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The Accurate Measurement of High Pressures and the Precise Calibration of Pressure Balances

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IN CONNEXION with the work of the National Physical Laboratory on the P–V–T relationships of gaseous mixtures, attention has been given to improvement in the accuracy of high-pressure measurements and in particular to the precise calibration of pressure balances.

The paper describes the progress made in this work with special reference to the 'similarity' method developed at the N.P.L. for the measurement of the variation of the effective areas of pressure balances under the influence of elevated pressures.

INTRODUCTION

THE ACCURATE ESTIMATION of the pressure-volume-temperature relations of gases or gaseous mixtures involves the precise measurement of four quantities, namely the pressure, the volume and the temperature of the substance, and in certain cases the measurement of the mass. For a considerable time now measurements of mass, volume and temperature have been on a very precise basis, as will be clear from reference to the standards maintained at centres such as the National Physical Laboratory and other similar standardizing laboratories. The measurement of pressure, however, is in a very different category. It is true that measurements of considerable precision are available in the region of 1 atm. but at higher pressures the precision of pressure measurements falls considerably short of those of the other quantities mentioned.

In most investigations involving the accurate measurement of high pressures, the measuring instrument employed is the pressure balance, or dead-weight gauge, in which the fluid pressure acting on a piston of known area is balanced by a load derived from a set of accurately calibrated weights. For practical use, no other instrument can compare in precision with the pressure balance, and its only serious rival is the mercury manometer, the direct use of which at high pressures would involve serious complications. In practice therefore, the precise determination of a high pressure virtually reduces to the problem of the determination of the effective area of a pressure balance, and the dependence of this quantity on the pressures to which it is subjected. Most past attempts to calibrate pressure balances have relied on the use of high-pressure mercury columns, but the published investigations have given very variable results, to such an extent that when the Laboratory became concerned in this problem some three or four years ago, it was difficult to arrive at any consistent indication of the changes in effective area which pressure balances were likely to exhibit under the influence of elevated pressures. Michels (1923, 1924)† discussed this matter at length in his earlier papers and indicated by calculation the order of magnitude of the probable effect, but did not provide any confirmatory measurements. Beattie and Edel (1931), using a high-pressure mercury column, attempted to measure these variations up to pressures of about 500 atm. but found no measurable effect. Ebert (1935), on the other hand, using a method of a different character developed at the P.T.R.,‡ reported changes of effective area which were considerably in excess of those indicated by elementary theory. Recently, Professor Newitt and his co-workers at Imperial College (Bett and others 1954) have described a very carefully constructed high-pressure mercury column intended to act as a fundamental standard for pressures up to the region of 2,500 atm. but no results of any measurements of the effective areas of pressure balances made with this instrument seem to have appeared as yet.

In these circumstances it was decided to devote effort at the N.P.L. to the development of new methods for establishing the effective areas of pressure balances

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† *An alphabetical list of references is given in Appendix 1.VI.*

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with the general objective of reaching an accuracy of the order of a few parts in 10^5, at any rate over the first few thousand atmospheres. A preliminary description of the early stages of this work has been given by the author (Dadson 1955), but the investigation has now proceeded a good deal further and the present paper summarizes the progress made up to the present time.

It is convenient to divide the work into two separate parts; firstly the establishment of an accurate value for the effective area of a pressure balance at low pressures where distortion has no appreciable effect, and secondly the determination of the variation of this effective area consequent on the distortion of the balance when subjected to high pressures.

For the present purpose the condition with the piston of the balance freely rotating as recommended by Michels (1923, 1924) ~~has been consistently adopted~~.

ABSOLUTE DETERMINATION OF EFFECTIVE AREA AT LOW PRESSURES

In the determination of the effective area of a pressure balance at low pressures it has been possible to use two distinct and independent methods, one depending on measurement of the dimensions of the components, and the other depending on comparison with a low-pressure mercury column.

If the piston-cylinder assembly consists of a perfectly straight and uniform piston moving in a perfectly straight and uniform cylinder, both being accurately circular in cross-section, then a simple calculation indicates that the effective area of the combination should be equal to the mean of the cross-sectional areas of the piston and cylinder bore. In practice, cylinder bores are not likely to be sufficiently perfect for this simple result to be applicable, although the piston itself can normally be made with much greater uniformity. For the most part, accurately ground and lapped cylinder bores show some degree of bellmouthing near the ends and this may be sufficient to upset the validity of the simple formula. Recently, however, very accurate methods have been introduced for the measurement of the internal diameters of cylinder bores in which a notable contribution has been made by the Metrology Division of the N.P.L. (Taylerson 1955). Provided the diameter is accurately determined as a function of the position along the axis and any small departures from circularity are measured, the effective area of the assembly can be calculated, though by a somewhat more complicated formula than in the simple case. It may be shown by analysis of the flow of the pressure transmitting fluid through the gap between piston and cylinder that the effective area A_0 of the assembly may be represented by the formula

$$A_0 = \pi a^2 \left(1 + \frac{2}{a}\int_0^l h^{-2} dx \bigg/ \int_0^l h^{-3} dx\right) \quad \ldots\ldots \quad (1.39)$$

where a is the radius of the piston, $2h$ the width of the gap at distance x along the axis, and l the total length of engagement of the piston and cylinder. This evidently reduces to the result formerly given in the case where the gap between piston and cylinder is a constant. With an assembly of nominal diameter of the order of 0·5 inch, the expression (1.39) may be arrived at with a standard error of the order of only 1 part in 10^5.

The second arrangement employed for the measurement of an effective area at low pressures is that of direct comparison with a mercury-in-glass manometer of the general type frequently employed in previous work, and of which no detailed description need be given. If proper attention is given to various details of design such as the accuracy of scales, verniers and sighting arrangements, the choice of appropriate diameters for the tubes, and careful correction for the effects of any temperature variations on the mercury, the subsidiary oil columns, or the scales, it is possible to obtain measurements at pressures of the order 3-5 atm. ~~with a standard~~ error which is again in the region of 1 or 2 parts in 10^5.

Mercury Column

These two independent methods have been used at the N.P.L. to calibrate gauges of the direct-loading type with piston diameters of about 0·5 in., the two methods giving results which agree to within 2 parts in 10^5. The following example relates to a certain assembly of nominal area 0·2 sq. in. at 20 deg. C.:

effective area from diametral measurements	0·199897 sq. in.
effective area from comparison with mercury manometer	0·199894 sq. in.

The validity of the first method—that depending on calculation from the measured dimensions of piston and cylinder—may be checked independently of the mercury manometer by comparing the relative effective areas of different piston-cylinder assemblies with the relative values obtained by direct balancing of the assemblies one against another. The determination of the ratio of two effective areas by direct balancing can, with suitable precautions, be ~~carried out with~~ considerably higher accuracy than an absolute determination, and is limited only by the sensitivity of the balances to small changes in load and the accuracy to which the loading weights and any other factors affecting the load are known. Provided the load is not too small, so that the piston and load assembly can rotate without interference for sufficient time for a precise balance measurement to be made, such balancing experiments can normally be carried out to an accuracy of a few parts in 10^6. Table 1.28 ~~shows some~~ examples ~~of comparisons~~ between the ratios of the effective areas of different pairs of assemblies determined in this manner with the corresponding ratios derived by calculation from the piston and cylinder dimensions.

Table 1.28. Comparison of Effective Areas at Low Pressure by Two Methods

Nominal areas of assemblies compared, sq. in.	Ratio of effective areas	
	Computed from measured dimensions	From direct balancing test
0·2 and 0·125 (*a*)	1·59927	1·59923
0·2 and 0·125 (*b*)	1·59931	1·59933
0·125 and 0·125	1·00002	1·00001
0·05 and 0·05	1·00002	1·00004
0·02 and 0·02	1·00005	1·00004

DETERMINATION OF VARIATION OF EFFECTIVE AREA AT ELEVATED PRESSURES

The method recently developed at the N.P.L. (which may conveniently be named the 'similarity' method) is completely independent of the high-pressure mercury manometer, and depends on the application of a principle of similarity to the distortions of a pair of piston-cylinder assemblies of the same nominal dimensions but constructed of materials whose elastic constants are in a known ratio to one another. Provided that a number of conditions which are considered below are satisfied, the distortions of two such assemblies will remain proportional to one another throughout a range of pressure appropriate to the particular design of assembly. This state of affairs leads very directly to a method of determining the absolute value of the distortion of each assembly. Denoting by A_P and B_P the effective areas of the two assemblies at a high pressure P, and A_0 and B_0 the corresponding areas at zero pressure we may write

$$A_P = A_0\left[1 + \Delta(\alpha, P)\right]; \qquad B_P = B_0\left[1 + \Delta(\beta, P)\right] \quad \ldots\ldots (1.40)$$

where the quantities Δ (α, P) and Δ (β, P) represent the effects of distortion under the applied pressure, and are functions of P and of the constants α and β representing the elastic constants of the two materials. Ignoring small quantities of the second order—since the Δ values are very small compared with unity—this leads to the expression

$$\frac{A_P}{B_P} = \frac{A_0}{B_0}\left[1 + \Delta\,(\alpha, P) - \Delta\,(\beta, P)\right] \ldots\ldots (1.41)$$

As already discussed above, the ratio A_P/B_P may be determined to a high degree of accuracy by balancing the two assemblies directly against one another, in fact apart from trivial corrections the ratio is simply the ratio of the loads on the two balances when these are in equilibrium.

It is now necessary to consider under what conditions the distortions of the two assemblies may be taken to be proportional to one another. In the distortion of a system as complex as a practicable piston assembly, two independent elastic moduli will be involved. The expansion of the cylinder under internal pressure is largely determined by the modulus of rigidity of the material provided the external diameter is reasonably large compared with the diameter of the bore (Newitt 1940). In assemblies similar to Fig. 1.23 *a*, the piston

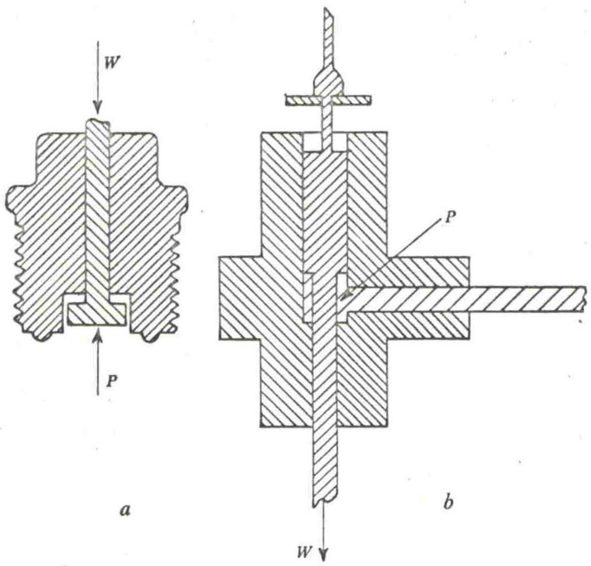

Fig. 1.23. Two Types of Piston–Cylinder Assembly

a Simple piston. *b* Differential piston.
The effective area of the assembly is the ratio of the load W to applied pressure P when the balance is in equilibrium.

is subjected to a compressive stress along its length due to the load and the pressure acting at its ends, and to a varying stress at right angles to its axis along the flanks due to the fluid pressure in the gap. The distortion of the piston will thus involve both the modulus of rigidity and the other independent modulus of the material. Strict similarity between the distortions of the two pistons therefore requires both moduli to be in the same ratio, or in other words that the values of Poisson's ratio for the two materials should be substantially the same.

Certain conditions must also be imposed on the mechanical forms of the components. For instance, the forms of the internal bores of the cylinders and of the pistons must be closely similar in contour, in order to ensure that the distribution of pressure in the gap between piston and cylinder is the same in the two assemblies. The similarity condition also requires that the widths of the gaps between piston and cylinder for the two assemblies at zero pressure should be inversely proportional to the elastic moduli of the materials. It is also clearly necessary that the elastic moduli should be constant over the range of stress concerned, and that the materials should be adequately isotropic as regards deformation under stress.

Provided these several conditions are adequately met, the distortions under pressure should be proportional to one another and should be in the inverse ratio of the elastic moduli of the two materials.

It is of interest to note that the distribution of pressure along the gap may be influenced by the manner in which the coefficient of viscosity of the fluid depends on pressure. For a given total flow of fluid and a given width of gap, the pressure gradient at any point will be proportional to the local value of the coefficient of viscosity. Since in turn the precise form of the distortion of the boundaries of the cylinder and piston depend upon the distribution of pressure along the gap, it is clear that the resulting distortion will depend in a complicated manner on a number of interacting factors. Under the similarity condition, however, all these complications are avoided.

Under the conditions postulated the distortions may be expressed in the form

$$\Delta(\alpha, P) = \alpha f(P); \; \Delta(\beta, P) = \beta f(P) \tag{1.42}$$

where $f(P)$ is an unknown function of the pressure but is the same function in the case of both assemblies, and α and β are, as before, two constants inversely proportional to the elastic moduli of the two materials. In these circumstances equation (1.41) may be written

$$\frac{A_P}{B_P} = \frac{A_0}{B_0}\left[1+(\alpha-\beta)f(P)\right] \tag{1.43}$$

The experimental procedure is now firstly to determine the quantity $(\alpha - \beta)f(P)$ by direct balancing over the range of pressure concerned, and secondly to determine the ratio α/β either by direct measurements of the elastic moduli or by any other method which may be available. The data provided by these two procedures clearly enables the distortion of each assembly to be derived in absolute terms.

In the present series of experiments the two materials employed are a very hard tool steel and a special bronze of high tensile strength, the elastic modulus of which was known to be about two-thirds that of steel which is a convenient ratio from the experimental point of view. Measurements of the ratio α/β have been made by two substantially different methods. In the first place direct measurements of the elastic constants have been carried out in the Engineering Section of the Physics Division using the customary static methods for determining these quantities over a wide range of stress. The stress-strain relationships were determined both for the modulus of rigidity, using the torsion method, and for Young's modulus, using the standard extensometers employed at the N.P.L. The values were also checked by the ultrasonic wave velocity method, where the stresses were considerably smaller than those involved in the pressure experiments. The ultrasonic measurements, however, provided a useful check of the degree of isotropy of the materials, and it may be noted that wave measurements in three directions at right-angles to one another gave practically indistinguishable results. One difficulty in the determination of α/β by the direct measurement of elastic moduli lies in the choice of samples of material for the construction of the necessary test pieces. The component parts of a piston-cylinder assembly are not themselves ideally shaped for extensometer measurements, and it is therefore important that test specimens should be chosen as far as possible from the same batch of material as that from which the assemblies themselves were constructed. If this is not possible care must be taken to ensure that the constitution of the material, and any heat treatments which may be necessary, are identical in both cases. Certain discrepancies in the elastic constant measurements which were encountered in the early stages of the work are thought to have been due to uncertainties in composition or heat treatment of some of the samples of steel which were employed.

In order to avoid the slight hazard attaching to the sampling of test pieces, and in an attempt to determine the ratio α/β on the actual assemblies themselves, an independent method has also been developed. This method makes use of precisely the same principle of similarity as has been discussed above. The general procedure is to measure the rate of flow of the pressure transmitting fluid through the gap between piston and cylinder in the case of each assembly at a series of corresponding pressures. Bearing in mind that the rate of flow for a given pressure distribution, and therefore a given distribution of the coefficient of viscosity, is a function of the width of the gap and the manner in which this is distributed along the axial length, a detailed examination shows that these measurements provide sufficient data for the calculation of the ratio of the distortions of the two gaps in terms of their original values at zero pressure. This method has given agreement as regards the value of α/β to within about 2 per cent with the value afforded by the direct measurements of the elastic constants, the mean being very close to 1·50.

Reference also needs to be made to the possible effects of inaccuracies or non-uniformity in the dimensions of the pistons and cylinders. The precise application of the principle of similarity as outlined above was at first thought to necessitate very accurate similarity between the contours of the components under the condition of zero pressure, and in the earliest measurements great care was taken to conform with this requirement. This proved to be possible owing largely to improvements which have been made in the Metrology Division of the Laboratory in the measurement of the form and diameters of cylinder bores (Taylerson 1955). In view of the resulting difficulties in construction, however, it was decided to investigate directly to what extent small departures from ideal similarity were likely to affect the results. For this purpose assemblies were constructed using alternative pistons of the 'wrong' diameter, so arranged that the initial dimension of the gap was incorrect by amounts as much as 20 per cent. These supplementary measurements showed that departures of this order of magnitude caused no significant difference in the rate of change of effective area with increasing pressure, and it may therefore be concluded that great exactitude in the choice of the initial condition is not as critical as might appear at first sight. Experience in the calibration of groups of piston-cylinder assemblies of the single-piston type of Fig. 1.23 *a* also confirms that the normal manufacturing tolerances of these assemblies do not cause any material departure from the similarity conditions postulated in the above treatment.

Important

RESULTS OF MEASUREMENTS OF VARIATION OF EFFECTIVE AREA

Fig. 1.24 *a-d* show a selection of the results which have been obtained on a number of different types of piston-cylinder assemblies. Fig. 1.24 *a-c* illustrate the results for three different ranges of pressure on assemblies of the same construction, differing only in the diameters of the pistons, these being some of the actual steel assemblies on which the basic similarity measurements were carried out. The type of assembly is a standard manufactured product and is similar to that illustrated diagrammatically in Fig. 1.23 *a*. This type of assembly is normally screwed into the top of a steel column and the load is supported on a carrier of the overhang type which in turn is supported on the top of the piston through the medium of a steel ball. The measurements all correspond to the condition with the piston rotating freely in the cylinder at a speed of the order 35-40 r.p.m. It will be noted that the changes of effective area of these assemblies were in most cases substantially linear functions of the pressure, the slope being nearly constant for the three pressure ranges which together cover the range up to about 3,000 atm. It has been found that assemblies of this general type show very consistent results as regards the rate of change of effective area, so that the examples shown may be regarded as reasonably typical.

Attention has, however, been drawn to the fact that the dependence of effective area upon pressure may be a function of the manner in which the coefficient of viscosity of the transmitting fluid depends on pressure. It has been found experimentally that variations from this cause do in fact occur, and an example is shown in Fig. 1.24 *b* in which the changes of area for a particular piston-cylinder assembly are shown over the range up to 2,000 atm., using three different transmitting fluids. As might have been expected, the dependence on the nature of the transmitting fluid is greatest over the upper part of the pressure range, this presumably

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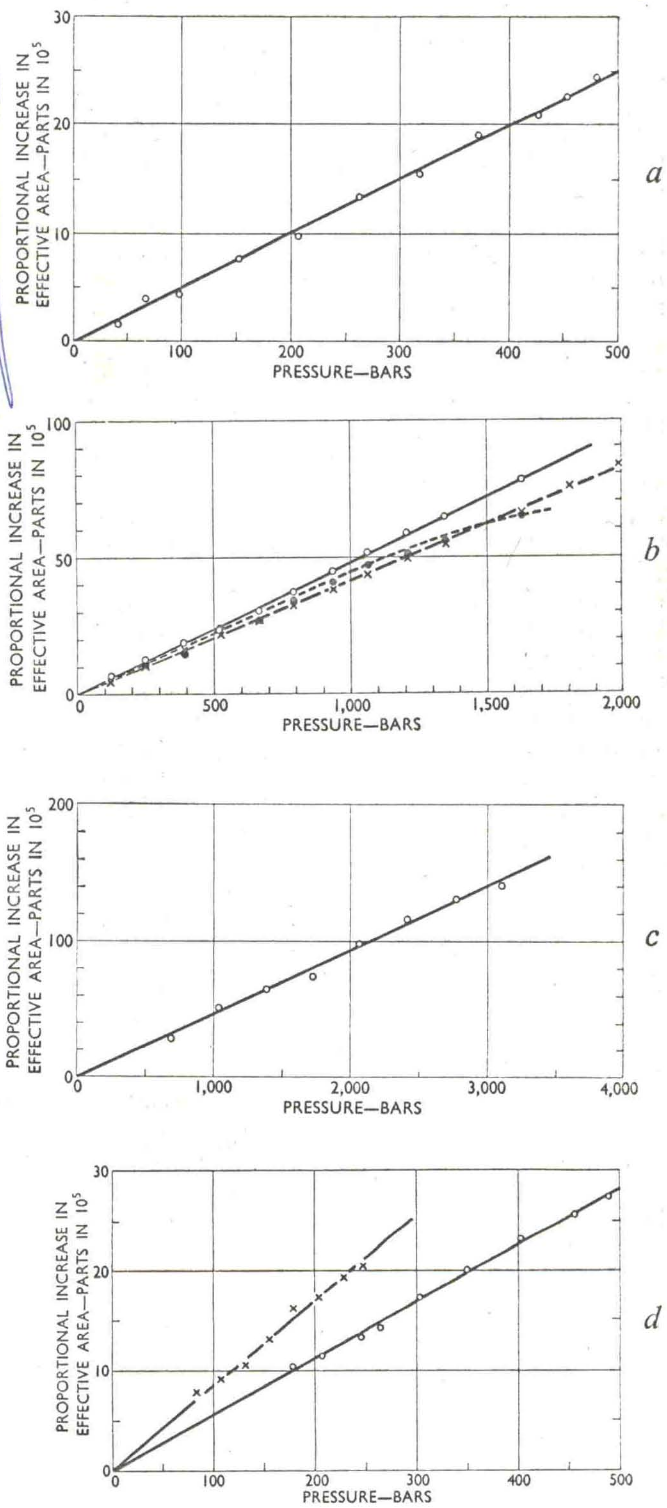

Fig. 1.24. Dependence on Pressure of Effective Area of a Steel Piston–Cylinder Assembly

a Nominal area 0·05 sq. in.
c Nominal area 0·01 sq. in.
d Differential piston cylinder.
× Nominal area 1 sq. cm.
○ Nominal area 0·5 sq. cm.

b Nominal area 0·02 sq. in.
○ Light mineral oil.
× Castor oil.
● Liquid paraffin.

being the range over which the dependence of viscosity on pressure is most variable as between the different fluids. Whilst the variations shown in Fig. 1.24*b* are not excessive in relation to the precision of the measurements, there is no reason to suppose that the full scope of these variations is necessarily covered by the three particular fluids which happen to have been used. It seems clear therefore that at high pressures it is necessary to associate any precise calibration of the effective area of a pressure balance with the particular transmitting fluid employed. It will be observed that in one case illustrated in Fig. 1.24*b*, where liquid paraffin was used as the transmitting fluid, the dependence of effective area upon pressure does not exhibit the same degree of linearity as in the other two cases—a light mineral oil and castor oil—in which the relationships can be represented by straight lines to within an accuracy of the order ± 1 part in 10^5. Whilst therefore the variations in the ~~case of assemblies~~ of this type seem to be for the most part linear, this condition cannot necessarily be assumed to apply in the case of all fluids.

In Fig. 1.24*d* are shown some comparable results for piston-cylinder assemblies of the differential type (Fig. 1.23*b*), in which the pressure acts upon the difference in the areas of two coaxial piston-cylinder units rigidly connected together with the load suspended from the base of the lower piston. These results have been obtained by comparison with the assemblies calibrated by the similarity method. It will be seen that the rate of change of effective area with pressure for different assemblies of this pattern, covering different pressure ranges, shows a greater degree of variability of slope than in the case of the simpler type of assembly, but it is not yet known whether these results are typical of differential-piston assemblies in general.

CONCLUSIONS AND FURTHER WORK

The methods described above seem to provide a basis for greatly improved calibrations of pressure balances. In particular, the similarity method for measuring the changes of effective area at elevated pressures relies on the properties of the balances themselves and is thus entirely independent of the use of the high-pressure mercury column.

The N.P.L. also proposes to instal a high-pressure mercury column of the general type recently described by Bett, Hayes and Newitt (1954), and this may be effected by the transfer of the mercury column already erected at Imperial College to the N.P.L. for maintenance as a pressure standard. The existence of these two independent methods is likely to prove very advantageous in that there will be ample means for the investigation of any uncertainties or discrepancies which may subsequently be encountered.

Preparations are being made for an additional check of the validity of the similarity method by the use of additional materials. It is clear that if a further comparison could be made, using either one or preferably two materials substantially different from those already employed, this would afford a very crucial test of the accuracy of the entire procedure. In addition, efforts are being made to extend the range of application of the method to pressures above 3,000 atm., and it is hoped that eventually an extension to the order of 6,000 atm. may be possible. This extension, however, will probably involve greater difficulties than have been encountered hitherto.

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APPENDIX 1.VI

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