Pramāṇa – J. Phys., Vol. 27, No. 3, September 1986, pp. 417–434. © Printed in India.

Accurate measurement of pressure

J K N SHARMA and K K JAIN
National Physical Laboratory, New Delhi 110012, India

MS received 3 December 1985; revised 28 April 1986

Abstract. This paper briefly describes the fundamental principles of the instruments used for accurate measurement of hydrostatic pressure and in particular the use of piston gauges as primary pressure standards. Different methods for the calibration of secondary standards have been discussed and in particular, emphasis has been given to the calibration of secondary piston gauges against the primary standards by the cross-float method along with the evaluation of uncertainties attached to different correction factors associated with the measurement of pressure from these gauges. The importance of secondary pressure standards in the region 0·1 GPa to several GPa has also been defined.

Keywords. Pressure standards; piston gauge; pressure measurements; transducers.

PACS Nos **47·80; 07·10**

1. Introduction

Accurate measurement of high pressures is of fundamental importance because of the recent spurt in the applications of high pressures in the field of high pressure science and technology. Reliable pressure measurement in different industrial processes such as, ammonium synthesis (Comings 1956), polyethylene manufacturing (Marano and Jenkins 1977), diamond synthesis (Bundy 1977), hydrostatic extrusion (Alexander and Lengyel 1967), jet cutting and hydrothermal growth (Schwacha 1961), chemical and petrochemical industry and power plants, etc is essential to understand and optimize these processes. Though very high accuracy may not be required in some of the actual industrial processes, there is nevertheless the need to standardize pressure over a wide range and for establishing pressure standards for calibration of pressure measuring instruments.

The range of pressures measured in science and in industry covers more than twenty decades, as shown in figure 1. However, most of the industrial applications of high pressure are upto about 5 giga pascal (GPa); therefore, in this article we confine our discussion to the pressure region from 100 kPa–5·0 GPa.

Over much of the pressure range to be discussed here, pressure is homogeneous in space, hydrostatic and independent of time. It can therefore be defined by a scalar quantity as force (F) per unit area (A)

$$P = F/A, \tag{1}$$

which is often simply referred to as the force or as energy density

$$P = (\partial F/\partial V)_T, \tag{2}$$

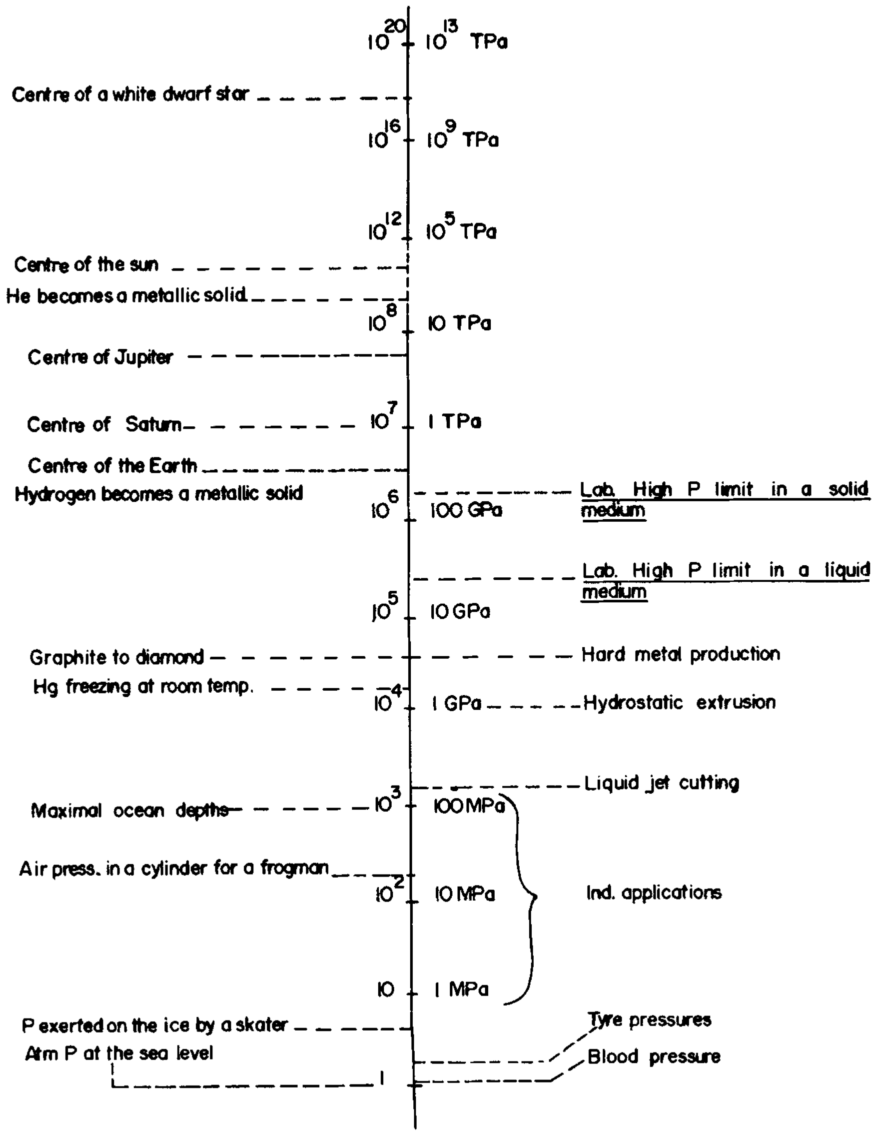

Figure 1. The range of pressures available to science and industry.

where F is the Helmholtz free energy with

$$F = U - TS \quad \text{and} \quad dF = PdV - SdT. \tag{3}$$

At high pressures when the viscous liquid or even solids are used to transmit pressures one has to be more specific in the definition of hydrostatic pressure because then the systematic stress tensor is introduced which is defined at each point in space. Measurable stress over finite areas can then be calculated by averaging which has been discussed in detail by Decker *et al* (1972).

The official unit of the pressure in the System International is Newton/squaremeter or N/m^2. This unit also carries the name Pascal (Pa).

2. Measurements

Regardless of the design of instruments used for pressure measurement, two classes of gauges must be distinguished; primary and secondary or transfer gauges.

In general, the primary pressure gauge establishes the pressure scale in terms of the basic units of mass, length and time or energy and volume using basic thermodynamic relations and does not need calibration whereas in the secondary transfer gauges the pressure is measured in terms of some other suitable physical parameters whose relation to pressure is determined by calibration, i.e. in comparison with another piston gauge or manometer of higher accuracy. Resistive, piezo-resistive, piezo-capacitance gauges and the Ruby fluorescence lineshift or the sodium chloride equation of state with adjustable parameters are all examples of transfer gauges. An exception to this is the simple piston gauge which is also used as a secondary/transfer pressure standard in the medium pressure range.

There are two commonly accepted independent prime methods by which pressures may be measured or established. In the first, the pressure is determined in terms of the column height of a liquid of known density under known gravitational conditions, for example, the mercury manometer (Michels *et al* 1942; Bett *et al* 1954). In the second method, pressure is measured in terms of force per unit area; for example, the dead weight piston gauge, also known as piston manometer or pressure balance, in which the force due to the pressure-transmitting fluid acting on the base of a cylindrical piston, free to rotate in an accurately matching cylinder to relieve friction, is balanced by a known gravitational force derived from known masses suitably positioned on the piston. For the sake of clarity and convenience, the term piston gauge would be used hereafter.

These two fundamental methods are also directly applicable to pressure measurement in the near atmospheric region and form the basis of the laboratory primary standards; comparisons made in meteorological laboratories have shown that the two methods agree well within the limits of experimental uncertainties (Peggs and Lewis 1977).

In practice the use of the column height of a mercury manometer for the measurement of pressure has been limited to the pressure region between 0·1 Pa and 200 kPa. A very elaborate system was designed and built by Bett *et al* (1954), for use upto 230 MPa though no measurements above 70 MPa have been reported by these researchers. Therefore, as an alternative, the free piston gauge is in common use to measure pressures.

The piston gauges can be classified as follows: simple—piston gauge; coaxial—piston gauge; re-entrant piston gauge; controlled clearance piston gauge.

2.1 *Simple piston gauge*

In a simple free piston gauge the loading weights are mounted directly on a platform attached to the top of the piston (figure 2), whereas the coaxial type simple piston gauge allows the possibility of alignment of the load in relation to the axis of the assembly (figure 3), and in some recent models the cylinder is made rotatable instead of the piston. The simple piston gauge is not recommended for high pressures because of the increasing distortion of the cylinder containing the measuring piston.

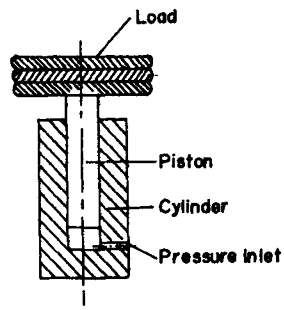

Figure 2. Simple form of piston-cylinder assembly with top loading.

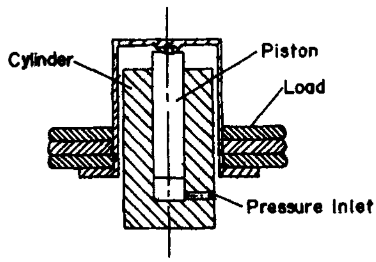

Figure 3. Simple form of piston-cylinder assembly with overhang carrier.

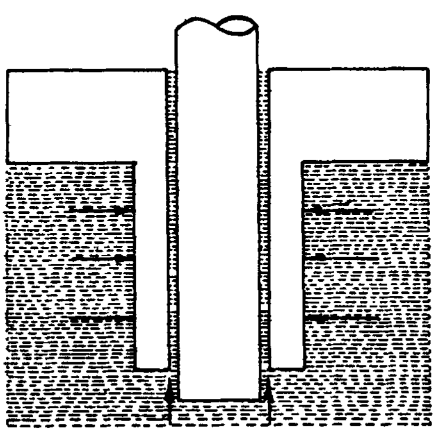

Figure 4. Re-entrant type piston-cylinder assembly.

2.2 *Re-entrant piston gauge*

Re-entrant piston gauge, shown in figure 4, was first developed by Bridgman (1911) and had been used to measure pressures upto 1·3 GPa. In the re-entrant type of piston gauge, pressure is applied on the outside and at the end of the cylinder which decreases the leak rate with increasing pressure because of the throttling of the piston due to the cylinder, thereby setting up an upper limit of the pressure to be measured.

2.3 *Controlled clearance piston gauge*

Johnson and Newhall (1953) have designed and built the controlled clearance piston gauge (figure 5) which constitutes a fundamental improvement in free piston gauge

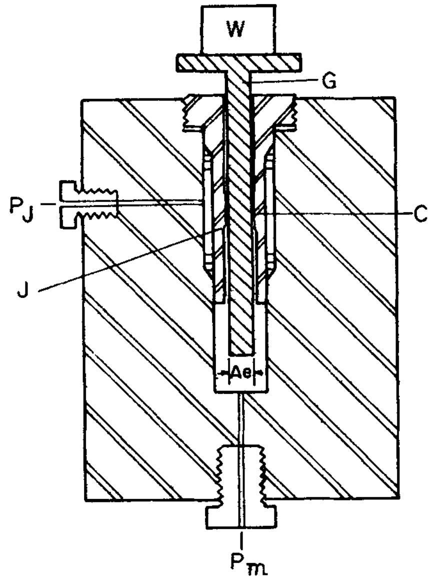

Figure 5. Schematic diagram of the controlled clearance piston gauge. P_j is the external pressure that controls the clearance C between piston G and cylinder J. P_m is the measured pressure. W is the weight applied on the piston.

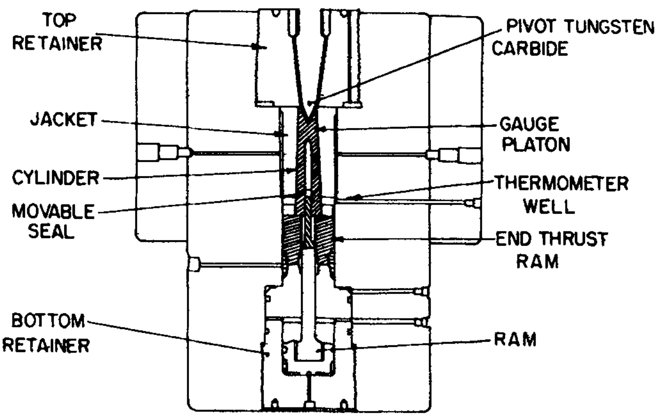

Figure 6. NBS 2·5 GPa controlled clearance piston gauge. 2 mm diameter cemented tungsten carbide gauge piston. Pressure generated by hydraulic ram. Longitudinal support of cylinder from end thrust ram. Radial cylinder support from jacket.

design by controlling the leak rate of the fluid past the piston, as well as the elastic distortion of the cylinder by jacketing the cylinder, in which the piston floats, with a separate and independent source of pressure. Thus, only the distortion of the piston needs to be considered and the more complex problem of treating with the elastic distortions of the piston and cylinder is avoided.

The pressure of the order of 1·4 GPa can be easily measured by controlled clearance piston gauge; however, hydrostatic pressure as high as 2·5 GPa, inside the high pressure cylinder of a controlled clearance piston gauge, has been generated by Johnson and Heydemann (1967) using hydrostatic intensifiers, and the pressure is measured with a cemented tungsten carbide piston of about 2 mm diameter as shown in figure 6.

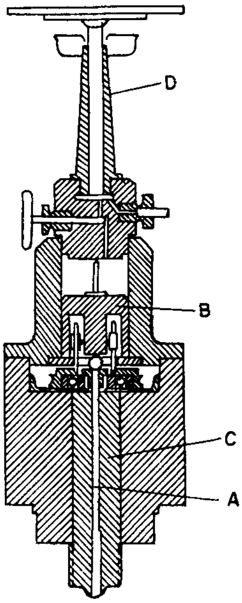

Figure 7. Schematic diagram of the Konyaev simple piston gauge. The high pressure piston (A) is pushed by ram (B) into cylinder (C) piston gauge (D) generates the ram pressure.

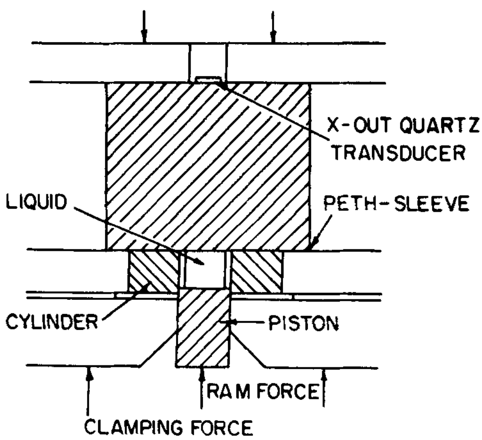

Figure 8. 5 GPa piston and die with polyethylene (PETH) liner.

Further, Konyaev (1961), was also able to measure the hydrostatic pressure by the simple piston gauge (figure 7) in the region of 2 GPa.

Two major constraints appear to limit the dead weight loaded piston gauges to pressures below about 2·6 GPa, the compressive strength of the rather long pistons and the large load to be applied to the piston. However such high pressures had been obtained (Kennedy and La Mori 1962; Jayaraman and Maines 1971) with rather short pistons with hydraulic loading and commonly known as piston die device. Heydemann and Houck (1969) had shown by using the polyethylene liner in piston die device that pressures as high as 5 GPa can also be measured with this device and with the measurement uncertainties as low as in the primary pressure standards (figure 8).

The pressure generated by controlled clearance piston gauges and the simple and re-entrant type piston gauges, at the reference level, is given by (4) and (5) respectively,

$$P = \frac{\sum_{1}^{i} M_i g(1 - \rho_{\text{air}}/\rho_M) + \gamma C + T_w}{A_0(1+bp)[1+(\alpha_c+\alpha_p)(T-T_r)][1+d(p_z-p_j)]} \tag{4}$$

$$P = \frac{\sum_{1}^{i} M_i g(1 - \rho_{\text{air}}/\rho_M) + \gamma C}{A_0[1+bp][1+(\alpha_c+\alpha_p)(T-T_r)]} \tag{5}$$

where M is the mass of the load applied to the piston, g is the local acceleration due to gravity, ρ_{air} and ρ_M are the densities of ambient air and of the weight respectively, γ is the surface tension of liquid, C is the circumference of the piston where it emerges from the fluid, b is the pressure coefficient of the area, A_0 is the effective area of the piston at standard pressure and temperature, α_C and α_P are the thermal expansivities of the cylinder and the piston respectively, and T the temperature of the assembly and T_r the reference temperature (standard temperature), d, the jacket pressure coefficient, p_j is the jacket or controlled pressure, p_z the jacket pressure at which the clearance is reduced to zero and T_w is the Tare weight. The coefficients of d, p_z and p_j are determined experimentally without the need for another calibrated pressure gauge.

Force and area are readily measurable at standard temperature and pressure. Methods have been developed to determine and account for various corrections such as air buoyancy $(1-\rho_{\text{air}}/\rho_M)$, temperature $[1+(\alpha_c+\alpha_p)(T-T_r)]$, surface tension, γ_c, elastic distortion $(1+bp)$ etc. Besides all these corrections, the most important parameter is the change of effective area with pressure i.e. A_{eff}, which can be defined in terms of the effective area of the piston A_p and the cylinder A_c as follows (Johnson and Newhall 1953; Johnson *et al* 1957),

$$A_{\text{eff}} = (A_p + A_c)/2. \tag{6}$$

For a cemented tungsten carbide piston and a steel cylinder with a ratio of outer to inner diameter as 3, the value of the pressure coefficient b is $2{\cdot}42 \times 10^{-3}$ at 0·7 GPa which is quite large and must be taken into account while measuring the pressure from the piston gauge.

In most cases the slow transport of leakage of the pressure fluid through the narrow interspace between the matching surfaces of the components is often misinterpreted as a source of measurement uncertainty in these gauges. But in case of the controlled clearance piston gauge, by appropriate adjustment of the jacket pressure it is possible to vary the radius of the cylinder until the radial separation between this and the piston becomes effectively zero at some position along the length of engagement. In this condition, there will be no transport of fluid through the system and if the radius of the piston in the region or band of zero clearance is known to be r, the effective area under these conditions is given by $A_{\text{eff}} = \pi r^2$, a condition which is neither recommended nor followed in practice, as it would result in damaging the piston.

A comprehensive report on the treatment and measurement uncertainties associated with different parameters of (4) has been discussed in detail by Heydemann and Welch (1975) and Lewis and Peggs (1979). Apart from the different parameters already mentioned which contribute some finite value of uncertainty in the measurement of pressure, the elastic distortion of the piston and cylinder is the leading cause of introducing the uncertainty in the measurement of high pressures and this effect has been reported at pressures as low as 160 kPa and hence the need for their calibration against the primary standard having relatively low uncertainty in the measurement of pressure.

3. Calibration of the piston gauge

The calibration of a piston gauge essentially consists of determining the effective area of its piston, which at low pressures does not envisage any problem and can either be done by dimensional metrology (Lewis and Peggs 1979), or by direct comparison with a mercury manometer (figure 9) or by comparison with a standard piston gauge. However, attempts made to find out the effective area by comparing the piston gauge with the pressurised mercury column at high pressures have not been very successful. Here are some of the details of the commonly used method for determining the effective area of the piston.

3.1 *Similarity method*

The experimental determination of the distortion coefficient has been reviewed by Dadson *et al* (1965) and has since been followed at the National Physical Laboratory, U.K., for calibration of piston gauges. The basic principle adopted in this method is first to determine the ratio of the effective area of the steel piston in cylinder assembly of a given type, at a series of pressures, to that of a precisely similar assembly constructed of a material having substantially different elastic modulus. This procedure determines the difference between the distortion factors of the two assemblies; the quotient of the two distortion factors, is obtained from measurements of the elastic moduli of the materials. The combination of these results allows the distortion factor of each assembly to be derived as a function of pressure in absolute terms. The effective area of the piston at a pressure p is connected with that at zero pressure by an expression of the form

$$A_p = A_0[(1+\lambda f(p)](bp = \lambda), \tag{7}$$

where λ may be termed as the distortion coefficient, involving for example, the elastic constant and dimensions of piston and cylinder, and $f(p)$ is an unknown function of

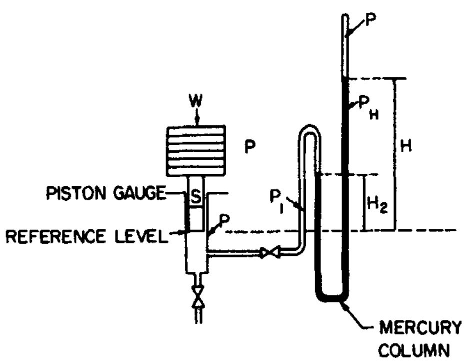

Figure 9. Measurement of effective area of a piston gauge with a mercury manometer.

pressure assumed to be the same for gauges made of different materials but with exactly the same dimensions.

Experience shows that the distortion is commonly a linear function of pressure and $f(p)$ may then be replaced by p. If two gauges of precisely the same dimensions are at hand, the ratio of their effective areas can be expressed as (Lewis and Peggs 1979),

$$\frac{A_p}{B_p} = \frac{A_0(1+\lambda_A p)}{B_0(1+\lambda_B p)} \approx \frac{A_0}{B_0}[(\lambda_A - \lambda_B)p] \tag{8}$$

$$= \frac{A_0}{B_0}[1+(1-K)\lambda_A p], \tag{9}$$

where $K = \lambda_B/\lambda_A$. From a cross float of the two gauges the ratio of the elastic moduli can be determined from the results of measurements of the elastic constants using either quasi-static or ultrasonic measurements. One of the essential conditions in this method is that the Poisson's ratio of the two materials should be the same.

An alternative method is the flow method, where flow of a pressure transmitting medium through the annulus between the piston and cylinder is first determined for an assembly, and then for the same assembly but with a piston having a small and well-defined different diameter. Agreement between the two methods is found to be within the experimental uncertainties. The final uncertainty in the measured distortion coefficients is reported (Dadson *et al* 1965) to be about ± 1 part in 10^5 parts at 100 MPa and increasing in proportion at higher pressures.

3.2 *Refinement in the theoretical treatment of the distortion factors on the basis of the elastic theory*

The elastic distortion of the piston and cylinder depends upon the ratio of the pressure in the annulus between them and to the pressure under the piston. Heretofore, as the proper value of this ratio or a method to determine it was unknown, the practice had been to assume a value of 0·5 when calculating distortion effects whereas Bass (1978) calculated this value as 0·8. Recently, Welch and Bean (1984a) have verified this value experimentally on the basis of the clearance between the piston and the cylinder by measuring the pressure between them. Their studies show that the appropriate value of the pressure ratio for calculation of the distortion is marked by a sharp decrease in the slope of the pressure ratio curve (figure 10). Further they have suggested that the

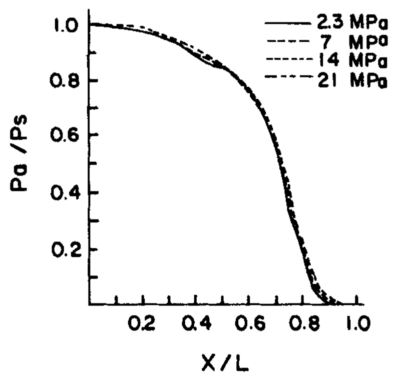

Figure 10. Ratio of the pressure in the annulus between the piston and cylinder to the pressure in the system measured at four system pressures as a function of the ratio of the position along the cylinder to its length for liquid 34.

corresponding value on the length axis is the appropriate location to calculate the area of the gauge from the dimensional measurements.

3.3 *Cross float method*

For any free piston gauge, when the pressure is balanced, the piston will slowly fall due to pressure-transmitting fluid past the piston. For the controlled clearance gauge this fall rate may be controlled by the jacket pressure. Therefore the area of the cylinder can be determined in two ways. The first is from elastic theory; $A_c = A_p[1+d(P_z - P_j)]$, where the parameter d, can be calculated (Yasunami 1967) as

$$d = \frac{4k^2(1 \times \sigma^2)}{E(k^2 - 1)},$$

where σ is Poisson's ratio, k the wall ratio and E the Young's modulus of the cylinder. Normally the correction is small enough to neglect σ^2. Another approach is to experimentally determine the change in the apparent pressure with jacket pressure (Heydemann and Welch 1975).

The effective area of the piston can as well be found experimentally by comparing the same with that of the piston gauge, whose effective area of the piston is already known, to a sufficiently high accuracy by connecting them to a common pressure system as shown in figure 11. When the loads on the two pressure gauges have been adjusted such that both are in equilibrium, the ratio of their total loads represents the ratio of the two effective areas at that pressure considering the common reference level and the establishment of the equilibrium conditions, there being no net fluid flow through the pressure line. For high accuracy work it is necessary to have an on/off valve preferably of the constant volume type (Markus 1972) in the pressure line between the two assemblies and some means of monitoring the vertical movement and position of the floating element of each assembly as shown in figure 12. For this purpose a null indicator along with the differential pressure cell is quite useful during the use of two different pressure-transmitting fluids and only null indicator in case a single pressure-transmitting fluid is used.

The accurate gas/oil cross float also requires that the hydrostatic pressures generated by the relatively dense oil be accounted for, which needs the monitoring of the height of the oil piston and the height of the gas/oil interface with an uncertainty as small as 0·1 mm. For this purpose, a conventional diaphragm-type differential pressure transducer which fixes the height of the gas/oil interface is used; however, the

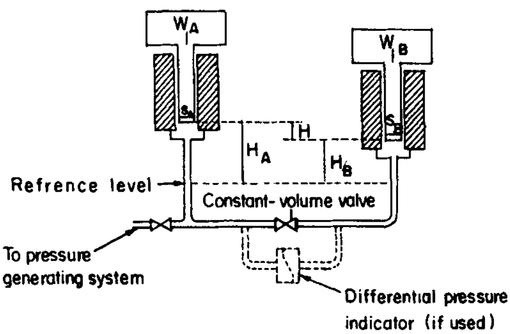

Figure 11. Schematic set-up of the cross float for calibration of piston gauges.

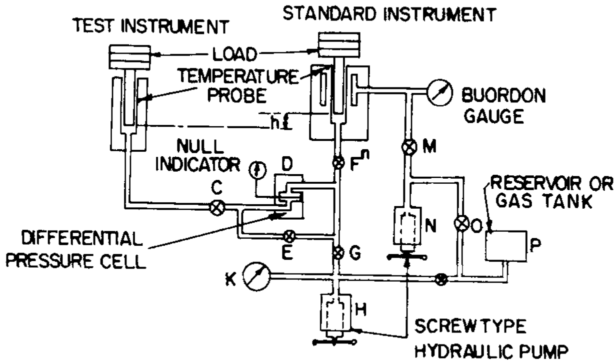

Figure 12. Experimental arrangement for comparison of effective areas of piston gauges.

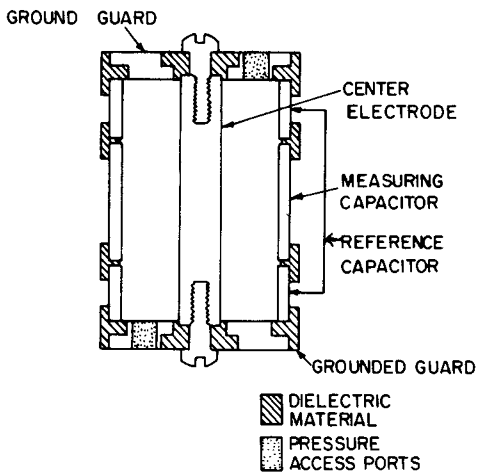

Figure 13. Cross-section of the cylindrical capacitance cell.

establishment of maintenance of its zero the differential pressure indicator still requires the periodic use of a free-surface interface. In order to achieve better resolution of fluid head correction, the conventional diaphragm-type pressure transducer has been replaced by the more recently developed cylindrical capacitance cell by Tilford and Martin (1984), as shown in figure 13 for free surface gas/oil interface to aid in the comparison of gas piston gauges with oil piston gauges wherein the coaxial three-terminal capacitor partially immersed in the oil and partially in the gas permits the determination of hydrostatic heads and differential pressures between the piston gauges. The standard deviation of 4·5 Pa over the range 0·5–4 MPa has been achieved from this cell.

Calibration should be performed at different pressure values in ascending and descending orders of weights to avoid any force error, and with clockwise and anticlockwise rotation of the piston to eliminate any cork screwing effect due to helical micro-scratches. After determining all the values associated with different parameters corresponding to the terms in (4) which contribute to the total uncertainty which is the sum of the random uncertainty from the cross float and the systematic uncertainty of the measurement (calibration) of standards, the fractional uncertainty in pressure measured can be expressed as the root mean square of the total differential,

$$\frac{\mathrm{d}p}{p} = \left[\sum_{1}^{i} \left(\frac{1}{p} \frac{\partial p}{\partial x_i} \mathrm{d}X_i \right)^2 \right]^{1/2}, \tag{10}$$

where X_i is the individual parameter of (4). On examination of (4) the contribution made by each parameter to the total uncertainty in the pressure measured from this gauge, it is found that the largest contribution to the uncertainty in the measured pressure is due to A_0, P_z and d. The determination of A_0 is the dimensional metrology problem. However the values of P_z and d depend upon the condition under which the gauge is used/operated, for example, the nature of pressure-transmitting fluid, temperature and speed of rotation of the piston and the way of measurement of its fall rate etc.

In the past it was assumed that the fall rate in a controlled clearance piston gauge is independent of the pressure-transmitting fluid provided the fluid did not freeze (Johnson and Newhall 1953; Newhall *et al* 1979). It was also assumed to be independent of rotational rate of the piston as long as it did not add extra heat to the piston and cylinder assembly and maintained a good lubricating film between them to prevent static friction. Detailed studies by Sharma *et al* (1983, 1984) on the characterization of the controlled clearance piston gauge as a function of viscosity of the different pressure transmitting fluids (figure 14) clearly show that the value of P_z is nearly independent of viscosity over a wide range upto some critical value of the order of 60 CP. Above this critical value, P_z depends strongly upon viscosity. The best choice of fluid is the one having the lowest viscosity at the operating pressure (Sharma *et al* 1983, 1984) which can be selected on the basis of having the most linear plot of P_z as a function of pressure (curves C and D in figure 14) and not the fluid having the variation of P_z with pressure as represented by curves A and B in figure 14. These results had been further confirmed by recent experiments carried out at PTB, Germany by Jäger and Bandyopadhyay (1985).

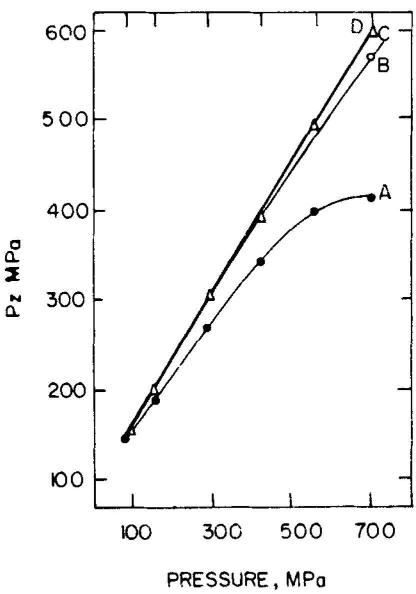

Figure 14. P_z as a function of pressure for the four fluids having zero heptane $H(0)$ to 90% heptane ($H90$) in a mixture of 100% Univis J-13 oil, $H0$, 100(A), $H50$, 50(B), $H65$, 35(C), and $H90$, 10(D) at 177 rpm and 297 K.

An additional evidence that the operation of piston gauges may be independent of viscosity over some range is found in other results as well (Welch and Bean 1984b). However a clear understanding of the role of viscosity of the working fluid in piston gauges is not yet available as it is known (Welch and Bean 1984a) that the viscosity changes by a factor of 2 for different fluids over the pressure range of study whereas the theory regards viscosity to be a constant.

The total fractional uncertainty in the pressure measured from the piston gauge can be further improved by monitoring the temperature of the piston and the rate of rotation. It has been reported (Sharma *et al* 1984) that for a variation in the temperature of the piston from 296–291 K, the change in effective area due to P_z thermal effects, introduces an uncertainty in the measured pressure by 26 ppm at 427 MPa and it increases to 37 ppm when the temperature of the piston is increased from 296–302 K with a low viscosity pressure transmitting fluid. However these values would be quite large, of the order of 71 ppm and 168 ppm, when the temperature of the piston is changed from 296–291 and 296–302 K respectively using high viscosity pressure-transmitting fluid. In addition, these studies offer the optimum value of the rotation rate of the piston in a controlled clearance piston gauge during measurement on the basis of the smooth variation of P_z as a function of rate of rotation with a particular type of the pressure-transmitting fluid. This is in contrast to the rather unusual effects reported by Newhall *et al* (1979) for DTE 24 oil at rotation rate under 50 rpm. The above studies (Sharma *et al* 1984) point the need to use piston gauge under the same operational and environmental conditions as prevailing during characterization.

The merits and demerits of different approaches for the calibration of piston gauge i.e. theoretical, similarity, flow and the cross float method demand constant comparison and analysis of the data obtained by these techniques to provide a valuable check over them.

Further, attempts have also been made to calibrate the piston gauge in the pressure region as high as 1 GPa in the static balancing conditions instead of the dynamic balancing conditions as usually adopted. This has been achieved by the lever-type controlled clearance piston gauge by Yasunami (1968) of 11·3 mm diameter piston wherein the state of no leak and no friction is achieved with an application of the external pressure calculated theoretically. Yasunami feels that the added difficulty of determining the lever arm ratio is offset by the greater ease with which large piston area can be determined. Moreover, a slight temperature rise at the effective area point is observed even after a few hours of operation in such conditions. The estimated limit of uncertainty of this instrument is less than $\pm 1{\cdot}5 \times 10^{-4}$ which is quite below the accepted uncertainty of any instrument over this range to be used for calibration of secondary/transfer standards and hence it has not been used any further.

Recently Yamamoto *et al* (1985) designed and built a new 500 MPa controlled clearance piston gauge with the larger diameter piston to work as the national primary pressure standards at NRLM (Japan). They have measured the pressure with non-rotating piston to avoid fluctuations in the pressure which according to them are of the order of 10 ppm in case the pressure is measured with a rotating piston. Preliminary studies showed that ultimate stability and reproducibility of 1 ppm over the pressure range upto 500 MPa can be achieved with this instrument. An extensive investigation regarding its stability, reproducibility and the frictional effects etc has to be carried out before it could be used as a pressure standard by pressure metrologists.

4. Precautions

For the attainment of optimum performance of the piston gauge one should be quite careful about the different factors such as tilting of the gauge, frictional effects between the piston and the cylinder, cork screwing etc as they may result in the extraneous vertical component of force on the floating system.

It is imperative to keep dirt out of the pressure-transmitting medium. It is also essential that leaks in all parts of the system attached to the gauge be avoided, for line drops of pressure between the gauge and the point of leak could lead to error in the pressure measurement.

During calibration, observations should be recorded in increasing and decreasing order of pressure to see the hysteresis and reproducibility; this would further recheck the load applied to the piston gauge.

Since, primary standards are unlikely to be used for direct measurement or calibration of working instruments which are normally made with secondary standards, the calibration of secondary standard instruments needs to be established at some stage by reference to the instruments of higher accuracy which may be the primary standards. Hence during calibration of the secondary or transfer standards against the primary piston gauge, as mentioned earlier, it is essential that the primary piston gauge be used in the same operational and environmental conditions as the one used in normal operations.

5. Transfer standards

As discussed earlier, by comparing the results of dimensional metrology with those obtained experimentally, a considerable confidence level is added in the evaluation of the effective area of a piston in a particular laboratory. But for promoting the uniformity in high pressure measurements and to facilitate inter-laboratory comparison of pressure dependence phenomena, a large number of international inter-comparisons have been organized by BIPM under the chairmanship of Dr G F Molinar from 1980 onwards upto 100 MPa pressure using piston gauges (Molinar and Vattasso 1976; Peggs *et al* 1980). The inter-laboratory differences of the effective area at 100 MPa lie within $\pm 78 \times 10^{-6}$ and within the combined laboratory uncertainties (uncertainties of individual laboratories in effective area determinations at 100 MPa range from $\pm 42 \times 10^{-6}$ to $\pm 88 \times 10^{-6}$) on the basis of 3 times the standard deviations which are of the order of $\pm 30 \times 10^{-6}$ in the low pressure region (Molinar 1985).

In the region below 280 MPa, the transfer standard piston gauge is portable and relatively robust whereas the pressure standard beyond 280 MPa is usually restricted to one site which limits the intercomparison of piston gauge standards beyond this region.

The most important problem faced by pressure metrologists these days is the selection of transfer pressure standards in the range of 0·1 GPa to several GPa.

The necessary requirements for the transfer standards are that it must be easy to employ, should have minimum possible hysteresis, high reliability and stability to make it useful for the reproducible pressure measurements. The subject of transfer pressure standards had already been discussed in detail by Peggs (1980). However, it would be appropriate to summarize here the performance and accuracies of some of the generally used accurate and reproducible transfer pressure standards.

A large variety of the high pressure transducers, as already mentioned, are available for accurate pressure measurements upto about 1 GPa but of these gauges the manganin-resistive and strain gauges which are quite accurate and reproducible are discussed below.

The piezo-resistive manganin gauges are extensively used as transfer pressure standard above 0·1 GPa. The main principal advantage of the manganin-resistive transducer over other transducers is because of the large amount of research work carried out in studying the behaviour of manganin coils under different ambient and hydrostatic pressure conditions (Yamamoto 1972a, b; Morris 1978; Molinar *et al* 1980; Houck *et al* 1983). It is found that these gauges are stable within an accuracy of 150 ppm at 1·3 GPa over two months but their long-term stability over 2 years gives the inaccuracy of the order of 1500 ppm at 1·4 GPa. Similar long-term stability has also been observed by Johnson (1963), Yamamoto (1972b) and Jäger and Wanninger (1976) whereas the manganin gauge used by Klingenberg (1981) shows poor stability over a period of 14 years. All the manganin gauges show a parabolic behaviour with temperature and gives the maxima around 304 K and when calibrated against the primary controlled clearance, the piston gauge (Sharma *et al* 1986a) shows the overall reproducibility of the data upto a pressure of 457 MPa better than $\pm 5 \times 10^{-4}$ and for the pressure measured by the manganin gauge was well within $\pm 4 \times 10^{-4}$. Moreover the hysteresis effect in such gauges, which is an inherent characteristic of the gauge, is quite low at high pressures and becomes prominent at low pressures. Yamamoto (1972b) performed several experiments which gave valuable information on the various constructional techniques and methods of stabilizing the resistance characteristics of the coil whereas the manganin-resistive gauge manufactured by Bean (1975) of National Bureau of Standards differs with the manganin coil made by Yamamoto (1972b) and shows quite a low hysteresis effect.

Strain gauge transducers (Birks and Gall 1984; Jäger and Wanninger 1976) are beginning to be used to measure pressures upto 1 GPa but they show a prominent hysteresis effect (Sharma *et al* 1986b) of the order of $\pm 1{\cdot}07 \times 10^{-3}$ to $\pm 0{\cdot}56 \times 10^{-3}$ but the overall reproducibility of the data over a full pressure range is always better than 5×10^{-4} similar to the manganin gauges (Sharma *et al* 1986a). When the results of the pressure-measured values of NPL are compared with those found at IMGC using the same transducer it is found (Molinar *et al* 1986) that these values agree well within 1×10^{-3} for the strain gauge transducer and $\pm 5 \times 10^{-4}$ for the manganin gauge transducer which is the acceptable limit of accuracy of any device to be used to measure pressure in this region.

Further it is well established (Jäger and Wanninger 1976; Sharma *et al* 1986a) that the shift in the initial zero of these transducers is always less than the 0·1 % full scale of the pressure transducer which is well below 10 % of the full scale of the gauge; hence even if there is a slightest shift in the initial zero, the calibration equation of the transducer still remains valid.

Moreover it is often reported that the calibration equation of the transducer can very well be represented by linear fitting; however Molinar *et al* (1980) found that while calibrating the manganin gauge against the mercury melting line and the primary pressure standard over a pressure range of 1 GPa, the best calibration equation can be represented with a polynomial fitting. They have found that the polynomial fitting shows deviation upto 1·3% in the pressure region 1·0–1·3 GPa whereas the deviation increases to + 1·7% if the linear fit calibration equation is used over the same pressure range which clearly show that polynomial fitting is preferred to linear fitting. On the basis of the extensive investigation carried out on these transducers it can well be stated that these transducers can be used as working pressure standards upto a pressure of 1 GPa.

Apart from the manganin-resistive and the strain gauge transducers (Darling and Newhall 1953; Bock and Wisniewski 1977) the gold-chromium and the semiconductor resistance gauges (Connell 1969; Vyas 1974) are also being tried but could not yet be adapted in practical use as transfer standards due to the large temperature coefficients in the former and the low resistance measurement and poor temperature characteristics of the latter. However the zeranin-resistive gauges, an alloy of the germanium, manganese and copper, seem to be a good candidate to replace the manganin-resistive gauges in future. Preliminary results show (Birks and Gall 1973; Wisniewski and Bock 1976) quite a promising future for this material with particular reference to hysteresis, linearity with pressure and high pressure coefficients etc.

As all the primary pressure standards against which the transfer pressure/secondary pressure standard are calibrated, are bulky and expensive pieces of equipment, they are not quick to use, as in any balancing operation the time required for the fine adjustments is large. It is possible that these gauges suffer from ageing effects for the first few years after manufacture, although it is by no means clear whether all gauges suffer from this disadvantage. A history of a gauge over an extended period of time has recently been given by Reamer and Sage (1969). Therefore, a considerable accuracy in the calibration of the pressure equipment can be attained when calibrated using the pressure-fixed points on the pressure scale over the pressure range of few GPa.

6. Pressure scale/fixed points

The mercury melting line has been investigated by a number of workers (Dadson and Greig 1965; Lloyd 1971; Decker *et al* 1972; Molinar *et al* 1980) upto a pressure of 1·2 GPa. Some of them have detected the melting of mercury by change in resistance whereas others have done it by measuring either the latent heat or the change in volume. Morris (1978) reported the new pressure value for the mercury melting which differs considerably from the prediction of the equation based on the Simon equation (Simon and Glatzel 1929) and to some extent by the value reported by Michels *et al* (1942). However Morris adopted the third-order polynomial to fit the experimental data and the values found agree with earlier values reported by Houck (1970). Subsequent detailed investigations carried out by Molinar *et al* (1980) have confirmed that the mercury melting pressure at 0°C is 756·84 ± 0·16 MPa which is in close agreement with the values obtained by Dadson and Greig (1965). However a systematic difference has been observed when compared with the calibration equation of Bodganov *et al* (1971) upto 1200 MPa and a good agreement with the values obtained by others (Houck 1977; Morris 1978). The experimental pressures observed by Molinar *et al* (1980) have been fitted very closely to a third-order polynomial in temperature. This equation fits the melting line data much more closely than the Simon type, and therefore, they have recommended its use upto 1200 MPa to increase the accuracy of a practical pressure scale based on the melting line of mercury. In recent times, to recheck the accuracy of the mercury melting pressure and to re-establish the pressure scale, detailed studies have been made at PTB (Jager and Bandyopadhyay 1985) using the volumetric method, and the melting pressure of mercury at 0°C is found to be in close agreement with earlier reported values within experimental uncertainties.

But as we go to still higher pressures of the order of 1·5 GPa and above, the mercury melting pressure scale is no longer valid and in that region the necessity of other fixed points does arise. An extensive literature on the fixed points is available and the need to establish the fixed points, their accuracies and for adding new fixed points over the wide range of required pressure are clearly emphasized. In general at higher pressures bismuth, barium and tellurium transitions are being used for pressure calibration and at still higher pressures the frequency shift of the Ruby fluorescent line is used. These have been discussed in detail by Barnett *et al* (1973), Piermarini *et al* (1975) and Jayaraman (1983).

References

Alexander J M and Lengyel B 1967 *Proc. Inst. Mech. Engr.* **180** 317
Barnett J D, Block S and Piermarini G J 1973 *Rev. Sci. Instrum.* **44** 1
Bass A H 1978 *J. Phys.* **E11** 682
Bean V E 1975 *Proceedings of the II Int. Conference on High Pressure Engineering, Brighton, UK* (London: Institute of Physics) p. 29
Bett K E, Hayes P F and Newitt D M 1954 *Philos. Trans. R. Soc. (London)* **247** 59
Birks A W and Gall C A 1973 *Strain* **9** 1
Birks A W and Gall C A 1984 The Queen's University of Belfast, Belfast, UK Report No 1566
Bock W J and Wisniewski R 1977 *Rev. Sci. Instrum.* **48** 336
Bodganov V S, Levin Yu L, Sekoyan S S and Shmin Yu I 1971 *Accurate characterization of the high pressure environment* (ed.) E C Lloyd, NBS Special Publication (Washington DC: US Govt Printing Office) **326** 297
Bridgman P W 1911 *Proc. Am. Acad. Arts Sci.* **47** 321
Bundy F P 1977 *High pressure technology* (eds) I L Spain and J Paauwe (New York: Marcel Dekker) **2** 321
Comings E W 1956 *High pressure technology* (New York: McGraw Hill)
Connell G A N 1969 *High Temp. High Pressures* **1** 77
Dadson R S and Greig R G P 1965 *Br. J. Appl. Phys.* **16** 1711
Dadson R S, Greig R G P and Horner A 1965 *Metrologia* **1** 55
Darling H E and Newhall D H 1953 *Trans. ASME* **75** 311
Decker D L, Bassett W A, Merrill L, Hall H T and Barnett J D 1972 *J. Phys. Chem. Ref. Data* **1** 773
Heydemann P L M and Houck J C 1969 *J. Appl. Phys.* **40** 1609
Heydemann P L M and Welch B E 1975 *Experimental thermodynamics* (eds) B LeNeindre and B Vodar (London: Butterworths) **2** 147
Houck J C 1970 *J. Res. Nat. Bur. Std.* **74** 51
Houck J C 1977 *J. Appl. Phys.* **48** 605
Houck J C, Molinar G F and Maghenzani R 1983 *J. Res. Nat. Bur. Std.* **88** 253
Jäger J and Bandyopadhyay A K 1985 (internal report)
Jäger J and Wanninger W 1976 *Feinwerktech. Messtech.* **84** 387
Jayaraman A 1983 *Rev. Mod. Phys.* **55** 65
Jayaraman A and Maines R G 1971 *Proc. Symp. Accurate Characterization of the High Pressure Environment*, NBS Spec. Pub. 326 (Washington DC: US Govt Printing Office)
Johnson D P 1963 *High pressure measurements* (eds) A Giardini and E Lloyd (Washington DC: Butterworths)
Johnson D P, Cross J L, Hill J D and Bowman A H 1957 *Ind. Engg. Chem.* **49** 2046
Johnson D P and Heydemann P L M 1967 *Rev. Sci. Instrum.* **38** 1294
Johnson D P and Newhall D H 1953 *Trans. Am. Soc. Mech. Eng.* **75** 301
Kennedy G C and LaMori P N 1962 *J. Geophys. Res.* **67** 851

Klingenberg 1981 *PTB Mitteilungen* **91** 33
Konyaev Yu S 1961 *Prib. Tekh. Eksp.* **4** 107
Lewis S and Peggs G N 1979 *The pressure balance; A practical guide to its use* (Teddington, UK: National Physical Laboratory)
Lloyd E C (ed.) 1971 *Accurate characterization of the high pressure environment*, NBS Special Publication (Washington, DC: US Govt Printing Office) **326** 313
Marano J P and Jenkins J M 1977 *High pressure technology* (eds) I L Spain and J Paauwe (New York: Marcel Dekker) **2** 61
Markus W 1972 *Rev. Sci. Instrum.* **43** 158
Michels A, Wassenaar T and Blaisse B 1942 *Physica* **9** 574
Molinar G F 1985 Report on the activity of the BIPM Working Group on High Pressures
Molinar G F, Bean V, Houck J and Welch B 1980 *Metrologia* **16** 21
Molinar G F, Sharma J K N and Jain K K 1986 Intercomparison of pressure standard upto 500 MPa using pressure transducers, IMGC Tech. Rep. R 228
Molinar G F and Vattasso M 1976 *High Temp. High Pressures* **8** 259
Morris E C 1976 *Aust. J. Instrum. Control* **32** 77
Morris E C 1978 *Metrologia* **14** 105
Newhall D H, Ogawa I and Ziberstein V 1979 *Rev. Sci. Instrum.* **50** 964
Peggs G N 1980 *High Temp. High Pressures* **12** 1
Peggs G N and Lewis S 1977 *J. Phys.* **E10** 1028
Peggs G N, Lewis S and Legrass J C 1980 NPL Report MOM 39
Piermarini G J, Block S, Barnett J D and Forman R A 1975 *J. Appl. Phys.* **46** 2774
Reamer H H and Sage B H 1969 *Rev. Sci. Instrum.* **40** 183
Schwacha B C 1961 US Patent No 2 985050
Sharma J K N, Jain K, Bean Vern E, Welch B E and Lazos Ruben J 1983 *Proc. IX AIRAPT Conference held at Albany, Sunny, New York*
Sharma J K N, Jain K, Bean Vern E, Welch B E and Lazos Ruben J 1984 *Rev. Sci. Instrum.* **55** 563
Sharma J K N, Jain K and Molinar G F 1986a NPL (India) Research Report NPL-86-A.3-0062
Sharma J K N, Jain K and Molinar G F 1986b Sensors and actuators (to be published)
Simon F and Glatzel G 1929 *Z. Anorg. U. Allg. Chem.* **178** 309
Tilford C R and Martin D F 1984 *Rev. Sci. Instrum.* **55** 95
Vyas M K R 1974 *High Temp. High Pressures* **6** 237
Welch B E and Bean Vern E 1984a *Rev. Sci. Instrum.* **55** 1901
Welch B E and Bean Vern E 1984b *High pressure in science and technology* (eds) C G Homan, R K MacCrone and E Walley (New York: Elsevier)
Wisniewski R and Bock W J 1976 *Arch. Elektrotech (Warsaw)* **25** 789
Yamamoto S 1972a *Bull. Nat. Res. Lab. Metrol. Tokyo* **24** 23
Yamamoto S 1972b *Bull. Nat. Res. Lab. Metrol. Tokyo* **25** 1
Yamamoto S, Ooiwa A and Ueki M 1985 National Research Laboratory of Metrology (Japan) (Private communication)
Yasunami K 1967 *Proc. Jpn Acad.* **43** 310
Yasunami K 1968 *Metrologia* **4** 168