
VOLUME 62 2002

Volume measurement of wool samples

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ABSTRACT

A rapid procedure for measuring the volume of wool samples was developed, using a gas pycnometer. Volume was measured on midside wool samples, harvested monthly, from thirteen sheep breeds representing a range of wool medullation categories. Clean sample weights between two and ten grams and the fourth sequential volume measurement are recommended. Volume was also calculated, using the sample mass and the theoretical density of wool. Measured and calculated volumes were the same for non-medullated wools ($p=0.20$), but not for highly medullated wools, the measured volume was 25% greater than the calculated volume ($p<0.001$).

Keywords: medullation; wool mass; wool volume; gas pycnometer.

INTRODUCTION

The volume of a wool sample can be estimated from sample mass, using the specific gravity of wool keratin (1.305 g/cm^3, King, 1926). Previously, specific gravity has been measured by displacement of liquid, using a benzene pycnometer (King, 1926; Van Wyk and Nel, 1940) and density gradient columns (Connell and Andrews, 1974) but these methods are slow, taking in excess of sixteen hours for sample measurement. Inaccuracies in calculated volume can arise if the density of the keratin varies from 1.305 g/cm^3 or non-keratin components, with a different density, form a significant portion of the fibre. This is likely to be the case for medullated fibres, which have a central core of vacuolated cells (Auber, 1950).

A gas displacement method can measure volume directly and should be quicker than established liquid displacement methods. The following experiments were designed to develop a procedure, using the gas pycnometer, to directly measure the volume of a wool sample. The method was then used to test the hypothesis that measured volume will be less than the calculated volume when medullation is present.

MATERIALS AND METHODS

Effect of sequential measurement

The helium pycnometer can be set to make up to five sequential measurements on one sample. In the first experiment, sixty four midside wool samples were harvested from eight sheep breeds (Halfbred (English Leicester x Merino), English Leicester, Merino, Poll Dorset, Romney, Drysdale, Suffolk and Lincoln) after four weeks growth in winter. The breeds, and individual animals within the breeds, were chosen to represent a range of wool types varying in medulla content and fibre growth rate. Greasy samples were scoured using a three minute wash in a warm aqueous solution of Teric GN9 (0.15%), then rinsed twice in warm water. The scoured wool samples were oven dried (45°C) overnight and conditioned for twenty four hours at 65% relative humidity and 20°C. Mass was recorded on each sample prior to five sequential measurements of volume in the helium pycnometer.

The ability of the five sequential measurements to predict volume was investigated using regression analysis (GENSTAT5, Rothamsted Agricultural Research Station). Individual volume measurements, the average of the first two, three, four or five measurements, a ratio between the first and fifth, second and fifth, third and fifth or fourth and fifth measurements, and prediction of actual volume using an exponential curve were considered. To establish when the volume measurement reached a plateau, a rate of decay (*R*) was calculated by fitting an exponential function (*e*) between the first and fifth sequential measurements, according to the equation:

$$V_{\times} = V_F + (V_0 - V_F) * e^{-Rt}$$

where: $V_{\times}$ = predicted volume
V_0 & V_F = first and fifth volume measurements, respectively
t = sequential measurement number

Effect of mass

In the second experiment, fleece samples of twelve months growth for four animals from each of thirteen sheep breeds (Halfbred, English Leicester, Merino, Poll Dorset, Romney, Drysdale, Suffolk, Lincoln, Polwarth, Shropshire, Wiltshire, Cheviot and feral Merino (Arapawa)) were measured over a range of nine masses (0.2, 0.4, 0.6, 0.8, 1, 2, 3, 6 and 10 g). The volume of the clean wool was measured at 65% relative humidity and 20°C using the fourth sequential value. The ten gram sample was measured first, a six gram subsample of the ten grams was measured next, then a three gram subsample of the six grams and so on. At each mass, the relative variability was calculated from the standard deviation of the mean of all samples, and expressed as the coefficient of variation.

Analysis of variance and regression, using GENSTAT5, were used to determine the effect of mass.

Volume measurement and medullation

In the third experiment, midside wool samples were harvested monthly through winter, spring and summer, from the same area, of approximately one hundred square centimetres, over two years. At each sampling, between two and ten animals represented each of ten of the breeds used in the second experiment. A total of one hundred and twenty one animals were sampled, each having a clean sample weight greater than two grams. Volume was measured at 65% relative humidity and 20°C, using the fourth sequential value. Volume was also calculated, using the sample mass and the specific gravity of wool.

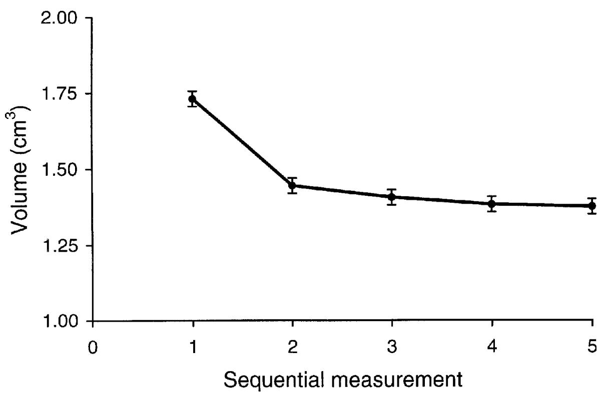
A two and a half gram sample from a twelve month fleece was measured for medullation (% medullation by fibre volume) for four animals from each breed, using the WRONZ Medullameter (Lappage and Bedford, 1983). From these results, the breeds were divided into the following categories - no medullation (<2%; Merino, Polwarth and Halfbred), low medullation (2-10%; Romney, Poll Dorset, Lincoln, English Leicester and Shropshire) and high medullation (>10%; Drysdale and Cheviot).

RESULTS

Sequential measurement

Volume declined exponentially with sequential measurement ($p<0.001$) and had reached a plateau by the fourth measurement, at which point there was a 20% reduction in volume from the first measurement (Figure 1).

FIGURE 1: Decline in mean volume of sixty four midside wool samples over five sequential measurements (error bars indicate the 95% confidence interval)



Volume measurements obtained from individual sequential measurements and combined values were highly correlated with predicted volume estimates ($r^2=0.94$ to 1.00). The third, fourth and fifth individual sequential measurements were all very good predictors ($r^2=1.00$ for each). The residual variances for the individual measurements were: first 0.0090, second 0.0015, third 0.0006, fourth 0.0003 and fifth 0.0005 cm^3. The fourth measurement was used in subsequent experiments as it was the most stable measurement and a compromise on the length of measurement time. The rate of decay in volume between the first and fifth measurements was similar ($p=0.07$) for wools with high, low or no medullation. The length of time to make five measurements was eight and a half minutes, individual measurements taking ninety nine seconds.

Effect of mass

There was a linear relationship between mass and volume for masses greater than one gram (Figure 2). The overall slope of the line was 0.80 ± 0.01, representing a density of 1.248 g/cm^3. The relative variability of measured volume decreased exponentially as mass increased. The coefficient of variation reduced from 180% at 0.2 grams to 19% at ten grams ($p<0.001$), (Figure 3).

FIGURE 2: Increase in volume with increasing mass, measured on twelve month fleece samples (n=420) from thirteen sheep breeds.

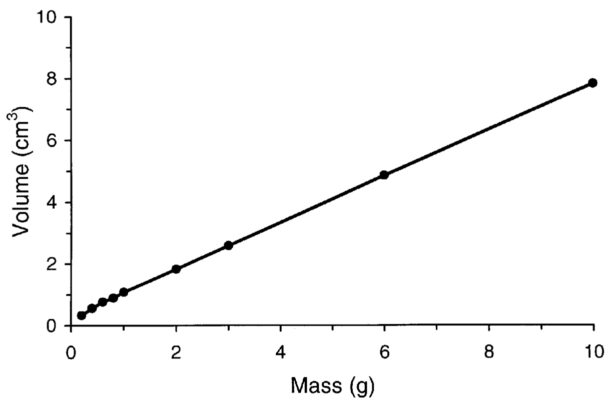
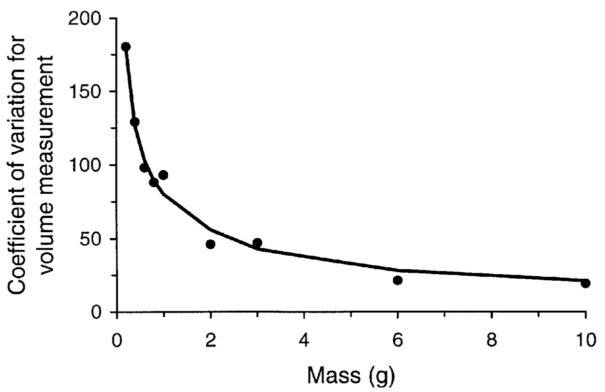


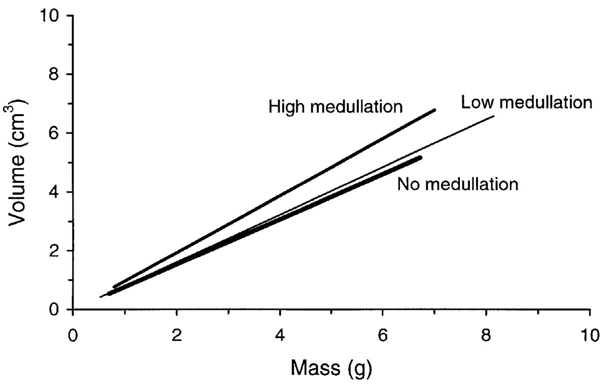
FIGURE 3: Decrease in relative variability of volume measurement as mass increases, measured on twelve month fleece samples (n=420) from thirteen sheep breeds.



Measured volume, calculated volume and medullation

Measured and calculated volume was comparable for non-medullated wool (2.43 and 2.44 cm^3 respectively ($p=0.20$)) but for highly medullated wool, the measured volume (3.05 cm^3) was 25% greater than the calculated volume (2.45 cm^3), ($p<0.001$). There was also a 25% increase ($p<0.001$) in the measured volume of medullated wool over the measured volume of non-medullated wool, with only a 1% difference in weight ($p=0.16$). Calculated volume was similar for all medullation categories ($p=0.16$). Average mass of the samples was 3.19 g for highly medullated wool and 3.17 g for non-medullated wool. The change in measured volume in the different medullation categories was reflected in the different slopes of regression lines fitted to mass and measured volume (Figure 4). The slopes were 0.77± 0.01 for non-medullated wool, 0.81± 0.01 for wool with a low degree of medullation and 0.97± 0.03 for highly medullated wool. This translated to densities of 1.307 g/cm^3 for non-medullated wool and 1.035 g/cm^3 for highly medullated wool ($p<0.001$), while the density of wool with a low degree of medullation was intermediate (1.241 g/cm^3).

FIGURE 4: Regression lines fitted to mass and measured volumes for monthly midside wool samples (n=1067) with high, low or no medullation.



DISCUSSION

The helium pycnometer provides a rapid method to measure the volume of wool samples. Several sequential measurements are required to obtain an accurate estimate of the volume of a sample. The automated calibration sequence of the pycnometer ignores the first measurement and averages the second, third and fourth measurements. From this investigation, the fourth volume measurement is recommended for use, as it was the least variable measurement for wool samples. The variability in the first measurements could be due to the presence of moisture in the samples, which are conditioned at 65% relative humidity and moisture in the air, which enters the pycnometer when the measurement chamber is loaded. As the dry helium gas is pumped into and flushed from the measurement chamber during the measurement sequence, moisture would be removed from between and within the fibres. Therefore, several measurement cycles would be required for the helium to displace the air and moisture in the chambers. By the fourth sequential measurement, the volume had reached a plateau. Use of this value avoids the need to adjust the measured value to actual volume. The similar decay rate (*R*) for the different medullation categories indicates that the reduction in volume estimates during sequential measurement was not affected by the presence or degree of medullation.

Mass constraints are recommended when using the helium pycnometer to measure the volume of wool samples. The non-linear relationship between mass and volume, when mass is less than one gram indicates measurement error. This, and the high coefficient of variation at low masses, leads to the recommendation to use sample masses between two and ten grams. Measurement of masses greater than ten grams is physically limited by the size of the measurement chamber, although midside patch samples with a mass greater than ten grams could be measured in two or more portions.

Considerable differences in the degree of medullation can occur between seasons of the year, between individuals within a breed, and between the breeds studied (Skårman and Nõmmera, 1954; Story and Ross, 1960; Scobie *et al.*, 1993). The recorded variations in medullation highlight potential inaccuracies created by broadly dividing wool samples into medullation categories on the basis of breed alone. Such broad division allows only gross effects of medullation to be examined. However, the difference between calculated and measured volume for wools with a high degree of medullation reflects the effect of medullation on fibre volume. The vacuolated cells of the medullae are not filled with keratin so they contribute less mass per unit volume than cells in other parts of the fibres. The results show that calculated volume will be underestimated when a wool sample is medullated.

Similarly, density calculated from measured volume will be lower for medullated wools than non-medullated wools. The density of non-medullated wools, calculated using volume measured by the gas pycnometer (1.307 g/cm^3), was comparable with specific gravities measured using a benzene pycnometer (1.305 g/cm^3) (Van Wyk and Nel, 1940) and density gradient column (1.304 g/cm^3) (Connell and Andrews, 1974). The density of wools with high medullation was much lower at 1.035 g/cm^3.

Potential sources of error in measurements of density or volume by displacement methods are absorption of the displacement medium by the fibre and penetration of the medium into the medulla. The similar density values for non-medullated wools, obtained using a benzene pycnometer, density gradient column or gas pycnometer suggest similar degrees of absorption of gas and liquid, if absorption is occurring. The divergence between measured volume and calculated volume indicates that the gas was not fully occupying the medulla, although the possibility of some penetration of the medulla by the gas cannot be ruled out.

Further investigation could lead to the development of a method for the measurement of medullation, derived from differences between volume measured using the helium pycnometer and that calculated from mass. The Optical Fibre Diameter Analyser (OFDA) provides rapid and inexpensive measurements of fibre diameter (IWTO, 1995) and offers the potential for measurement of medullation at the same time, but the Airflow method of measuring fibre diameter (IWTO, 1993) remains the method on which most of the international trading of wool is based. The helium pycnometer could be used in conjunction with Airflow equipment to provide medullation, density and fibre diameter measurements on the same two and a half gram wool sample.

ACKNOWLEDGMENTS

This research was supported by the New Zealand Foundation of Research, Science and Technology.

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