

Biomass Energy Production Alfalfa Supply System

# Rapid Analysis of Hay Attributes Using NIRS

MINNESOTA AGRIPOWER PROJECT  
TASK II RESEARCH REPORT

Neal P. Martin, James L. Halgerson, Salli J. Weston, and  
Mark Matteson

University of Minnesota  
Department of Agronomy and Plant Genetics

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October 24, 1997

Final Report of  
Rapid Analysis of Hay Attributes Using NIRS  
Task II Alfalfa Supply System

by Neal P. Martin, James L. Halgerson, Salli J. Weston, and Mark Matteson

The quality of alfalfa hay varies considerably due to differences in growing conditions, soil fertility, stage of maturity, alfalfa varieties, harvest methods and equipment, and storage conditions. Because of the wide variation in alfalfa hay quality, a dependable method is needed to accurately determine leaf stem ratio and nutrient concentration (energy, protein, fiber, xanthophyll) of alfalfa feedstock as the basis of payment for producers and to establish quality control systems to provide consistent quality for processing alfalfa stems for fuel and leaves for alfalfa leaf meal products for MAP operations.

Near infrared reflectance spectroscopy (NIRS) is a nondestructive, nonpolluting instrumental method for fast, accurate, and precise evaluation of the chemical composition and associated feeding value attributes of forages and other foodstuffs provided proper procedures are followed, Marten et al., 1989. The analytical method has been used at hay auctions and in commercial laboratories for rapid determination of forage quality and is well suited for describing quality traits of the alfalfa biomass feedstock and of processed alfalfa products.

With the purchase of the Pilot Plant facility in Priam, Minnesota MnVAP purchased a state-of-the-art NIRS instrument, NIRSystems model 6500. This new piece of equipment had not been calibrated and was not in service due to the lack of trained technician to operate the equipment and the need for a specialized component that was not part of the original equipment.

Our tasks were to:

- To develop an NIRS driven analytical system for analysis of alfalfa hay and processed alfalfa products
- Assist in hiring a qualified NIRS technician and recommend changes in testing equipment necessary to provide accurate analysis
- Calibrate the NIRS instrument for accurate analyses
- Develop prototype equipment and sampling procedures as a first step towards development of a totally automated sampling system that would rapidly sample and record incoming feedstock and outbound product.

## Forage Quality Analysis Protocol Established at Pilot Plant in Priam, Minnesota

MnVAP staff assisted by University NIRS technicians developed an accurate analytical system of testing incoming and outgoing alfalfa products via NIRS analysis. The analytical system required qualified trained personnel, equipment modifications, computer software modifications, and establishment of a testing protocol. Equipment additions to the Pilot Plant included hay sampling probes (Forageurs), electric drill and ladder. These equipment items were needed to ensure sampling of truckloads was done in a random manner to best represent the load and to minimize ability of haulers to "stack the load" to influence sampling analysis. A new NIRS instrument sample cup system was purchased. The NIRS calibrations used by NIRS Forage and Feed Testing Consortium were purchased in addition to enrolling MnVAP into the NIRS forage and Feed Testing Consortium.

A job description for the NIRS technician was written and a technician was hired. After hiring a highly qualified, dedicated NIRS operator the University NIRS staff trained the instrument operator. The NIRS technician received additional training as new tasks or new tests are added. Sample preparation equipment and the NIRS equipment were modified to establish a system of forage quality analysis. Analysis was initially done on unground samples using a transport chamber to present the sample for NIRS analysis. After a preliminary test with 10 alfalfa hay samples and 6 alfalfa pellets the staff decided to use a sample preparation system that analyzed a microwave-dried sample cyclone ground sample for analysis. The new method requires an extra step in analysis; but it was chosen to provide the highest possible precision of testing, Table 1. Though the accuracy of the ground NIRS prediction did not match that of the unground prediction at the preliminary test further monitoring and calibration updates were proven necessary to represent the hay population. Using this system of analysis provided the smallest subsampling error and allowed use of the NIRS Forage and Feed Testing hay calibrations, Martin et al. 1996 and 1997 (developed from a national calibration base and adjusted to 25 commercial testing labs).

Table 1. Means and standard deviations 10 alfalfa hay samples tested by chemistry, NIRS of unground, untested calibration and NIRS of ground verified hay calibration.

Test	Chemistry	NIRS (unground)*	NIRS (ground)**
	-----Mean of tests-----		
Crude protein	20.83	21.58	19.69
Acid detergent fiber	41.51	41.89	43.65
Neutral detergent fiber	53.91	52.07	51.63
	-----Standard deviations of tests-----		
Crude protein	2.25	3.49	2.67
Acid detergent fiber	5.74	6.28	5.42
Neutral detergent fiber	7.06	8.14	7.14

\* Unground samples analyzed with NIRS using a large rectangular cup in the transport and company supplied calibrations.

\*\* Ground samples analyzed with NIRS using a small ring cup in the transport and proven calibrations.

The computer software has been modified to provide the test information MnVAP wants presented to its producers. Producers are paid on a crude protein basis, so the produce report forms were formatted to show dry matter, moisture, and crude protein. Later ADF, NDF, TDN, ENE and RFV index (TDN, ENE and RFV index are attributes predicted from other tests) were added. As new tests are needed for producer payments, they are added to the report format.

New protocols for collection of samples and NIRS analysis have been established at the Pilot Plant. These protocols follow:

- A total of 15-20 hay core samples are collected from random locations on each lot that enters the plant.
- A dry matter procedure is performed on the combined sample by weighing before and after microwave drying. This minimizes moisture loss in the grinder so the correct moisture can be determined.
- The sample is fine ground using a cyclone grinder with a 1-mm screen to improve precision in ground NIRS vs. unground NIRS analysis
- Hay samples are analyzed by the NIRS for dry matter (DM), crude protein (CP), acid detergent fiber (ADF), neutral detergent fiber (NDF), ash, total digestible nutrients (TDN), energy digestion (ENE), and relative feed value (RFV) index. The microwave DM and NIRS DM are combined to calculate the 100% DM of the hay.
- Fine grinding is also performed on the pellet samples to improve precision of analysis.

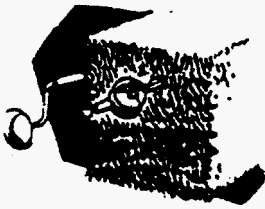
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## NIRS: FAST, EFFICIENT AND ENVIRONMENTALLY FRIENDLY

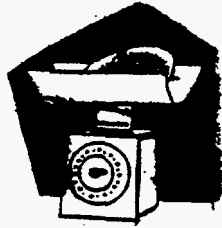
As fast as one hour  
photofinishing

30 times more efficient than  
chemical methods

No hazardous chemical  
waste



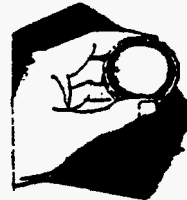
15-20 cores  
are taken per  
load to  
provide a  
representative  
sample.



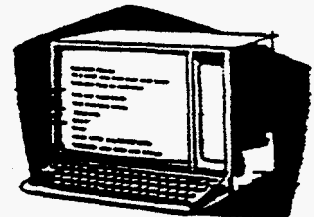
Hay samples  
are weighed  
and dried in a  
microwave  
oven.



Dried samples  
are finely  
ground.



The ground  
sample is  
mixed and  
placed in a  
sample cup.



The cup is  
placed in the  
NIR. Two  
minutes later  
the results are  
printed out.

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### Calibration of NIRS Analysis of Hay and Pellets

NIRS analysis is successful when four cardinal rules are followed:

- Ensure the calibration samples adequately represent the unknown samples in the population to be analyzed
- Conduct accurate laboratory analyses (chemical or bioassay) of the calibration samples
- Select the correct data processing techniques to extract the maximum information from the NIRS spectra
- Select the correct wavelengths.

The University of Minnesota cooperates with the University of Wisconsin to operate a National NIRS Forage and Feed Testing Consortium, Martin et al., 1996, which develops and monitors NIRS calibrations for commercial and public agency forage testing laboratories. The Consortium has developed an expertise of matching the spectra output for like NIRS instruments which allows the transfer of calibrations created by a Master Instrument to other Slave Instruments. The chemistry and NIRS analysis performed by the Consortium has been granted honor role status for accuracy for 1997 by the National Forage Testing Association (NFTA), a National voluntary forage testing certification organization. Therefore, MnVAP and the MAP project can obtain accurate NIRS calibration equations without the expense and time required developing an accurate calibration. (It would cost MnVAP at least \$72,000 for the laboratory chemistry alone to duplicate the number of samples within the Consortium hay calibration library). Therefore operation of a calibrated NIRS instrument is simplified; the calibration and instrument matching expertise is supplied by the NIRS Forage and Feed Testing Consortium of which MnVAP is a member. The selection of the calibration samples and calibration development has been done as directed by the NIRS analytical handbook, Shenk et al., 1989 using software of Infracsoft International. To use another calibration a representative set of samples representing the population of samples being analyzed must be analyzed by chemistry monitored against the existing NIRS calibration.

The MnVAP NIRS instrument was standardized to the Consortium NIRS Master Instrument in May of 1994. More detailed information is available about the instrument performance and the matching procedure upon request. The statistics used to compare the analysis of the same 14 sealed hay samples by both instruments is shown to Table 2. The SEP (C) shows that the average difference of the test values of the same sample analyzed at MnVAP vs. Consortium Master is 0.07, CP; 0.18, ADF; and 0.19, NDF which exceptional for lab to lab variation of hay tests.

Table 2. Statistics from monitoring 14 sealed hay samples run on the MnVAP vs. the NIRS Consortium Instrument.

Hay	NIR Master Mean	NIR MnVAP Mean	Bias <sup>†</sup>	Bias <sup>‡</sup> Limit	SEP(C) <sup>§</sup>	SEP(C) <sup>¶</sup> Limit	r <sup>2#</sup>	Slope
CP	20.01	19.66	0.35	0.55	0.07	1.20	1.00	0.99
ADF	33.58	33.87	-0.29	0.99	0.18	2.15	1.00	1.02
NDF	46.15	45.73	0.42	1.58	0.19	3.42	1.00	1.00

† - Conventional chemistry mean minus NIR mean.

‡ - Indicator that equation is satisfactory if absolute value of bias is less than bias limit.

§ - Standard error of performance corrected for bias.

¶ - Indicator that equation is satisfactory if SEP(C) is less than SEP(C) limit.

# - Squared simple correlation of conventional chemistry values vs. NIRS values.

Thirty (30) hay and 30 pellet samples were randomly collected from the Pilot Plant during fall of 1996 and winter of 1997 to determine the accuracy of NIRS analytical calibrations.

The monitoring procedure, Windham et al., 1989, requires a paired comparison of the same samples analyzed via chemistry and NIRS. The calibration used is acceptable if 2 criteria are met: 1) the NIRS predicted samples are not biased from the chemistry values, and 2) the samples fit the sample population represented within the calibration set. The statistics in Table 3 show the accuracy of the hay calibration equation used by MnVAP is exceptional. The mean of samples run by chemistry agree well with the same sample analyzed by NIRS, see small bias values for CP, ADF, and NDF, (bias is the difference between chemistry and NIRS means). Figures 2 thorough 4 show the graphic representation of NIRS analyzed values vs. chemistry values for CP, ADF, and NDF. The second test of performance of the calibration equation is the Standard Error of Performance (SEP). This value represents the average difference of the predicted values; if the SEP (C) exceeds the SEP (C) limit the samples are not represented within the calibration set and recalibration is required. If the SEP (C) is below its statistical limit, the samples predicted represent the calibration; thus, samples being analyzed at the Pilot Plant fit the calibration.



Table 3. Statistics of conventional chemistry vs. NIRS for hays.

Hay	Chemistry Mean	NIR Mean	Bias <sup>†</sup>	Bias <sup>‡</sup> Limit	SEP(C) <sup>§</sup>	SEP(C) <sup>¶</sup> Limit	r <sup>2#</sup>	Slope
CP	21.13	21.29	-0.16	0.47	0.53	1.02	0.88	1.07
ADF	38.08	38.44	-0.36	0.87	1.78	1.88	0.92	0.85
NDF	48.21	47.78	0.44	1.35	1.54	2.92	0.98	0.92

† - Conventional chemistry mean minus NIR mean.

‡ - Indicator that equation is satisfactory if absolute value of bias is less than bias limit.

§ - Standard error of performance corrected for bias.

¶ - Indicator that equation is satisfactory if SEP( C ) is less than SEP( C ) limit.

# - Squared simple correlation of conventional chemistry values vs. NIRS values.

The pellet analyses were also monitored against the Consortium hay calibrations, Table 4. The monitoring results were not as favorable as the hay calibrations; but show the hay calibration can be used with reasonable accuracy (Figures 5-7). The monitoring showed that a bias exists for CP, ADF and NDF, but that none of the three analyses showed the SEP( C ) exceeded the statistical limits. Hence, we have biased all the equations to correct for the over prediction of both CP and NDF and under prediction of ADF. MnVAP should consider having us develop a separate pellet calibration for all pellet products in the future.

Table 4. Statistics of conventional chemistry vs. NIRS for pellets.

Pellet	Chemistry Mean	NIR Mean	Bias <sup>†</sup>	Bias <sup>‡</sup> Limit	SEP(C) <sup>§</sup>	SEP(C) <sup>¶</sup> Limit	r <sup>2#</sup>	Slope
CP	18.73	19.65	-0.91	0.47	0.66	1.02	0.80	0.82
ADF	39.80	38.89	0.90	0.87	1.04	1.88	0.96	1.00
NDF	50.49	51.42	-1.03	1.35	2.29	2.92	0.91	0.87
ASH	11.09	11.06	0.03	0.61	0.61	1.33	0.90	1.62

† - Conventional chemistry mean minus NIR mean.

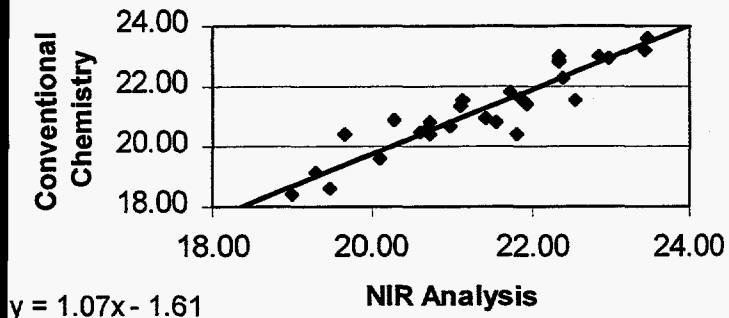
‡ - Indicator that equation is satisfactory if absolute value of bias is less than bias limit.

§ - Standard error of performance corrected for bias.

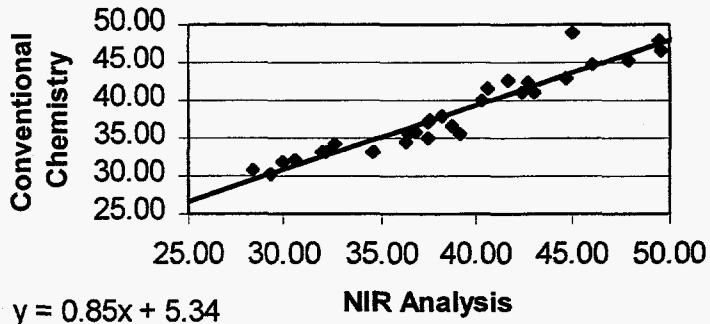
¶ - Indicator that equation is satisfactory if SEP( C ) is less than SEP( C ) limit.

# - Squared simple correlation of conventional chemistry values vs. NIRS values.

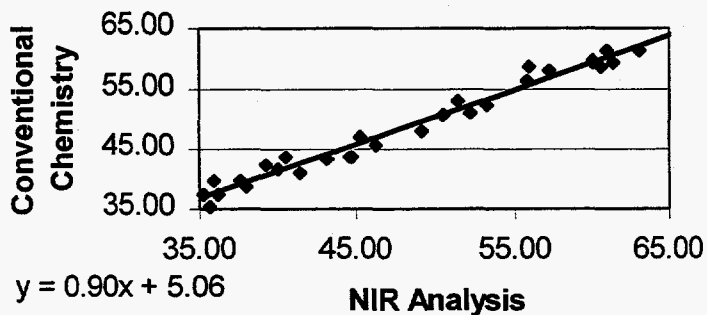
**Fig. 2 Paired Hay Sample Analysis:  
CP**



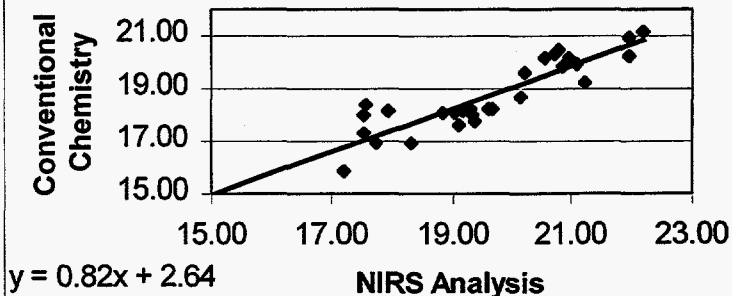
**Fig. 3 Paired Hay Sample Analysis:  
ADF**



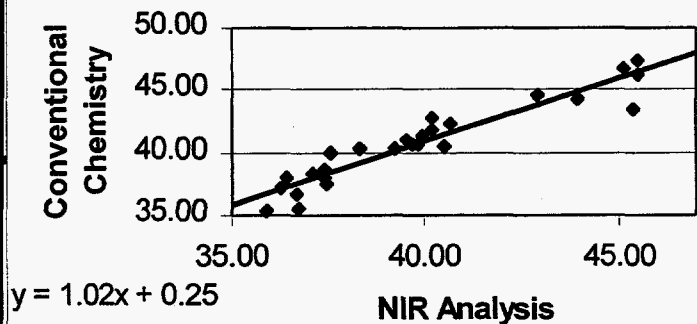
**Fig. 4 Paired Hay Sample Analysis:  
NDF**



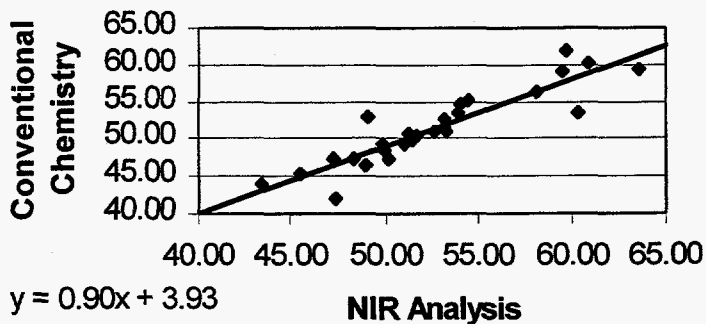
**Fig. 5 Paired Pellet Sample Analysis:  
CP**



**Fig. 6 Paired Pellet Sample Analysis:  
ADF**



**Fig. 7 Paired Pellet Sample Analysis:  
NDF**



## Significance of successful monitoring of NIRS Calibrations

Successful NIR calibration of a open population of samples coming into the Pilot Plant, requires extensive time, expertise, and resources. The calibration must represent samples from a variable extensive population; samples that represent different locations, seasonal variation, species mixtures, harvest and storage variations, etc. The calibration developed by the NIRS Forage and Feed Testing Consortium has been developed from locations throughout the U.S. on hay samples representing many different environments and many other variables possible in hay harvest. The current calibration has 755 samples (started 3 years ago with 1200 samples) which were downsized from a larger national set to fit samples tested within the North Central and Northeastern United States, Martin et al., 1997. Most important, the accuracy of this calibration is excellent; it has received honor role status by NFTA when compared to 140 other commercial laboratories within the United States.

### Leaf-Stem Calibration

The Pilot Plant will be processing leaf meal products. Development of a test to estimate the percentage of leaves in alfalfa hay will provide a quality control parameter with broad applications. It will be able to estimate the amount of leaves: 1) in the standing alfalfa crop, 2) harvested in the field, or lost from various field operations, 3) present before processing, and 4) retained during processing.

NIRS prediction of percent leaf has been used for several years on oven-dried whole herbage research samples. However, these prediction equations are not adequate for sun-cured farm samples from baled hay that is sampled using a small probe. Therefore, a protocol was needed to routinely handle this sample type. A commercially available probe (Forageurs) and a 4 inch diameter lab probe were used in developing the protocol (see separate sheet, Protocol for Leaf-Stem Separation of Cored Samples), which uses standard soil sieves and a mechanical shaker. A mechanical device should reduce variation associated with changes in personnel when processing samples.

A calibration file of sun-cured alfalfa samples included samples collected in the winter of 1994 throughout the hay shed of western Minnesota, in 1996 from the Minnesota Valley Alfalfa Producers (MnVAP) cooperative and in 1996-1997 from a bale stacking study from University of Minnesota, Morris Experiment Station. Samples were taken from small (70 lb.), mid-size (1000 lb.) and large (2000 lb.) square as well as round bales. Spectra were collected using a NIRSystems 6500 Monochrometer with Infrasoft International software version 4.0. A math treatment of 1,4,4,1 was used with the multiple partial least squares option (Table 5). For validation, handmade mixtures ranging from 10% leaf to 90% leaf were created in steps of 5% from several different hay samples (Table 6 and Figure 8). The equation shows good performance on the handmade mixtures, but may underestimate the error associated with the open population of hay

samples. Therefore, additional samples from the 1997 hay shed are being collected for further validation and possible update of the equation.

This equation will be transferred to the MnVAP pellet plant for inclusion in their overall analysis of incoming hay. As the Minnesota Agri-Power energy project proceeds, this equation may become part of the basis of payment for incoming biomass.

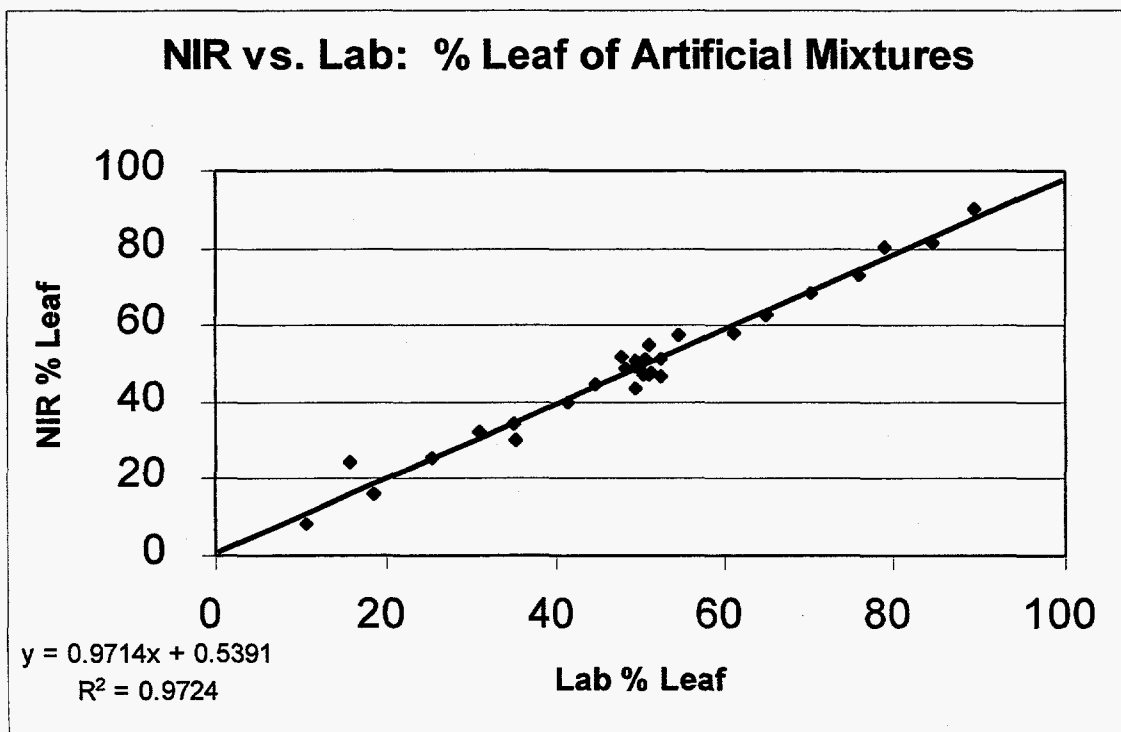
Table 5. NIR statistics for percent leaf prediction equation.

N	X	Range	SEC	R <sup>2</sup>	SECV	1-VR
150	53.4	0.01-100	2.8	0.99	3.4	0.99

Table 6. Monitoring statistics of percent leaf in alfalfa using artificial mixtures.

N	Bias	SEP( C )	Slope	R <sup>2</sup>
31	1.2	2.6	0.98	0.97

Figure 8. Comparison of percent leaf lab values to those predicted by the NIR using artificial mixtures of leaf and stem.



The importance of accurately sampling hay packages has increased throughout the industry and is especially important to the Minnesota Agri-Power Project. The question arises as to the accuracy of a small hay probe on large hay packages of 800 to 2000 lbs. The most common commercial hay probe available in Minnesota is the Forageurs probe, which has a ¾ inch diameter and a 14-inch length. To test the accuracy of this probe other sampling methods were tried, such as sawing into the bale, which caused excessive shattering of leaf material. A 4-inch diameter stainless steel probe (Megaprobe) with a 24-inch length was built at the University of Minnesota Scientific Apparatus Shop and was successful in taking a large sample from 12 ~1000 pound bales. Using a ½-inch electric drill, both probes penetrated to a depth of 14 inches. To compare the probes, three ¾-inch cores were taken within a 4-inch diameter, and then the 4-inch probe was taken directly over that area. Finally, the ¾-inch probe was used to take six cores around the outside of the 4-inch core. The samples were dried and divided. Half the sample was separated into leaf and stem fraction according to the attached protocol for leaf-stem separations. The other half was reserved as a whole plant sample. The fractions were weighed to determine percent leaf and stem.

Within an individual bale, the three coring methods gave variable results due to the variability in the hay package (Table 7). However, there appears to be no bias between Forageurs probe and the Megaprobe for leaf percentage when comparing the means and standard deviations, which are nearly identical.

Table 7. Statistics comparing the two probes, Megaprobe vs. Forageurs probe.

Bale Number	Megaprobe -----	Outside ---% Leaf---	Inside -----
1	41.9	38.5	41.0
2	47.7	44.9	44.1
3	35.5	41.5	38.3
4	39.6	42.9	36.6
5	45.1	41.4	36.8
6	34.1	35.2	35.8
7	42.7	40.7	36.4
8	38.5	35.2	35.6
9	39.0	40.5	45.1
10	35.7	40.1	46.8
11	45.2	45.1	48.7
12	33.4	28.1	36.1
Mean	39.9	39.5	40.1
Std. Dev.	4.7	4.8	4.8

The Forageurs probe appears to do an adequate job in determination of percent leaf or percent stem on large hay packages. The Minnesota Agri-Power Project has adopted the Forageurs probe as its standard sampling tool for hay.

### Quality of Alfalfa Hay Received by MnVAP

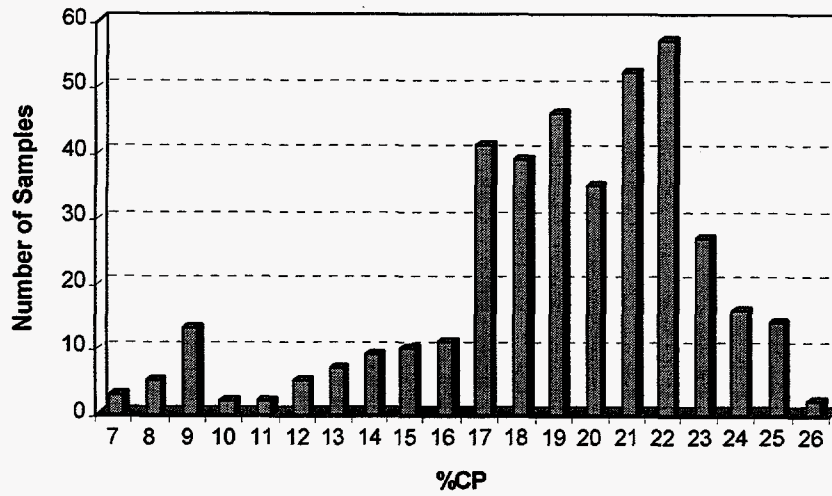
Hay samples analyzed between July 1996 and January 1997 (400 test samples were selected from MnVAP's NIRS files - the starred samples were not selected) show the quality of alfalfa hay received for processing. (Samples analyzed from Feb 1 thorough October 17, 1997 are currently being summarized.) Alfalfa standing in the field before harvest will analyze between 15 and 23 percent crude protein (CP), Table 8. The distribution of samples by percent CP shows the majority of the samples analyzed between 17 and 22 % CP, Figure 9. Hay samples could analyze lower than 16 % CP from excessive field losses or excessive weather damage. However, the few samples analyzing below 12 % CP could be hay containing a significant amount of grass, which reduces the CP level and increases the fiber levels. Ruminant livestock feeds are higher in quality if CP is high and fiber is low; fiber is inversely correlated with intake and digestibility.

Table 8. Expected quality values of alfalfa at various stages of maturity.

Stage of maturity	Crude protein	Acid detergent fiber	Neutral detergent fiber	Relative feed value
	-----% of dry weight-----			index
Vegetative	>22	<25	<34	>189
Bud	22-20	25-31	34-41	189-147
Early bloom	19-18	32-36	42-46	146-123
Late bloom	17-16	37-40	47-50	122-107
Seed pod	<16	>41	>50	<107

The quality tests that describe the variation within the hay population should be examined. Figure 10 shows the range of RFV index values at each whole percent CP. There the feeding quality (RFV) can vary greatly at a specific level of protein. Relative Feed Value is an index representing the potential intake of digestible dry matter of a lactating dairy cow (Table 9). Fiber tests are used to predict RFV index. The use of RFV index by MnVAP would provide an added test which separates different hays on a quality basis better than CP.

**Fig. 9 MNVAP Hays Ranked by %CP (July 96 - Jan. 97)**



**Fig. 10 MNVAP Hays: RFV Variations Within CP**

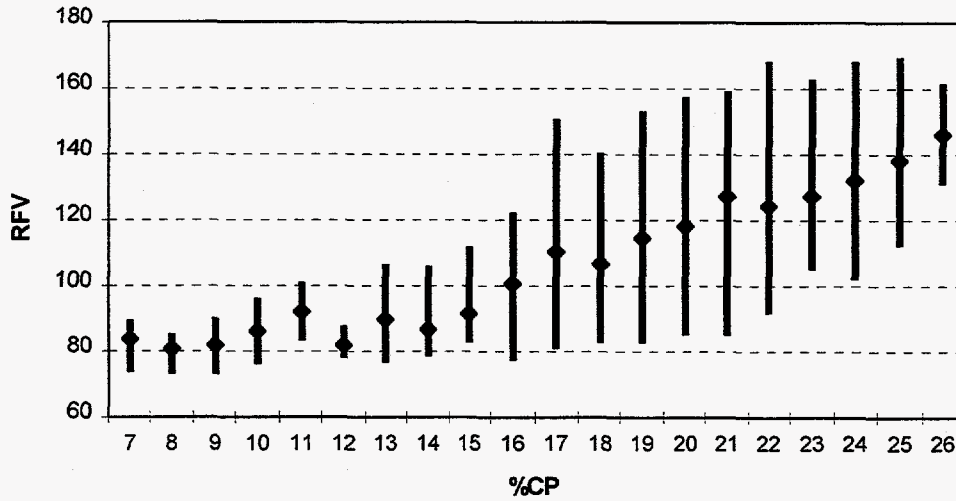
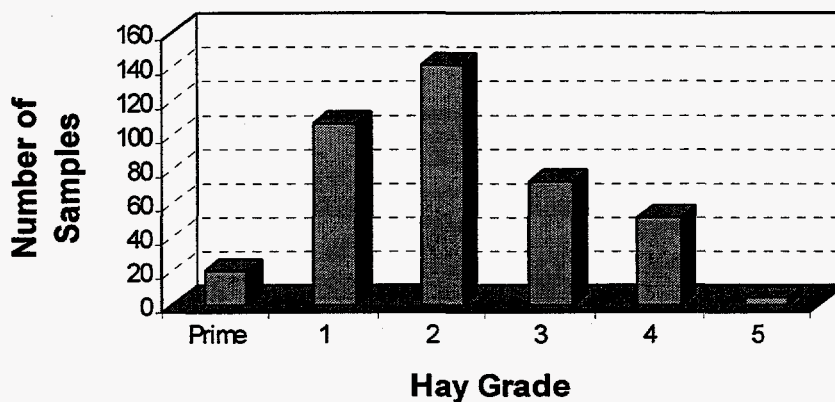


Table 9. The American Forage and Grassland Council description of the quality of hay by test standards.

Hay Grade	Relative Feed Value Range
Prime	>151
1	151-125
2	124-103
3	102-87
4	86-75
5	<75

Prime and standard 1 hays are hays of high enough quality to be preferred by high producing dairy cows. Standard 3 hay or RFV index of 100 is slightly below average. Hays received by MnVAP for the period from July 1996 through June 1997 are ranked by grade in Figure 11, below.

**Fig. 11 MNVAP Hays Ranked by Hay Grade (July 96 - Jan. 97)**





## Large Bale Sampling Study

Several sampling studies have been initiated to examine the proper method of sampling truckloads of large round, mid-size and large sized square bales. A preliminary review of the data collected to date shows a hay probe of at least 5/8 - inch internal diameter and a length of 12 to 18 inches is an effective sampling device. It represents the leaf stem ratio of any of the package sizes or shapes tested. The data also indicate the cardinal rule of sampling from 20 bales at random still best represents that load or lot. We have extensive sampling data currently under statistical analysis to support these findings and support MnVAP's sampling protocol.

## Evaluation of Pilot Plant Pellet Manufacturing

Two studies involving a quality control analysis of pellet processing were conducted at the Pilot Plant, Priam, MN. One investigation was done to determine the temperature generated during pelleting and to see if the heating occurring during pelleting reduced protein content. A second evaluation was done with a Consulting Engineer and MnVAP to determine losses of leaves during processing from grinding, mixing, through to pelleting.

### Hardness/temperature lab test

Two tests were conducted with a laboratory-scale pelletizer to determine the temperature produced during pelleting and the hardness of the pellet. As the pelleting machinery at the Pilot Plant increases its daily throughput, the hardness and temperature of the pellets increase. The temperature at the Pilot Plant was 190 °C, determined using an infrared gun. It was suspected that a temperature this high maybe causing a loss of CP. The highest temperature we achieved in the laboratory was 180 °C when both samples and pelletizer were pre-heated. The best pellets were made using 12 % moisture alfalfa. Our conclusion from this test was that no significant loss of CP occurred during pelleting.

The second test was run on pellets manufactured at the Pilot Plant. In this test the shiny outside layer was shaved off (0.0025 in layer). This layer tested 0.4 % CP less than the remaining pellet, but this layer does not represent a large portion of the overall volume. Therefore, we concluded there was no significant loss of CP during pelleting at the Pilot Plant in Priam, Minnesota.

## Plant Process Testing

Five separate tests have been performed with MnVAP at the Pilot Plant, Priam. The conclusions of those tests are:

- Approximately 2-3 % loss of material (shrink) occurs during processing.

- When inserting bales of different quality into the line, very little mixing of quality component occurs within the equipment. A dramatic change in quality of output can be observed in the less than a minute when sampling the pelletizer during processing of different quality bales.
- Pelleting decreased the protein of the hay by 1.3 %. This may be attributed to the loss of high quality dust escaping from the plant or to the leaves burning in the dehydrating drum.
- Good repeatability was shown on hay core sampling between samples taken off the truck at the weigh - scale vs. additional samples taken after unloading.
- Samples coming out of the drying drum have approximately 12 % moisture and pellets directly out of the pelletizer have 8.5 - 9.0 % moisture. The drying drum reduces the moisture by at least 6.0 percentage points.

Table 10. Thirty-nine (39) round bales were cored 4 times each and analyzed via NIRS

Test	Min	Max	Average
DM (NIRS)	94.96	96.67	95.89
CP	16.65	21.95	19.76
ADF	36.95	45.45	41.44
NDF	43.01	59.02	51.65
RFV index	84.31	129.89	102.57

### Summary and Conclusions

- MnVAP has developed an accurate hay-testing program.
- The NIRS Forage and Feeding Consortium hay calibration equations will accurately analyze alfalfa hay and sun-cured alfalfa pellets. At a future time MnVAP should consider cooperating with University of Minnesota to develop a separate alfalfa pellet calibration which should involve other processing plants and could set the example of how alfalfa processed products should be analyzed.
- The leaf stem calibration developed is worthy of testing on a broader scale.
- A total quality control program should be implemented by MnVAP to insure quality of alfalfa hay delivered to the processing plants contains the potential value-added components necessary to develop quality alfalfa products.
- We are willing to cooperate with DOE to develop NIRS calibrations for evaluation of materials for gasification.

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## Protocol for Leaf-Stem Separation of Cored Samples

1. Dry samples overnight at 50 °C in Blue M oven or in big dryers in threshing room.
2. Remove sample from paper bag and place in a labeled plastic ziploc bag.
3. Massage bag between palms for approximately 5 minutes to break leaves into a fine powder; and to break up any remaining cores.
4. Setup soil sieve screens in the following sequence: 8, 10, 16, 20, holding pan. Place screens on shaker set to 300 rpm.
5. Dump sample from plastic bag onto shaker. If it's a large sample only dump part of the sample on the screen at a time. Make sure there are no unbroken cores or leaves. Remove and discard any visible burrs or pieces of twine. Turn the shaker on.
6. After approximately 30 seconds, or when sample appears to be separated, turn the shaker off.
7. Put the contents of screens 8 & 10 into stem fraction bag. Always pour screen contents into three-corner pan to reduce loss of material, before moving to storage bag.
8. Put the contents of screen 16 into a plastic bag and crunch again if alfalfa leaves can be seen. Otherwise shake again at 400 rpm for 20 seconds through screens 16 and 20. Put contents remaining on screen 16 into stem fraction bag.
9. Remove screen 20 and its contents. If there is visible stem material place only screen 16 on holding pan. Pour contents of screen 20 through screen 16 and shake at 400 rpm for 15 seconds. If there is no visible stem material in screen 16 put contents of both screens into leaf fraction bag.
10. If screen 16 has visible stem material pour into stem fraction bag.
11. Pour the contents of the holding pan into leaf fraction bag if there is no stem material visible.
12. If holding pan has visible stem material pour through screen 16 at 400rpm for 15 seconds.
13. Weigh the leaf and stem fractions on a tared balance and record the weight.