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Report No. 7

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## The Use of Variable Test Specimen Mass when Measuring Mean Fibre Diameter by Airflow

By

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### SUMMARY

*The development of a technique is outlined for the measurement of Mean Fibre Diameter (MFD) by Airflow using a test specimen which can vary in mass from 2.400g to 2.600g. The new technique requires additional calibration steps resulting in an individual calibration equation for each value of specimen mass. Measurement of MFD is the same as the current fixed mass procedure, except that an appropriate calibration equation must be selected based on the specimen mass. Trials show equal performance between MFD measurements made using the Standard fixed mass (2.500g) and the variable mass techniques for wools less than 32 $\mu$ m. A recommendation is made to change IWTO-28-93 to accommodate this variable mass technique.*

### INTRODUCTION

The Airflow method of measuring mean fibre diameter utilizes the principle that, for a given pressure difference, the air flowing through a mass of fibres is an indirect measure of the MFD of the sample. A fixed mass of fibre (2.500g  $\pm$ 0.004g) is used when measurements are made in accordance with the IWTO Standard procedure for testing fibre diameter of core material using Airflow, IWTO-28-93<sup>1</sup>.

Other authors<sup>2,3</sup> have reported using variable, rather than fixed, test specimen masses when testing fibre fineness using the Airflow. For reasons of testing convenience and calibration advantages when testing either superfine or very coarse wool, Jackson and Engel<sup>1</sup> tested wool fibre diameter with a variable test specimen mass ranging from 1.0g to 2.0g using a Sonic Fineness tester<sup>4</sup>. More recently, Textest AG<sup>3</sup> is marketing an Electronic Micronaire which uses a variable test specimen mass ranging from 3g to 5g.

In practical testing of fibre diameter, weighing a conditioned test specimen to within  $\pm$ 4 mg is relatively time-consuming, as a number of small corrections is typically made to the test specimen to achieve the required mass. This extended weighing procedure adds to the cost of testing for a high volume test house. In addition, compared to the Standard procedure, a variable mass technique could improve measurement precision because the individual test specimen mass is used rather than test specimens with a range of masses from 2.496 to 2.504g.

In order to improve the efficiency of measurement and the precision of the test specimen mass, AWTA Ltd has been investigating a procedure to adjust for the effect of small (0.1g) changes in test specimen mass in Airflow testing. The aim of this report is to examine whether or not the Variable mass procedure is equivalent to the Standard fixed mass procedure in IWTO-28-93. The report presents details both of the Variable mass procedure and of a comparison between fixed (2.500g) and variable mass (2.400g - 2.600g) measurements of MFD for a range of wools. An examination is also included of the theoretical aspects of this procedure.

**THEORY**

• The Relationship between Air Flow and Sample Mass

An understanding of the expected relationship between rate of air flow and test specimen mass can be obtained from an examination of the relevant theory. The generally accepted explanation for the relationship between flow rate of air and MFD in a Standard Airflow test is based on the Carman-Kozeny<sup>5</sup> theory. Although it does not provide an accurate prediction of measured air flow values this theory is used as an aid to interpretation of the effects of various parameters in Airflow testing since it describes the relationship between various parameters. The theory which was applied to wool testing by Cassie<sup>6</sup> is summarised in the following equation:

$$Q = \frac{AP}{L} \cdot \frac{\epsilon^3}{k\eta S^2(1-\epsilon)^2} \dots\dots\dots(1)$$

- where Q = the flow rate of air through the plug of fibres;
- A = the cross-sectional area of the fibre plug;
- P = the pressure drop across the fibre plug;
- L = the length of the fibre plug;
- S = the specific surface area of fibres in the plug;
- k = a 'constant', which is dependent on the size and shape of the pores;
- η = the viscosity of the air;
- ε = the free air space per unit volume of the plug.

For fibres with a circular cross section,

$$S = \frac{6}{d(1+c^2)} \dots\dots\dots(2)$$

where d = the mean fibre diameter, and,  
c = the fractional coefficient of variation of diameter (CVD).

Further, if V = the volume of the plug;  
M = the mass of the test specimen; and  
ρ = the specific gravity of wool,

$$\text{then } \epsilon = 1 - \frac{M}{\rho V} = \dots\dots\dots(3)$$

for a Standard Airflow meter, where

V = 7.96 cm<sup>3</sup>, and, substituting the generally accepted value for the specific gravity of wool, ρ = 1.31 gcm<sup>-3</sup>.

From equations (1), (2) and (3) the following relationship can be derived between rate of air flow and (test specimen) mass:

$$Q = \frac{Kd^2(1+c^2)^2(10.428)}{M^3/M^2} \dots\dots\dots(4)$$

where,  $K = \frac{AP}{Lk\eta}$

Thus, over the feasible ranges of fibre diameter and mass for wool testing, the air flow is dependent on the mass in a relatively complex manner. In order to obtain a clearer understanding of the relationship between air flow rate and mass for **small changes** in mass, a series of these relationships was derived using equation (4) for six wools with MFD's ranging from 15µm to 40µm and mass varying between 2.400g and 2.600g. In each case the values of standard deviation of fibre diameter, and thus c, were estimated from the following relationship<sup>7</sup> which is based on a large number of Interwoollabs calibration tops:

$$SD = -1.788 + 0.299 d, \text{ where}$$

SD = the standard deviation of fibre diameter.

The values of  $c$  (or CVD) for each wool are shown in Table 1. This analysis makes the (reasonable) assumption that the value of the Kozeny constant,  $k$ , remains fixed, i.e. when testing 15 $\mu\text{m}$  to 40 $\mu\text{m}$  wools with a sample mass between 2.4g and 2.6g the shape and size of the pores within the plug of fibres are not altered beyond the bounds normally associated with Airflow testing.

Table 1. Values of Coefficient of Diameter for the six (6) wools being analysed

Mean Fibre Diameter ( $\mu\text{m}$ )	Coefficient of Variation of fibre diameter (%)
15	18.0
20	21.0
25	22.7
30	23.9
25	24.8
40	25.4

Figure 1 shows the essentially linear relationships between the relative flow rate,  $Q/K \times 10^{-4}$ , and sample mass,  $M$ , for each of the six (6) wools. The square of the correlation coefficient for the linear regression analyses for each of the six (6) relationships was very close to unity, 0.9986, indicating that, over the narrow range of mass under investigation, a straight line is a very good approximation of the theoretical relationship between flow rate and mass for any given value of MFD. This result implies that there is a unique, linear relationship between flow rate and mass for each MFD. Figures 2(a) and (b) show graphs of the predicted values for the slope and intercept of these relationships.

In theory, the information contained in Figures 1 and 2 could be used to obtain the MFD of a sample of wool from measured values of air flow and mass for small (0.1g) variations in mass from the Standard fixed mass of 2.5g. Before examining the application of this theory in practice, an investigation is presented of the theoretical effects of medullation on variable mass in Airflow testing.

- The Effect of Medullation

The Carman-Kozeny theory of Airflow includes an assumption that the specific gravity of wool is a constant value. This assumption can be breached if wools contain different values of medullation<sup>8</sup>. The effect of medullation on variable sample mass can be examined by assuming that  $\rho_m$  is the specific gravity of a sample of medullated wool. For the fixed volume,  $V$ , of wool in an Airflow test,

$$V = M^* \rho_m, \text{ where } M^* \text{ is the 'apparent' mass of medullated wool in the test.}$$

Since,  $V = Mp$

$$M^* = \frac{Mp}{\rho_m}$$

For a medullated wool,

$$\begin{aligned} \epsilon &= 1 - \frac{M}{\rho V} \\ &= 1 - \frac{M^*}{\rho} \dots\dots\dots(5) \end{aligned}$$

If we assume that the medullation in a fibre is cylindrical along the length of the fibre, is centred on the fibre centre, has a diameter,  $d_m$ , and that,

$$\frac{d_m}{d} = 0.5$$

there is 25% less material in a medullated fibre compared to an equivalent fibre with no medullation. Using this mass correction and equations (4) and (5), values of the Mass versus Relative Air Flow relationship have been calculated for a 40 $\mu\text{m}$  wool with (i) 3.5% and (ii) 20% of medullated fibres. The lower value of medullation, 3.5%, is equivalent to results<sup>9</sup> obtained from the coarsest Interwoollabs calibration tops (Series 10) measured using an OFDA. Results of this analysis are shown in Figure 3, along with results for equivalent 35 $\mu\text{m}$  and 40 $\mu\text{m}$  wools with 0% medullation. The effects of moderate and of high levels of medullation are a reduction in measured MFD. Assuming a linear change in flow rate between 35 $\mu\text{m}$  and 40 $\mu\text{m}$ , the reductions in measured MFD are approximately 0.6 $\mu\text{m}$  and 2.8 $\mu\text{m}$  for 3.5% and 20% medullation, respectively, compared to an equivalent fibre with no medullation. Both the slope and offset of the relationship between air flow and mass are changed by the degree of medullation of the wool.

### Theoretical Effect of Variable Mass on Air Flow Rate

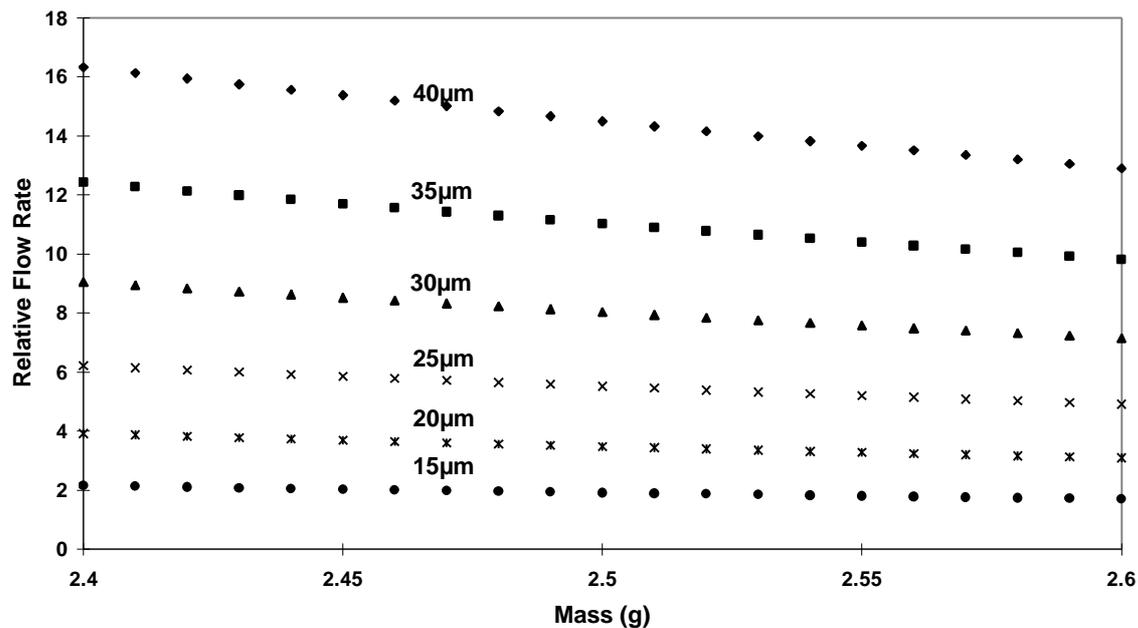


Figure 1. The theoretical relationships between (relative) Air Flow Rate and sample mass for six (6) wools with fibre diameters ranging from 15 $\mu\text{m}$  to 40 $\mu\text{m}$  in 5 $\mu\text{m}$  steps.

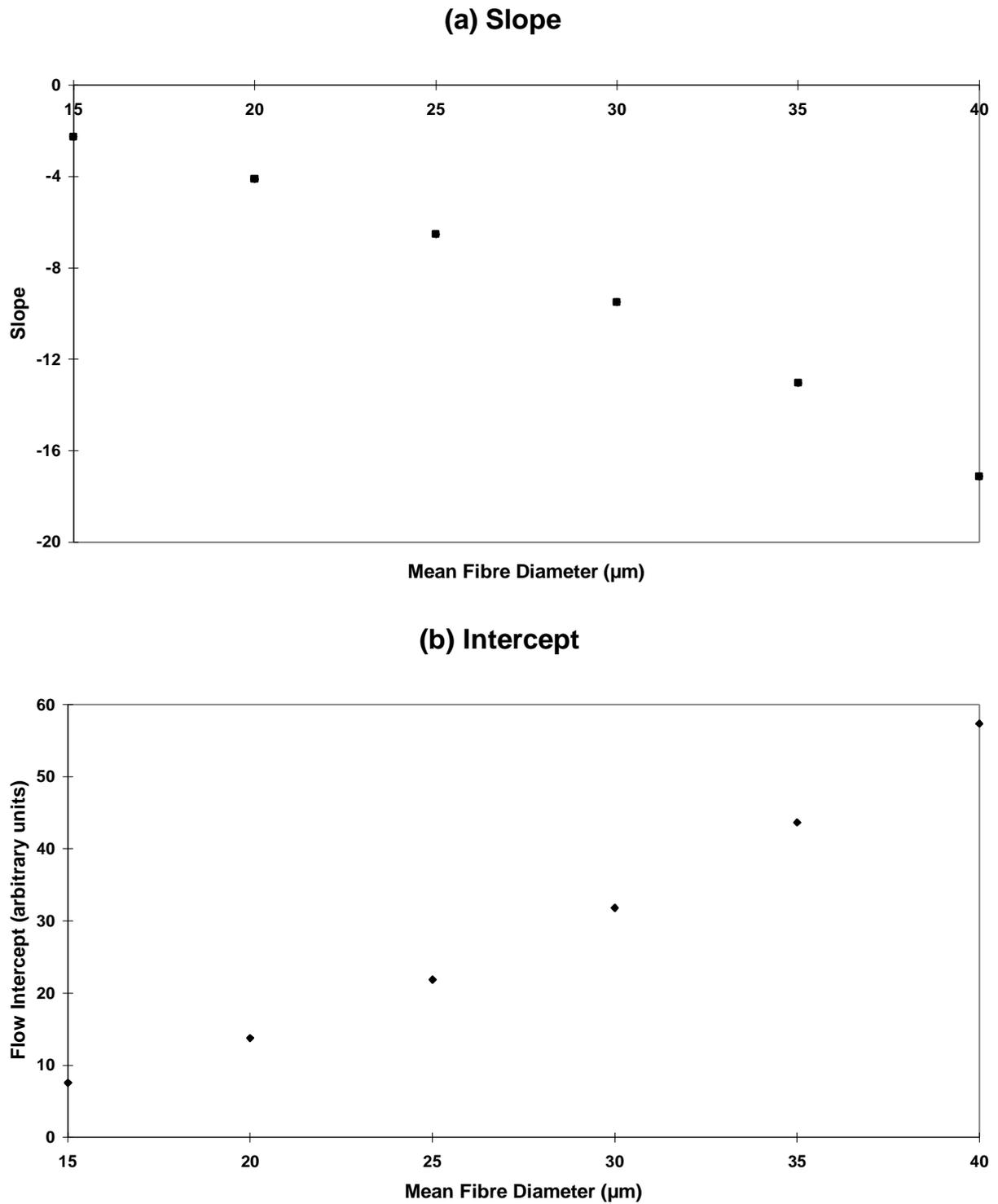


Figure 2. Predicted values for the linear relationships between Air Flow Rate and Sample Mass defined in Figure 1 - (a) Slope and (b) Intercept.

### Effect of Medullation on Airflow

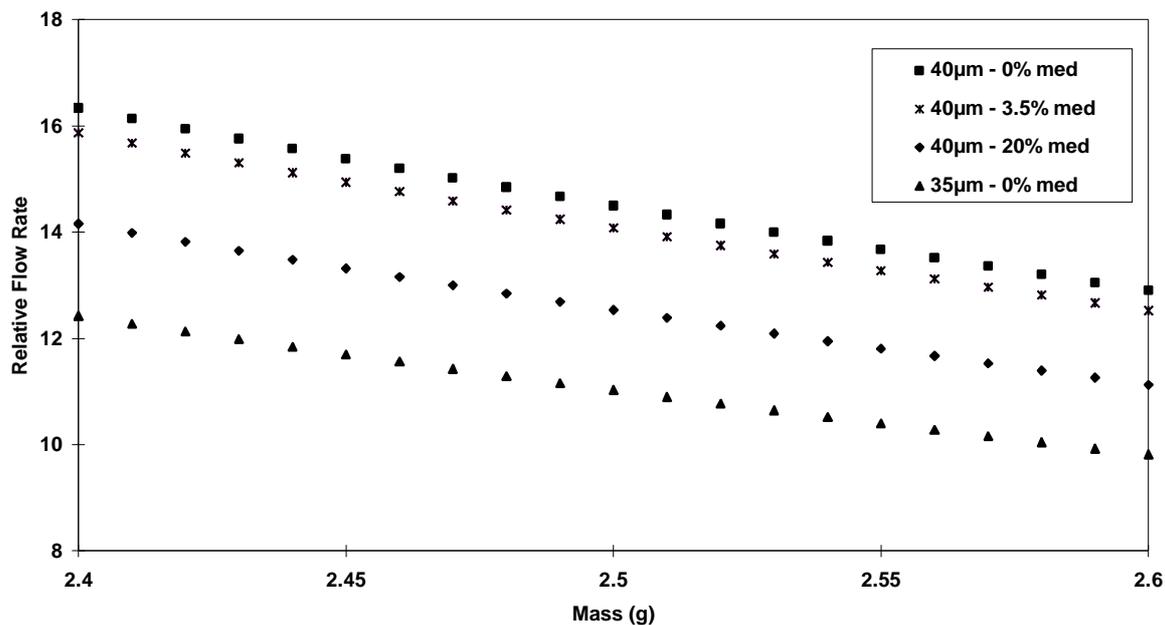


Figure 3. The theoretical effect of 3 levels of medullation (0%, 3.5% and 20%) on the relationship between mass and air flow rate for a 40µm wool. The relationship between mass and air flow for a 35µm wool with 0% medullation is shown for comparison.

## METHOD

- Description of technique

The technique of measuring MFD using variable mass requires an additional calibration procedure to be performed. This produces a series of 201 calibration equations, 1 for each milligram increment from 2.400g to 2.600g.

The measurement process requires measurement of both mass and flow rate of the test specimen. MFD is determined by substituting test specimen flow values into a specific calibration function, according to the measured specimen mass.

- Experimental Method
  1. Flow versus mass regression equations

Interwoollabs Harmonisation (IH) Standard Tops were prepared as per the method described in IWTO-28-93. Four specimens from each Top were weighed to  $\pm 1$  mg at each of 11 different masses spread over the range from 2.325 to 2.675 grams. Two of each 4 specimens were measured on Airflow Meter 1 and the remaining two specimens were measured on Meter 2. So, for each of the eight IH wools, 4 flow readings at each of 11 masses were recorded per Airflow Meter.

For each Top, the 4 flow readings at each mass were averaged and plotted against mass. This produced a series of 8 regression equations for individual Airflow Meters. Linearity of the relationships was assessed.

## 2. Comparison of Variable and Fixed mass techniques

### a) Calibration of Meters

A pair of Instruments was chosen for the comparison. A total of 12 specimens, 4 x 2.400g, 4 x 2.500g and 4 x 2.600g was taken from each IH Top, then measured according to the method for calculating regressions described in the previous Section, 'Flow versus mass regression equations'.

Regression equations relating flow and mass were generated for each IH Top. Masses in the range from 2.400g to 2.600g were assigned to regression equations to obtain flow values. Flow and MFD values were used to calculate 201 quadratic functions.

### b) Validation of technique

160 wools were selected to cover the MFD range from 15 $\mu$ m to 40 $\mu$ m. Two specimens were drawn from each wool and weighed to 2.5g ( $\pm$  0.004g). Two separate specimens from the same wool were also drawn and weighed to within a range from 2.400g to 2.600g. Each set of two specimens was measured in accordance with the procedures in IWTO-28-93. Fixed mass and Variable mass flow values and Variable masses were recorded. Flow rates were substituted into the appropriate calibration function, according to the measured specimen mass. MFD values obtained from the fixed and the Variable mass techniques were compared.

## RESULTS AND DISCUSSION

- Flow versus mass regression equations

IH Tops were used to establish 8 regression equations relating flow and mass. This was performed separately for Meters 1 and 2. These regression equations are shown in Figures 4a and 4b.

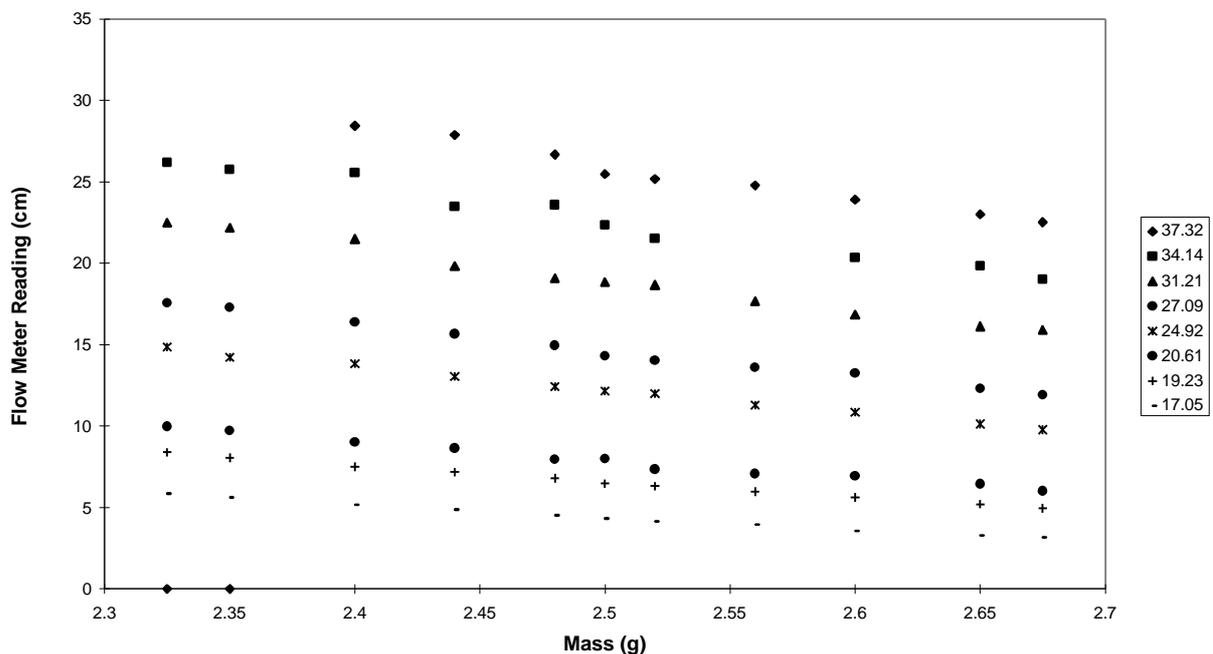


Figure 4a. Flow versus mass relationships for each of eight IH Tops on Airflow Meter 1.

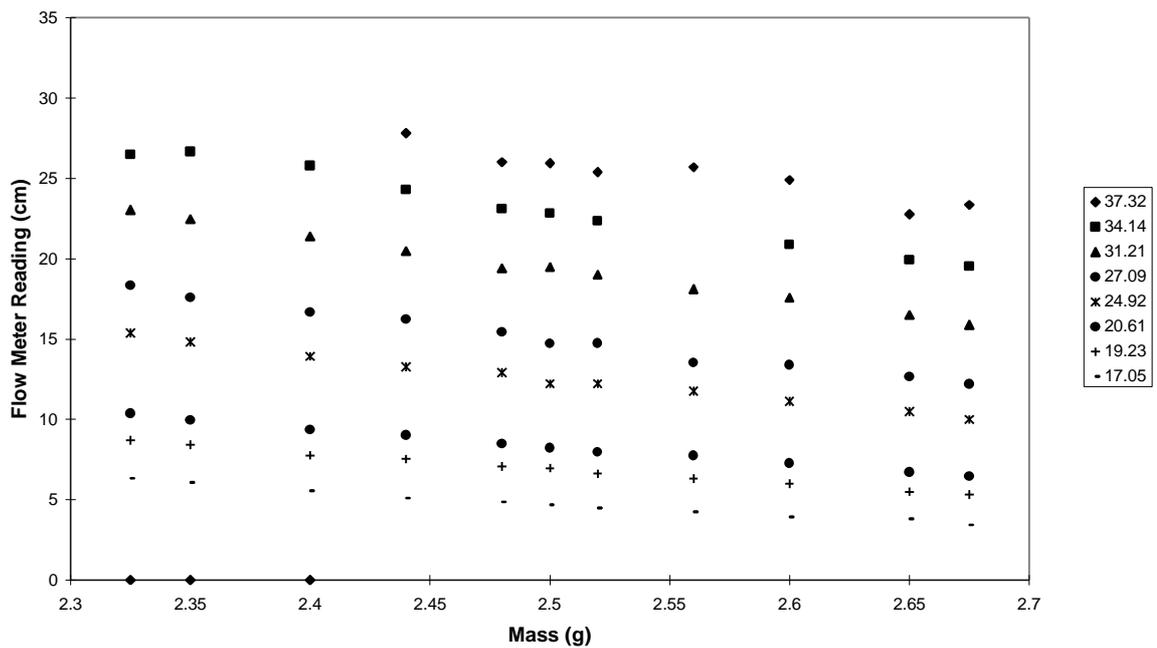


Figure 4b. Flow versus mass relationships for each of eight IH Tops on Airflow Meter 2.

In Figures 4a and 4b, average flow values for two and three test specimens, respectively, for the coarsest Top in the low mass range are missing. Flow rates for these specimens exceeded the measurement range of the rotameter. The regression equations are approximately linear over the range of specimen masses tested, as predicted from the analysis of Airflow theory discussed previously.

The averages of Meter 1 and Meter 2 slope and intercept coefficients were then calculated for each Top. These values were plotted against MFD. Figure 5a shows a plot of slope coefficients versus MFD and Figure 5b shows intercept coefficients versus MFD.

The relationships for both slope and intercept versus MFD are linear up to approximately  $34\mu\text{m}$ , then they deviate from linearity at the extreme coarse end. This may be due to a medullation effect as described in the Theory section.

## 2. Comparison of Variable and Fixed mass techniques

### a) Calibration of Meters

Figures 6a and 6b show flow versus mass relationships based on three test specimen masses at 2.400g, 2.500g and 2.600g for Meter 1 and Meter 2 respectively.

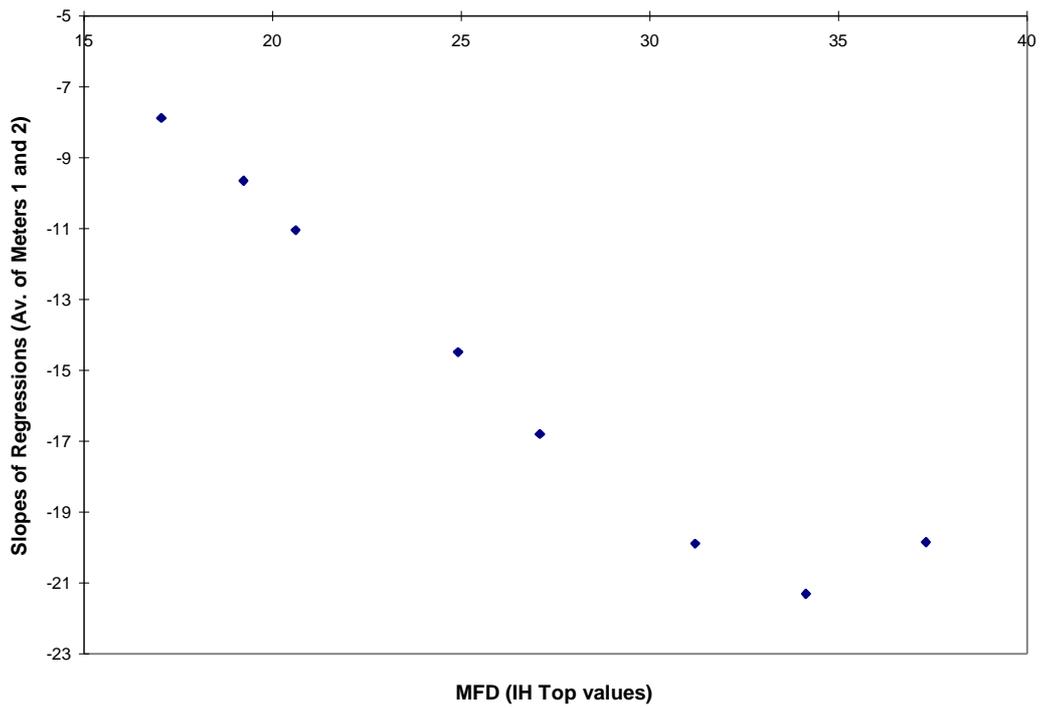


Figure 5a. Relationships between MFD and the average of the slopes of regression equations for Meters 1 and 2 for each IH Top.

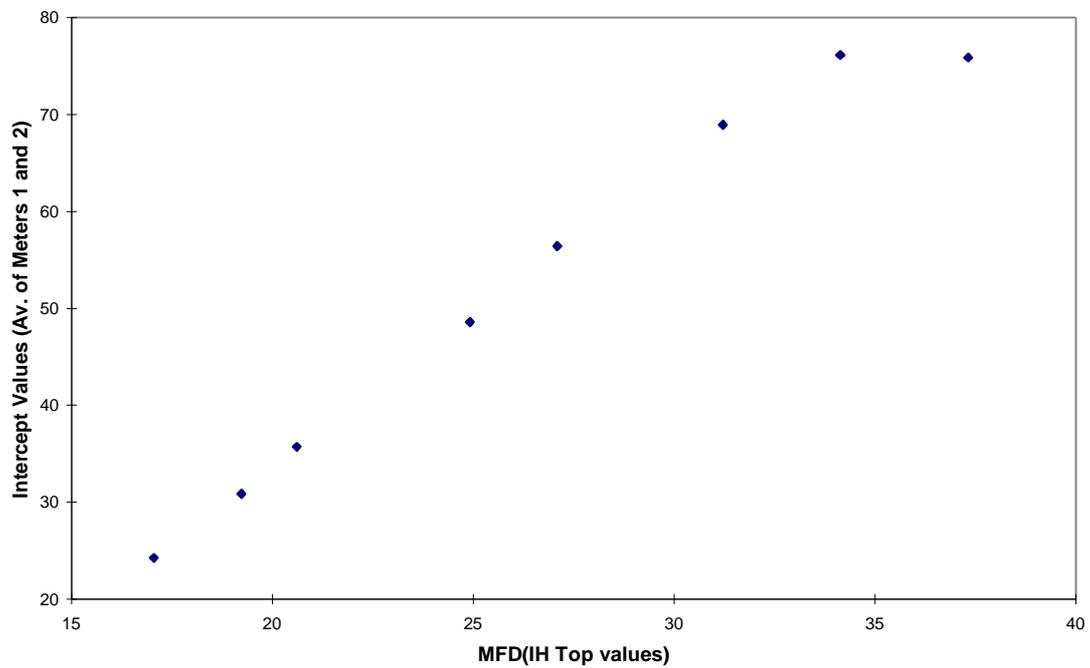


Figure 5b. Relationships between MFD and the average of the intercepts of regression equations for Meters 1 and 2 for each IH Top.

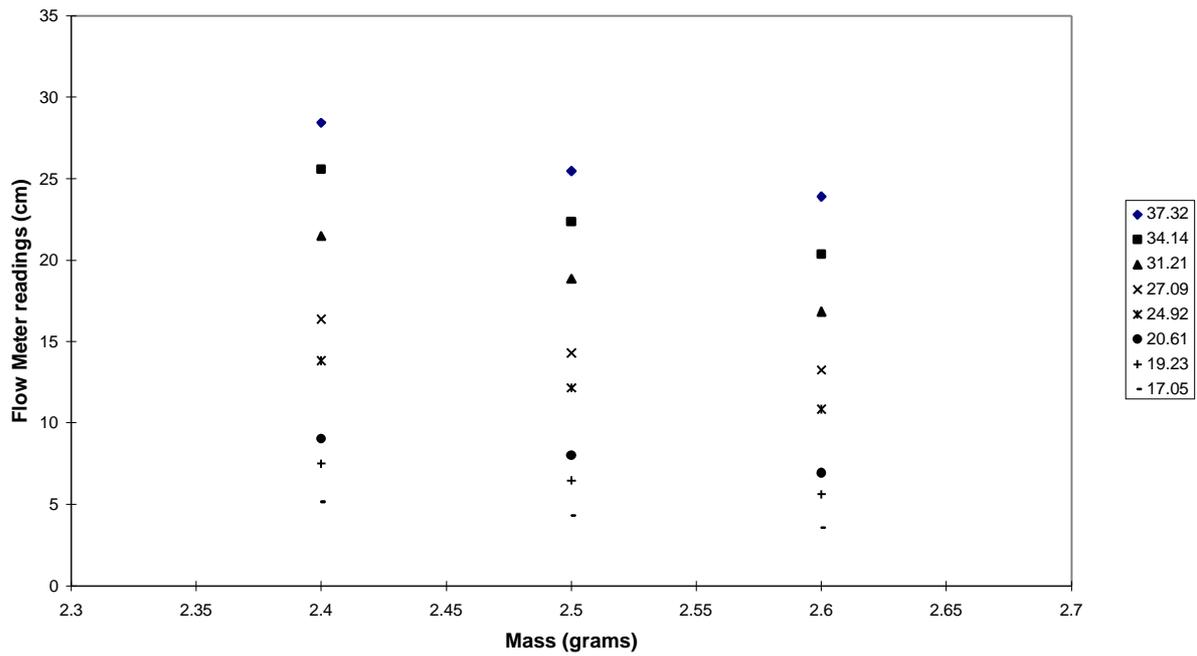


Figure 6a. Flow versus mass relationships for each of 8 IH Tops on Meter 1 using 2.400g, 2.500g and 2.600g test specimens.

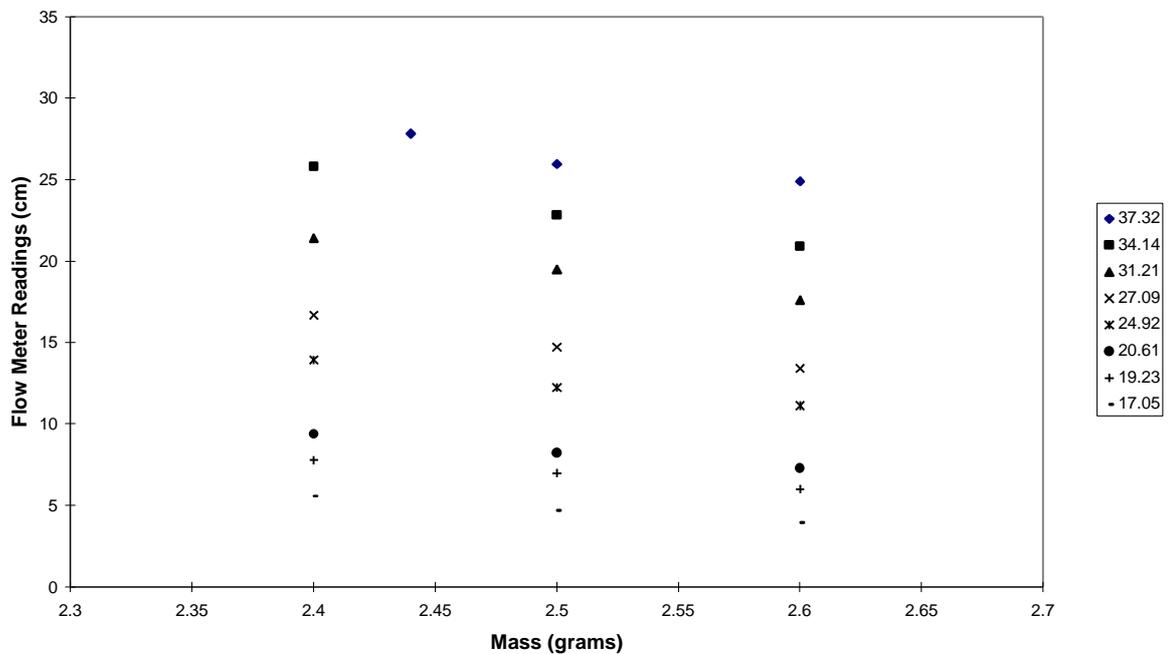


Figure 6b. Flow versus mass relationships for each of 8 IH Tops on Meter 2 using 2.400g, 2.500g and 2.600g test specimens.

Note: No average flow value could be obtained for the lowest mass specimens from Top 37.32. A 2.44 gram specimen mass was substituted in this case.

## b) Validation of technique

Figure 7 shows the difference between the Variable and Fixed mass techniques. Although the data show very small differences between these techniques up to 32 $\mu\text{m}$ , larger differences and a negative bias are apparent in the range from 32 $\mu\text{m}$  to 40 $\mu\text{m}$ . This may be due to higher than normal medullation of coarse wools used in this trial or anomalies in Variable mass calibration for coarse wools as indicated in Figures 5a and 5b.

If medullation is a contributing factor to this variation then a relationship should exist between the degree of medullation and the size of the Variable mass - Fixed mass difference. All wools above 32 $\mu\text{m}$  were subsequently measured for medullation using the OFDA. Only 1 wool had a percentage medullation (8.0%) which was higher than the values for the calibration Tops (2 - 4%) between 32 $\mu\text{m}$  and 40 $\mu\text{m}$ . It therefore can be reasonably assumed that medullation has contributed only a small amount, if any, to coarse end variation in Figure 7.

In Figure 7, data from 15 $\mu\text{m}$  to 32 $\mu\text{m}$  appear to be evenly distributed about zero. This result was expected since the data lie in the linear regions of the relationships shown in Figure 5a and 5b. The reason for the pattern of results above 32 $\mu\text{m}$  is unknown and therefore it was decided to limit the use of the Variable mass technique to MFD's between 15 $\mu\text{m}$  and 32 $\mu\text{m}$ .

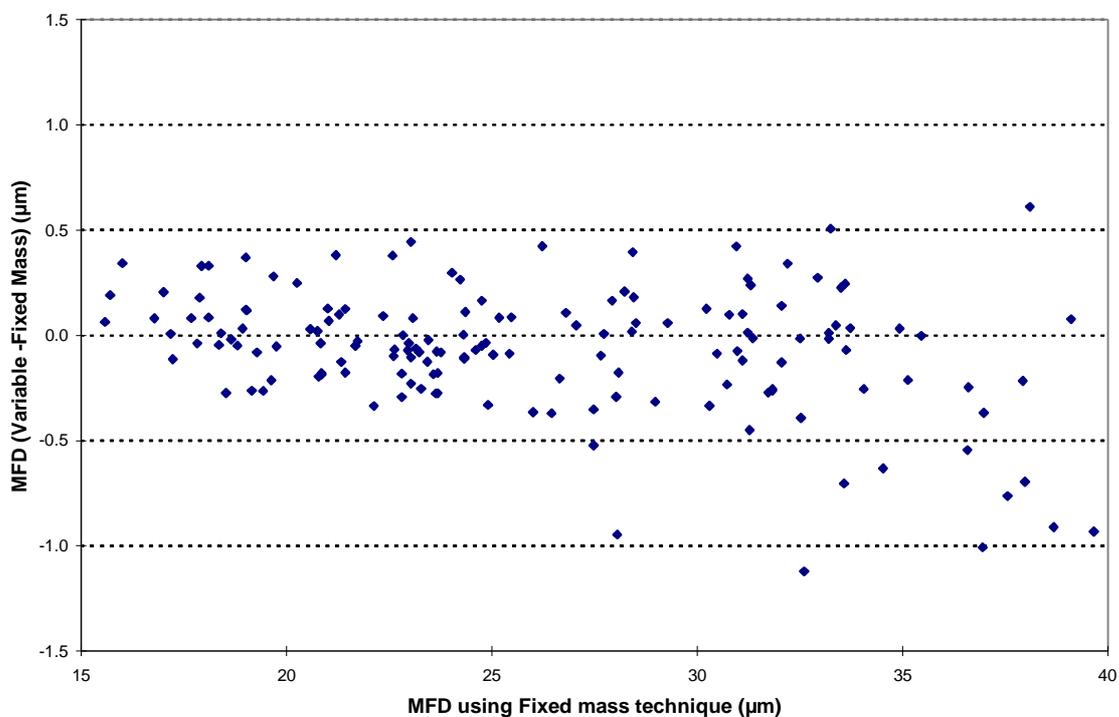


Figure 7. Differences in MFD between Variable and Fixed mass techniques

Figure 8a shows the distribution of differences between Variable mass and Fixed mass techniques for wools in the range from 15 $\mu\text{m}$  to 32 $\mu\text{m}$ . Figure 8b shows MFD results for Variable mass (Treatment) versus Fixed mass techniques (Control). Relevant statistical data are presented in Tables 2 and 3 respectively.

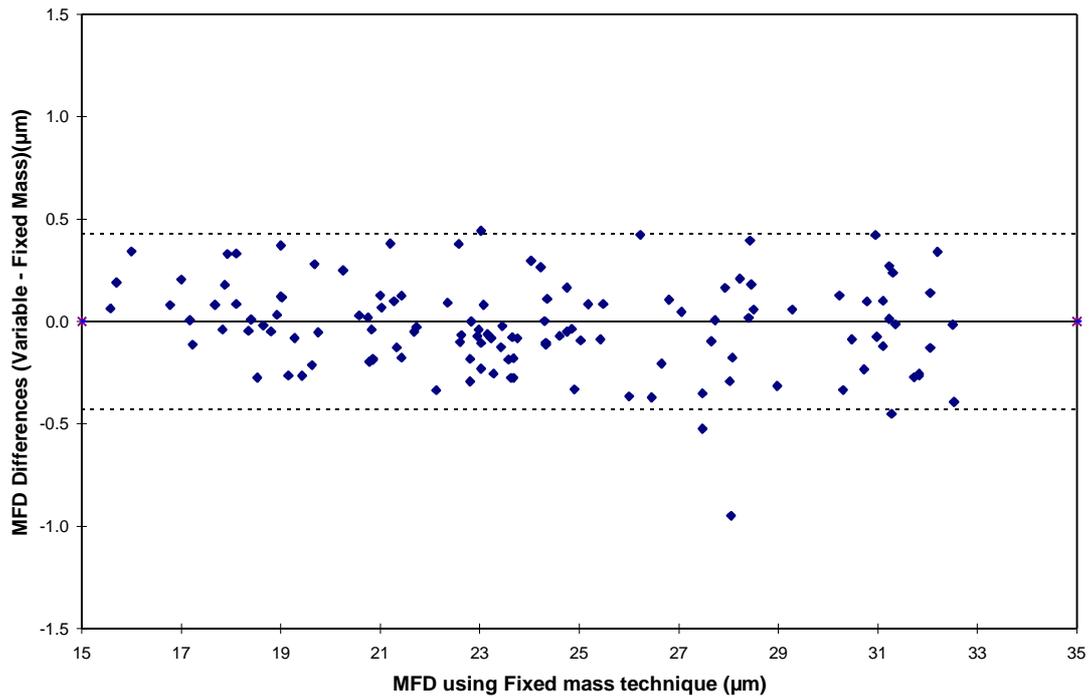


Figure 8a. MFD differences between Variable and Fixed mass techniques for wools between 15 mass and 32 μm.  
Note: “.....” limits indicate Standard Deviation of differences (SD) multiplied by 1.96 (SD=0.22).

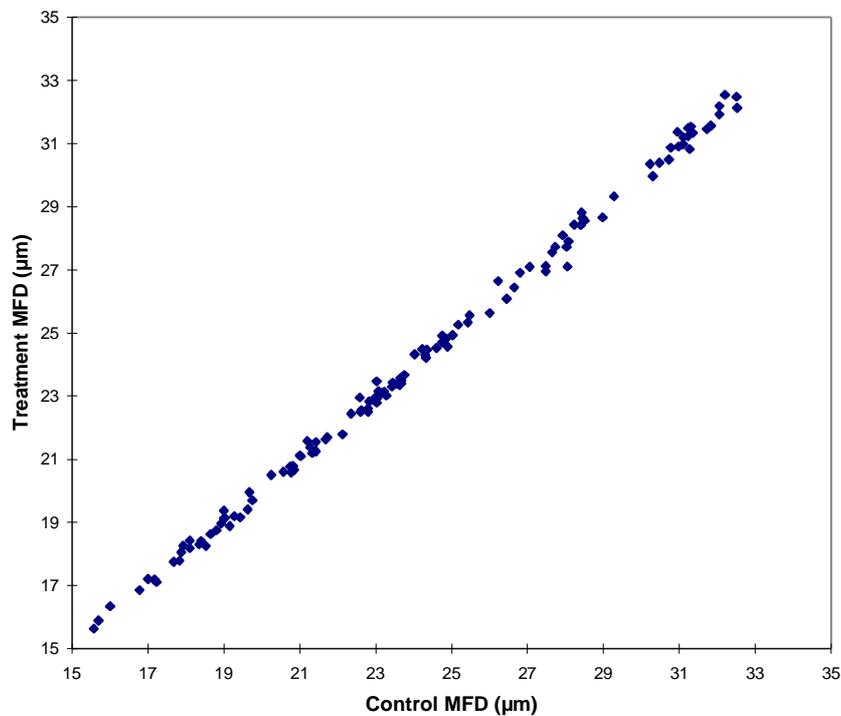


Figure 8b. MFD results for Variable (Treatment) versus Fixed mass (Control).

Table 2. Test for Overall Relative Bias and Paired t-test

	Overall Bias		Paired t-test
	Control	Treatment	
Number	127	127	127
Average	24.1319	24.1063	-0.0256
SD	4.6019	4.5679	0.2227
SE	0.4083	0.4053	0.0198
t value	59.0962	59.4725	-1.2937
p value	0.0000	0.0000	0.1981 <b>NS</b>

Test for Level Dependent Bias

Regression	Slope	SE of Slope	t-Value	p-value	Rsq
GM	0.9926	0.0043	1.7323	0.0857 <b>NS</b>	0.9977
DVA	-0.0074	0.0043	1.7259	0.0868 <b>NS</b>	

Table 3. ANOVA of the Variable and Fixed mass techniques

**Fixed mass technique**

Source	Sum of Squares	Df	MS	F-Ratio	P-Value	Variance Estimate
<b>MAIN EFFECTS</b>						
<b>A: Wools</b>	10678.70	126	84.75	2868.56	0.00	
<b>B:Meters</b>	1.48	1	1.48	50.02	0.00	0.72
<b>RESIDUAL</b>	11.23	380	0.03			0.03
<b>TOTAL</b>	10691.40	507				

**Variable mass technique**

Source	Sum of Squares	Df	MS	F-Ratio	P-Value	Variance Estimate
<b>MAIN EFFECTS</b>						
<b>A: Wools</b>	10515.70	126	83.46	2729.30	0.00	
<b>B:Meters</b>	0.01	1	0.01	0.40	0.54	0.00
<b>RESIDUAL</b>	11.62	380	0.03			0.03
<b>TOTAL</b>	10527.30	507				

Table 2 shows no overall bias or level dependent bias for the difference between the Variable and Fixed mass techniques. Table 3 gives a separate ANOVA for each technique using a Two Nested classification with Replication. Note that in this analysis there is a between-Test Specimen variance component included in the between-Meters variance. This is because, for each technique, two specimens were sampled for each wool, one specimen for Meter 1 and the other specimen for Meter 2.

The results in Table 3 indicate that the between-Meters/Specimen variance is significant for the Fixed mass technique but is not significant for the Variable mass technique as indicated by the probability (P) Values. These results also show that there is a relatively large difference between techniques for the between-Meters/Specimen variance estimate. Since the variance estimate is lower for the Variable mass technique, it will have a higher precision than the Fixed mass technique.

Improved precision for the Variable mass technique could be expected as this technique relies on calibration equations specific to individual specimen mass whereas for the Fixed mass technique, one calibration equation is used over the range 2.496g to 2.504g.

- Application of flow versus mass regression equations to other Airflow Meters

If flow versus mass relationships are the same for all Meters, then calibration of Meters for Variable mass measurement would require calibration only at 2.500g. Values for the slope of the flow versus mass relationship could be obtained from Figure 5a. and an instrument specific intercept value could be calculated from the flow values at 2.500g.

An investigation was performed where new regression equations were calculated on two additional pairs of Airflow Meters. The value of the slope of flow versus mass was obtained from Figure 5a. A comparison was made between the Variable and Fixed mass techniques for a set of 60 wools.

The Analysis showed significant differences between Variable and Fixed mass techniques. This result implies that flow versus mass relationships differ between Airflow Meters. Meter specific regression equations must therefore be calculated when using the Variable mass technique.

## **CONCLUSION**

The Variable mass technique for measurement of MFD has been shown to be equivalent to the Fixed mass technique for wools less than 32 $\mu$ m. The difference between Variable and Fixed mass data was shown to be not significant. The between-Meters/Specimen precision was found to be higher when using the Variable mass technique.

## **RECOMMENDATIONS**

It is recommended that IWTO-28-93 be modified to include the option of using variable test specimen mass when measuring MFD for wools which do not exceed 32 $\mu$ m in diameter.

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