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Development of a portable greasy wool yield instrument

By

Peter Baxter, Gavin Wallace

SGS Wool Testing Services
GNS Science

PO Box 15062, Wellington, New Zealand
PO Box 31312, Lower Hutt, New Zealand

SUMMARY

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This report summarises development of an innovative X-ray-based portable yield instrument for measuring greasy wool samples. The aim was to produce a yield measurement within 30 seconds on a single midside sample, with an accuracy and precision similar to that obtainable with conventional fleece testing methods (approximately $\pm 1\%$ accuracy and 95% confidence limits of ± 3 to 4%). The development spanned some 5 years and progressed through several interesting phases, from a stand-alone X-ray device, to an integrated three-component instrument in which data was transmitted directly to the OFDA2000 classing software.

A working commercial prototype was produced and tested. The evaluation revealed satisfactory performance on core material and on New Zealand fleece samples, but there were some calibration issues with Australian fleece samples that, whilst almost certainly capable of resolution, could not be achieved without further work. The capital cost of the proposed instrument had by that stage significantly exceeded the original specification. At the time the work was commenced, there were several million on-farm measurements being performed annually across Australasia, but due to economic factors, these numbers had fallen to probably less than 1 million in 2005. It was therefore considered that further refinement could not be justified in this economic environment. The project was thus suspended, and a decision made to make the results publicly accessible.

INTRODUCTION

Market research, funded by SGS and IWG Pty Ltd, and carried out in Australia and New Zealand in 1999, established that growers were keen to gain access to on-farm measurement of wool diameter. It was also established that of those growers who already utilised laboratory-based fleece measurement services, approximately half also had yield measurements carried out. In parallel with the introduction of the OFDA2000 service to satisfy the need for rapid on-farm determination of fleece mean fibre diameter, a research program was commenced to design and develop an instrument to rapidly measure wool yield.

The design constraints were that the instrument must be:

- Portable
- Work at the same cycle rate as the OFDA2000 (approximately 2 samples/minute)
- Produce similar accuracy and performance to conventional fleece test washing yield measurements
- Have a capital cost not greater than a specified amount.

Initial investigations concentrated on technology that was familiar – near infra-red analysis (NIRA). Preliminary experiments confirmed that it should be feasible to predict yield using NIRA technology to

sufficient accuracy and precision to comply with the specification. Portable NIR instruments were also available as an offshoot of the space-based remote sensing terrain-evaluation projects. However, at prevailing prices, it seemed most unlikely that the technology could be harnessed within the capital cost specification.

Following lengthy discussions with Geological and Nuclear Sciences (GNS) in Lower Hutt, it was considered that the use of ionising radiation to determine radiation absorption could provide a feasible route. Since GNS already had a project team working on another wool industry application, they had to construct a suitable "firewall" to ensure that there was no leakage of IP in either direction.

Initial investigations at GNS showed promise and a research project to develop a prototype instrument was commenced in 2000.

PRINCIPLES

Raw wool is a mixture of components, such as grease and lanolin, that are removed in the scouring process. Wool yield is basically the percentage weight of pure wool remaining after the additional components have been removed; this factor contributes to the value of the fleece.

Previous work had been undertaken to evaluate transmission measurements of x-rays as a method of discrimination between pure wool and other components. The components have varying elemental compositions, and should therefore exhibit different x-ray attenuations (Franklin, 2000)

	% distribution of four main raw wool components				
	H	C	N	O	Other
<i>wool</i>	5	38	22	21	14
<i>veg. matter</i>	11	4	1	84	0
<i>gravel</i>	0	0	0	53	47
<i>animal fat</i>	12	77	0	11	0

The prototype instrument was based on measurement of the transmission of x-rays through wool. An x-ray beam of intensity I_0 will be attenuated to an intensity, I , after passage through an absorber, and the intensities are related thus:

$$I = I_0 e^{-\mu\theta}$$

where: μ is the mass attenuation coefficient for the absorbing material, and θ is the area density (equal to density x path length).

Where the material is composed of different components, the mass attenuation coefficient will be a combination, by weight fraction, of coefficients of the components. Wool can be considered as a two-component mix: pure wool (represented by what remains after scouring) plus non-wool components (removed by scouring) i.e.

$$\mu\theta = \mu_w\theta_w + \mu_c\theta_c$$

with:

$$\theta = \theta_w + \theta_c$$

If the components have significantly differing mass attenuation coefficients μ_w and μ_c , then a measurement of the x-ray attenuation through a weighed sample of standard dimensions can be used to estimate the relative proportions. In this two component model, the wool yield Y can be expressed as:

$$Y = 100 \cdot \frac{\theta_w}{\theta_w + \theta_c}$$

from which:

$$Y = 100 \cdot \frac{(\mu_c - \mu)}{(\mu_c - \mu_w)}$$

Preliminary measurements indicated that $\mu_w = 0.950 \text{ cm}^2/\text{g}$ and $\mu_c = 1.737 \text{ cm}^2/\text{g}$.

FIRST PROTOTYPE INSTRUMENT

DESCRIPTION

The method adopted for the prototype instrument required samples of about 5g mass to be packed into a plastic container 70mm long and 25mm diameter. The sample was tamped down so that the 5g of wool occupied less than half of the container. Following weighing, the container was slid into a brass block that was dropped into a well to intercept the x-ray beam. The beam was normally shuttered off, and opening of the shutter was only possible with the loaded block. There was therefore no ambient radiation to pose a hazard. The low energy x-rays (14-21 keV) were supplied by a 30mCi Cm-244 radioisotopic disc source. The transmitted x-rays are measured using a 25mm diameter NaI (TI) detector. GNS electronics was used to amplify and count the x-ray events. The counter was controlled by a HP360 handheld computer (HPC). The software on this, running under Windows CE, was written specifically for this application. It combined the sample mass and measured count rate to produce an estimate of wool yield based on calibration from known samples. Counting times were user-set, normally 1 or 2 seconds. The touch-sensitive HP360 screen allowed easy operator use. Each measured sample could be coded and stored in a text file for later analysis, for instance for loading into a spreadsheet.

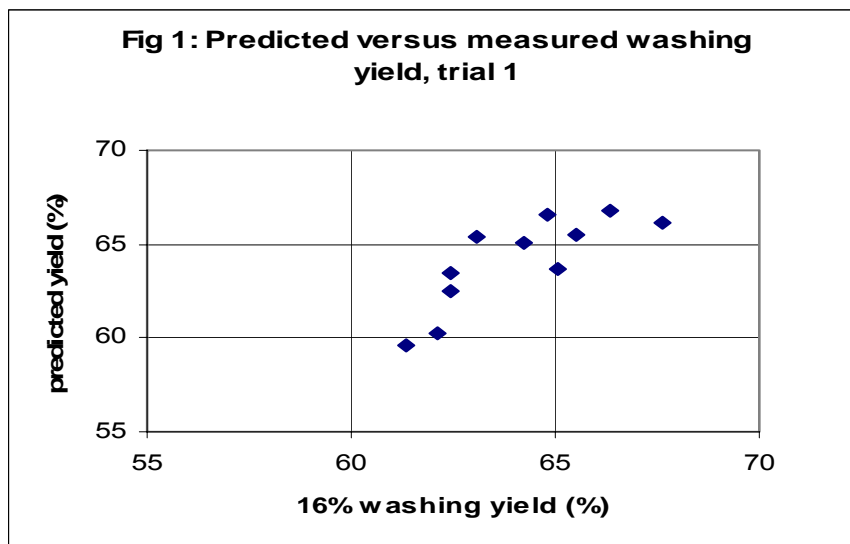
The instrument was mains-powered through a detachable cable. A balance (AND EK-200G) was part of the instrument. This read to 0.01g, adequate for the requirement. An optional serial interface could be purchased with this balance to transfer measurements directly to a computer. In this part of the project, it was decided to concentrate on the technique, rather than the hardware, and the instrument was based on a standard aluminium-clad briefcase. In addition to security offered by the case locks, removal of the sample block (and access to the x-ray well) required a separate key.

The prototype instrument was successfully built as was initially envisaged. The length of time required to process samples was principally due to sample handling, even though this consisted merely of packing the plastic container and weighing. The x-ray measurement took one second, and the results were displayed on the HPC, and could be logged.

INITIAL TRIALS

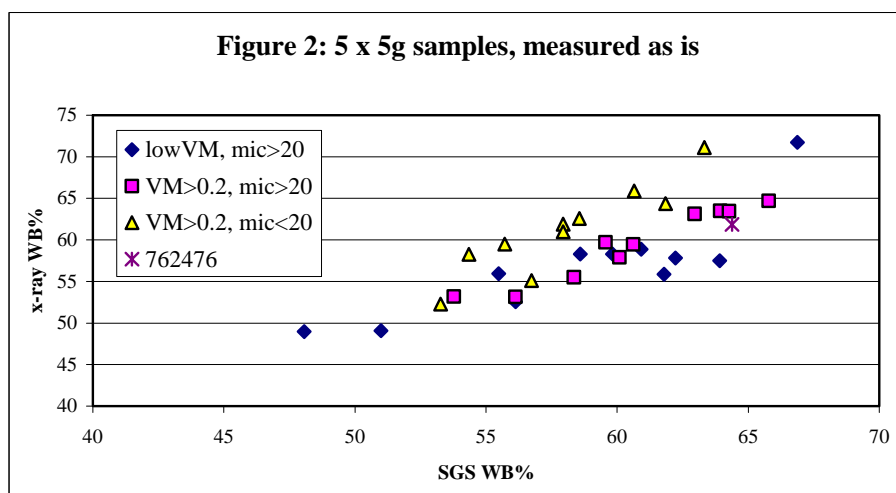
Reference values were initially provided from conventional fleece testing methods in accordance with AS/NZS 4492. Calibration of the instrument with mid-side samples had, however, simply confirmed that both the wool samples and this measurement method were very highly variable. The fleece test method has a published precision of $\pm 3.0\%$ within a single laboratory (Morgan, 1990). It was necessary to carry out the initial evaluation with blended core material to reduce the uncertainty of the reference values. Reference values for the core material were subsequently provided from conventional IWTO core testing (IWTO-19), using the oven-dried scoured yield to determine the 16% washing yield.

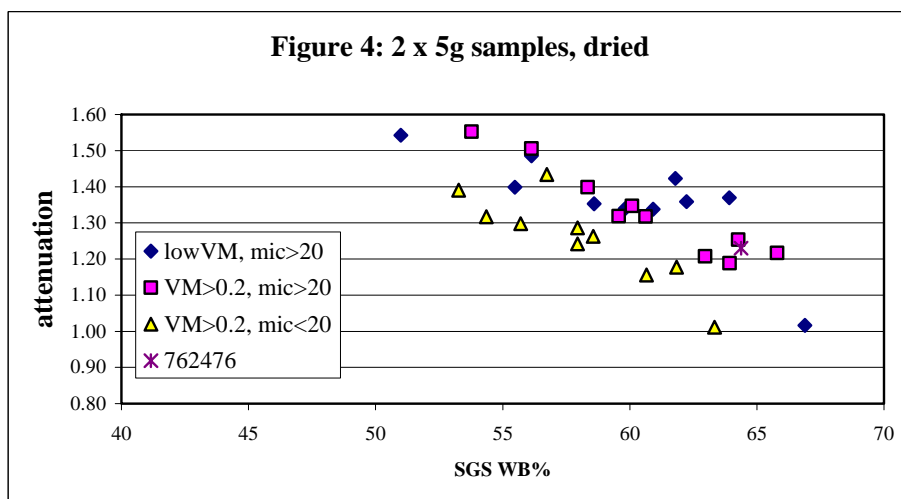
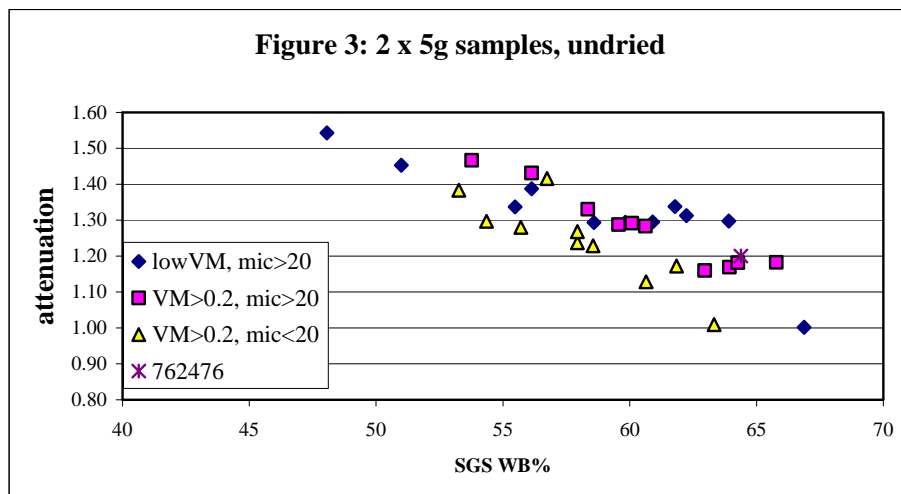
12 samples of fine wool with low vegetable matter were prepared from blended core material that was expected to be more homogeneous than the initial mid-side samples. Each sample was divided into 4 x 25g packets, and the 4 sub-samples were individually measured on the instrument. Eliminating one sample, the average standard deviation in estimated wool yield was 0.70% i.e. the reproducibility was very good. However, there were still systematic variations with the washing yields, as shown in Fig 1 below:



The intended wool yield range was not as wide as envisaged, and all samples were about 18 μm fibre diameter. It was also considered that the supplied values may not be as accurate as initially thought since no especial care had been taken to preserve moisture content. It was also noted that the original certification had been carried out using a total of 300g of wool, rather than the 25g supplied in this project. All in all, there were several valuable lessons to be learnt from this initial trial, but the limited results looked promising.

A broader set of 32 samples was provided with more stringent preparation procedures, handling controls and cross-checking. These were measured twice, first as 5 by 5g subsamples (Fig 2) and then as 2 by 5g subsamples (Fig 3). In the latter series, subsamples were also oven-dried before remeasurement (Fig 4) to check if water content was influencing results. Moisture contents were typically 5-10%.





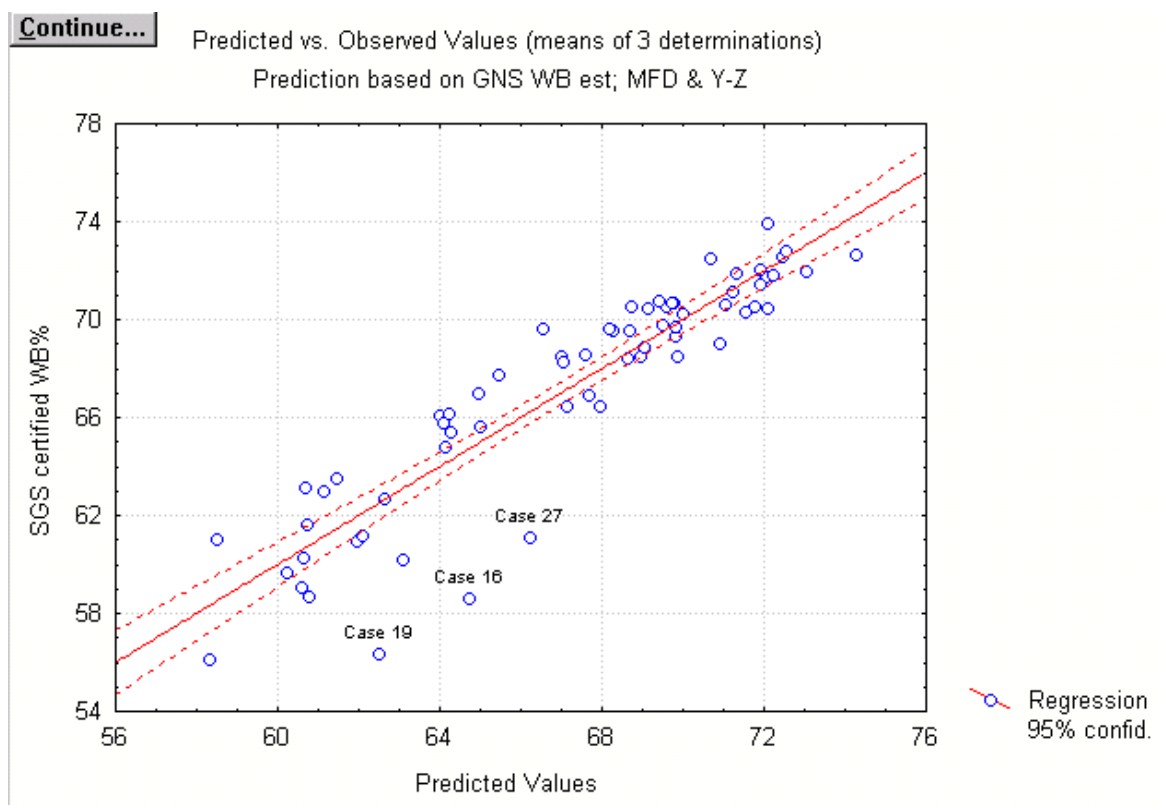
It appeared from this that rather than moisture content being a dominant variable, the mean fibre diameter (micron size) was significant. The reason for this was not understood.

The influence of fibre diameter shows in the estimated wool yields in Figs 2 to 5 above. Inclusion of a variable to take this factor into account improves the agreement between x-ray and gravimetric yields, but the agreement was still marred by unexpected outliers. X-ray fluorescence was carried out on some samples to try and identify whether mineral content might be the reason for anomalies, but this was inconclusive.

Two issues were considered to be causing uncertainty. The first was the fact that woolbase (or yield) is measured with reference to the sample as received at the laboratory. Wool absorbs up to 30% of its weight as moisture, and whilst normally one would expect there to be less than 20% moisture on receipt, the woolbase value obtained is relative to the weight of (wool + water + grease + dirt + vegetable matter) at the time the sub-sample is taken. After the "seal" is broken, the moisture content, and hence the woolbase, can change. There is therefore always some uncertainty associated with extending a woolbase result to a sub-sample which may be transferred to another environmental condition. The second issue is the difficulty of sub-sampling a material which is naturally extremely inhomogeneous. This was alluded to earlier - the certification of woolbase takes place after the measurement of two independent 150g subsamples, whereas the work being carried out here was performed on much smaller aliquots of relatively unknown representativeness. Whilst sub-sampling in the laboratory is performed on blended material, the level of blending is designed to ensure the representativeness of 150 g sub-samples, not of 5 g sub-samples. The laboratory test based on 150 g sub-samples is designed to ensure that woolbase is certified with a precision of $\pm 1\%$ expressed as 95% confidence limits.

In order to minimise the effect of change in conditions from sampling, the instrument was set up adjacent to the core sample blender at SGS's Wellington core test laboratory, and representative samples (as far as could be judged by eye) were taken directly from the blended product. The results from this exercise were encouraging when diameter and colour (yellowness) were incorporated in the prediction:

Figure 6: Results using blended core samples



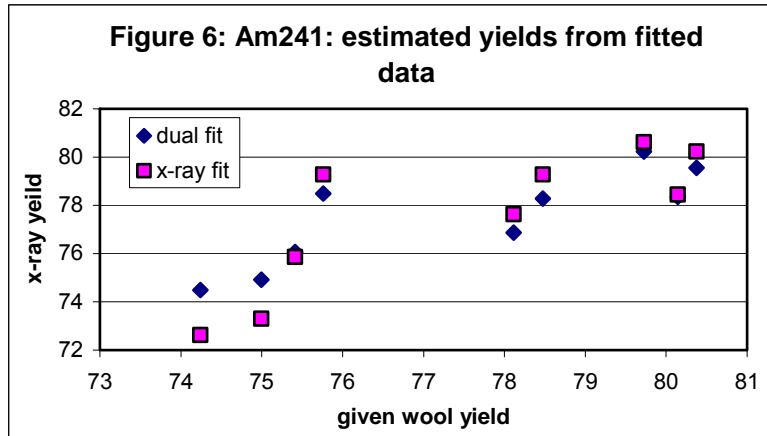
The three cases highlighted as outliers were all wools that would be regarded as atypical. If these outliers were removed, the standard error of prediction diminishes to approximately 1.5%, which is regarded as being within range of an acceptable level of precision.

In order to trial the instrument on a wider variety of samples more typical of what would be encountered on farm, it was moved to Timaru in early April 2001. Whilst there, it was used on wools from five separate farms and, with one exception, the results proved unsatisfactory, in that the precision was very poor. It should be noted that in this trial the results were being compared with fleece testing yield results, which are typically much less precise than core test results (95% confidence limits of $\pm 3\%$). Nevertheless, the standard deviation of the differences was well in excess of what could be expected even given this handicap.

There were three conclusions from all of these trials:

1. the high variability of wool was inadequately described in terms of the two-component model
2. the moisture content did not seem to have a significant influence, other than on potential uncertainty in the reference result.
3. the fibre diameter did seem to be important

It was therefore resolved to dismantle the prototype instrument so that the source could be changed to Am-241, which emits a 60 keV gamma ray in addition to x-rays similar to those from the original Cm-244 source. Although the attenuation of the gamma rays through wool would be much less, it was considered that the dual energy approach may enable anomalies in wool base to be identified (Bartle, 1999). This change also required major alteration to the detector electronics; basically, a 'lashup' of electronics and software was put into use. Initial trials with this modified system were carried out on the previously supplied 'low & fine' series of samples:

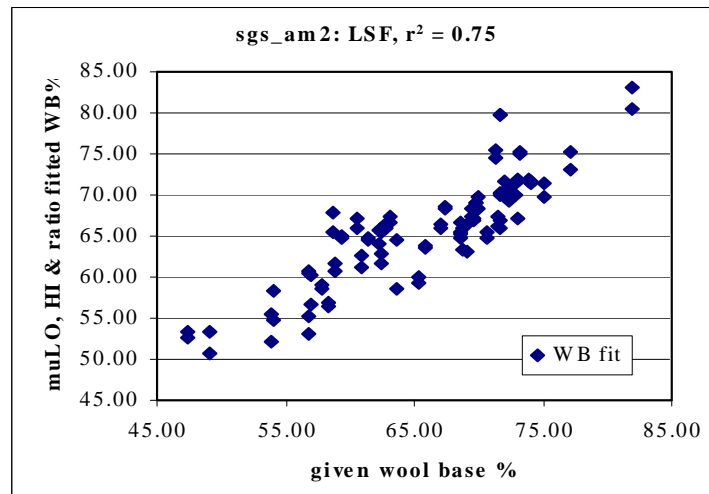


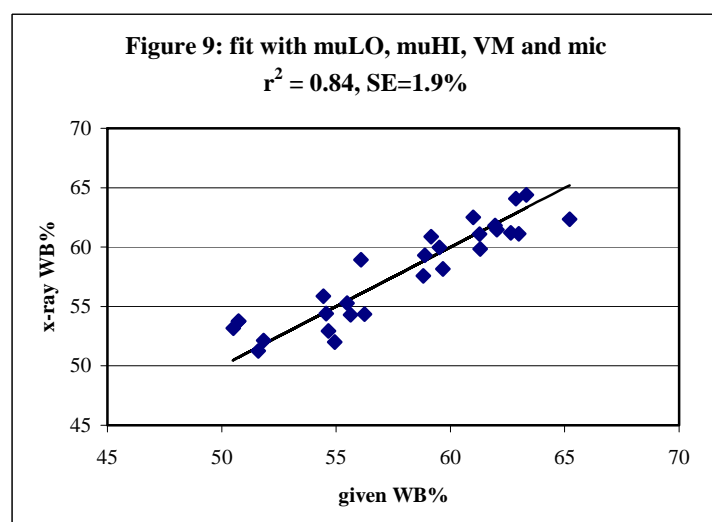
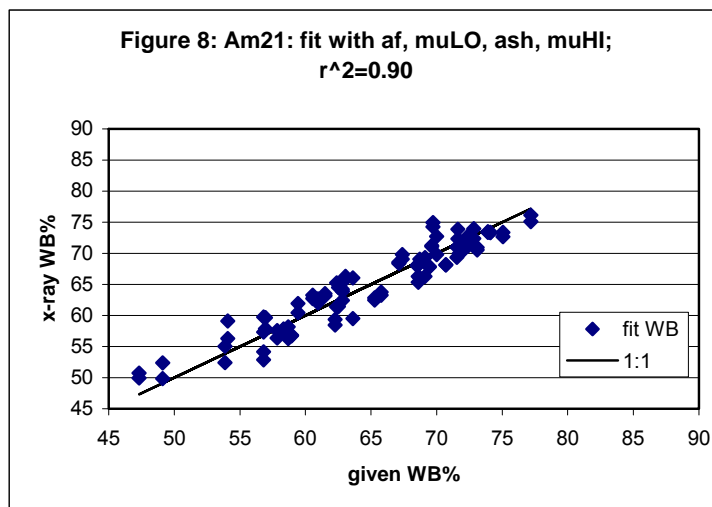
The dual fit demonstrated an improvement. To test this, a fresh set of 50 blended samples were analysed, using two subsamples of each. The results are plotted in Fig 7.

Clearly, the agreement between pairs of samples was good, but the overall standard error of 3.7% was still inadequate. On review, it was found that some slipe wool samples had been included. These were removed, and other wool parameters, particularly wool diameter were introduced (Fig 8).

The standard error from fitting, at 2.2%, was improved, but still too high. In addition, the wool samples used were of relatively high fibre diameter - in the range 26-43 μm , and parameters such as ash content which had been used in the prediction would not be available in the farm environment in which the instrument was to work. A final suite of blended fine wool samples was therefore submitted for measurement (Fig 9).

Figure 7: Results on fresh set of 50 blended samples

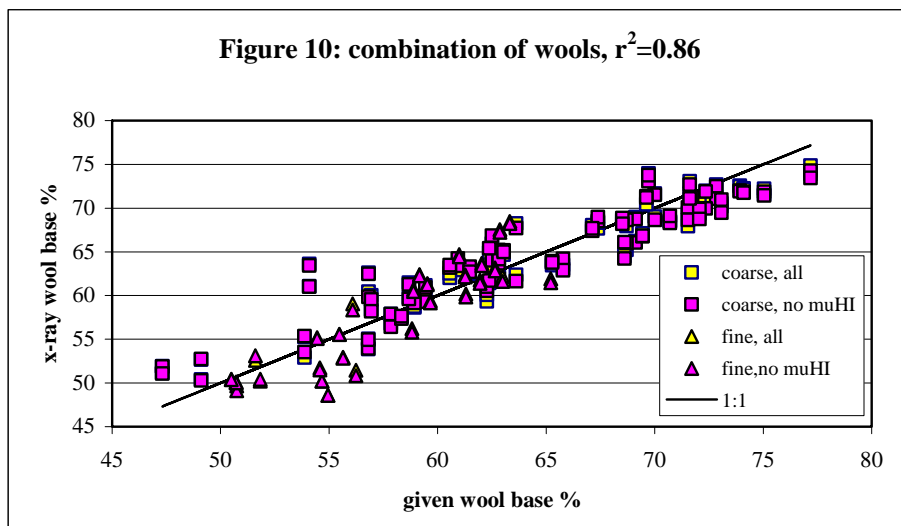




Here, *VM* refers to vegetable matter, ranging from 0 to 6.1%, and *mic* is the mean fibre diameter, ranging from 16.6 to 24.9 μm . The standard error was 1.9%, and, as indicated earlier, this is verging on what commercial fleece testing laboratories are capable of. This represents an improvement on the earlier results on blended samples, since here the standard error of prediction is based on individual measurements, whereas the earlier plot used the average of 3 X-ray determinations (the equivalent of $\pm 1.5\%$ on 3 measurements is $\pm 2.6\%$ on a single measurement).

DISCUSSION

Measurements of the last two suites of samples indicated that the technique based on x-ray attenuation in weighed blended wool samples could provide acceptable determinations of wool base if the x-ray data was combined with estimates of vegetable matter and fibre diameter. Vegetable matter can be appraised visually, and an estimate could therefore be included when making the wool base measurement. The reason why fibre diameter is important was not understood. Its inclusion does not pose a problem, however, because it was always intended that the on-farm wool yield analyser would be used in conjunction with another portable instrument to measure fibre diameter. The last two suites of samples shown above were distinct as fine and coarse groups. These have been combined in Fig 10.



The overall standard error in this linear regression has increased to 2.7%, and the removal of ash content from the data on the coarser samples worsened the estimates. In addition, vegetable matter for this group was unavailable, and was set to zero. This could explain why some of the outliers have deteriorated in the fit. However, it is clear that fine and coarse wools can be combined. It is also clear that although the change to include the high energy gamma ray (muHI variable) has improved accuracy, and reduced the standard error, the improvement is quite small. However, the muHI variable did help to reject some samples such as crutchings and slipe wools that were included in the groups by accident.

CONCLUSIONS OF THE PRELIMINARY WORK

1. A rapid, on-farm portable prototype instrument based on x-ray attenuation was built.
2. The reproducibility of x-ray measurements of blended wool samples was good.
3. The high variability of wool, including within a fleece, has caused significant problems.
4. X-ray measurements alone are not sufficient to accurately analyse samples for wool yields; combination with fibre diameter and vegetable matter is required. The reasons for this are not well understood.
5. True dependency of the X-ray result on moisture content seemed insignificant.
6. The overall accuracy and precision on blended samples is close to that provided by laboratory analysis of fleece samples.
7. The sum of deviations in estimated wool yields from laboratory values over the last two groups of samples was zero i.e. overall, there was no systematic difference in mean values.
8. The prototype instrument needed to be changed to facilitate measurements on larger samples and thus avoid the need to blend samples.
9. It may be useful to analyse a range of scoured wools to check for variability in x-ray transmission, and to see if fibre diameter has a real influence.

SECOND PROTOTYPE

DESCRIPTION

The second prototype was primarily a bench-top device, not intended for use outside GNS. The principal aim was to use a larger sample size, since this had been identified as one of the shortcomings of the previous work.

Multiple Measurement Technique

Because of the limited x-ray beam and detector diameters, it was suggested that multiple measurements would be necessary to cover all of a sample. A measurement is of the x-ray attenuation as it passes through the sample. If the transmitted x-ray beam intensity in the absence of the sample is I_0 , and I_i is measured with the sample intercepting the beam, then the mass attenuation coefficient μ is

$$\mu = -\frac{1}{\theta} \ln \left[\frac{I_i}{I_o} \right]$$

where: θ is the areal density (mass/area) of the sample. μ is a combination of coefficients representing the various components of the wool, and will change according to the wool yield. It is also dependent on the energy of the x-rays used, and is higher (more absorption) for lower energy x-rays.

With multiple measurements on different parts of a variable sample, it was asserted that rather than average the transmitted beam intensities, it was more accurate to average the calculated μ 's. This was the basis of this trial.

Samples

SGS supplied 37 samples each in 6 bags over a wool base range of 44.5% to 62.4%. Each bag contained 30g of wool, and the samples had been blended so that each bag should have had the same wool characteristics. In addition, the fibre diameter and vegetative matter values were supplied.

Equipment

Phase 1 of this part of the project covered the design and assembly of gear suitable to conduct this trial. The focus was on proving the technique, and not on producing a prototype instrument. Therefore, laboratory equipment was "bread-boarded" for the measurements. A step motor drive was fabricated to enable a plastic cosmetic jar (62mm outside diameter) to be rotated through an off-centre x-ray beam. Throughout these trials, 8 x-ray measurements were made at 45° intervals through the sample contained within the jar. A five-second x-ray detection period was used for each.

The intention was to use the radioisotopic source Am-241 that emits both low energy x-rays and a 60 keV gamma ray. This had produced the best results in the previous trial when combined with fibre diameter and vegetation matter data. The only suitable strength of source available was 100 mCi, and this proved too intense for the detector and electronics. To restrict counting rates, some lead washers were used to collimate the beam, and two suites of measurements were taken. However, reproducibility checks on attenuation (μ) indicated that the collimated beam area was insufficient to fully cover all the sample within the jar. For three samples examined, the standard deviation of measurements on the same packed bag was typically $\leq 0.8\%$ with the jar offset 9°, 18°, 27°, 36°; up to 8% when the same bag was repacked into the jar; and up to 6% over all 6 bags of a sample. Clearly, the measurement technique was accurate, but the sampling with the narrow x-ray beam was not.

All subsequent measurements were therefore made using a Cm-244 x-ray source which had 1/3 of the activity. All collimation was removed so the off-centre beam intercepted as much of the sample in the jar as possible. This may have meant that measurements were over a slightly annular area.

Measurement process

Typically, the octet of measurements made whilst rotating the jar provided a standard deviation of up to 18% for the μ 's. With the sample left in the jar, but the octet repeated at offset angles of 9°, 18°, 27°, and 36°, the standard deviations were $< 0.32\%$. When the same sample was repacked into the jar, the standard deviation was typically 2% for middle and high woolbase samples, but increased to 6% for the low wool base samples. Over all 6 bags of a sample, the standard deviation was about 2% for middle and high woolbase values, but increased to 4% for the low wool base samples. The conclusion was that the measurement technique was accurate, and that the combination of multiple measurements brought significant improvement.

Attenuation measurements were made on bags 2 and 3 of all the samples. From these, the given wool bases were fitted to the linear expression

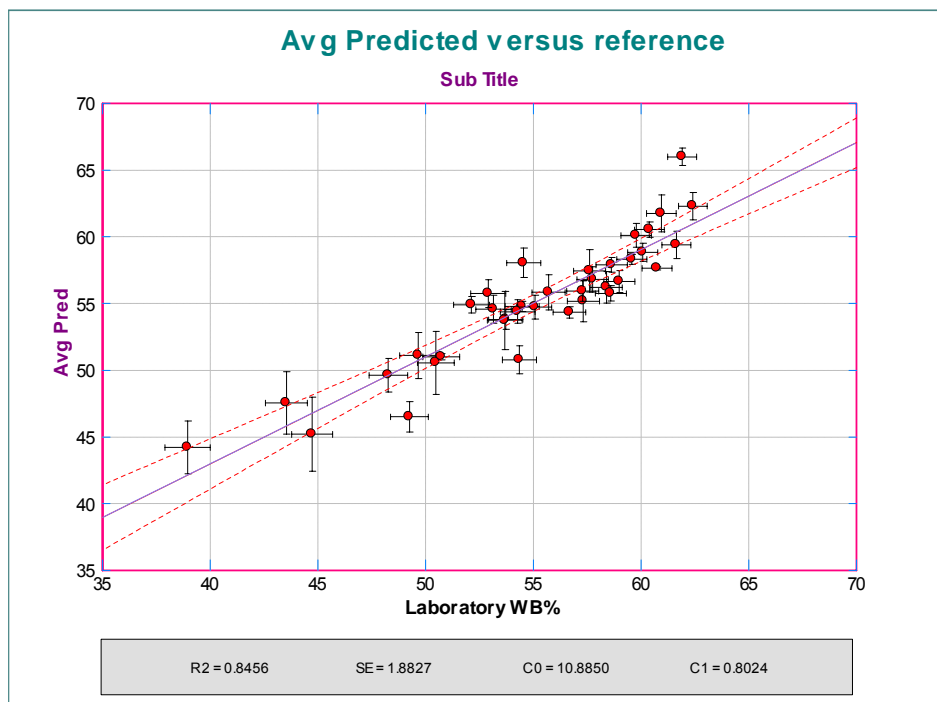
$$WB = A + B\mu + C.VM + D.MIC$$

where: μ is the average over the eight measurements on the jar.

TRIALS

Calibration was undertaken using 37 core samples split into 5 replicated 30g subsamples, covering a woolbase range of 48 to 65%. Estimates were made of the standard deviation (precision) of the certified core test woolbase values using data from IWTO-19. The standard deviation of the predicted woolbase was obtained from 4 replicate measurements. The calibration outcome is shown in Fig 11.

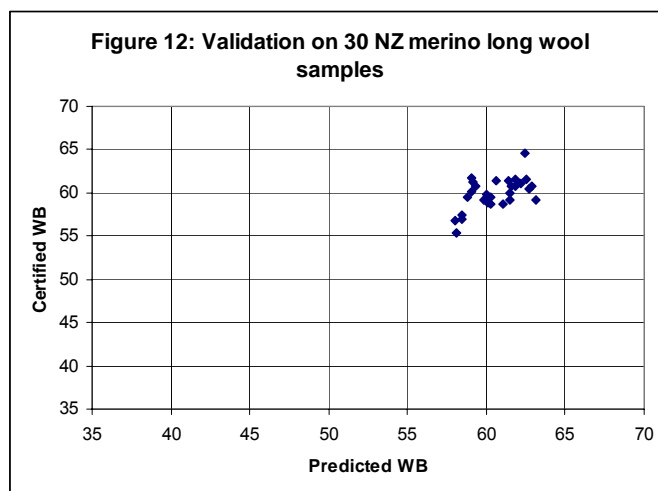
Figure 11: Calibration – predicted versus reference woolbase values



The response was deemed sufficiently satisfactory for the project to proceed to validation. (Note that the regression shown in the above plot appears biased simply because the predicted values were plotted against the reference values rather than the other way round.)

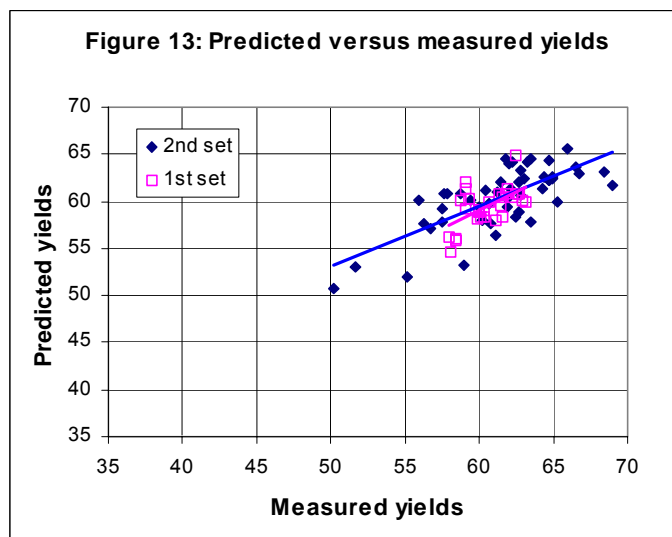
Validation part 1

In part 1 of the validation, GNS were supplied with 5 x 30g replicates of 30 long-wool samples, in order to validate the calibration on an independent set. Unfortunately, because the yield results were not known before the samples were selected, the range of woolbase values in this set of New Zealand merinos was limited to 58 to 63%. The samples were obtained by carefully assembling sets of tufts from length and strength sampling. Once the X-ray measurements had been made, the subsamples were returned to the SGS laboratory, where they were cut into core-size lengths and then carefully scoured using conventional methods. The results of these measurements are shown in Fig 12, with the same scales as Fig 11. It was concluded that with virtually no bias and an estimated precision of prediction of woolbase of $\pm 3.4\%$ (compared with $\pm 3\%$ for washing yields by conventional fleece testing), the project should proceed to evaluating performance on a wider range of long-wool samples.



Validation part 2

Following this initial validation, an attempt was made to select a wider range of wools, and to include some from Australia. 48 grab samples were selected, comprising 30 NZ and 18 Australian merino and half-bred wools. Fig 13 shows the validation results on the total of 78 long wool samples from both series, with the 2 sets identified - the pink squares show the initial 30 validation results:



It was observed that whilst the two sets of data seemed to be consistent, the second set (which contained some Australian wools) was more variable, with some errors of as great as 7%. The data was therefore re-examined for the influence of source. Analysis of variance on the 'errors', and tests for heterogeneity of regressions both indicated that at the 0.05 level, the source effect was not statistically significant. Homogeneity of variance tests indicated similar levels of variance in both sets. However, there were only 18 Australian samples in total, and the significance levels in the ANOVA and HoR tests were 0.088 and 0.055 respectively, and therefore this conclusion of an insignificant effect was regarded as tentative. Whilst two validation regressions could be produced, the 95% confidence intervals overlapped and it was concluded that there was insufficient evidence at this stage to suggest that source was an important variable.

Accuracy & precision

Whilst it was unfortunate that the validation set still did not cover as wide a range of woolbase values as the calibration set, the actual range was probably representative of the majority of fleece wools.

There are two primary issues to be considered when looking at new instruments or methods - accuracy and precision. **Accuracy** is a feature of calibration, and all this work has demonstrated that provided the instrument is calibrated with a wide range of wools, it seemed "reasonably" accurate. However, the validation response with all 79 wools would fail any strict criteria such as those in IWTO-0 (although it should be noted that the instrument was never intended to be used for IWTO measurements). It similarly would fail with just the NZ long wools, suggesting that either the wool state (core samples or long wool samples) has an effect, or there was some change in the system between the initial calibration and the two validation periods. It could also be simply an artefact of the reduced range of the validation samples.

In an effort to examine possible causes for this anomaly, the changes in sub-sample masses were examined from when originally weighed at GNS and subsequently at SGS (for the woolbase determination). Whilst statistically significant, the average mass change was only 0.01g, which is trivial compared with the effects sought here. For a number of the samples, the original certification data was able to be obtained. This would not have been determined on the long wool samples themselves but on the associated core sample. The woolbase values obtained in this trial were on average 1% lower than had originally been certified - however, there was no level-dependent effect that could have explained the failure of the GM regression slope criterion in IWTO-0. (The fact that the woolbase figures were lower on the long wool samples is not unsurprising. These samples would have been handled several times in the process of taking the sub-samples, and, for the purposes which these samples are required, there was no necessity to control moisture content - hence the reason for the woolbase being determined on these samples after the GNS measurements have been completed.)

A possible contributor to the slope validation error might be that the calibration set contained wools with a narrower range of diameters (16 to 22 μm , compared with 16 to 31 μm), and a wider range of VM (0.2 to 5.3%, compared with 0 to 2.3%). In comparing the original calibration equation with regression equations calculated from the long wool validation data, it was noted that VMB did not play a significant part in the latter, whereas diameter seemed to play a more significant effect, judging by the size of the coefficients.

Clearly, if the decision was to be made to move to Stage 3, of producing a commercial prototype, more care would be needed to resolve this issue. However, the effect, whatever the cause, appeared to be simply level-dependent and should therefore have been correctable.

Evaluating the instrument with respect to **Precision** involves a number of issues. Replication allows an assessment of sub-sampling variance, and comparison with the reference method allows an overall assessment of sampling plus prediction variance. However, the reference method is in itself is not very precise. The techniques used in this project, of measuring the whole sample, are of some assistance, but still leave us with a degree of uncertainty.

Examining firstly the reproducibility between 30g sub-samples (which is a combination of instrument performance and sampling error variances, although mainly the latter), a simple analysis of variance (ANOVA) confirmed that the average error variance (ie. within-sample) was 2.02 %², compared with 0.81%² on the calibration core samples (and 1.68 %² obtained on replicates of core samples in the original 5g instrument). In other words this instrument had significantly reduced this component of variance compared with the 5g instrument, as intended, and long-wool samples are more variable than core samples, as is obvious.

If we now examine the relationship between the average predicted results and the reference values, the average variance of the differences ranges from 4.0 %² ('re-calibrated' set using the validation data) to 5.3 %² (using the original calibration formula). However, a portion of this variance is due to imprecision in the reference method, and this is where some assumptions had to be made. In general terms, one expects a standard core test to have a total error variance of approximately 0.25 %² over the range of woolbase values with which we are concerned (IWTO-19, table G1). However, this estimate relates to the average result of at least two 150g sub-samples, but in this trial, only one sub-sample could be measured, and this comprised a chopped long-wool sample, which would not be as uniform as core-sample material.

Another way of estimating the variance is to examine the comparisons between these measurements and the original certified values, for which the average variance of the differences was 2.0 %² in this trial. If the original core test in this case can be assumed to have the published error variance, it would suggest that the residual variance associated with the 'reference' result in this trial is approximately 1.75 %². However, this estimate overlooked the component of variance that would be associated with the

imprecise relationship between the core sample and the long wool sample, for which we had no estimates. A modest (compromise) estimate of the precision of the 'reference' results in this trial, used for the remainder of this discussion, was 1 %². The above discussion draws attention to the need to also have replication in the reference measurement.

If the estimated reference imprecision of 1 %² is deducted from the residual variance of the comparisons between predicted and measured, the residual is approximately 3 to 4 %² for the variance associated with the instrument result, indicating a probable 95% confidence limit of $\pm 3.5\%$ to 4.0 %. It must, however, be born in mind that the instrument result in this case was actually the mean of 5 individual sub-sample measurements, for which it had already been deduced that the within-sample or between-subsample variance was 2.0 %². The contribution of this component to the average was therefore $2.0/5 = 0.4\%$ ², and if this was removed from the estimate, the residual (instrument + prediction + sample error) variance was approximately 2.6 to 3.6 %². For a measurement carried out on a single long wool subsample of 30g, this calculation suggested a total variance of 4.6 to 5.6 %² on such a single measurement, implying 95% confidence limits of $\pm 4.3\%$ to 4.7 %. This did not compare very favourably with the published 95% confidence limit of $\pm 3.0\%$ for conventional fleece testing washing yields. Looked at in another way, in order to obtain a similar level of precision, this work suggested that 4 or 5 midside samples would need to be measured from each animal, which was clearly impractical.

Discussion

The contributions from the various sources of error were examined with a view to deducing whether this situation could be improved:

Source	Variance component % ²	% of total
B'tween subsamples	2.0	36 to 43
Sample+prediction+instrument	2.6 to 3.6	57 to 64

Clearly the subsample variance had a significant contribution to total variance, and whilst this could be reduced further by taking more sub-samples, or even considering a yet-larger instrument, there was a logistical boundary for in-field use of the instrument for fleece testing. The use of more sub-samples would clearly be of assistance if the instrument were to be used in store or by country buyers to assess private sale lots, and indeed the total precision for this application would be somewhat better anyway, since this application would use core samples.

The second component, (sample + prediction + instrument), largely comprised variance due to the prediction process, as can be seen from Fig 11. Data produced by GNS showed the instrument to be very reproducible on the same sub-sample, and since the same sample was in each case used for both the prediction and woolbase measurement, the 'sample' contribution was virtually nil in this work. Hence the conclusion that the prediction process itself was the major contributor to the total variance. In other words, the physical principles used did not seem to be adequate to explain enough of the variability in woolbase.

Interim Conclusions

The conclusions at this stage were:

- Whilst there were some accuracy issues, we considered that these could be overcome
- The precision of the instrument for determination of woolbase on individual fleece samples was estimated to be between $\pm 4.3\%$ to 4.7%. A major portion of this imprecision arose from the inability of the physics of the instrument to fully model the nature of greasy wool.
- The precision of the instrument for determination of woolbase on core samples was of the order of $\pm 3.5\%$, which could be improved by measuring more than one subsample.

By comparison, the precision of woolbase determination by certification core testing is $\pm 0.9\%$ to 1.7%. Precision of washing yield on midside samples (determined by the conventional fleece testing methods) was documented by P. Morgan, from analysis of the 1988/89 Woolplan trials, to be $\pm 2.9\%$ to 3.4% within an individual laboratory, or $\pm 3.7\%$ to 5.1% across a number of laboratories. In each case the lower figure was from fleece testing carried out by the major core test laboratories, rather than from laboratories that only tested fleece samples.

Purely on the basis of this data, the instrument in its current form did not appear to be of adequate precision for measuring individual fleece samples.

ADDITIONAL VARIABLES

In early February 2003, a meeting was held at GNS to consider the report and evaluate options for further work. It was agreed that a further trial would be carried out in which, in addition to the X-ray measurements, full OFDA2000 parameters on the greasy wool plus airflow measurements on the greasy wool would also be utilised. The logic for this was that airflow on the greasy wool may present additional data related to the amount of dirt and grease in the sample, and that the full suite of OFDA2000 measurements may give additional fibre surface roughness information that might also assist.

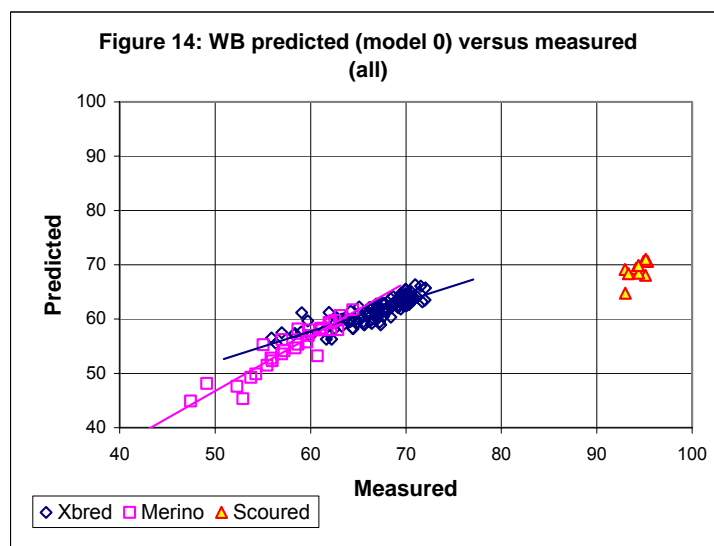
Following the agreement of a trial procedure, core samples were then sought to cover a wide range of diameters and yields. Unfortunately the samples could only be sourced in two stages. The initial set comprised only crossbred samples, and merinos and scoured samples were added slightly later. In total 140 samples were measured in duplicate using all 3 instruments.

Results

For the purposes of comparison, the data was first examined using the original prediction model, which utilised the X-ray data, diameter and vegetable matter, which was of the following form, where A to D are coefficients:

Model 0: $\text{Pred WB} = A - B \cdot \text{X-ray attenuation} - C \cdot \text{VMB} + D \cdot \text{micron}$

It can be seen from Fig 14 that there were some significant biases apparent using this model on this data set:



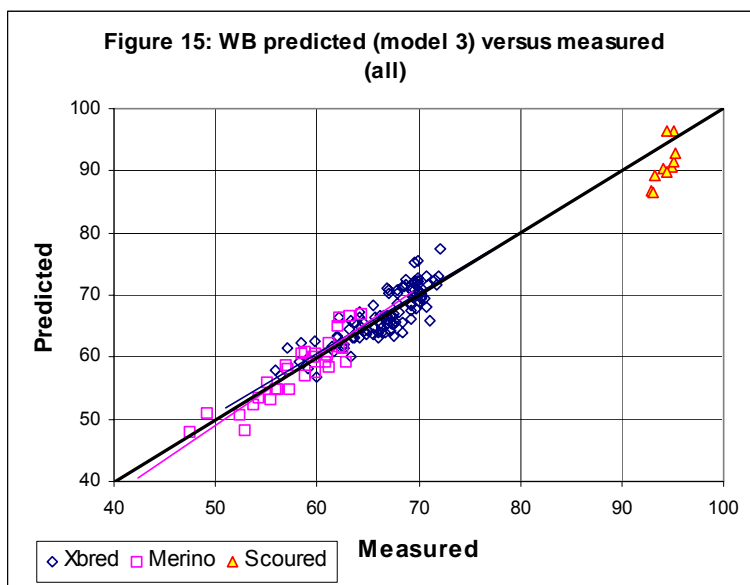
The reasons for the biases were not clear. Nevertheless, if this simple model were used only on merino samples, it could produce a similar level of precision of prediction to previous work, with a standard deviation of the residual errors of 1.7 %. The bias of 3% could be easily corrected. However, it was of concern that as the woolbase levels increase the prediction appears to fail to a progressively greater degree, so that on virtually clean wool, the error was of the order of 25%.

The next stage to be examined was the use the ancillary OFDA2000 greasy measurements together with the X-ray data. Variables which were known to be strongly correlated were transformed to less correlated variables. The total list of OFDA2000 variables explored included: mean fibre diameter (mfd), CV of diameter (cvd), mean curve (cr), CV of curve (cvcr), SmallBlob% (SmIB) and CV SmallBlob% (cvSmIB), LargeBlob% (LrgB) and CV LargeBlob% (cvLrgB), SD of diameter along (sdDA) and CV of sdDA (cvsdDA). Stepwise linear regression produced an equation utilising:

- X-ray attenuation

- Vegetable matter base (VMB)
- cvLrgB, sdDa and cvSmIB

Interestingly, diameter did not play a role in this equation, but 3 different measures of surface roughness and fibre unevenness were included. It is possible that since these parameters tend to be diameter-dependent, MFD was acting as a proxy variable. The overall performance of the prediction was considered moderately acceptable, with an overall standard deviation of differences of 2.5 %, and 2.2 % on the merinos only.

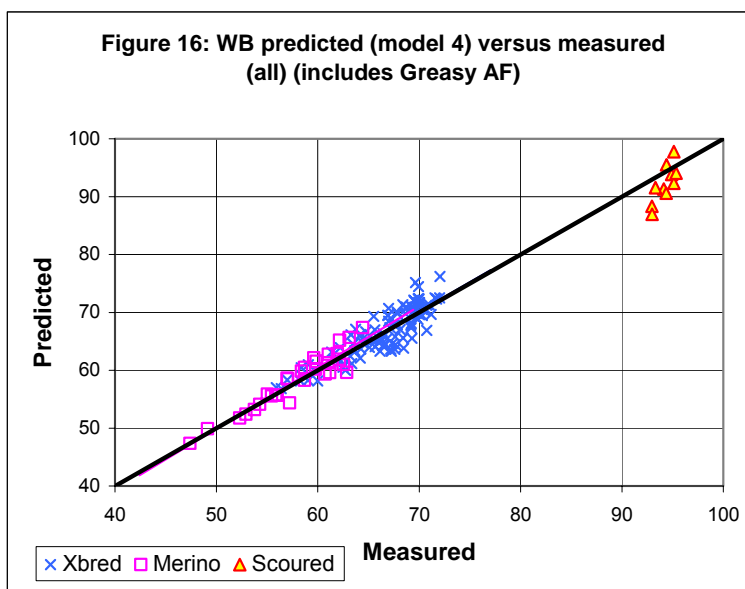


As can be seen, this model was satisfactorily linear over the full range, but the standard deviation of the differences was not as good as desired. The next stage was to add in the greasy airflow measurements. For the purposes of this trial, an existing airflow instrument designed to take 5g specimens (twice the normal mass) was calibrated using standard calibration tops (using a quadratic relationship between flowrate and mean fibre diameter), so that the numbers produced bore some relationship to measured fibre diameter. The intent of these measurements was to give an indicator of the difference between greasy and clean diameters. When compared with clean airflow measurements, the difference assisted significantly in reducing the residuals, but in practice, of course, only the OFDA2000 diameter data would be available (which is calibrated against clean mean fibre diameter), and so the parameter included in the stepwise regression was greasy airflow minus OFDA mean fibre diameter (GAF-mfd).

With GAF-mfd included, the regression equation (Model 4) included the following variables:

- X-ray attenuation
- VMB
- cvLrgB, sdDa and cvSmIB
- (GAF-mfd)

The standard deviation of differences reduced to 2.1 % for the full data set and 1.6 % for the merino samples. As can be seen from Fig 16, this represents a significant improvement, particularly in the merino segment.



It is worthwhile examining the along-fibre parameters that have been included in models 3 and 4. These were:

- | | |
|--------|---|
| cvLrgB | coefficient of variation of the percentage of large blobs on the fibre - normally interpreted as the variability of dirt on the fibre |
| cvSmlB | coefficient of variation of the percentage of small blobs on the fibre - more closely related to the variability of grease or possibly scale edge elevations |
| sdDa | standard deviation of diameter along the fibre - relates to the overall variability of fibre diameter along a fibre segment, which is usually an indicator of environmental effects on fibre growth |

Since all three parameters are potentially indicative of some aspect of fibre surface roughness or diameter variability, there is a degree of correlation between them, and it would be desirable to reduce the number of variables in the model. The relatively poor precision of these variables also contributes significantly to the overall precision available. In an attempt to simplify the model, the analyses were run again using the composite variables TotalBlob (sum of large and small blobs), and cvB (variability of total blobs). This model was less successful in explaining variation, and gave standard deviations of residuals of 2.4% for the full data set and 1.9 % for the merino segment. This suggests that each of these along-fibre parameters does seem to have a distinct effect on the model, although there is no obvious physical explanation for this, other than the fact that they all relate in some way to lack of fibre surface smoothness, which is, of course, largely the result of dirt and grease on the fibre.

A further analysis was undertaken to establish whether fibre bulk (related to resistance to compression) could contribute to the prediction. This was affected by using the composite variable (mfd * curve), which has been successfully utilised to predict bulk (Sumner et al, 2005). However, the analysis indicated that this variable provided no assistance in refining the model.

Model 4 was therefore taken as being the best that could be achieved with the full data set and linear regression.

Analyses of variance

All the measurements were undertaken on replicated subsamples, so that full analyses of variance could be undertaken. However, it should be noted that these were all independent subsamples, so that, for example, subsample A used for X-ray measurements was not the same physical entity as subsample A for greasy airflow, or subsample A for OFDA2000 measurements. This introduced a significant element of uncertainty into the analysis (i.e. the between-subsample component of variance), which was not easily quantified. If the assumption were to be made, for example, that all the measurements on subsample A were undertaken on the same physical sample, then the precision of the predicted values

using the standard combination of uncertainties in model 4 becomes unrealistically high. The analysis below therefore follows a similar logic to that which was used in the earlier work, with the only assumption being that the measurements of woolbase and the predictions of woolbase are totally independent. This is a reasonable assumption under the circumstances.

An examination of the plot for model 4 (Fig 16) suggests that the precision of prediction may be heteroscedastic. This is known to be the case for conventional woolbase measurements, although in this case, the precision normally increases with reducing woolbase level, which is not the trend suggested by this plot. However, this may be because the X-ray instrument is measuring the quantities of impurities rather than the quantities of clean fibre. An analysis has therefore been carried out on a level by level basis:

WB range	Woolbase variance	sd diffs (WB-pred)	Variance differences	Predictive variance	95%CL of prediction	
					single	pair
< 56	0.139	0.611	0.373	0.235	1.34	0.95
56.1 - 60.9	0.102	1.484	2.201	2.099	4.02	2.84
61 - 65.9	0.090	2.444	5.971	5.881	6.72	4.75
66 - 70.9	0.114	2.264	5.128	5.014	6.21	4.39
> 71	0.848	2.764	7.638	6.790	7.22	5.11
all	0.210	2.067	4.272	4.062	5.59	3.95
merino	0.099	1.581	2.501	2.402	4.30	3.04

The last two columns indicate the estimated precision of prediction using measurements on a single subsample and the average of a pair of subsamples respectively.

The results in the first column of this table suggest that the assumptions concerning the precision of the reference method used in the earlier work were not accurate. In that work it was assumed that the precision of the reference method on long-wool samples was $\pm 2\%$, whereas in this analysis the worst precision for the reference method on core samples (of greasy wool) was $\pm 0.7\%$. On just the merino samples, the precision was $\pm 0.6\%$. In consequence the earlier analyses probably slightly underestimated the precision of prediction. Using a corrected estimate for merino wool, the precision of prediction should have been closer to $\pm 5\%$ for long wool, and $\pm 4\%$ for core samples. Unfortunately, this new work indicated that on a single subsample basis, the precision had not improved even with the additional complexity of the prediction process. The system, did, however, appear to be much more linear over a wider range of wools types and diameters. The calculations indicate that the precision could be improved to approximately $\pm 3\%$ by using the mean of 2 subsamples.

Alternative statistical approaches

1. Partial least squares

Since many of the variables used are correlated, it seemed logical to explore whether these could be reduced using partial least squares regression. An additional variable was included - sample type (merino, crossbred or scoured). This was moderately successful in that the standard deviation of differences became 1.65% on the overall data set. This should be compared with the figure in bold (2.067 %) in the table above. The slight disadvantage to this method is the marginally complicated formulae required.

2. Generalised regression model

With the use of the additional variable, this again produced a satisfactory outcome, with a standard deviation of differences across the entire data set of 1.71 %.

3. Neural networks

The use of neural networks was slightly less successful than the two approaches noted above, with a standard deviation of differences of the test set (the technique uses training, validation and test sets selected at random from the total data set) of between 1.8% and 2.1%. Neural network analysis requires

large data sets in order for the outcomes to be optimal, and this set would certainly be on the margins of acceptability, which could be a factor in the limited success. The implementation of NN is not trivial, and in view of the slightly less than optimum performance, it was not considered further.

4. Discrete regression treatments

Whilst it is clearly desirable to have a single calibration covering all types of greasy wool, one alternative would be to have separate calibrations for merino and crossbred wools (in other words, less than and greater than 26 μm). This is, in effect, what the generalised regression model is doing by using the 'type' variable. An analysis of this approach was undertaken, based on the type being preselected, and there seem to be some advantages with this option in terms of performance.

Using stepwise linear regression, using individual subsample results rather than sample means, two equations were obtained (models 6A and 6B) which utilised the following variables:

Merino:

- X-ray attenuation
- VMB
- (GAF-mfd)
- lrgB

Xbred:

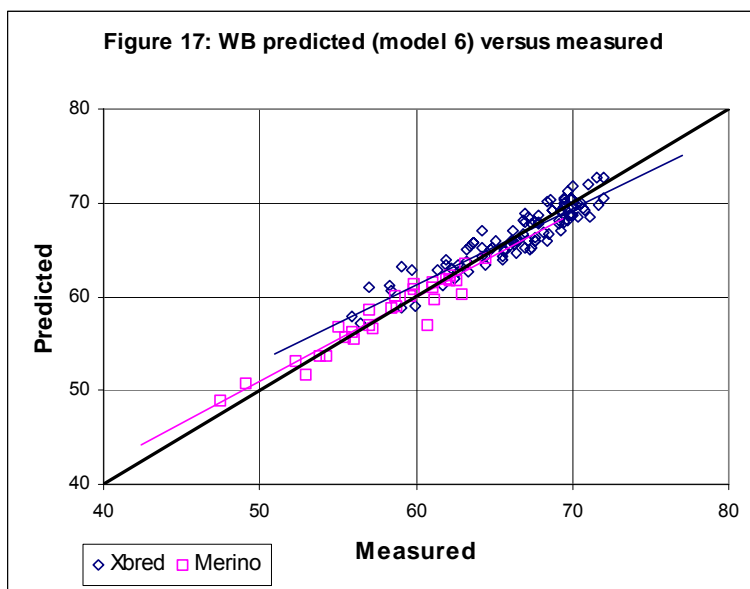
- X-ray attenuation
- Curve
- VMB
- (GAF-mfd)
- Cvd
- cvlrgB and cvsmIB

Neither of these equations were suitable for scoured wool, but this is not considered to be a potential application for the instrument.

The analyses of variance yielded the following results, and a plot of the predictive results on sample averages is shown in Fig 17:

Wool type	Woolbase variance	sd diffs (WB-pred)	Variance differences	Predictive variance	95%CL of prediction	
					single	pair
merino	0.099	1.224	1.499	1.400	3.28	2.32
Xbred	0.132	1.436	2.061	1.929	3.85	2.72

Clearly this option appeared to have significant benefits in terms of precision of prediction. The figures shown in the preceding table indicated that the required outcomes had been achieved using single samples in the case of merinos. It was encouraging to see that the form of the equation for merinos was similar to model 0, except that the diameter term has been replaced with a combination of (GAF-mfd) and LrgBlob. Both equations validated satisfactorily on sample means as shown in Fig 17.



The GAF variable

In virtually all the analyses, the (GAF-mfd) variable played a statistically significant part, albeit at the 3rd level. It should be remembered that airflow actually measures specific surface area rather than fibre diameter, and it is possible that the along-fibre OFDA parameters and the (GAF-mfd) variable are actually estimating similar aspects (in the later commercial prototype, in which the same specimens were being measured for all parameters, it was found that GAF played less part, and this may be an explanation). For merino, values ranged from 3 to 12 μm , corresponding to an equivalent woolbase effect of 1.5 to 6.5%. Whilst the range for crossbred wools was slightly wider (1 to 16 μm), the regression coefficient was somewhat less, and the equivalent effect on woolbase was of the order of 0 to 5%. This would suggest that an accuracy of 1 unit (1 μm) would probably be adequate. However, it was noted that the variable was a difference between two independent measurements, and the OFDA mfd itself was subject to an imprecision of at least this order of magnitude (expressed in 95% confidence limit terminology). Taken together, this suggests that at a typical merino mean fibre diameter of 20 μm (clean), a precision would be required from the greasy airflow measurement of approximately $\pm 1 \mu\text{m}$ - i.e. approximately 5% of the measurement value. A further complication was that the airflow measurement is the product of a preset pressure gradient and a flowmeter reading.

It was suggested by GNS that a measurement of packed fibre density might be simpler, and it was agreed that it should be tested as an alternative approach. 20 samples covering the entire range of woolbases were selected for a 'proof of principle' test. Unfortunately, analysis of this limited set of data indicated that this alternative measure would not substitute for (GAF-mfd). Indeed, in various stepwise regression analyses undertaken with and without some of the variables, this result played no useful part.

Conclusions to Stage 2

The use of a more complex measurement and predictive process appeared to offer the opportunity to develop a calibration that covered a wide range of wools. However, in the merino range, this 'unified' model did not give significant gains in terms of predictive precision or accuracy. When the wool types were investigated separately, and separate calibrations were used, satisfactorily precise predictions appeared possible.

It therefore seemed that the next logical step was for a new prototype device to be constructed which included greasy airflow measurement, with the ability to combine the outputs from this, the X-ray instrument, and some of the OFDA2000 parameters.

COMMERCIAL PROTOTYPE

DESCRIPTION

The Electronic Wool Yield (EWY) instrument (Fig 18) consists of five components:

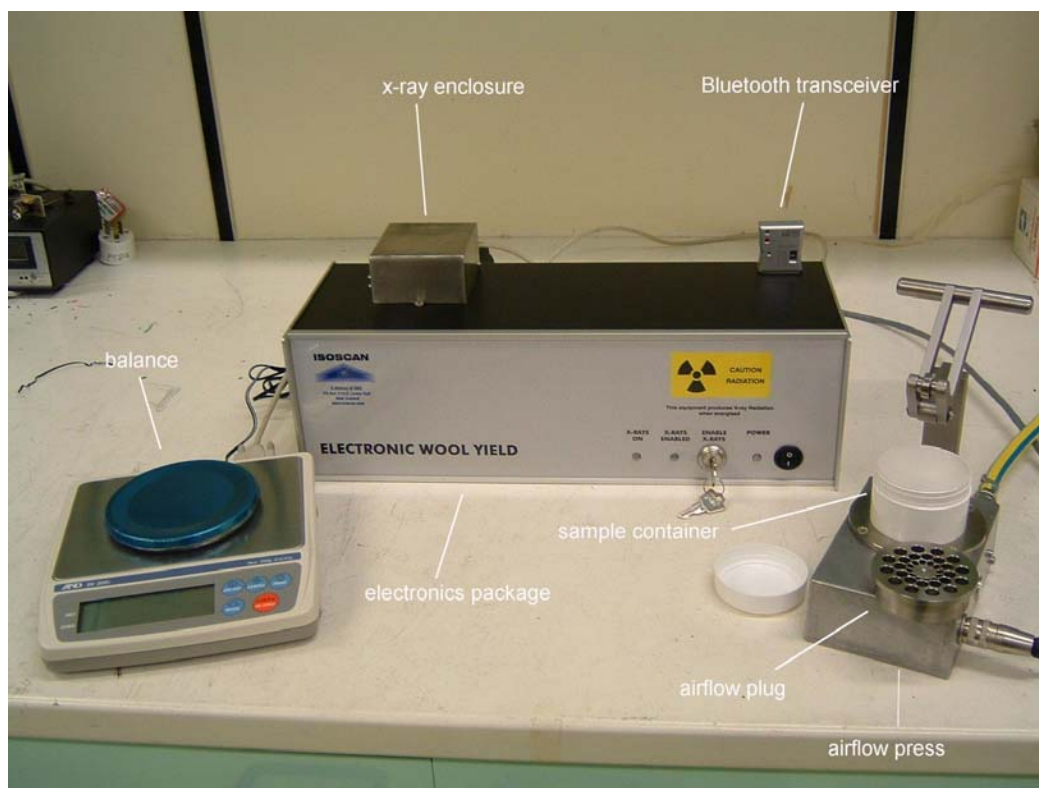
- 21 litre domestic air compressor
- air flow gauge
- electronics package – this has a microprocessor and incorporates the x-ray attenuation gauge; it is mains-powered, and also has serial communication input by cable
- digital balance – mains-powered
- pocket PC (PPC) – battery or cradle (mains) powered; the PPC is used as an intelligent remote control.

Additionally:

- An OFDA2000 with modified communication software

In the instrument under test, the components were tied together with a Bluetooth wireless network.

Figure 18: The Electronic Wool Yield Instrument



EWY uses a low power x-ray tube that produces a continuous range in x-ray energy that peaks at about 10 keV. Unlike conventional x-ray tubes, this tube does not have a filament; instead, electrons are produced by field emission from a carbon nanotube cathode. It has the potential for much greater lifetime, provided it is powered up and used appropriately. In EWY, the tube is being run at half power for life extension. Unfortunately, because this was cutting-edge technology at the time, and the original tube was damaged in transit, and a replacement took some months to arrive, the whole project was delayed at the build stage.

The transmitted x-rays generate a tiny current in a detector, approximately 10 x 20mm in area. The detector has a slight amount of leakage current, present in the absence of x-rays. This is the 'dark' current, I_d , due to thermal noise, and this is subtracted from the current detected with transmitted x-rays.

I_d is measured during execution of the **Setup/X-Rays/Null** procedure for determining the x-ray beam without a sample I_0 . These measurements are carried out on initialisation, prior to running samples. They should be periodically checked. Because of possible drifts in the x-ray tube output, measured detector currents are referenced to the ratio of the measured i_m , and set anode i_s , currents:

$$\phi_0 = (I_0 - I_d) \frac{i_s}{i_m}$$

Wool packed in the sample container will not be uniform in x-ray attenuation. Therefore, the x-ray beam that is used is smaller in cross section than the container, and this is rotated through the off-centre beam so that most of sample is scanned separately. The period of revolution is 7 seconds, during which time about n=700 measurements of I_i are taken (every 10 msec). These are averaged:

$$X = -\frac{1}{n\theta} \sum \ln\left(\frac{\phi_i}{\phi_0}\right)$$

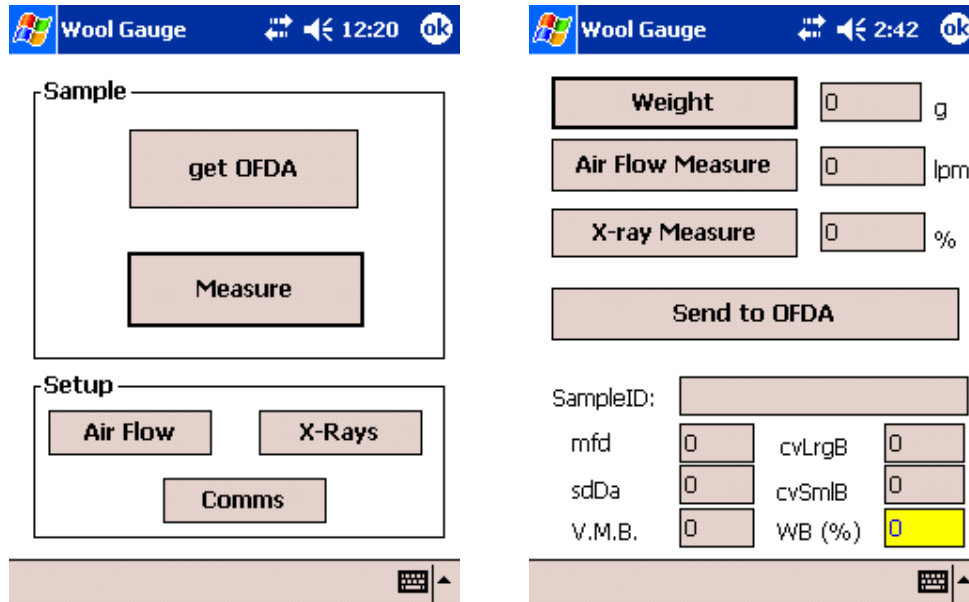
The areal density θ is calculated from the weight of wool, w , and the cross sectional area of the sample container, A :

$$\theta = \frac{W}{A}$$

It is assumed that θ is a constant over the cross section of the sample: this is an approximation. The estimate X is used in the calculation of wool yield. The averaged percentage transmission $T = 100 \cdot X$ is reported at the conclusion of the x-ray measurement.

The whole instrument is controlled by an handheld PDA with touch-sensitive screen running GNS software. Typical screenshots are shown in Fig 19. In a production instrument, this interface would be simplified further.

Figure 19: Typical control PDA screens



In operation, the operator would measure the staple on the OFDA2000 in the normal manner whilst a 2nd operator would visually assess the sample against typical VM photo-references, load the pre-tared sample canister with approximately 30g of wool, weight it, place it in the airflow device, and then finally transfer the canister to the X-ray unit. The data is then sent back to the OFDA for display alongside the diameter data. With practice, the whole operation can be completed in the required cycle time.

Calculation of Wool Base

The wool base is calculated from the empirical equation:

$$WB = w_0 + w_1X + w_2VMB + w_3cvLrgB + w_4sdDa + w_5cvSmlB + w_6(GAF - mfd)$$

The coefficients w_0 to w_6 are determined by calibration using known samples. The variables X and GAF (greasy air flow = Q_{sw}) are measured using EWY. The other variables are measurements made by the OFDA instrument, and are read by the EWY PPC. These are:

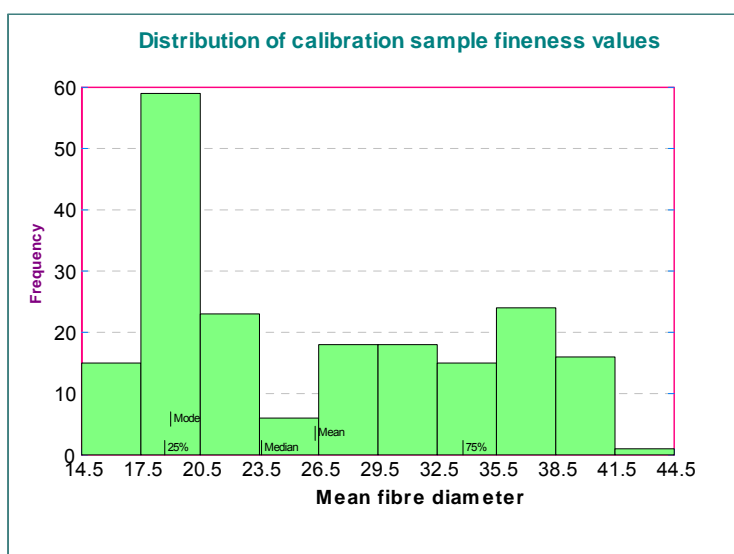
variable	explanation
VMB	Vegetable matter base
cvLrgB	Coefficient of variation of large blob %
sdDA	Standard deviation of diameter along fibre
cvSmlB	Coefficient of variation of small blob %
GAF	Greasy airflow
mfd	Mean fibre diameter

CALIBRATION

195 cores samples were selected from New Zealand fleece wools and used for calibration, comprising 96 greasy fine wool samples, 84 greasy crossbred samples and 15 scoured wool samples (including 2 merino samples). Each of the samples was measured twice for woolbase and vegetable matter base (ie 4 subsamples in total) in accordance with IWTO-19. From the replication, it was estimated that the average precision (95%CL) for each reference woolbase measurement was $\pm 0.5\%$. The scoured oven-dry mass percent was also used to calculate the washing yield at 16%, which was used for a subsidiary calibration.

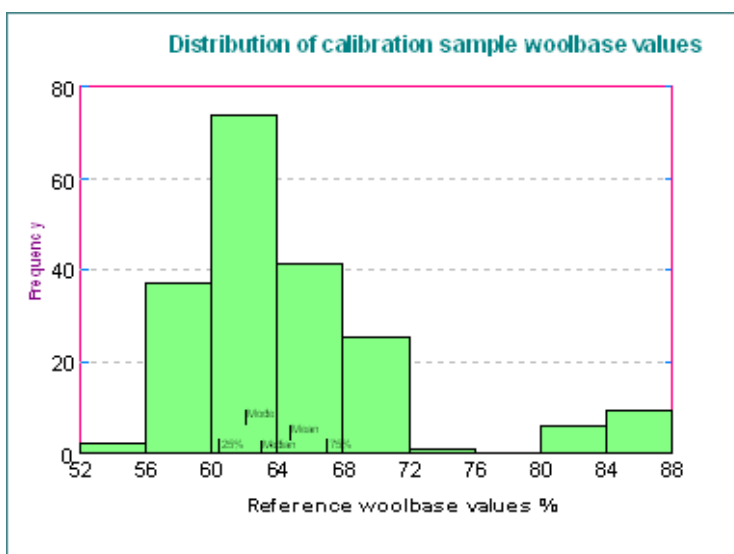
The distribution of fineness values in this population is illustrated in Fig 20:

Figure 20: Calibration samples – distribution of mean fibre diameter values



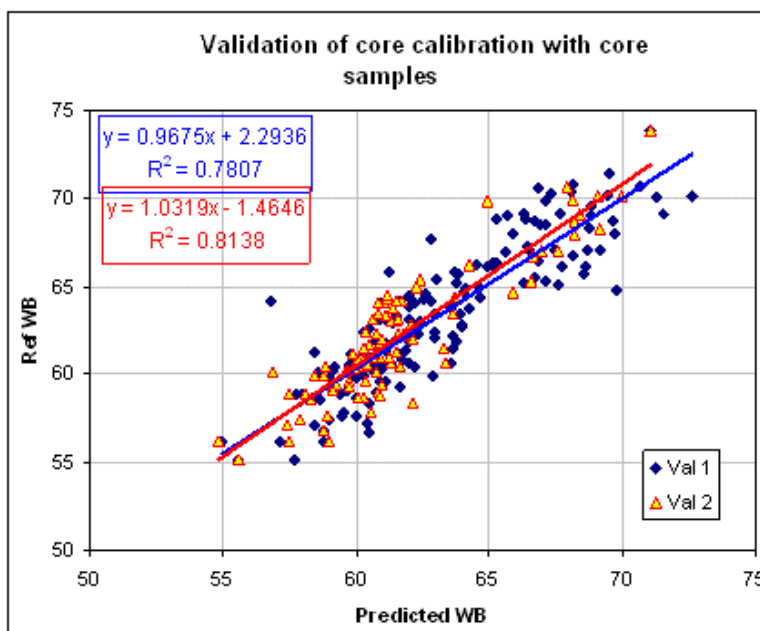
The samples encompassed vegetable matter bases from 0 to 3%, and woolbase values from 55 to 74%, as indicated in Fig 21.

Figure 21: Distribution of woolbase values in calibration set



In the final version of the core calibration, only the greasy samples were included, as it was clear, confirming earlier trials with the early prototypes, that the scoured samples comprised a different population for the purposes of prediction. The results of 2 sets of validation, using cores, are illustrated in Fig. 22 below:

Figure 22: Core calibration results



It can be seen that the level of performance was not unduly dissimilar to that illustrated with the earlier prototypes. This was confirmed when the precision values were estimated from this data. The 95%CL of the repeatability of the predicted individual woolbase values was $\pm 1.6\%$, whereas the estimated 95%CL of the predicted woolbase values (based on a single measurement) was $\pm 3.5\%$, which is very similar to the values reported for the earlier prototypes. The difference between these two values relates to the imprecision of the predictive model. It was therefore concluded that the commercial prototype was producing equivalent performance to the earlier "breadboard" model. It was also recognised that the GAF measurement was only playing a minor part in the calibration and could probably be eliminated from

future versions of the instrument. However, for purposes of this evaluation, the measurement continued to be included.

VALIDATION WITH MIDSIDE SAMPLES

In the original proposal for the validation phase, this was to have taken place in two stages – the first with New Zealand samples and the second using a wide range of Australian samples. Due to various delays, the two stages were eventually combined. An initial set of NZ validation samples produced very poor results, which at the time was attributed to problems with the laboratory scouring and drying system. (The system that was used for this work was a rebuilt laboratory tape scour that had been in use as part of a reliable fleece testing process some years ago, but had been mothballed in the interim. After some refinement of the procedures the kit was brought up to suitable specification). Unfortunately, at the time the trials were carried out, it proved difficult to replace these samples, and therefore a smaller NZ sample set had to be incorporated in the final evaluation. All measurements were carried out in duplicate to allow for precision data to be obtained.

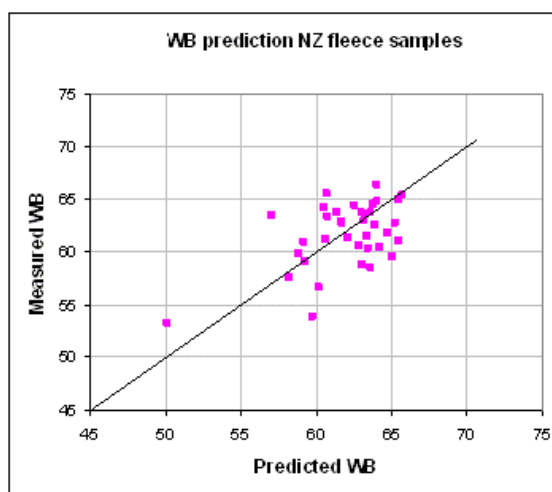
The final evaluation was carried out with 36 New Zealand samples and 361 Australian samples before the project was halted. The measured Australian samples included 24 from Queensland, 82 from West Australia, 80 from New South Wales, 46 from Tasmania and 129 from Victoria, therefore reasonably representing a cross-section of wools. All samples were of fleece wool.

There had been some concern expressed (during peer review of the trial proposal) that traditionally fleece testing yield is reported as "16% washing yield", yet this project was focussed on woolbase. The reason for this had been that reference woolbase measurements were available for the core calibration, and that washing yields or any other yields could be recalculated if necessary. With this in mind, residuals measurements were also undertaken on the scoured fleece samples so that woolbase results could be estimated. Alongside this work, the calibration had also been recalculated for washing yield and this had also been used in examining the data. The effect of using washing yield was to marginally diminish the precision, but, when the other issues were taken into account, this became a secondary consideration. Hence, whilst the data remains available, washing yield will not be considered further in this report.

Validation results on NZ fleece samples

The results on the limited number of available NZ samples were initially encouraging. Satisfactory replicate repeatability was obtained for both the rapid yield prediction and the scoured woolbase values (95%CL of 1.7% and 2.0% respectively). The predictive accuracy also appeared satisfactory as far as could be ascertained from the limited range of samples, as indicated in Fig 23.

Figure 23: Validation on NZ fleece samples



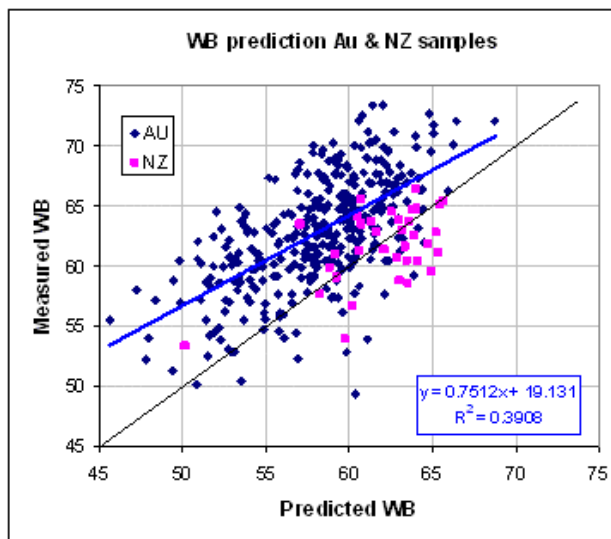
Clearly the limited range of woolbase values amongst these samples was restrictive, but the results were considered sufficiently acceptable to allow continuation with the Australian samples, especially given the substantial delays already experienced and the pressure to meet revised milestones agreed with the

funding agencies. The decision was partly based on the appearance of the above plot, where the scatter was judged satisfactory compared with Fig 22. However, it was overlooked that each individual point in Fig 23 was the mean of replicate results. Consequently, when the variances were subsequently calculated it was estimated that the precision of prediction of a single measurement was somewhat higher than expected at $\pm 5.5\%$. This was unfortunately not immediately obvious at the time.

Validation results on Australian fleece samples

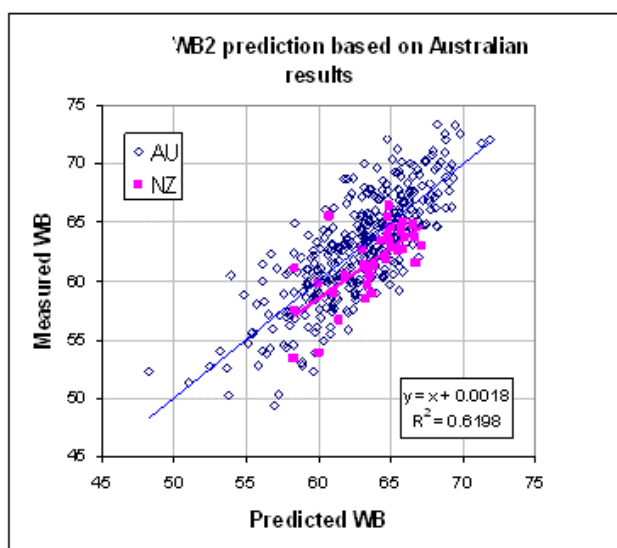
The results on the Australian samples continued to appear very reproducible between subsamples and therefore the measurements proceeded apace. Unfortunately, for various reasons, they were not plotted until approximately $\frac{3}{4}$ had been processed. The results are shown in Fig 24.

Figure 24: Validation results on Australian & New Zealand Fleece samples



Clearly there was something quite different about these samples and the work was halted. Various attempts were made to "calibrate out" the problem using the Australian results. Fig 25 illustrates the comparison using a calibration based solely on the Australian data

Figure 25: Verification using an "Australian" calibration



Whilst this considerably improved the relationship, and showed only a relatively small bias on the NZ samples, the precision is still relatively poor. The estimated overall precision of prediction of a single

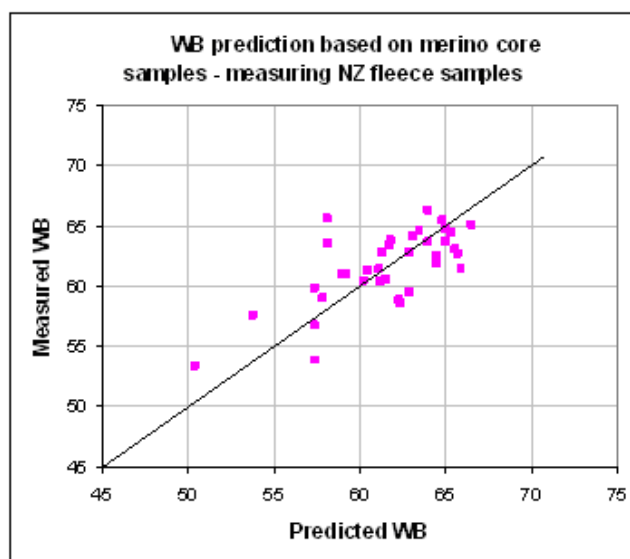
measurement on this calibration is $\pm 5.0\%$. An analysis of variance showed both State and Property to be significant factors. By correcting for the State (ie overall geographic area), the 95%CL of prediction could be reduced to $\pm 4.5\%$. Correcting by Property reduced the precision to $\pm 4.2\%$. Sex and age of the animals showed no detectable effect.

These observations tend to suggest that the instrument is more sensitive to the nature of the mineral components of the dirt than was originally anticipated.

Further analyses

Earlier work has suggested that different calibrations for merino wools and crossbred wools might be appropriate. The core sample dataset was therefore reduced to merino-only samples and a new calibration produced, which was then used on the NZ merino fleece samples. This produced an improved validation, as shown in Fig 26.

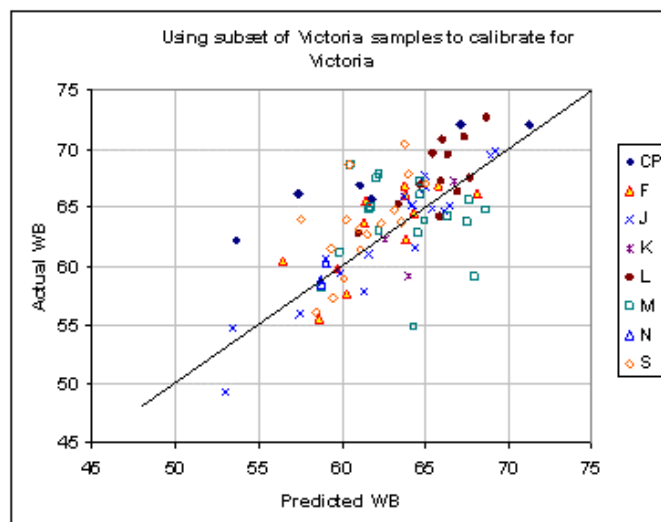
Figure 26: Validations using merino-only calibration data



Comparison with Fig 23 suggested that this represented a significantly improved validation. Analyses of variance indicated that the precision of prediction was of the order of $\pm 3.7\%$ on this data set, which is much closer to the performance observed on cores. Performance on the Australian midside samples was, however, poor.

A subset of the Victorian midside samples was selected, comprising a random selection from each of the 8 properties, totalling 40 of the available 129 tested samples. This set of 40 was used to develop a calibration, which was then applied to the remaining 89 results. Fig 27 shows that whilst the overall precision of prediction was poor ($\pm 6\%$), two properties in particular (CP and M) were responsible for most of the extreme values. If these two were excluded, the precision of prediction reduced to $\pm 4.5\%$, confirming that local calibrations could help to resolve the issue.

Figure 27: Attempted verification using a subset of Victorian fleece samples for calibration



CONCLUSIONS

1. Compared with the earlier prototypes, the commercial prototype gave better repeatability, and predictive performance similar to expectation, on core samples and on NZ midside samples when calibrated with NZ merino cores.
2. Performance on cores of both crossbred and merino types was also similar to expectation when using a single calibration based on NZ core samples. The estimated 95% confidence limit of $\pm 3.7\%$ on a single midside sample was similar but slightly higher than the published precision for conventional midside testing ($\pm 3.0\%$ within one laboratory). It was considered probable that the between-instrument performance should be better than the between-laboratory performance of fleece testing laboratories, such that the overall objective of providing similar or better total precision to conventional midside testing could still be achievable.
3. Performance on Australian samples, using a calibration based on NZ cores, whilst similarly repeatable, was of much poorer predictive power. On this basis, the instrument could not be regarded as ideally robust in its present operating mode. However, analyses of the results by source of sample (ie Property, or location), indicated that the performance would, not unreasonably, be improved if specific calibrations were developed for different locations. This conclusion implies that precision within the NZ environment might also feasibly be improved, since sample source had never been taken into account within the NZ set.
4. The GAF measurement would almost certainly be eliminated from any future instrument build, and GNS have suggested a further option to investigate whether an improved detector configuration might add additional physical data that could be used to mitigate the location (i.e. soil type) issue. This would, however, involve an additional research project.

Whilst the technical issues are now understood, unfortunately the estimated costs for manufacture of commercial instruments in this configuration are almost twice the original estimates that were made at the commencement of the project, and when taken together with the R & D amortisation, total costs are considered excessive in terms of the potential returns in the current market. With a possible need to also develop local calibrations, it was not considered that the instrument was currently commercially viable. However, should the market conditions improve, this conclusion may change.

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