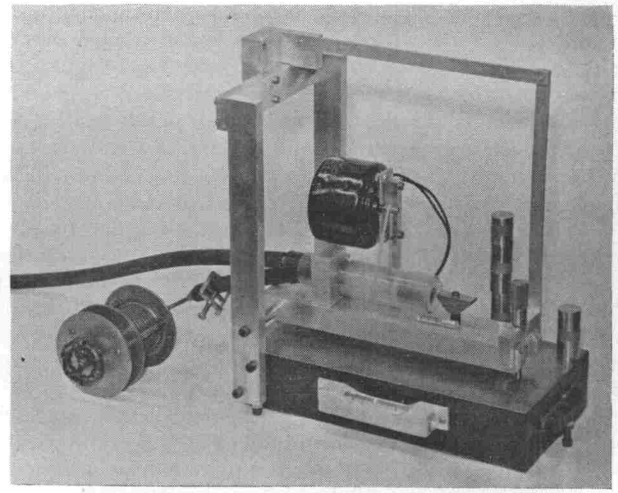

Figure 5. Experimental model of N.B.S. tilting air piston gauge, useful for pressures below 15 mm Hg. *The instrument is in the zero position*

For reasons of safety, we would not care to use a gas-lubricated piston gauge above 100 atm, particularly since at these pressures the oil-lubricated piston gauge is nearly as good.

For differential pressures less than a few mm of mercury the National Bureau of Standards has constructed an air-lubricated piston gauge in which the axis of the piston and cylinder is inclined to apply an appropriate component of the load along

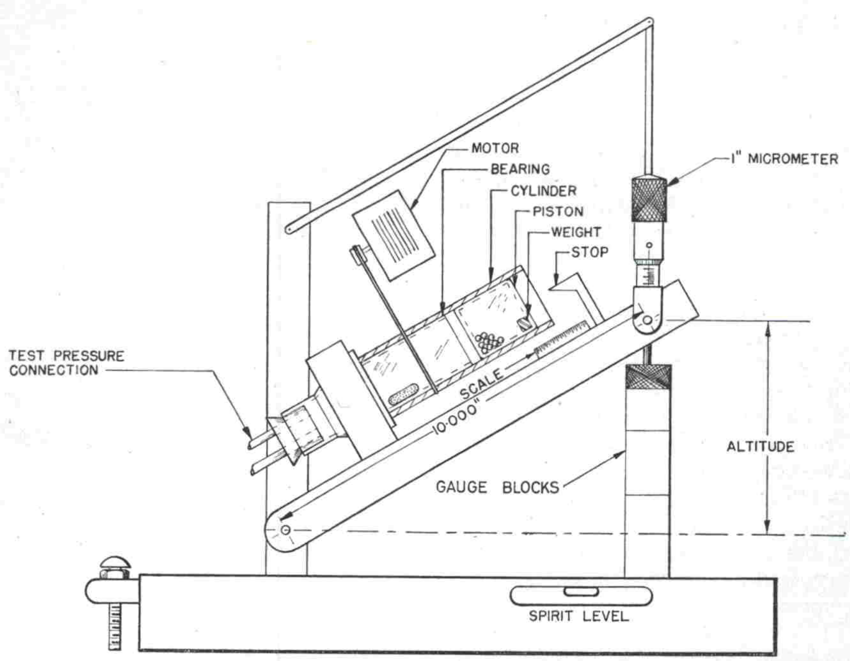

Figure 6. Schematic arrangement of N.B.S. air-tilting piston gauge showing the instrument in an inclined position. The pressure developed is directly proportional to the altitude as measured by gauge blocks and micrometer

the axis. This instrument is the 'tilting air-lubricated piston gauge'[3], an experimental model of which is shown in *Figure* 5. In this photograph the piston axis is horizontal, and gauge blocks used for accurately elevating the right-hand end of the piston are shown stacked on the base plate of the instrument. The cylinder is rotated, and the piston is stationary. The piston comprises the only weight load, and is constructed with its centre of mass off-centre in order that it shall not rotate in the air bearing formed with the cylinder. *Figure* 6 is a schematic drawing of this instrument. It is seen that an air bearing also is used to support the cylinder and to serve as a seal. In use it has been found that the gauge axis can be elevated to an angle of about 80° before friction causes the piston to rotate with the cylinder. It may be possible to operate the gauge for short times in the vertical position for calibration purposes, thus giving a check at both zero and maximum pressure independent of the altitude measurement. This experimental instrument, designed for pressures up to 12 mm of mercury, has exhibited sensitivity and reproducibility of 1 part in 100,000 of the range, and an absolute accuracy better than 1 part in 10,000. It is believed that this performance can be improved by giving close attention to the shape and fit of the piston and cylinder. One source of error found, not significant in more conventional piston gauges, is due to forces between electrostatic charges built up on the glass piston and cylinder. This has been satisfactorily controlled by maintaining the air within the cylinder at greater than 50 per cent relative humidity or by the use of conductive surface coatings.

Although the tilting air-piston gauge was originally intended for differential pressure measurement, experiments indicate that it can be used with one side evacuated, thus providing a primary instrument for absolute measurement in the pressure range of a few mm of mercury. Extension of the range of the gauge to several cm of mercury with comparable accuracy may be expected by selection of the most favourable parameters such as the piston diameter, the speed of rotation, and the precise fitting of the cylinder to the piston and its support bearing.

Equipment using solid-pressure transmitting media

The upper limit of pressures attainable with fluid-pressure transmission appears to be on the order of perhaps 25,000 atm; higher pressures involve the use of solid pressure media. At present the pressures obtained in various devices using solid pressure media are not precisely known, particularly above 25,000 atm. The National Bureau of Standards is working with such equipment with a view towards the establishment of reference pressures or fixed points based on polymorphic transformations or changes of state.

Figure 7 is a sectional view of a tetrahedral-anvil device constructed at the National Bureau of Standards[4]. A conventional hydraulic press is used with this equipment to apply force to each of the four faces of a tetrahedron, generating pressures well in excess of 100,000 atm in the tetrahedron. It will be seen from *Figure* 7 that although external force is applied to only one of the anvils, wedge-reaction forces act on the other three anvils so that all four anvils move equally towards the centre of the tetrahedron. In sliding along the internal conical surface of the ring, the three lower anvils are lubricated by a thin sheet of Teflon (polytetrafluoroethylene). *Figure* 8 is a photograph of this equipment with the top anvil removed, showing the appearance of a charge after removal of pressure. The lubricating Teflon sheets are visible; these also serve as electrical insulation between the anvils and the conical ring. *Figure* 9 is a record obtained with this equipment showing the electrical resistance of a specimen of bismuth as a function of pressure up to about 120,000 atm. It is hoped that further work will enable us to determine more accurately the pressures

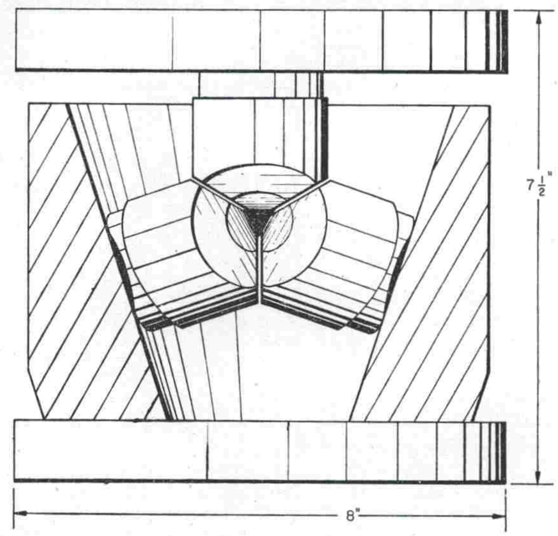

Figure 7. Sectional view of N.B.S. tetrahedral high-pressure generator with one anvil removed. The apparatus is used with a conventional hydraulic press to apply compressive force between the upper and lower bolster plates. Equal motion of the four anvils results from the sliding of the lower anvils along the conical surface

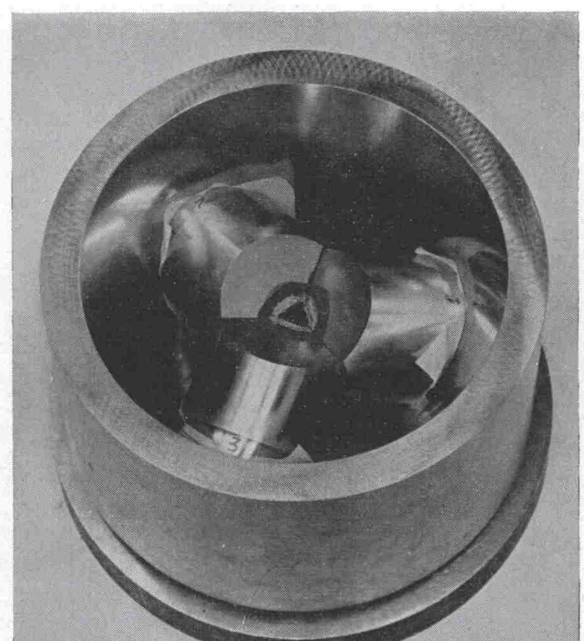

Figure 8. N.B.S. tetrahedral anvil high-pressure generator with upper anvil removed, showing appearance of a charge after removal of load. Lubrication between the lower anvils and the conical ring is provided by pieces of sheet Teflon shown

of these transitions, and of transitions in other materials to provide pressure fixed points useful in pressure calibrations. Even before these reference pressures are more accurately determined, however, the availability of equipment to permit

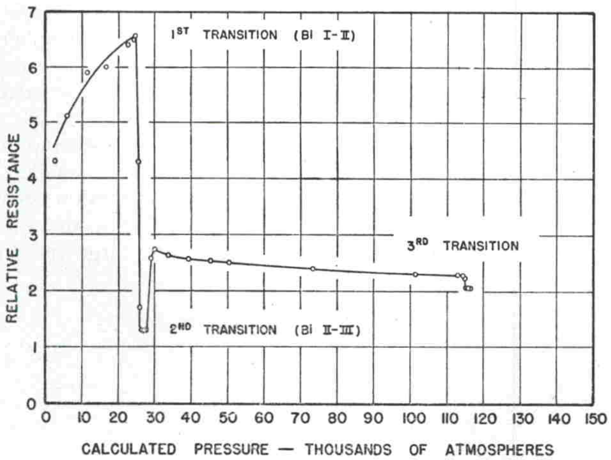

Figure 9. *Pressure* versus *resistance of a specimen of bismuth showing the bismuth transitions, obtained by use of the tetrahedral anvil apparatus. Pressures shown were calculated from measurement of applied force and area of the charge, with Bridgman's value for the first transition* (24,800 *atm*) *used to determine a correction factor due to force supported by the gasket and internal friction in the pyrophyllite*

them to be readily reproduced should facilitate the measurement of relative pressures and thus provide a means for better comparison of the results of different laboratories.

Dynamic Calibration of Gauges for Measurement of Transient Pressures

Pressure transducers of many types intended for use in measurement and recording of transient pressures are available commercially. These include piezoelectric gauges, strain-gauge instruments, and the like. However, the accuracy of results obtained with these devices, particularly in the measurement of high transient pressures or high-frequency components of transient pressures (pressures above 65 atm or frequencies higher than 500 c/s) is usually limited by incomplete knowledge of the performance of the pressure transducer. Securing adequate knowledge of the performance requires accurate dynamic calibration; this difficult type of calibration has received some attention at the National Bureau of Standards. For pressures below 65 atm use has been made of the shock

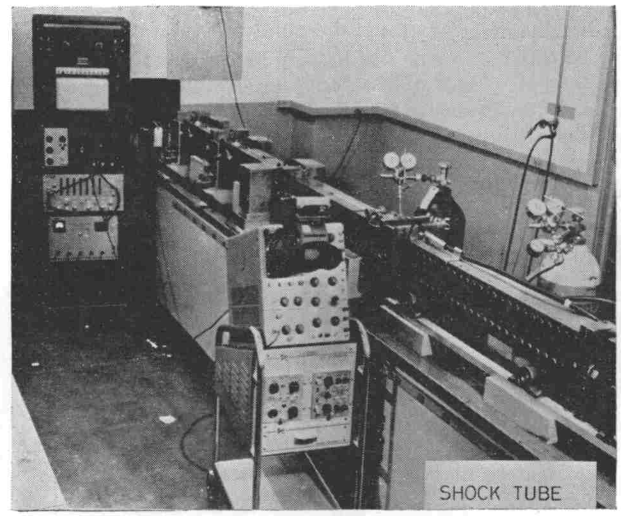

Figure 10. *View of shock tube facility for dynamic calibration of pressure transducers to* 65 *atm*

tube for generation of fast pressure steps of known amplitude. *Figure* 10 is a general view of the shock tube facility. The shock tube has a working cross section 3 in. × 3 in. square, a compression chamber length variable from 3·4 ft to 12 ft, and an expansion chamber length fixed at 8 ft. The tube is constructed of flanged sections, each section comprising a 4-piece bolted assembly. The material is steel. The expansion chamber sections are carefully finished and chrome-plated on the interior surfaces to minimize 'noise' due to shocks reflected from surface discontinuities. The diaphragm station is visible just to the right of the oscilloscope, and it is seen that the compression-chamber sections are arranged to move on rollers to permit insertion of diaphragms.

The tube is equipped with three pairs of optical glass windows, each pair provided with a schlieren system and photomultiplier tube arranged to give a pulse at the time of passage of the shock wave.

The tube is designed to provide pressure steps having a duration of 4 to 5 msec at a shock-wave Mach number of 1·36. Using air on both sides of the diaphragm, shock step heights may be generated up to about 40 atm, and by using helium in

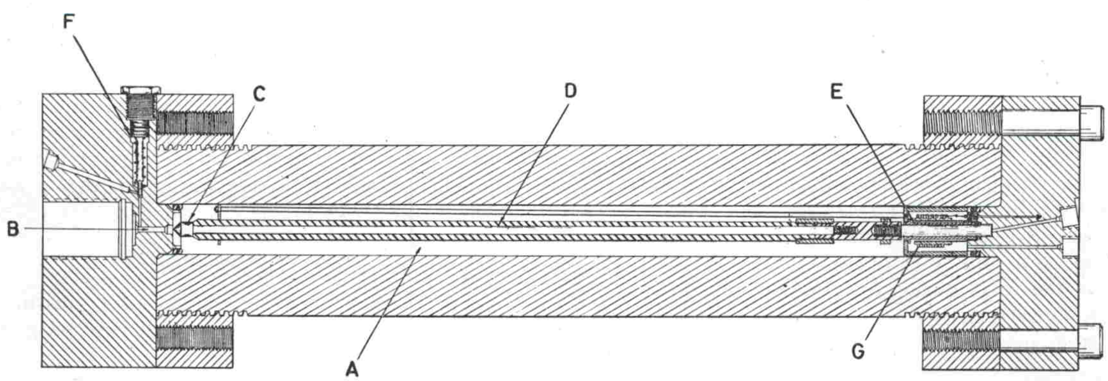

Figure 11. *Sectional view of N.B.S. quick-loading device for dynamic calibration of pressure transducers to* 4,000 *atm. The valve separating a large volume of oil at high pressure and a small volume of oil at low pressure is opened very rapidly by the spring action of the long valve stem in tension*

(A) Large chamber *(B) Small chamber* *(C) Main valve* *(D) Valve stem* *(E) Valve piston* *(F) Bleeder valve* *(G) Manganin gauge*

place of air in the chamber at an initial pressure of 65 atm shock step heights up to 65 atm at the rigidly closed down-stream end may be obtained. The rise time of the pressure step usually is less than 10^{-8} sec. The amplitude of the pressure step is calculated by means of ideal-gas theory from the velocity of the shock-wave and the temperature and pressure of the air before the diaphragm is burst*. The method appears to be capable of an accuracy within 1 or 2 per cent of the step pressure.

Figure 12, obtained with the shock tube, is typical of the response of a pressure transducer to a pressure rise occurring fast enough to excite the natural frequency of the gauge. These records may be interpreted in terms of frequency response of the transducer if an electrical analogue can be constructed whose response to an electrical step function sufficiently resembles the response of the gauge to the shock tube pressure step. This can be done if the gauge response is linear or if it is non-linear only with respect to damping forces. A technique of harmonic analysis utilizing repetitive playback of a magnetic recording of a transient response promises to facilitate the analysis of gauges vibrating in three or more modes. In addition, analytical methods of analysis of transient response records are available[5].

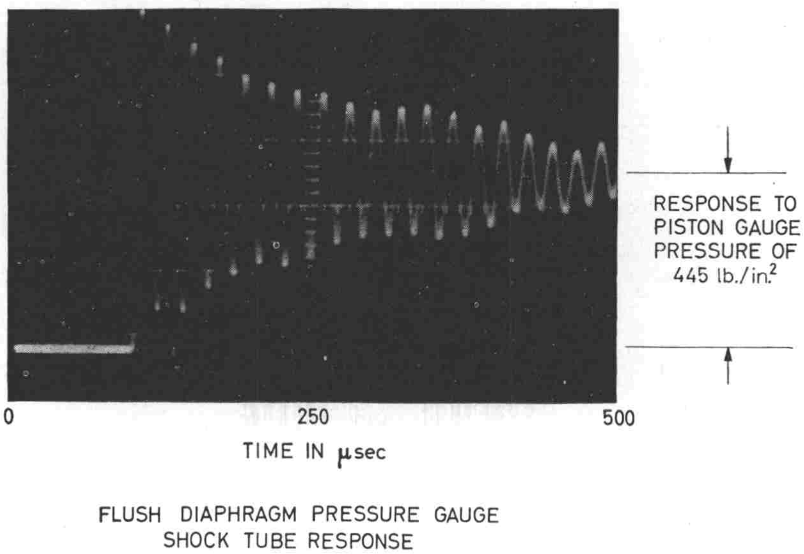

Figure 12. Typical records of the response of pressure transducers to step-pressure changes generated by the equipment of Figures 10 and 11. These records are analysed to determine frequency response and linearity of the pressure transducers

For dynamic calibrations at pressures up to 3,500 atm we have constructed the quick-loading device shown in *Figure* 11. The device consists of two chambers, one of small volume into which the gauge is connected, and the other a large reservoir. The volume of the reservoir is about 1,000 times that of the small chamber and its length about 40 times as great. When the valve is opened, the two chambers are connected. Quick opening of this valve is accomplished by making the stem of the valve long, and pulling it open from the far end. The acceleration of the valve during opening is rapid if there is a large amount of elastic energy per unit of mass, i.e. if the stress levels in the stem are high. The chamber and the reservoir are both filled with liquid and the valve is closed by pressure under the piston. The gauge chamber is vented to atmospheric pressure and closed, and the pressure in the reservoir is then raised to the desired high value. The valve is held closed by the pressure in the reservoir so that the stem must be put in tension in order to open it. This is done by reducing the pressure under the piston. The stem of the valve is smaller in area than the port so that it is highly stressed in tension before the valve begins to open. As the pressure below the valve rises the valve is accelerated. Because of the large ratio of volume between reservoir and chamber, the pressure at equilibrium is less than 1 per cent lower than the pressure before the valve is opened. The opening time to 90 per cent of the final pressure on the gauge is estimated from transducer response records to be 100 μsec.

It is to be noted that while the shock tube is quantitative as to rise time of the pressure step, the calculation of pressures developed depends on the characteristics of the gas and is uncertain if these characteristics are not known well. The quick-opening valve apparatus, on the other hand, is not intended to be quantitative as to opening time; that is, the time to open is not important provided that it is sufficiently short for the gauge to respond. This device is intended to be quantitative as to the amplitude of the pulse. The two types of devices are therefore complementary and each is useful in obtaining information on pressure transducer performance. Further work is planned in the concurrent use of the shock tube and quick-loading apparatus in the pressure range covered by the shock tube.

Fixed Points on the Pressure Scale

The establishment of fixed points on the pressure scale is an important part of the pressure-measurement programme at the National Bureau of Standards.

At lower pressures, use has been made of the vapour pressure of carbon dioxide at 0°C (about 26,140 mm of mercury). This has been measured by the National Laboratories of several countries, and has been used as a calibration point in the range of 10–100 atm. We are considering measurement of the vapour

* The relationship used is $Pr/P_0 = 7/3\ (M_s^1 - 1)(2 + 4M_s^2)/(5 + M_s^2)$, where Pr/P_0 is the ratio of the pressure step amplitude to the initial static pressure in the expansion chamber and M_s is the shock-wave Mach number. In the use of this expression the principal uncertainty in step amplitude is due to viscous losses.

pressure of CO_2 with a dynamic hypsometer, of the type used at the National Bureau of Standards in establishing temperature fixed points (water boiling point and sulphur boiling point), working directly against a gas-lubricated piston gauge, thus eliminating all other fluids from the system.

Higher up the pressure scale the triple point on the water phase diagram (Ice I–Ice III–liquid) provides a reference pressure at about 2,000 atm. A preliminary experiment indicates that during a very slow first melt from a mixture of Ice I and Ice III, the pressure is extremely stable. Our provisional value was 2,093 ± 5 bars, about 18 bars greater than Bridgman's value.

There is an important gap in the fixed points between the CO_2 vapour pressure and the first water transition at 2,000 atm. Attention is needed to the selection of suitable transitions that could be used in this range between 35 and 2,000 atm.

Many workers have used the melting pressure of mercury at 0°C (Bridgman's value is 7,640 kg/cm^2), or the melting pressure of Ice VI at 25°C (the value 9,630 bars was used in the Geophysical Laboratory of the Carnegie Institute). Our measurements on this mercury point[2], and on the Ice VI point by extrapolations with manganin-wire gauges calibrated at lower pressures, give values greater than Bridgman's by not more than 1 per cent.

At pressures above 10,000 atm the bismuth transitions involving changes in electrical resistance provide useful and reproducible pressures. Further work on these and transitions in other materials in this pressure range is planned. In particular we shall attempt to observe the first bismuth transition with the free piston gauge, if a satisfactory pressure medium (preferably less flammable than petroleum ether) can be found which remains liquid at that pressure.

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Summary

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Equipment developed or used at the National Bureau of Standards for pressure calibrations is described. For static calibrations this includes the controlled-clearance oil piston gauge at high pressures, the air piston gauge, the newly developed inclined air piston gauge, and precision mercury columns. Use of a compact form of the tetrahedral anvil apparatus for investigation of polymorphic transitions to pressures above 100,000 atm is described. For dynamic pressure calibrations at pressures up to 3,500 atm a pressure-pulse generator of high amplitude accuracy is used, and for lower pressures the range of this equipment is overlapped by pressure pulses generated by a shock tube. The methods of use and the sources of error, and relative accuracies of these instruments are discussed.

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