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DESIGN AND EVALUATION OF A NOVEL AIR PYCNOMETER FOR TRUE VOLUME MEASUREMENT OF SOLID PARTICLES

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ABSTRACT

Precise measurement of volume, true density and specific gravity of small grains and food particles is a challenging task in the laboratories without sophisticated equipment. Therefore, a low-cost gas pycnometer was designed and evaluated for true volume measurement of solid particles as an alternative for expensive air comparison pycnometers. The theoretical principle is based on the Boyle's law of volume pressure relationship. The instrument was designed with three main components: a sample cup, two 20 mm diameter liner pneumatic cylinders and a differential pressure gauge. Two identical pneumatic cylinders were used to convert the liner displacement of the piston to the proportional volume change in both cylinders. The reading accuracy of displacement was increased by using a 0.01 mm dial indicator gauge. As the reading of the instrument is the liner motion of the piston shaft, the volume is calculated using an equation developed through calibration using some known volumes. In the calibration process, seven known volumes were used with three replications. The co-efficient of determination value for the calibration data set was 0.86. Two known volumes of 4.49 cm^3 and 17.21 cm^3 were used to evaluate the optimum volume to be measured and the standard deviations (SD) of linear measurements were calculated as 0.2658 cm and 0.2163 cm, respectively. Volume errors were calculated based on the SD values and the error percentages were calculated as 3.7% and 0.78% for 4.49 and 17.21 cm^3 objects, respectively. Results revealed that the percentage error for the larger volume object was less than 1%. The accuracy of the instrument can be enhanced further using gases like helium or argon which, are more closed to ideal gases. Temperature influence on the reading has to be minimized by conducting the test in a temperature controlled chamber. Unlike commercially available instruments this can be fabricated at very low-cost using readily available components in the local market. The device is portable and no need of external power supply for the operation.

KEYWORDS: Air Pycnometer, Boyle's Law, Particle Volume, Specific Gravity, True Density

INTRODUCTION

Physical properties of agricultural materials is very important for designing and operating agricultural processing systems. Volume, true density and specific gravity of food materials are important parameters in desigining, operating and quality evaluation in food processing systems.Volume can be defined as the amount of three dimensional space occupied by an object, usually expressed in units of cubes of length units(Sahin & Sumnu, 2006). Volume is one of the most frequently used measurement carried out in any analytical experiment (Prichard, 2003). Volume measurements for liquids or gases are made using a graduated container, for instance; a graduated cylinder. For solids, the volume can be obtained either from the measurement of dimensions of the solid or by displacement of either liquid or air(Prichard, 2003).

Density is another physical attribute that distinctly identifies any type of material; solid, liquid or gas. Density is merely calculated as the ratio of mass to volume; mass being invariably measured on a discrete device, usually by weighing(Simbun, 2010). But in volume measurements, solids have some distinctive attributescompared to liquids. Solids may have void spaces in-between particles that occupy the space and while measuring the 'true volume' those void spaces have to be distracted from the reading. The irregular patterns on solid surfaces may also interrupt the accurate measurements. Volume measurements of particulate solids and materials with irregular shape can be determined by displacement of gas or air in a pycnometer (ASTM, 2014). Pycnometry word was derived from a Greek word pyknós, which means 'dense' (Instruments, 2016).

The theory of gas displacement instruments is based on Boyle-Mariotte's law of volume pressure relationship. By this method volume of substances that react chemically or physicochemically with water can be determined by this method. Further, the problem of air entrapping, which is common in a liquid pycnometer does not exist in this measurement(Tamari & Aguilar-Chavez, 2004).

To obtain air volume readings, the pycnometer uses Boyle's ideal gas law as shown below.

$$PV = nRT$$

Where; P = pressure, V = volume, n = number of moles of gas, R = gas constant, T = temperature (in Kelvin)

In gas pycnometry, the volume of solids are not influenced or interrupted by the open pores or internal cavities, thus leads to have precise true volume measurements(Tamari & Aguilar-Chavez, 2004). The method is based on the fact that the used gas is displaced by the solid object. If helium is used as the media, the gas particles can penetrate in to open pores larger than 0.1 nm (Tsotsas & Mujumdar, 2009). So it can be assured that all most all the pores are detected and the final reading has a much higher accuracy.

TYPES OF PYCNOMETERS

- **Constant Volume Gas Pycnometer**

Constant volume Gas Pycnometer (Figure 1) is consists of a sample chamber, a reference chamber and an absolute pressure transducer (P) (Tamari & Aguilar-Chávez, 2005). Reference chamber and sample chambers are pneumatically connected by a tube with a coupling valve 01.

Gas or air can be supplied to the system through a tube connected to the reference chamber with a main coupling valve 02. Here thevolume of the reference chamber should be defined by the fixed volume (via calibration) or internal volume andsample chamber volume depend on his sample size and mass.

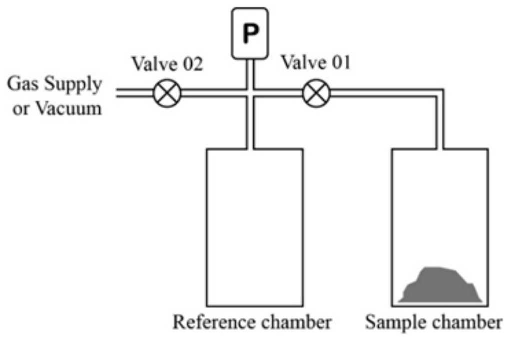
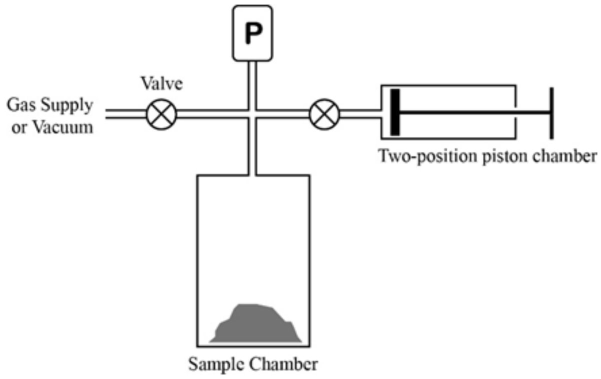


Figure 1: Diagram of Constant Volume Pycnometer

- **Variable-Volume Pycnometer**

The variable-volume gas pycnometer (Figure 2.) is composed ofa sample chamber, a piston chamber, and a pressure sensor. These three components are pneumatically interconnected. Thepycnometer can also be connected to a gas supply through a tubewith a coupling valve(Parmar, et al., 2016).

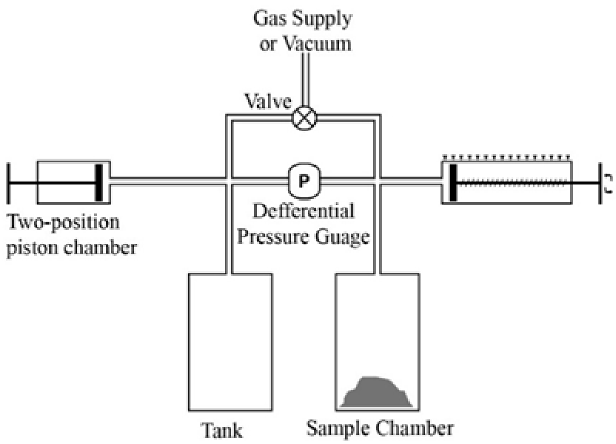


Source: (Tamari & Aguilar-Chávez, 2005)

Figure 2: Diagram of Variable-Volume Pycnometer

- **Comparative Pycnometer**

Basically, a comparative gas pycnometer is composed of a samplechamber with a screw cap, a tank, a two-position piston chamber, avolume controller (i.e., a piston chamber whose internal volume isknown for any piston position), a differential pressure transducer,and a valve(Tamari & Aguilar-Chávez, 2005). The pressure transducer measuresthe difference between gas pressures in the sample chamberand in the tank (Figure 3). Both the sample chamber and the tank canbe connected at the same time to a gas supply through a tube witha coupling valve.



Source: (Tamari & Aguilar-Chávez, 2005)

Figure 3: Diagram of a Comparative Pycnometer

General Drawbacks

Most of the commercially available gas comparison pycnometers can typically measure volumes with an accuracy of ± 0.1 cc(Philip, et al., 1978). However, higher precision is desirable since volume uncertainty is the limiting factor in the determination of density and specific gravity. Three major criticisms of known commercially available gas comparison pycnometers can be made. First, some devices require that the ideal gas law be precisely obeyed. Any departure from ideality of the gas being used results directly in an uncertainty in the inferred volume. Secondly, many devices are not stable against thermal variations. Changes in room temperature due to ambient variations can influence the pressure measured in the sample chamber. This in turn can cause a spurious variation in measured volume. Thirdly, in the balanced or zero condition, the number of moles of gas is different from that in the pycnometer when a sample is present. Thus, volumes are not calculated from measurements made under identical conditions(Philip, et al., 1978).

One of the main drawbacks of existing commercial air pycnometers is the cost. Therefore, they are very rare in most of the laboratories in poor countries. In addition, air used in commercially available instruments are not normal atmospheric air and the manufactures recommend to operate such equipment with helium or argon, which behaves similar to ideal gases, but cost of operation is high and in some cases difficult to find them as well.

Therefore, the objective of this research was to design and evaluate a low-cost air comparison pycnometer for true volume measurement of small grains and food particles.

METHODOLOGY

Boyle's law states that at constant temperature for a fixed mass, the absolute pressure and the volume of a gas is inversely proportional(Majumdar, 1995). The law can also be stated as the product of absolute pressure and volume is a constant.

$$\frac{Pressure \times Volume}{Temperature} = k$$

Many gases behave like ideal gases at moderate pressures and temperatures. Boyle and Mariotte derived the law solely on experimental grounds. Based on this theorem, gas pycnometer was designed.The device is composed of three main parts; the piston and cylinder, sample chamber and the differential pressure gauge.

In the design four fundamental assumptions were made.

- Normal air behaves ideally; it does not absorb to solids and its compressibility is negligible.
- The pycnometer is air tight and expanding gas quickly reaches a static equilibrium.
- The pycnometer's components are rigid and does not expand at the operating low pressures.
- Gas temperature is uniform and remains constant while measurements are performed.

Piston Cylinders

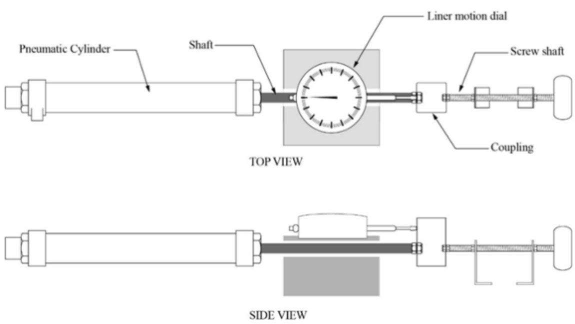


Figure 4: Top and Side Views of the Sample Cup Connected Cylinder and Fittings

Commercially available two pneumatic cylinders were usedas the variable volume chambers.Pneumatic pistons of different sizes of cylinders are available in the market and the necessary fittings are also readily available for making connections. Pneumatic cylinderis amechanical device which is usedto compress gas to produce a force in a reciprocating linear motion(Majumdar, 1995).For the design, two pneumatic cylinders were used, one is coupled to the sample chamber and the other is the reference cylinder. The barrel of the cylinder is made of aluminum alloy and both cylinders had 20 mm bore and 125 mm stoke. Due to the uniform inner diameter, the volume change is proportional to the liner motion of the piston. The shaft of one of the cylinders was coupled to a screw shaft, which canfacilitate the uniform liner motion of the piston (Figure. 4). A dial indicator gagewas fixed on the shaft in a way that the liner motion can be conveniently measured with a precision of 0.01 mm as shown in Figure 4.

The shaft of the other piston (reference piston) was coupled to a rigid plate in such a way it can be moved10-15 mm linearly (Figure 5& Figure 6) to increase the air pressure in that side.Then the two cylinders wereinterconnected by a 4 mm diameter pneumatic tube with a valve in-between.

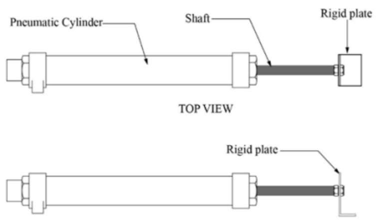


Figure 5: Top and Side View of the Secon Cylinder

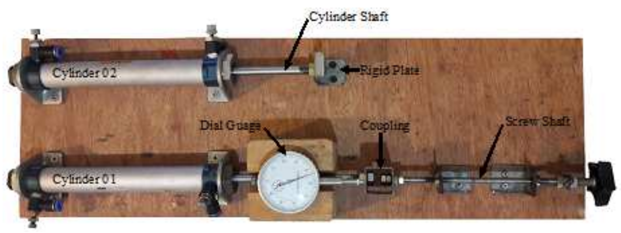


Figure 6: Top Veiw of the Two Cylinders

The Sample Chamber

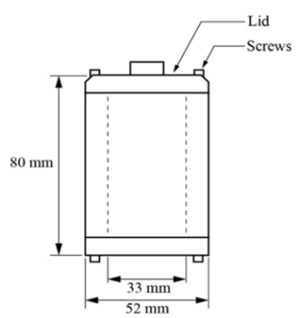


Figure 7: Sample Chamber

The sample chamber was made of aluminum. It has an air tight lid and a cup inwhich a sample can be placed (Figure 6). The internal volume of the sample chamber was 41.91 cm^3. Four screws were used to close the cup and to make it airtight, a small ‘O’ ring was used. The sample chamber was pneumatically connected toone cylinder using a rigid pneumatic tube.

Differential Pressure Gauge

A commercially available digital differential pressure gauge (model: HT-1890) was used and it can measure the differential pressure conveniently (Figure 7). The device has an accuracy of ±0.3%at 25 ˚C, and measures gauge/differential pressure in the range of±0.140 kg/cm^2 with a resolution of 0.001 kg/cm^2. The device isoperated by a 9 V battery.The pressure gauge measures the pressure difference between two pneumatic cylinders; cylinder coupled to the sample chamber and the reference cylinder.

Figure 8: Differential Pressure Gauge HT-1890

Device Setup

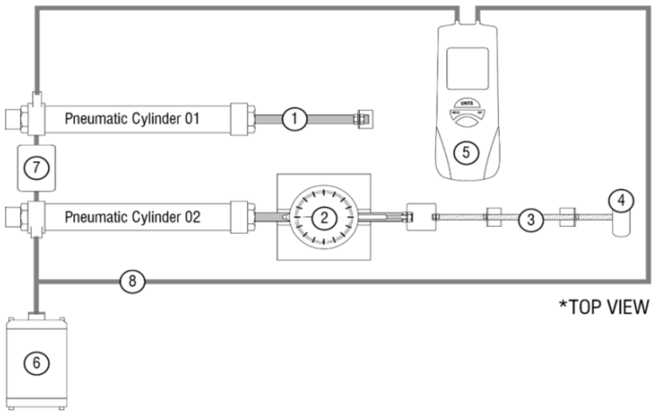


Figure 9: Design Setup of the Components

Pneumatic cylinders and other components were fixed on a wooden base plate as shown in the figure below (Figure 9). All the components were connected pneumatically using 4 mmdiameter pneumatic tubes.

1. Cylinder shaft
2. Liner dial gauge
3. Screw shaft
4. Screw turning knob
5. Differential pressure gauge
6. Sample chamber
7. Valve
8. Pneumatic tubes

Measurement of the Sample Volume

- The following steps were followed in the volume measurement of a sample using the instrument.
- The lid of the empty sample chamber should be tightened and the valve (7) should be kept open.
- The differential pressure gauge and the liner motion dial gauge should be set to their zero mark.
- Then the valve (7) should be closed and the shaft of the cylinder 01 should be moved few millimeters forward (preferably 6-8 mm) and then the piston rod should be tightened. At this moment the differential pressure gauge indicates a +/- pressure value corresponding to the pressure developed inside the cylinder 01.
- While keeping the valve closed, the shaft of the cylinder 02 should also be moved forward using the screw knob to compress the air in that side until it reaches the same pressure as in cylinder 01. At this moment, the differential pressure gauge should indicate zero.
- Then the liner movement of the second cylinder shaft should be read from the liner motion dial gauge (L_1). After that pull back the shaft to its original position.
- Open the lid of the sample cup and place the sample inside the sample chamber and close it tightly.
- Turn the screw knob again and move the cylinder shaft of the second piston and read the liner movement (L_2) when the differential pressure gauge indicates the zero pressure difference.
- (L_1-L_2) gives the liner distance which is proportional to the sample volume placed in the sample chamber. Using the calibration equation, the true volume of the sample can be calculated.

Calibration of the Device

Seven different known volumes were used to obtain the liner relationship between the two variables; volume and linear motion (L_1-L_2). Three replicates were done for each known volume and the respective L_1-L_2 values were measured. The means of three trials were used to plot the regression curve.

Standard glass beads were used as the known volumes as they are perfect spheres and the thermal expansion is also negligible. Average of five diameter readings were measured using a micrometer with an accuracy of 0.001 mm (Moore & Wright: 965) for the volume calculation of glass beads.

Accuracy of Measurement with Sample Volume

The device was tested toinvestigate the volume of the sample to be used in measurementsfor taking accurate readings using two different known volumes. Eight trials were carried out for each volume and the standard deviation of the sample was measured. Using the standard deviation and the inner diameter of the pneumatic cylinder, the error of the volume was calculated, which deviates from the mean value.

RESULTS

A summary of the data obtained in the calibration process is given in the following table (Table 1).

Table 1: Mean Values of the Measured Data for Calibration

Volume (cm^3)	L_1 (avg) (mm)	L_2 (avg) (mm)	L_1-L_2 (mm)
0.000	9.11	6.29	2.82
2.348	8.86	4.96	3.90
4.491	8.59	4.44	4.15
8.011	8.69	4.48	4.21
10.924	9.26	3.67	5.59
13.865	9.54	4.07	5.47
17.211	9.56	3.94	5.63

Using above data, a linerregression was done to establish the relationship between volume and the liner displacement difference (L_1-L_2)(Figure 10).

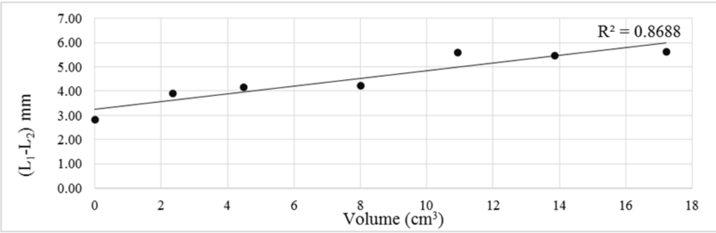


Figure 10: Calibration Curve of the Air Pycnometer

The equation for the calibration graph is as follows.

$$y = 0.1581x + 3.2524$$

The equation can be modified as;

$$Volume\ (cm^3) = \frac{Liner\ displacement\ difference\ (L_1 - L_2) - 3.2524}{0.1581}$$

Where, (L_1-L_2) is in mm.

Minimum Theoretical Volume

The instrument can be used to measure a minimum volume of 0.00314 cm^3 theoretically, since the cross sectional area of the pneumatic cylinder is 3.141 cm^2 and the minimum liner displacement that can be measured is 0.01 cm. But this value could be achieved from the developed instrument, thus a volume measurement range has to be optimized for a higher accuracy of a particular measurement. However, the accuracy of the measurement will be influenced by the sample volume under practical conditions. Therefore, the accuracy should be tested with increasing sample volume.

Effect of Smaple Volume on Measurement Error

The volume of the sample chamber is 41.91 cm^3. Following table (Table 2) shows the summary of the measurement data for two different test volumes. The standard deviations (SD) of the two samples were calculated separately using sample data and the error volume was also calculated. The inner diameter of the pneumatic cylinder two was 20 mm. Then the cross sectional area is 3.141 cm^2.

Table 2: Summary of the Data Used for SD

	Trial no	L_1 (mm)	L_2 (mm)	(L_1-L_2) (mm)
4.491 cm^3	1	7.27	2.84	4.43
	2	9.72	5.35	4.37
	3	8.88	5.13	3.75
	4	8.89	4.86	4.03
	5	9.85	5.40	4.45
	6	9.98	6.03	3.95
	7	8.90	4.95	3.95
	8	9.81	5.51	4.30
17.212 cm^3	1	8.45	3.00	5.45
	2	8.57	3.02	5.55
	3	8.68	3.07	5.61
	4	8.73	2.91	5.82
	5	8.71	3.22	5.49
	6	8.87	3.59	5.28
	7	9.08	3.97	5.11
	8	8.64	3.30	5.34

Calculated SD value for the 4.49 cm^3 and 17.212 cm^3 were 0.2658 cm and 0.2163 cm,respectively. Therefore, the respective volumes were calculated as 0.0835 cm^3 and 0.0679 cm^3 using the cross sectional area and the SD values. This can be simplify as while measuring a volume of 17.212 cm^3, the reading of the device can be in-between 17.045 cm^3 and 17.379 cm^3 with 95% accuracy (±2 SD). Therefore, larger the sample size, the reading is more accurate.

DISCUSSIONS

Since the available instruments for true volume measurements of small food grains and particles are expensive, the measuring device is important for laboratories. According to the literature, gas comparison pycnometry is one of the most reliable techniques for true volume measurements since it does not interfere with the material properties or any other conditions except the ambient temperature. According to the Boyle's law for a fixed mass, the absolute pressure and the volume of a gas are inversely proportional. Gas pycnometry is one of the applied technology of this phenomena.

Even though there are various types of gas pycnometersavailable, this study focused on a novel technique of taking measurements which applies the same theory on Boyle's law.

A separate sample chamber was designed and introduced for convenient loading of the sample to be measured. Two identical pneumatic cylinders were used as they are readily available and the necessary fittings can also be found locally. Pneumatic fittings are designed in a way that they are hermetically sealed when connected using standard tubes and very much convenient to make the connections. When the fittings were properly fixed, the air leakages could be detected from the pressure gauge and correct them.

Operation Principle

Initially two of the cylinders and the sample chamber opens to the normal atmospheric air, thus the pressure in both sidesare identical (P_1). By closing the valve (7), two cylinders are separated by each other pneumatically. Sample chamber is always connected with the cylinder 2. Then while the piston shaft of the cylinder 1 moves forward and the pressure will increase inside the cylinder chamber (P_2) and the difference (P_2-P_1) will be indicated by the differential pressure gauge. To compensate that pressure difference, the shaft of the piston 2 should also be moved forward (to the same direction) until the pressure gauge indicates zero pressure difference. The liner movement of the cylinder 2 can be measured within the range of 0-10 mm with a minimum reading of 0.01 mm. The liner displacement is proportional to the initial pressure increment of the cylinder 1 and the value should be recorded. Then the shaft 2 has to be pulledback to its original position. When the sample chamber is opened, that side of the device will open to the atmospheric pressure again and with the sample placement, the total free internal space will be reduced. The lid has to be tightened next and the shaft of the cylinder 2 should be moved again until the pressure gauge indicates zero value. But at that moment, the liner displacement is a lesser value than earlier because of the sample taking some space from the initial volume. Therefore, the pressure will develop with a lesser displacement of the cylinder shaft. The reduced volume (due to liner displacement reduction) is proportional to the sample true volume and the sample volume could becorrelatedto the liner displacement.

Accuracy and the Reliability

Results of the study reveal that the instrument has anacceptable accuracy. The standard deviation of 0.21 cm for the linear displacement for 17.21 cm^3 volume measurement resulted ±0.0679 $cm^3$of volume error. This error is within the acceptable range with respect to other available devices (Philip, et al., 1978). As a percentage, this is 0.789%. According to the data obtained, with the increasing of the volume being measured, the error percentage could be reduced. For a measurement of 4.491 cm^3 the error percentage was calculated as 3.719%, which is a higher value. With these data it is recommendedto use a larger sample size to get an accurate measurement; ideally more than 17 $cm^3$of sample volume. The instrument can be identified as a reliable low-cost measuring device for true volumes of particulate agricultural materials as it generates readings with an acceptable accuracy. The reliability of the used components in the design also at an acceptable level and all the components are readily available in the market at reasonable prices.

Cost Analysis

The device can be fabricated for a cost of around 21,000 Sri Lankan Rupees ($ 137) and that would the biggest advantage of this design. Cost of main components are listed in the table given below (Table 3).As commercially available air pycnometers are relatively expensive and difficult to be purchased with limited funding, this will be a viable option for many such laboratories.

Table 3: Cost of Main Components

Item	Unit Cost (LKR)	Quantity	Cost (LKR)	Cost (USD)
Pneumatic cylinder (20x125)	3,800.00	02	7,200.00	47.05
Differential pressure gauge	4,500.00	01	4,500.00	29.41
Dial indicator	1,050.00	01	1,000.00	6.53
Pneumatic valve	600.00	01	600.00	3.92
Sample chamber	1,800.00	01	1,800.00	11.76
6mm tubes	200.00/m	02 m	400.00	2.61
Pneumatic fittings	-	-	2,000.00	13.07
Machine works	-	-	1,500.00	9.80
Other	-	-	2,000.00	13.07
Total			**21,000.00**	**137.25**

(Note: prices of sri Lankan Rupees were converted toUSD with a conversion rate of SLR 153)

In the design process, itis importantto consider the following error sources as well. The Boyle's law is only applicable for ideal gasses and this device operates with normal atmospheric air.However, if the instrument can be operated with a gas like helium or argon, this issue can be minimized and the accuracy of the device can also be improved. During the calibration process, the temperature effect was neglected and assumed that there is no temperature variation while the measurements were taken. However, the temperature directly influencesthe accuracy of the reading; thus a calibration for temperature variation should also be incorporated or test should be done inside a temperature controlled chamber.

The re-designed pycnometer is light in weight, portable and no need of external power supply for the operation. Thus it can be used even as a field testing instrument apart from the laboratory measurements.

CONCLUSIONS

The air pycnometer designedis a cost effective and adequately precise for measuring true volumes of particulate materials when the sample volume is relatively large. Volumes larger than 17 cm^3 gives more accurate readings with an error of ±0.06 cm^3 or less than 1%. The separate sample chamber is an efficient design and the method of taking measurements is a novel concept in gas pycnometry. Simplicity and time saving of measurements are some major advantages of the instrument.

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