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Prediction of Clean Mohair, Fiber Diameter, Vegetable Matter, and Medullated Fiber with Near-Infrared Spectroscopy

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ABSTRACT: Four experiments were conducted in three separate years to test the utility of near-infrared spectroscopy (NIRS) to predict the clean mohair content of Angora goat fleece. Mohair fleece samples were obtained each year from yearling billies at the conclusion of the Angora Goat Performance Test conducted at the Texas A&M University Research Station, Sonora. In Exp. 1 (n = 293) and Exp. 2 (n = 256), fleeces were scanned with a Pacific Scientific (Silver Spring, MD) near-infrared spectrometer fitted with a fiber-optic probe, and calibrations were developed for clean mohair content. In Exp. 3, 59 mohair fleeces collected at the Texas A&M Research Station in San Angelo were sampled four times each. Each sample was scanned with the same spectrometer in reflectance mode fitted with a transport mechanism. This mechanism allowed the instrument to scan a 15-cm² segment of the fleece sample. Conventional procedures to determine reference values for mohair yield, vegetable matter content, fiber diameter, and percentage of medullated and kemp fibers were conducted. Prediction equations were developed that related NIR spectra to reference values for yield and diameter parameters and were used to predict mohair characteristics for each fleece sample. The pre-

dicted and reference values were subjected to a simple analysis of variance to determine variation within and across samples. In Exp. 1, mohair base was related to NIR spectra with $R^2 = .46$ and standard error of calibration (SEC) = 2.84%. In Exp. 2, similar repeatability errors for mohair base could be obtained for both reference- and NIRS-derived values. Fiber diameter and medullated fibers were poorly related to NIR spectra. When samples were scanned using the transport mechanism (Exp. 3), R^2 and SEC were .82 and 1.19% for mohair base and .93 and .98 μm for fiber diameter, respectively. The CV for mohair base and diameter were 1.0 and 1.4%, whereas those for predicted mohair base and diameter were 1.4 and 3.4%, respectively. The increased variation within samples for predicted values represents sampling error and lack of fit between NIRS and the laboratory determined values. When the samples from Exp. 1 and 2 were rescanned with the NIRS transport (Exp. 4), R^2 and SEC were .79 and 2.03% for mohair base and .52 and 3.49 μm for fiber diameter. The fiber optic probe would facilitate real-time analysis on the shearing floor, but our data indicate that the spectral limitations so far are too severe. A large sample device such as the transport gave excellent results for predicting mohair base and fiber diameter.

Key Words: Mohair, Clean Yield, Fibers, Spectroscopy

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Introduction

Clean yield of wool, mohair, and cashmere fiber is an economically important trait for both producers and buyers. Because yield of clean material ranges from 30 to 95% in wool, mohair, and cashmere, accurate evaluation is imperative. Standard methods for de-

termining yield are lengthy and time-consuming. They involve washing, drying, and weighing steps followed by three further analyses for residual grease, ash, and vegetable content of the washed fibers. Sabbagh and Larsen (1978) announced that wool yields could be determined accurately in less than 10 min using near-infrared spectroscopy (NIRS) analysis. However, subsequent research showed that the NIRS technique could not match the accuracy ascribed to the standard method ($\pm 1\%$) when applied to greasy wool (Connell and Brown, 1978; Connell, 1983). Further, NIRS calibrations for clean wool yield were somewhat specific to geographical location, and, for some areas, large errors in yield prediction were not attributable to vegetable content. This discouraging result changed the focus for researchers

¹Mention of a trade name or brand does not imply endorsement by the USDA to the exclusion of other suitable brands.

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using NIRS analysis, and emphasis was shifted to developing NIRS methods for determining residual grease and moisture contents of scoured wool (Ranford et al., 1985; Hammersley et al., 1992) and medullation (Ranford et al., 1990). However, rapid and reliable methods are needed to evaluate fiber quality at the site of shearing for accurate classing and packaging. The objectives of our experiments were to compare the relative precision of standard laboratory analyses and NIRS predictions of yield, fiber diameter, vegetable matter, and medullated fibers (med and kemp) of mohair and to develop feasible NIRS procedures for quality analysis.

Materials and Methods

Experiment 1. Fleece samples were obtained from yearling billies at the conclusion of the 1990 Angora Goat Performance Test conducted at the Texas A&M University Agricultural Research Station at Sonora. Fleeces were sampled with a fleece coring apparatus similar to the one described by Johnson and Larsen (1978). Mohair base (a measure of clean yield) was determined using a modification of American Society of Testing and Materials (ASTM) Test Method D 584 on duplicate subsamples. In the modified method, washed and dried fleece samples were pooled for subsequent determination of residual grease, ash, and vegetable matter because all animals in the study had been penned together and consumed the same diet.

Interactance spectra in the NIR region were obtained with a Pacific Scientific Model 6500 spectrometer (Silver Spring, MD) equipped with a fiber optic probe. The fiber optic mechanism limited the range of spectra that could be obtained in the NIR region from 1,100 to 1,700 nm. An aliquot remaining from laboratory subsampling (25 g) was scanned by placing the fiber optic probe directly on the sample contained in a polyethylene bag with a solid surface on the back side. Light was transmitted from a white light source through one set of fibers and into the fleece sample, and the interactance light was returned through the remaining fibers to a set of lead sulfide detectors. A standard reference made of Teflon was scanned after each 10-sample interval to correct for changing conditions, such as instrument drift.

Software developed by Infrasoft International (Port Matilda, PA) and described by Shenk and Westerhaus (1991) was used to select representative samples and to develop and validate calibration equations. A subset of 124 samples was selected based on spectral variance using the computer program SUBSET with the selection criteria set for number of samples (120). SUBSET computes a set of principal components based on the spectral characteristics and selects samples to represent neighborhoods set in multidimensional space. Prediction equations were developed using the selected samples, and the remaining 166 samples were predicted as a pseudo-validation set. This was not a true validation because the remaining samples were origi-

nally part of a parent set from which the selected samples were withdrawn.

The method of fitting spectra to laboratory values was partial least squares, a procedure that combines principal component analysis of the spectra with correlation analysis with laboratory values (Shenk and Westerhaus, 1991). The spectra were normalized for light scatter with the DETREND method described by Barnes et al. (1989) for yield and mohair base, but not for fiber diameter. The software package contained provisions to eliminate outliers during the validation procedure if the *t*-test between laboratory analysis and the value of sample predicted from the spectrum was greater than a predetermined value, which we set at 1.75.

Experiment 2. To further test and develop the technique for determination of yield and other fleece parameters, 198 fleeces were obtained in 1991 from a study similar to that described in Exp. 1 and subjected to spectral analysis in both the visible (700 to 1,100 nm) and the NIR (1,100 to 1,700 nm) range, again using the fiber optic probe. This time, samples were placed in 8 × 8-cm watch dishes and covered with plastic wrap for scanning. Fifty fleeces were then selected based on spectral characteristics using the computer program SUBSET for detailed spectral and chemical analyses to determine the relative errors of laboratory and NIRS analyses. Three subsamples of each of the 50 fleeces were analyzed for mohair base and vegetable matter base (ASTM D584). In addition, mean fiber diameter was determined on 1,000 per fibers sample using a Peyer Texlab (Spartanburg, SC) FDA 200 System (Lynch and Michie, 1976). Medullated fibers, including both med and kemp fibers, were determined with a projection microscope using ASTM Test Method D2968.

Four additional interactance spectra were obtained on one of the subsamples for each of the selected fleeces, each spectrum representing a different sampling area. Both NIR and visible spectra were obtained from each location. Calibration equations were developed for mohair base, vegetable matter base, fiber diameter, and medullated fibers (med and kemp).

Experiment 3. Four samples each from 59 fleeces collected at the Texas A&M Research Station, San Angelo, were subjected to monochromatic light in both the visible and the near-infrared region using the same spectrometer as in Exp. 1 and 2, but fitted with a transport mechanism for collecting reflectance data over a large sample size (4.5 × 20 cm; scanned surface was approximately 2.5 × 15 cm). Thirty-two scans from 400 to 2,500 nm at 2-nm intervals were collected and averaged for each of the four samples of each fleece. The samples were then subjected to conventional procedures to determine mohair yield, vegetable matter content, fiber diameter, and percentage of med and kemp fibers. Prediction equations were developed by regressing reference laboratory data to NIR spectra. The same samples were predicted with the equations, and the results, along with the conventional laboratory values, were

subjected to a simple analysis of variance to partition the relative sources of variation within and across samples.

Experiment 4. Samples from Exp. 1 ($n = 245$) and 2 ($n = 239$) were rescanned in the visible and NIR regions with the spectrometer fitted with a transport similar to that used in Exp. 3. The program SUBSET was used to select a calibration sample set from each experiment and from the combined experiments. All samples from each year were predicted using the within-experiment and combined experiment equations. The combined predictions were analyzed for discrimination between data sets in terms of bias and random error.

Results

Experiment 1. Spectral interactance occurs when monochromatic light enters a sample, the light interacts with the molecular and physical structure of the sample, and part of the light is collected and analyzed for intensity. It differs from both reflectance and transmission spectroscopy. The interactance spectra for greasy and washed (scoured) mohair are shown in Figure 1. Two rather distinct differences may be noted. In the scoured fleece, two rather dominant peaks occur at 1,196 and 1,504 nm. These correspond to the second and third overtone of N-H stretch from protein. In the greasy fleece, overall absorption was much greater due to carryover of the dark color from the visible. Also, a change in the shape of the two dominant peaks occurred. They may be observed as shoulders on the dominant peaks, one at 1,212 nm to the right of the smaller peak and the other a broad shoulder centered at 1,422 nm to the left of the 1,504-nm peak. Both of these are observed when fatty substances are scanned and reflect the C-H stretch and bend combination. It should be

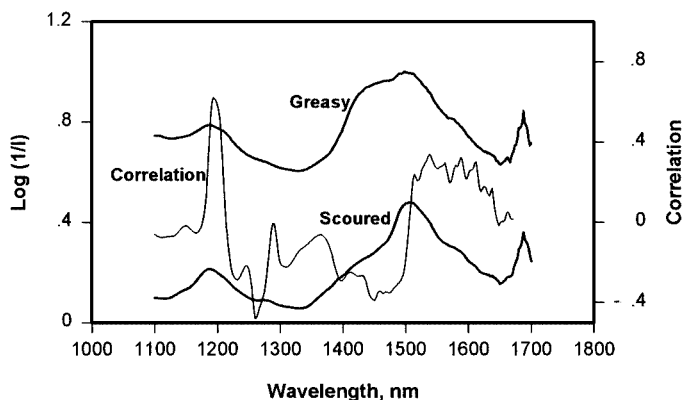


Figure 1. Near-infrared spectra, $\log(1/\text{interactance } [I])$ of clean and greasy mohair using fiber optics. Correlation is between each spectral data point and percentage of mohair base.

noted that all spectral information is repeated from its origin in the far infrared to the visible.

A correlation plot of mohair base with the first derivative of the spectra of the data set ($n = 243$) reflects the predictive information centered around 1,200 nm. It was surprising that the larger, more dominant peak did not correlate with mohair base, but observation revealed more variation existed at the 1,200-nm band. Correlation depends on range in both the dependent and independent data. With this data set, we were only trying to determine the feasibility of the methodology, and these procedures seemed to be adequate.

When the available spectrum (1,100 to 1,700 nm) was used for calibration using partial least squares procedures (Shenk and Westerhaus, 1991), the standard errors of calibration (**SEC**) for mohair base, fiber

Table 1. Calibration and validation statistics for analysis of greasy mohair with near-infrared spectroscopy (Exp. 1)

Item	Mohair base, %	Fiber diameter, μm	Medullated fibers, % ^a
Calibration samples ^b			
Mean ^c	64.8 \pm 4.05	42.4 \pm 5.01	3.0 \pm 3.45
SEC ^d	2.84	4.78	2.15
R ²	.46	.10	.05
Validation samples ^e			
Reference mean ^c	63.7 \pm 4.10	43.5 \pm 5.14	3.80 \pm 5.90
Predicted mean ^c	65.1 \pm 2.01	43.7 \pm 1.50	2.28 \pm .55
Bias ^f	-1.44	-0.2	-1.52
SEV(C) ^g	3.23	5.21	5.92
R ²	.40	.01	0
Slope	1.29	.35	.12

^aSum of med and kemp fibers.

^bCalibration statistics on 124 samples selected based on H-statistic (Shenk and Westerhaus, 1991). Calibration procedures were partial least squares.

^cMean \pm standard deviation of the data set.

^dStandard error of calibration.

^eValidation statistics based on 166 samples not selected because they were already represented by the selected samples for calibration.

^fBias = averaged difference of means by reference and predicted methods.

^gSEV(C) = standard error of validation corrected for bias.

Table 2. Root mean square errors of repeatability for mohair base determined with either reference or near-infrared spectroscopy (NIRS) methods (Exp. 2)

Analysis	NIRS				
	Reference		Error		
	n	Error	n	NIR ^a	VIS ^b
Mohair base, %	3	1.42	4	1.66	4.20
Fiber diameter, μm	—	—	4	2.47	6.03

^aNear-infrared region (1,100 to 1,700 nm).

^bVisible region (700 to 1,100 nm).

diameter, and medullated fibers were 2.84%, 4.78 μm , and 2.15%, respectively, and the coefficients of determination (R^2) were .46, .10, and .05 (Table 1). Validation statistics were reasonable for mohair base with a small bias (-1.44%) calculated as the difference between the laboratory and NIRS predicted means. The standard error of validation corrected for bias, SEV(C), was 3.23% and R^2 was .40. Because of the extremely low R^2 , we judged there was no relationship between NIR spectra and fiber diameter and medullated fibers. Fiber diameter has no chemical basis on which to base a spectral relationship; with forages, however, particle size can be predicted in reflectance mode because larger particles absorb more light (Norris et al., 1989). However, this is apparently not true with interactance. The proportion of medullated fibers was generally very small and probably has no spectral signature.

Experiment 2. In this experiment, we attempted to evaluate variation due to laboratory analysis and to spectral interactance. Residual standard errors for both laboratory determinations and NIRS predictions in both NIR and visible are shown in Table 2. Analytical error, represented by within-sample error, for triplicate analyses on the 50 selected samples for mohair base was 1.42%. Some quality control protocols specify less than 1.0% difference within and between laboratories. We believe that our analytical error is typical of actual industrial experience. Analytical error for NIRS analysis of mohair base was represented by repeated scan error on the same sample and averaged 1.66% in the NIR and 4.20% in the visible region (Table 2). The value in NIR region compares favorably with that for reference laboratory values (1.66 vs 1.42%). One limitation of a prediction procedure such as NIRS is that the errors of analysis cannot be reduced below the laboratory analytical error. Smaller errors for NIRS analysis of fiber diameter were also obtained in the NIR region over the visible, but laboratory reference errors were not available because analyses were conducted on pooled subsamples.

Because standard errors for laboratory determinations were larger than what the industry required, we further defined the sources of error. Of the three subsamples for lab analysis, one was sent to El Reno, OK for collection of NIR spectra and returned to San Angelo, TX for lab determinations. The other two "reps"

were held in San Angelo and analyzed without shipment. The analysis of variance indicated a significant rep effect ($P < .0001$) after sample variation was removed. Duncan's range test indicated that each rep was different from the other. However, if one simply analyzed for rep effect and allowed sample variation to be part of the error, then only the C rep was different (Table 3); the C rep was the one transported to El Reno for collection of spectra. Mohair base for the C rep was 2% less than that for the A rep and 1.7% less than that for the B rep. This indicates that handling and transporting the fiber samples resulted in changes in the composition, or possibly a change in moisture content. When each of the three reps was used as calibration data, the C rep was much better related to spectral interactance than either A or B rep, or the average. Because rep C was the one scanned with NIRS, it is reasonable to conjecture that a better relationship exists between reference values for it and NIR spectra. Errors associated with reps A and B were representative of sampling error. This suggests that raw mohair composition is subject to change during handling, and, for a sample to represent its fleece, it must be carefully handled during laboratory or scanning procedures. This would make analysis on the shearing floor using a rapid technique such as NIRS even more attractive because samples could be taken from the fleece and analyzed immediately without incurring changes due to handling or shipping.

Further analysis indicated the error between replicate C and the average of replicates A and B was positively related to sample number (Figure 2). The cause of this relationship is unknown, but it may have re-

Table 3. Replicate means for laboratory reference determinations for mohair base of Angora goat fleeces (Exp. 2)

Replicate	Mohair base, %
A	64.2 ^a
B	63.8 ^a
C	62.0 ^b

^{a,b}Means in the column with different superscripts are different ($P < .05$). Error term included sample and sample \times rep.

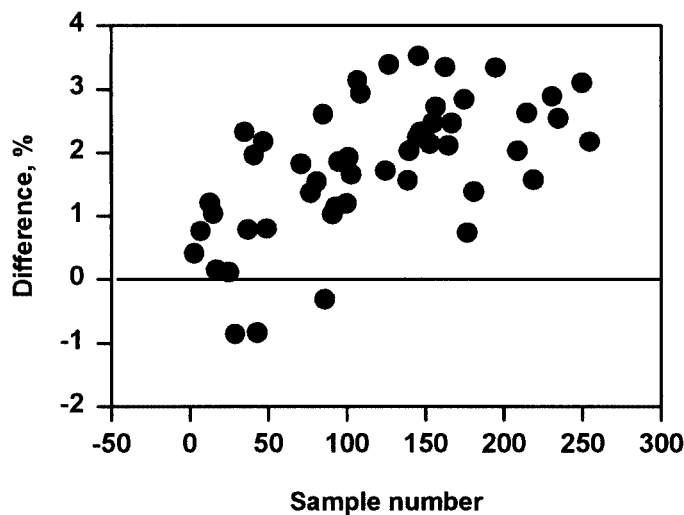


Figure 2. Relationship of sample number to deviations of near-infrared-predicted and laboratory-determined mohair base.

sulted from increased handling during the scanning process, the later samples having been moved or handled before scanning, whereas after scanning the samples were returned to a box and not disturbed further.

Calibration statistics comparing the utility of the spectra in the NIR region (1,100 to 1,700 nm) to those in the visible range (700 to 1,100 nm) are shown in Table 4. These data were obtained using the averaged spectra from the four scans of rep C for the 50 samples. For mohair base, R^2 was larger and standard errors for calibration and validation were smaller for the NIR spectra. It is possible that this was due to elimination of more outliers in the NIR range to develop satisfactory prediction equations. The causes of the outliers are un-

certain but were due to large t -values, indicating lack of agreement between laboratory and NIR values. Calibration and validation statistics for fiber diameter, vegetable matter, or medullated fibers indicated lack of fit with NIR spectra.

Connell and Brown (1978) observed that the visible spectra contained useful information for prediction of wool yield that actually complemented the NIR spectra. We were unable to combine spectra with our software to use both spectra in one equation. It was not possible to scan in both regions simultaneously with the fiber optic apparatus because the detectors for each region are different. It seems from our data that there is no advantage to using the visible spectra over the NIR spectra.

Standard error of validation in this experiment was the average of four standard errors of validation, each being generated by eliminating one-fourth of the samples from calibration and using these for validation. In four iterations, all samples are used once for validation. Validation statistics for mohair base were better than calibration statistics in the data summarized by M. J. Hammersley (1990, unpublished results). Analytical errors for repeated analysis for mohair base predicted from spectra in the NIR region for Exp. 2 were only slightly larger than laboratory error (1.66 vs 1.42, Table 2). This indicates that the NIRS procedure has the capability of being almost as precise as current laboratory methods. Sampling error is the major part of the error associated with the NIRS procedure.

The equation for mean fiber diameter was not as good as that of Larsen and Kinnison (1982), who obtained an SEC for mean fiber diameter of 1.0 μm , but validation statistics were not available. Wavelengths related to fiber diameter in their study were beyond 2,000 nm, whereas we were limited to 1,700 nm by the characteristics of the fiber optic probe.

Table 4. Statistics for prediction of mohair fleece characteristics of individual fleece predicted with equations developed from averaged scans (Exp. 2)^a

Parameter	Mohair base, %	Fiber diameter, μm	Vegetable matter base, %	Medullated fibers, %
Lab mean	63.3	38.8	1.23	2.51
SD	4.53	3.63	.78	3.64
Visible prediction				
Mean	63.4	38.7	1.05	1.68
SD	4.98	2.31	.38	1.08
SEV(C) ^b	3.94	3.9	.85	3.74
R^2	.43	.04	0	.02
Slope	.57	.32	.11	.54
NIR prediction				
Mean	63.3	38.7	1.05	1.62
SD	4.86	1.13	.17	.71
SEV(C) ^b	3.25	3.52	.8	3.67
R^2	.58	.07	0	.01
Slope	.71	.82	.11	.53

^aCalibration $n = 50$ fleece samples. Four scans per fleece were averaged for calibration. Individual scans were predicted ($n = 50 \times 4 = 200$). Actual $n = 185$ for NIR due to unacceptable spectra for some scans. Calibration performed using PLS (Shenk and Westerhaus, 1991).

^bSEV(C) = standard error of validation corrected for bias.

Table 5. Performance statistics of the equations from selected samples from Exp. 2 on extrapolated data sets from Exp. 1 and 2

Data set	Laboratory			Predicted			R ²
	n	Mean	SD ^a	Mean	SD ^a	SEV ^b	
1990 (Exp. 1)							
Mohair base, %	293	64.2	4.11	61.7	1.91	3.52	.27
1991 (Exp. 2)							
Mohair base, %	198	64.9	4.42	63.4	4.61	3.75	.41
Fiber diameter, μm	198	42.5	5.13	42.1	3.29	6.02	0

^aStandard deviation of the mean of the population.

^bStandard error of validation corrected for bias.

Vegetable matter base and medullated fibers could not be predicted with either spectral region. Vegetable matter base is the amount of oven-dried, scoured burrs, seeds, twigs, leaves, and grasses, free of mineral matter and ethanol-extractable matter expressed as a percentage of the original greasy mohair. The range was too small (.2 to 4.0%) and the variability too great to be predicted from a greasy sample. Medullated fibers were not related to spectra. Only 11 of the samples contained significant quantities of med and kemp fibers (> 2.0%; range = .1 to 18.1% of medullated fibers). M. J. Hammersley (1990, unpublished results) also identified similar problems in predicting medullated fibers with NIRS. Boguslavsky et al. (1992), however, found that fiber diameter was a confounding element in prediction of medullated fibers using NIRS. With fiber optics, we observed no particle size effect because transmittance rather than reflectance was used.

The 190 samples not chosen for calibration in Exp. 2 were predicted using the equations generated from the average scans of the 50 selected samples. Data with which to compare the predicted and laboratory determined values were only available for mohair base and fiber diameter (Table 5). Bias, representing systematic error, was small for both fiber diameter and mohair base (.40 μm and 1.56%, respectively). Even though standard error for validation (SEV), representing random error, was higher than desired for mohair base, we noted that standard deviation for the predicted data set was similar to that for the laboratory data (4.61 vs 4.42%). This is in contrast to the data from Exp. 1, in which the predicted data set was compressed and the standard deviation of the predicted values was 2.01%, as compared to 4.10% (Table 1) for the laboratory data. This suggested that the equation may be adequate for prediction of the 1991 data set and that extreme outliers contribute to the large SEV. Results for fiber diameter were of no value at all because of high random error (SEV).

Attempts to validate our equations with independent data sets were somewhat unsuccessful. The equations developed from the 50 samples of the 1991 data set were used to predict the 293 samples from Exp. 1 (1990). Comparison of the laboratory values and predicted estimates indicate that bias was a problem for mohair base

(Table 5). A more serious problem is the low R² (.27) and compression of the data set (SD = 1.91% for predicted; 4.11% for laboratory). Our conclusion was that the equation did not properly fit this data set. We were simply investigating the bounds of the robustness of the equation in these experiments. A robust equation for commercial use would require greater representation of the expected variation from all sources of interest.

Experiment 3. The objective of this experiment was to compare the variation among and within fleeces for both conventional laboratory analyses and in the spectral data collected using the more conventional reflectance mode (Figure 3). The transport mechanism allows for a large sample area (~15 cm²) to be scanned. Coefficient of determination (R²) between NIR spectra and laboratory values ranged from .93 for fiber diameter to .02 for kemp fibers (Table 6). We consider that mohair base and fiber diameter can be predicted with excellent precision using NIRS having R² of .82 and .93 with resulting SEC of 1.19% and .98 μm , respectively. Vegetable matter base and percentages of med and kemp fibers were marginal to poor in their relation-

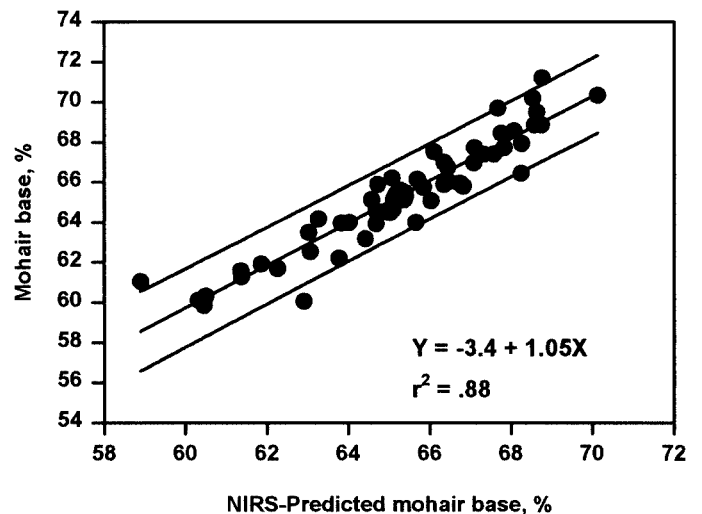


Figure 3. Relationship of near-infrared spectroscopy (NIRS)-predicted mohair base to that determined with laboratory reference methods (Exp. 3).

Table 6. Calibration statistics for mohair fleece characteristics using near-infrared spectroscopy (Exp. 3)

Trait	n	Mean	SD ^a	SEC ^b	R ²	SEV ^c	R ²
Mohair base, %	223	65.4	2.76	1.19	.82	1.26	.79
Vegetable matter base, %	214	.81	.40	.30	.48	.31	.44
Fiber diameter, μm	222	29.1	3.74	.98	.93	1.13	.91
Medullated fibers, %	218	.74	.40	.30	.20	.33	.08
Kemp fibers, %	186	.28	.22	.15	.02	.16	.00

^aStandard deviation of the mean of the population.

^bStandard error of calibration.

^cStandard error of validation within the calibration data set.

ships. During calibration, the number of spectra that were eliminated as outliers ranged from 13 for mohair base to 50 for number of kemp fibers. The reasons for outliers could be high *t*-statistic (difference in lab vs predicted value) or H-statistic (spectral characteristics do not match other samples).

The within-sample variance (s^2) obtained for laboratory analysis for mohair base was about one-half that obtained by NIRS (Table 7), but that for NIRS was .85%, less than the 1% desired by the industry. These are reasonable values because the NIRS method incorporates the error of the laboratory method, of sampling, and of agreement of NIR spectra with reference data. Therefore, NIRS precision can never be better on the same data set, even though repeatability of NIRS values on the same samples is often better than that for reference laboratory values for forage analysis (Barton, 1989). Variance for fiber diameter was approximately five times greater for NIRS, indicating additional increases in error over the laboratory method. Vegetable matter base, med fibers, and kemp fibers all had lower s^2 for NIRS, but the relationships between laboratory reference data and NIRS were very poor, and predictions were low. These data are in contrast to those of Boguslavsky et al. (1992) in South Africa for med and kemp fibers. However, they too were unable to obtain successful predictions of medullated fibers with dry mohair, but required suspension of the hairs in a liquid of similar optical density to reduce the effect of specular reflectance due to variation in fiber diameter.

Experiment 4. Calibration statistics for the combined and selected data set are presented in Table 8. When the remaining samples were predicted with the equations, reasonable agreement with the reference method of analysis was obtained for mohair base and fiber diameter. A marked improvement was made in calibration and validation of mohair quality using the transport mechanism over use of fiber optics (compare results from Table 8 and those from Table 1 [1990] and Table 4 [1991]). No evidence of preferential bias existed for either mohair base or fiber diameter in either year.

Equations developed from the selected samples over both years (combined) performed better than those developed from samples selected in either single year. For this equation, the 1991 samples were chosen with the program SELECT based on variation from the 1990 samples, whereas the 1990 samples were chosen based on variation within 1990 samples alone. All selections were based on Mahalanobis distance (H-statistic) of multivariate characteristics of the spectra (Windham et al., 1989; Shenk and Westerhaus, 1991). We used this technique because normal practice of developing equations follows such a plan. During the first sampling, a representative group of samples are chosen to represent that population. Subsequent samplings are then tested against the former selected samples to determine those necessary to represent the subsequent population. This eliminates multiple samples with redundant information from the calibration data set that contribute little to the equation's definition.

Table 7. Source of variation for mohair fleece characteristics analyzed with reference or near-infrared methods (Exp. 3)

Trait	Reference				NIRS ^a			
	Mean	Sample MS ^d	s^{2b}	CV ^c	Mean	Sample MS	s^2	CV
Mohair base, %	65.4	29.4	.42	1.00	65.4	23.3	.85	1.41
Vegetable matter base, %	.86	.71	.08	32.5	.82	.33	.01	12.9
Fiber diameter, μm	29.1	53.8	.18	1.44	29.0	49.6	.97	3.40
Med fibers, %	.78	.26	.12	44.6	.74	.07	.005	9.10
Kemp fibers, %	.27	.08	.04	69.5	.28	.003	.000	2.95

^aNIRS = near-infrared spectroscopy.

^bVariance due to replicates within sample.

^cCoefficient of variation.

^dMean square due to fleece.

Table 8. Calibration and validation statistics for mohair samples of Exp. 1 and 2 scanned with a transport mechanism

	n	Mean	SD _r ^a	SD _p ^a	SE ^b	R ²	Slope ^c	Bias
Calibration								
Mohair base, %								
1990	117	63.2	4.69	—	1.95	.83	—	—
1991	124	64.5	4.92	—	2.49	.72	—	—
Combined	240	64.0	4.47	—	2.03	.79	—	—
Fiber diameter, μm								
1990	116	43.4	5.59	—	3.35	.62	—	—
1991	127	42.6	4.87	—	3.88	.37	—	—
Combined	243	42.9	5.06	—	3.49	.52	—	—
Validation ^d								
Mohair base, %								
1990–1990	125	65.0	3.53	3.29	2.09	.66	.87	–.11
1991–1991	110	65.7	3.62	3.02	2.56	.52	.86	–.46
Combined–1990	125	65.0	3.53	2.92	2.02	.67	.99	.02
Combined–1991	110	65.7	3.62	3.20	2.62	.51	.81	–.37
Fiber diameter, μm								
1990–1990	125	43.2	4.93	3.94	4.29	.30	.69	.00
1991–1991	110	42.3	4.98	2.50	4.62	.15	.78	.08
Combined–1990	125	43.2	4.93	3.20	3.95	.36	.93	.11
Combined–1991	110	42.3	4.98	3.09	3.97	.37	.98	.02

^aStandard deviation of the population mean from reference (r) or predicted (p) values.

^bStandard error of calibration or validation.

^cSlope of line relating predicted to reference values.

^dValidation data set was samples from the respective year not used in calibration. First year (or combined) refers to data set on which equation was based; second year represents the validation samples.

The combined equations predicted the residual samples from each year with SEV of $\sim 2\%$ for mohair base and $4 \mu\text{m}$ for fiber diameter. The values for fiber diameter are probably still not accurate enough to be useful, but we suspect that variation in the sampling and laboratory analysis could be improved in such a way to improve the statistics. This conclusion is derived primarily from the results of Exp. 3 in which variation among analysis of four replicates of the same fleece was 1.44 and calibration with those data resulted in prediction errors of $\sim 3 \mu\text{m}$.

General Discussion. The use of the fiber optic probe in NIRS analysis of mohair characteristics would increase the utility, but the fiber optics we used limited the effective portion of the spectrum that can be used. Our results indicate that this limitation is too severe, and the reflectance mode using a transport or similar large sample attachment would be suitable for measuring mohair base and fiber diameter. The transport mechanism produced spectra with more precision than the fiber optics probe. New fiber optics are continually being developed, and it is possible that a fiber optic probe with more favorable characteristics would increase our ability to predict mohair quality. A range of wavelengths from 1,350 to 1,800 nm should give adequate resolution for grease, moisture, and ash. Scatter also seems to be a problem with fiber optic devices, further complicating the potential for developing a system based on these devices.

Using the transport, and thus direct reflectance, NIR spectra showed a strong relationship with mohair base

and fiber diameter. Vegetable matter base was marginally related, and kemp and med fibers constitute such a small fraction of the total that little relationship existed in these fleeces. The NIRS should be an excellent tool for rapid analysis of greasy mohair for yield and fiber diameter, and it could be easily adapted to real-time analysis at the shearing site. This would be an improvement in current assessment because it is likely that sampling error is large and that handling and shipping fleece samples contributes to this error. The failure to accurately predict med and kemp fibers may be a result of lack of precision in the reference method. New reference methods are being assessed, but they were not available to use for evaluation for calibrating NIRS.

Implications

Near-infrared spectroscopy has potential for rapid analysis of raw mohair fleeces. The use of fiber optics would provide more flexibility, but the accuracy and precision provided with the fibers used in our study was not sufficient for a recommendation. The use of a large sampling device such as our transport mechanism gave results that rival the reference methods for mohair base and fiber diameter. This technique could be implemented with current technology to provide objective and rapid measures of mohair characteristics.

Literature Cited

Barnes, R. J., M. S. Dhanoa, and S. J. Lister. 1989. Standard normal variate transformation and de-trending of near-infrared diffuse reflectance spectra. *Appl. Spectrosc.* 43:737–808.

- Barton, F. E. II. 1989. Considerations of chemical analyses. In: G. C. Marten, J. S. Shenk, and F. E. Barton, II (Ed.) *Near Infrared Reflectance Spectroscopy (NIRS): Analysis of Forage Quality*. pp 68–82. Agric. Handbook No. 643. USDA, Washington, DC.
- Boguslavsky, A., A. Botha, and L. Hunter. 1992. Measuring medullation in mohair with near infrared reflectance analysis. *Text. Res. J.* 62:433–437.
- Connell, J. P. 1983. Predicting wool base of greasy wool by near-infrared reflectance spectroscopy. *Text. Res. J.* 53:651–655.
- Connell, J. P., and O. H. Brown. 1978. The yield testing of wool by reflectance spectroscopy. *J. Text. Inst.* 69:357–363.
- Johnson, C. L., and S. A. Larsen. 1978. Clean wool determination of individual fleeces. *J. Anim. Sci.* 47:41–45.
- Larsen, S. A., and J. L. Kinnison. 1982. Estimating quality components of natural fibres by near-infrared reflectance. Clean wool base and average wool fibre diameter. *Text. Res. J.* 52:25–31.
- Lynch, L. J., and N. A. Michie. 1976. An instrument for the rapid automatic measurement of fibre diameter distribution. *Text. Res. J.* 44:653–660.
- Norris, K. H., W. E. Hruschka, M. M. Bean, and D. C. Slaughter. 1989. A definition of wheat hardness using near infrared reflectance spectroscopy. *Cereal Foods World* 34:696–705.
- Ranford, S. L., J. M. Hammersley, V. C. Acker, and F. L. Glassey. 1990. The measurement of medullation in wool using NIRA. *Proc. 8th Int. Wool Text. Res. Conf., Christchurch, N.Z.* Vol. 2:94–103.
- Ranford, S. L., M. J. Hammersley, and V. C. Patrick. 1985. Raw wool testing using near-infrared analysis. *Proc. 7th Int. Wool Text. Res. Conf., Tokyo, Japan.* Vol 2:167–175.
- Sabbagh, H. R., and S. A. Larsen. 1978. Wool yield by infrared reflection analysis. *Proc. West. Sect. Am. Soc. Anim. Sci.* 29:108–109.
- Shenk, J. S., and M. O. Westerhaus. 1991. Population definition, sample, and calibration procedures for near infrared reflectance spectroscopy. *Crop Sci.* 31:469–474.
- Windham, W. R., D. R. Mertens, and F. E. Barton, II. 1989. Protocol for NIRS calibration: Sample selection and equation development and validation. In: G. C. Marten, J. S. Shenk, and F. E. Barton, II (Ed.) *Near Infrared Reflectance Spectroscopy (NIRS): Analysis of Forage Quality*. pp 96–103. Agric. Handbook No. 643. USDA, Washington, DC.