THE INFLUENCE OF DRYING METHOD ON HAY QUALITY

By

Sultana Natho-Jina

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Department of Agriculture Engineering

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ABSTRACT

This project investigated the feeding value of alfalfa, dried with three different methods of drying : forced air heated by solar energy, forced air at ambient temperature and the conventional drying method.

Attention was given to the crude protein and the acid detergent fiber contents, since they are of particular concern to the dairy cattle production.

The measurements of moisture content or dry matter content were taken. The changes in the chemical composition such as vitamin A and other nutritive values were studied but not analytically. During the drying process, different measurements were taken such as temperature variations and weight losses through the bales. Three tests were carried out corresponding to three different cuttings of alfalfa. For the first cutting, dried samples from the bales were collected following the three methods. Whereas for the two other cuttings, samples were obtained from the two artificial driers. These samples were used for the chemical analysis in order to determine the feed value.

Statistical analysis was performed on the data after each test of drying. For the first cutting, dried hay did not show any significant difference in crude protein and acid detergent fiber. But for the second cutting, hay dried with forced air at ambient temperature gave higher protein and lower fiber and dry matter than hay dried with the other method. Finally, for the

matter than hay dried with the other method. Finally, for the third cutting, more fiber and less dry matter was obtained with ambient forced air. Crude protein was not different for the samples of both methods of drying.

In this research, although no best quality was found in using any particular method, it was learnt that more samples are required for determining the best method of drying to obtain top quality hay.

RÉSUMÉ

Ce projet essaie de determiner la qualité et la valeur nutritive de la luzerne séchée selon trois méthodes de séchage différentes qui sont le séchage à l'air forcé et chauffé à l'energie solaire, le séchage à l'air forcé à la température ambiante et le séchage conventionnel sur le champ.

Les mesures se rapportent particulièrement sur les protéines ainsi que sur les fibres en notant que ces éléments sont très importants dans la chaîne de la production laitière. Les mesures du taux d'humidité et de la matière sèche seront aussi étudiées mais on ne fera qu'évoquer les implications dans la composition chimique des éléments tels que la vitamine A de même que d'autres éléments particuliers.

Trois coupes de foin ont été réalisées au cours de l'été. Les balles de foin provenant de chacune de ces coupes ont été soumises aux différentes méthodes de séchage d'où des échantillons ont été prélevés en vue des analyses. Notons que seule la première coupe a été testée pour les trois méthodes. Par la suite, le séchage au champ a été abandonné.

Les résultats obtenus ont été assujetis à des procédures d'analyse statistique. Dans la première coupe, aucune différence significative pour les protéines brutes et les fibres n'a été observée. Dans la seconde coupe, le séchage à l'air forcé à la température ambiante a donné plus de protéines brutes, moins de fibres et moins de matières sèches que l'autre méthode. Enfin dans la troisième coupe, on a obtenu plus de

fibres, autant de protéines brutes et moins de matières sèches avec le séchage à la température ambiante qu'avec l'autre méthode.

Forts de ces résultats, nous ne pouvons distinguer la meilleure des trois méthodes de séchage. Mais nous sommes conscients que plus d'échantillons et des examens plus poussés nous amèneront sûrement à l'objectif souhaité.

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I. INTRODUCTION

Hay is a feed, produced by dehydrating green forage to a moisture content of 20 percent or less. This feedstuff is both quantitatively and qualitatively important from the economic and nutritional standpoints. Hay provides a considerable proportion of the energy and of certain other nutritional essentials of livestock feeds.

High quality hay is indispensable for maintaining health and thrift in young animals between pasture seasons, and for satisfactory breeding performance of mature animals.

The feed value of a hay crop can be attained by the retention of all leaves and by a rate of drying that quickly stops respiration, enzyme action and fermentation. The importance of leaf volume is readily appreciated by the fact that leaves contain most of the feed value of a hay crop. In alfalfa, leaves make up about 50 percent of the total weight but contains 70 percent of the protein and 90 percent of the carotene or pro-vitamin A, of the entire plant. Considering that the leaves are about 40 percent more digestible than the stems, the difference in protein, carotene and other nutrient content in leaves, as compared with stems, is still further increased. Most of these leaves are lost due to their shattering in the field and also by inefficient methods of curing hay. This is the reason why the conventional drying system (field curing) has been the least effective drying process, not only for the loss of leaves, but also for the

decreased rate of drying as a result of weathering and unpredictable stage of growth at which the crop has to be cut. The use of the forced air drying method was introduced in the Province of Quebec, in the early 1960's.

The first installation of forced air in barns was done with air heated by fuel energy. However with increasing costs of fossil fuels, supplementary maintenance, and the hazard of fire, it became a luxury system for the average farmer. The introduction of solar energy in drying, more specifically for low temperature hay drying, is of economic interest as a major source of energy for hay drying. Since solar energy is a "free" abundant source, the only costs involved are operation and capital whereas in hay drying units, using fossil fuels, an additional cost of energy must be taken into account. Part of the benefits to be derived from using solar energy in drying crops, should be an improvement in the quality of feed value of the final product.

In Quebec, 6 million tons of hay is harvested per year. Within the last ten years, the number of hay dryers has been increased to over 10 000 units. Despite this amount, only approximately 15 percent of hay is dried with forced ventilation. The remaining forage is dried by other methods before storage and feeding, the most common method being field curing.

In this report, three drying methods are compared using alfalfa hay (Medicago sativa) in terms of quality. Moisture content (MC), the percent of crude protein (CP) and the acid detergent fiber (ADF) are analyzed. Vitamins and other criteria of feeding value are examined but no chemical experiment was done

since they are of little concern in terms of cost of production for the dairy farmer. There have been cases where a large portion of the carotene has disappeared, even though the hay had retained its green color after a period of storage.

II. REVIEW OF LITERATURE

Many past experiments have been done for comparing drying methods. They have shown that low temperature hay drying with air temperatures up to 6 degrees above ambient, is a method which offers a great potential to reduce energy consumption, while allowing for a flexible harvest schedule and maintaining of good hay quality.

1) Field Curing

Bohstedt (1944) showed that certain losses in nutritive value inevitably take place during harvesting, processing and storage of hay. These losses averaged about 10 percent under near-ideal conditions of weather, properly operated machinery and adequate storage conditions.

Dexter et al. (1947) found that the moisture content of many materials at equilibrium is a function of the relative humidity of the surrounding atmosphere. This research stated that the mold growth was primarily a function of the relative humidity.

In another research, it was demonstrated that if hay is left in the field too long, the dry leaves will shatter and will subsequently be lost. If hay is brought in too soon from the field and cured slowly, sugars are lost. Similar problems exist with vitamins as they are directly affected by the ultra-violet portion of solar radiation incident on the field.

Other investigators such as Kurtz (1968) and Dobie et al.

(1961) noted that inclement weather damaged hay, and the feed value losses were carried from 25 to 40 percent. As most of the protein and total digestible nutrient are in the leaves, the feed value reduction must be a result due to leaf losses. These losses occur as the hay must remain in the field exposed to uncontrollable elements until it is dry enough to store. The leaves may be adequatly retained if the forage is dried and stored as quickly as possible after cutting. Ideally, this process should take place within a 24 hour period.
It was evaluated by Stanford et al. (1954) that good alfalfa hay may contain over 20 percent protein, but for same hay, exposure to rain or mechanical handling may drop to as low as 11 percent protein.

2) Artificial versus field drying

It has been recognized by several investigators that forced ventilation has overcome the problem of hay losses in the field. Miller and Shier (1943) reported from their studies on hay drying with forced natural air ventilation, that better hay quality, with minimal leaf loss, and more color can be obtained by this method. Farmers were satisfied with this method, since it eliminated the fire hazard attributed to spontaneous combustion from excessive respiratory heating.
Terry (1947) concluded that the use of artificial hay curing not only decreased the drying time but also reduced the damage done to the forage by mold and bacterial action. Respiration losses were also decreased when fan drying was used.

3) Heat generation from respiration

The heat generated by the hay has been a major problem for many farmers. Hendrix (1947) disclosed that respiration heat is continuously released by drying hay until a critical temperature is reached. Davis (1947) observed that the rate of heating increased when the initial moisture content was 45 to 50 percent. Above 50 percent, although it continued to increase, but at a less rapid rate. A higher rate of heat generation at higher moisture content, however, speeds up the rate of moisture removal in the mow.

From their experiments on respiration in hay as a source of heat for barn drying, Dawson and Musgrave (1946), drew two conclusions. First, respiration occuring in hay during barn drying liberates more than 25 percent of the heat absorbed by water evaporation. This occurs when the entering air has a low relative humidity and microorganisms development has not yet occured ; second, respiration liberates more than 60 percent of the heat absorbed by water evaporation when the entering air has a relative humidity of 75 percent or more, and microorganisms development has occured. The heat generated by the respiration combined with the high relative humidity, encourages the production of mold. This increases the loss of carbon dioxide present in the stack, thus promoting enzymes in the hay to consume starches and sugars, the final result is a reduction in the forage feed value. Rapid hay drying will not only eliminate the moisture for forage conservation, but it

will also reduce the bacterial populations which can severely deteriorate hay quality (Dexter et al., 1947).

4) Supplemental heat in drying process

A more recent improvement to the natural forced air method for drying is the introduction of supplemental heat into the system.

- High temperature drying

Very high temperature of 135° C (275° F) for drying has possible detrimental effects upon the nutritive value. Whereas Hart, Kline and Humphrey (1932) found that nutrients of green alfalfa exposed to temperatures 480 to 535° C (896 to 995° F) in a Koon-type drier for 40 seconds, were no less digestible than those of similar alfalfa dried in the field. Further research was undertaken by Bratzler et al. (1960) with drying temperature between 43 and 93° C (110 to 200° F). With the lowest drying temperature of 43° C (110° F), crude protein content was significantly higher than that of higher temperatures.

Due to higher energy cost, there has been little interest in using high temperature dehydration as a method of hay drying.

- Low temperature drying

Montford (1947) showed several distinct advantages in the use of fuel heated air, at low temperatures, in barn hay curing system. They are :

- a better quality of hay with higher protein and vitamin content due to a shorter precuring period in the field,
- less exposure to sun and rain,
- shorter curing time.

In this manner, hay can thus be dried regardless of weather conditions. Driers can be used effectively 24 hours per day. Equipment can be adapted to dry other crops such as grain, clover seed, corn, rice, etc. Montford also noted some disadvantages of using supplemental heat from natural and butane gas fuels. The more important of them being : 1) fire hazard, 2) higher initial cost of equipment and 3) higher cost of operation.

Davis et al. (1947) analyzed the factors affecting the use of supplemental heat in barn drying forage crop. Some of the factors studied, were the effect of the temperature, relative humidity and initial moisture content on the rate of drying, heat generated in the forage and the change in the chemical composition of the hay during drying. It was observed that the air exiting the hay reached its maximum relative humidity while passing through a height of three feet of forage.

In the use of continuously and intermittently ventilated air drying for hay, it was found that the overall reduction in quality during drying was negligible (Nellist, 1976). It was

noticed however, that there were considerable visual and analytical differences between random samples from heated and unheated areas of a bale. Respiration heating produced by alfalfa left the bale moist and discoloured.

A report from farmers on barn curing of hay with heated air by Strait (1944) noted distinct advantages of the use of heat : 1) a saving in labor and 2) an increase in capacity, enabling the system to operate day and night. In the latter, it is possible to load the barn with more hay at one time during a given harvesting period. In this research, it was found that feeding quality was about the same for a low temperature solar drier and for a high temperature gas drier. Lower drying temperatures resulted in more protein retention.

Tests undertaken by Clancy et al. (1976) compared different conservation methods with respect to nitrogen balance, digestibility and intake of alfalfa. One of the treatment used to preserve the forage was the use of heated air (50° C). Of the six treatments tested, this method yielded the highest values in crude protein, in digestible crude protein and in dry matter. This method of conservation had low quantities of lactic acid, neutral detergent fiber and acid detergent fiber. Average values of ash and apparent digestible energy were also observed. As far as the intake was concerned, hay dried with heated air had a high rate of voluntary intake and high meal sizes.

III. OBJECTIVE

The aim of this project was to compare alfalfa hay in terms of quality using the following three methods of drying :

1) Field curing

2) Drying with forced air at ambient temperature

3) Drying with forced air heated by solar energy.

IV. STANDARD CRITERIA IN EVALUATING HAY

In order to minimize cost and maximize profit, the quality of forage fed to the livestock is a dominant factor. The higher the quality is, the less concentrates will be used. The determination of the quality of hay in this research is based on the American Forage and Grassland Council and the Federal Grain Inspection Service (Hannaway, 1981). They proposed new grading standards for hay, based upon visual examination and chemical testing procedures. Visual examination evaluates leafiness , color, odor and condition. Chemical analysis evaluates the feeding value of hay by testing for moisture, crude protein and acid detergent fiber.

1) Organoleptic characteristics (sight, smell, touch) :

Leafiness : leafiness is extremely important since two thirds of the protein is found in the leaves. Plants having a high proportion of leaves will give a high value of protein and a low value of fiber resulting in high quality hay. The method of curing, the method of handling the hay from field to storage, and weather conditions during curing and baling, also influence leafiness.

Color : this is another definite indication of hay feed value. Bright green color is an indication of proper curing, high carotene content and good palatability. Any change from a

bright green color indicates loss in feed value. However, hay which has been slightly sun bleached or has had a small amount of rain damage will still be a high quality feed.

Odor and condition : the smell of a new mown hay is the standard for comparison. Any other odor, such as musty or rotten odor indicates lower quality. Odor problems usually result in lower acceptability by livestock.

Extension service of Oregon State University prepared a guideline for visually evaluating hay (Reference 1).

2) Chemical analyses :

Moisture or dry matter content : moisture determination is one of the most important and most widely used measurement in the processing and testing of feeds. Since the amount of dry matter in a feed is inversely related to the amount of moisture it contains, moisture content is of direct economical importance to the farmer. Moisture content must be known to express results of analytical determination on a uniform basis.

Crude protein : the quality of alfalfa and that of other legume hays is closely related to its crude protein content. Hay, high in protein, allows the dairyman to save money by feeding a grain ration low in protein. High levels of crude protein are generally associated with high A.E. content (available energy which is the amount of energy an animal can extract per unit of

dry matter consumed). However, the plant breeder should be concerned about crude protein levels mainly from the point of view of the nutrient content.

Acid detergent fiber : this is the plant fiber that remains after removing part of the digestible cell wall material with an acid detergent. It is highly correlated with animal digestible dry matter : as acid detergent fiber increases, digestibility decreases.

V. MATERIALS AND METHODS

A. Experimental Design

The drying tests were conducted at the experimental field station of the Brace Research Institute. Two experimental forced ventilation drying bins were identically constructed (Figure 1). Each of them contained six bales, simulating bales of dried hay at the center of a larger stack.

Two Delhi 400 centrifugal blowers of 1/2 HP each, were used to supply the air flow through the hay bales. The air used to dry the hay in one bin was preheated by solar energy while the other was not. The one used for ambient air was located in the ventilation plenum (open to outside air). The solar heated air crib drew its air from the solar wall collector (Figure 2).

The two bins were filled with six bales of hay of comparable quality and moisture content for each test.

The bins were constructed by sheets of 2.0 * 1.0 m. by 15 mm. plywood lined with polyethylene plastic to prevent the air from short-circuiting the system. Detailed drawings of these bins with the dimensions are shown in Figure 3.

These bins were mounted on mechanical scales and a record of the weight losses of the hay was obtained every hour during the daylight period. A plenum chamber of 0.3 m^3 underneath each bin (simulating the air duct of a conventional system) was connected to the blower by an air supply duct.

Air flow in the system was measured using an anemometer placed

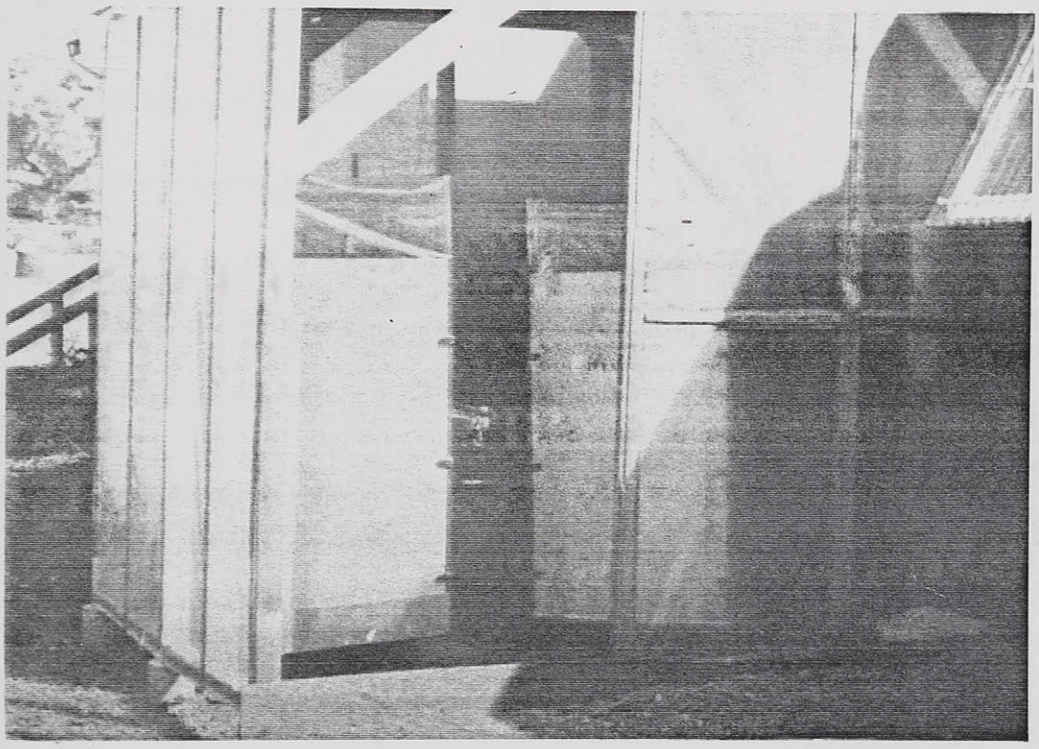


Figure 1. Two artificial driers.

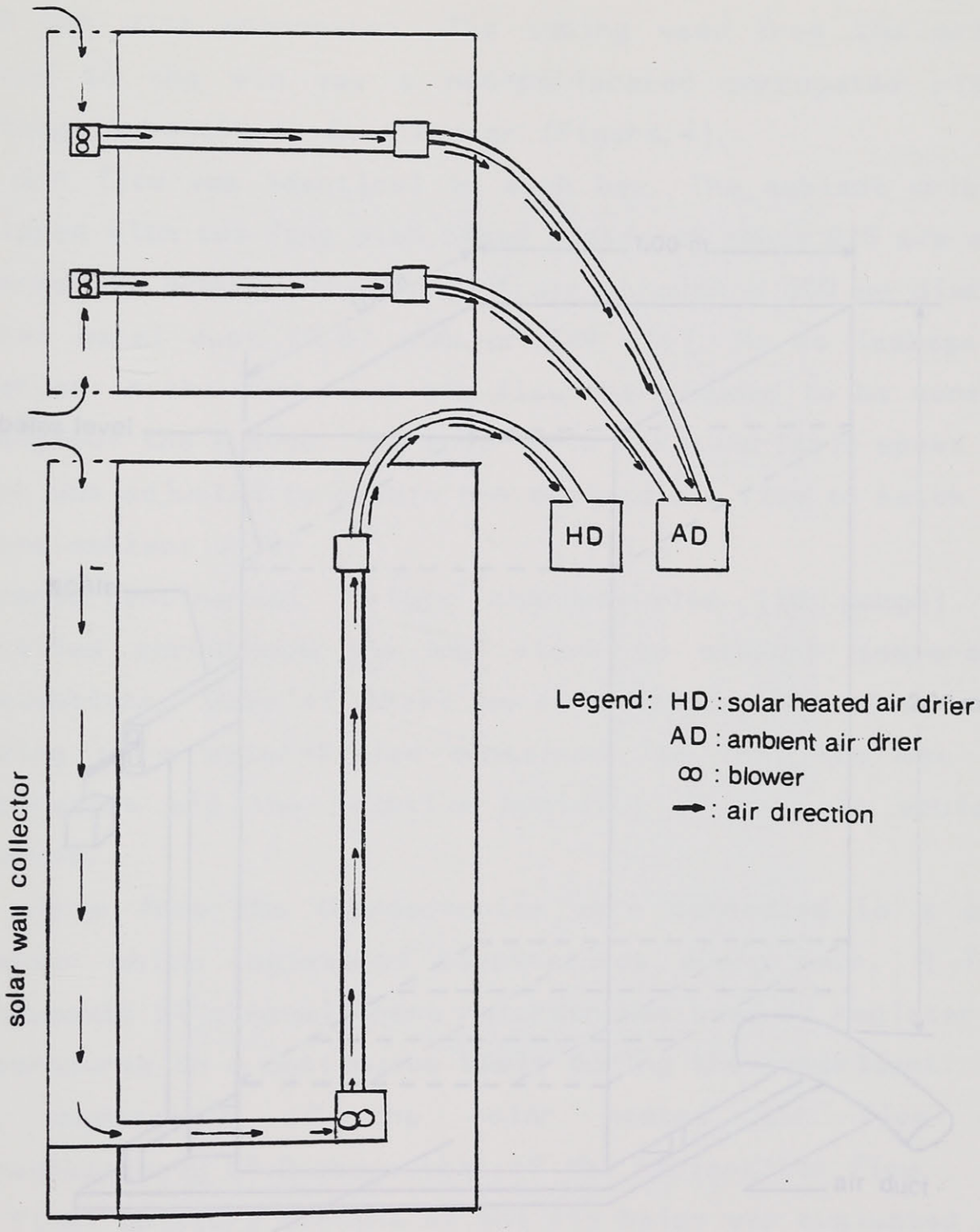


Figure 2. Schematic of the experimental set up.

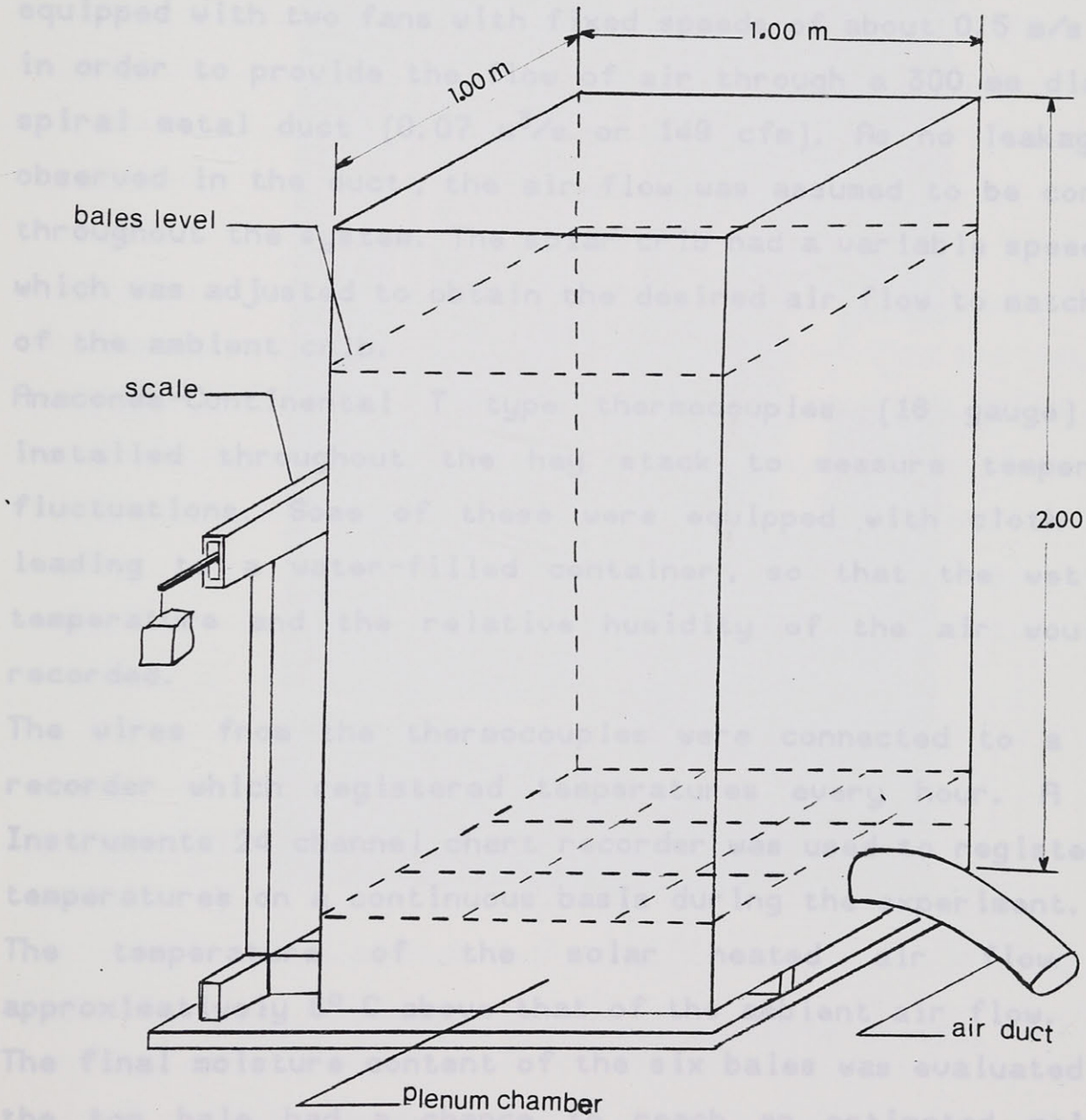


Figure 3. Detailed drawing of a typical experimental bin.

on a smooth circular duct. The instrument used was a TSI model 1000 hot wire anemometer. The tubing used from the orifice plates to the bin was a non-perforated corrugated plastic drainage tube 100 mm in diameter (Figure 4).

The air flow was identical in each box. The ambient crib was equipped with two fans with fixed speeds of about 0.5 m/s each, in order to provide the flow of air through a 300 mm diameter spiral metal duct (0.07 m^3/s or 149 cfm). As no leakage was observed in the duct, the air flow was assumed to be constant throughout the system. The solar crib had a variable speed fan, which was adjusted to obtain the desired air flow to match that of the ambient crib.

Anaconda-Continental T type thermocouples (16 gauge) were installed throughout the hay stack to measure temperature fluctuations. Some of these were equipped with cloth wicks leading to a water-filled container, so that the wet bulb temperature and the relative humidity of the air would be recorded.

The wires from the thermocouples were connected to a chart recorder which registered temperatures every hour. A Texas Instruments 24 channel chart recorder was used to register the temperatures on a continuous basis during the experiment.

The temperature of the solar heated air flow were approximatively 6° C above that of the ambient air flow.

The final moisture content of the six bales was evaluated once the top bale had a chance to reach an estimated moisture content of less than 20 percent.

Samples were taken in order to evaluate the dry matter and the quality of the dried hay.

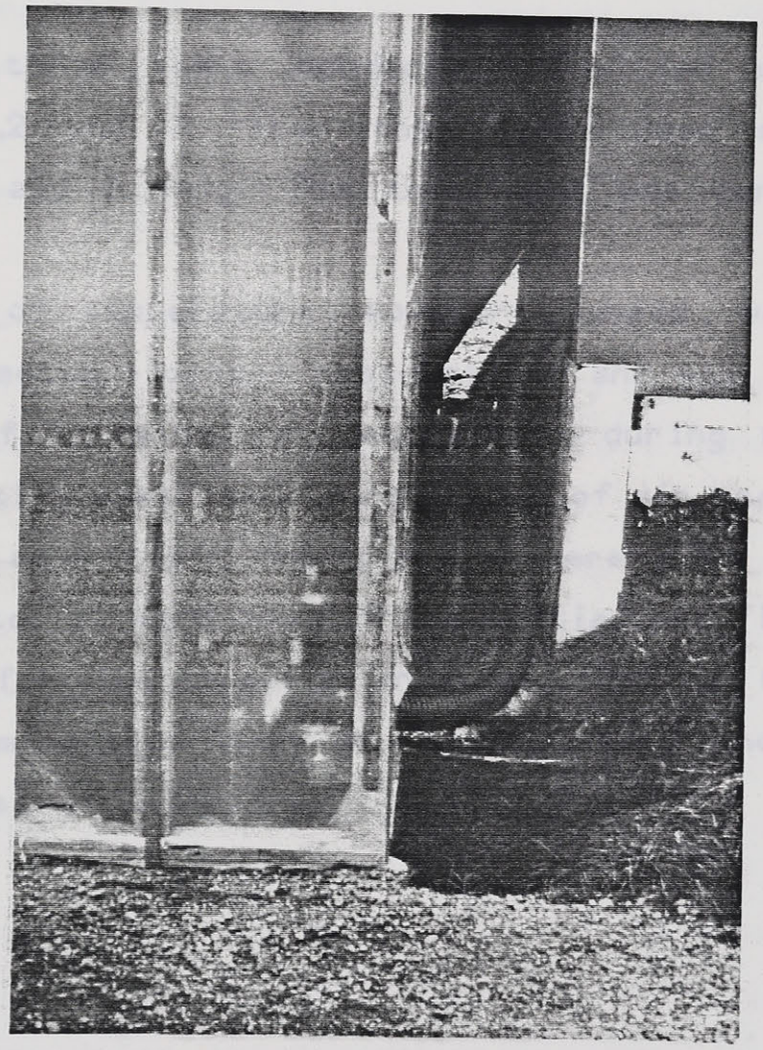


Figure 4. Air ducts connected to driers.

As mentioned above, the treatments are as follows :

1) Field curing
2) Ambient, forced air drying
3) Solar heated, forced air drying.

One of the three tests conducted, included samples from treatments 1,2 and 3. The other tests used samples from treatments 2 and 3 only. The experiment was carried out 24 hours a day.

The readings of temperature, moisture losses, variations in temperature, meteorological observations and wet and dry bulb temperature of incoming air, were taken during the day time period. The daily measurements consisted of air speeds in entry ducts. After each test, measurements were done for the air speed at the top of each bin, of the initial and final moisture content and for the quality analysis. Before the chemical analysis, examinations such as visual, smell and touch were done on the set of bales.

B. Procedure

The samples taken from different drying methods were used for the chemical analysis. The methods used were standardized and were taken from the book "Official methods of analysis of the Association of Official Analytical Chemist" (13th edition, 1980 ; Reference 2).

1) Sampling :

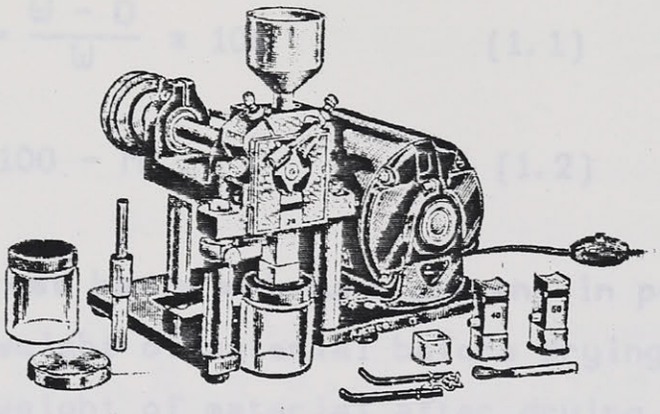
For purposes of determining forage quality, three replicates of samples collected were analysed for crude protein and acid detergent fiber. Moisture content was determined for each sample prior to subdivision.

2) Preparation of sample :

A small quantity was obtained from a batch. The sample was chopped, ground in a Wiley mill (Figure 5) and mixed.
The sample was ground small enough to fall through a 1 mm. diameter hole in a sieve. This speeds up the experiment. The Wiley mill was recommended because the sample is subjected to a minimum of heating during grinding, and because it is isolated from the contact of atmosphere.

3) Moisture content or dry matter (MC or DM) :

A small quantity (less than 10 g) of the prepared sample was weighed and placed into an appropriate container (aluminium dishes). It was dried in an oven until there was no further weight loss (at 60° C for a period of 24 hours). An alternative method of determining the absolute moisture content could have been done using a vacuum oven.
After heating, it was cooled for 20 to 30 minutes in a dessicator and reweighed (Figure 6).
Handling of dishes was done with tongs to avoid errors introduced through direct human contact.



Courtesy A. H. Thomas Co.

Figure 5. Picture of a Wiley Mill.

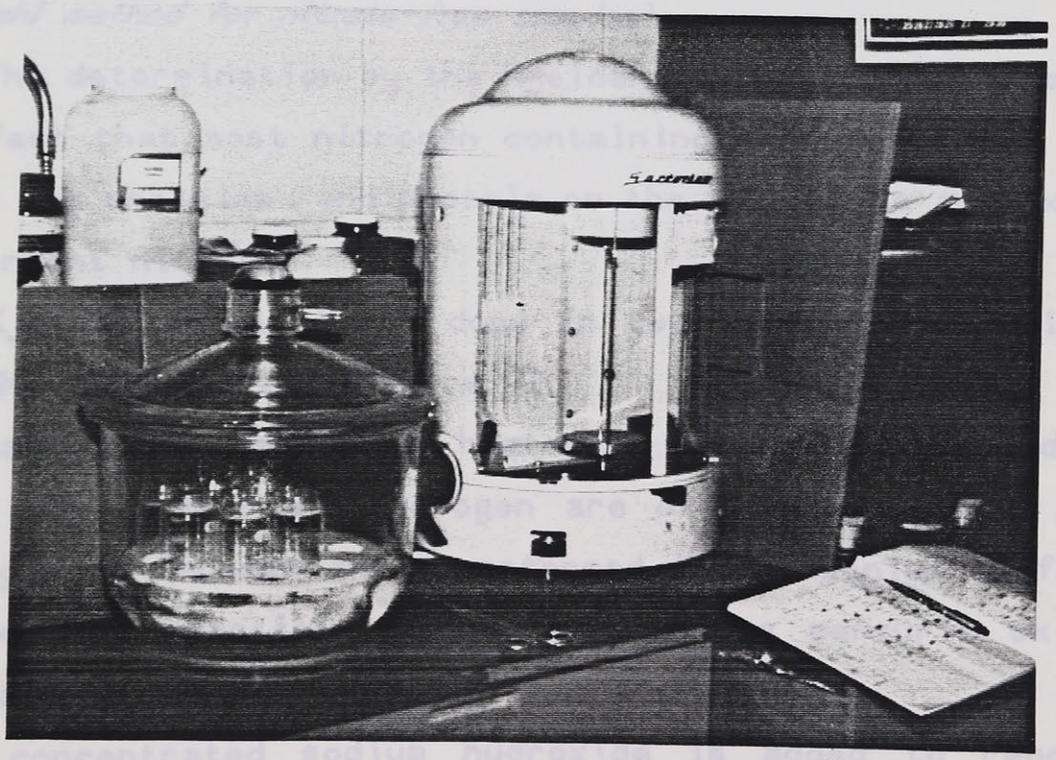


Figure 6. Dessicator and balance.

The percent of moisture on a wet basis (wb) was calculated :

$$MC_{wb} = \frac{W - D}{W} * 100 \qquad (1.1)$$

$$DM = 100 - MC_{wb} \qquad (1.2)$$

where MC_{wb} = wet basis moisture content in percent.
W = weight of material before drying in grams.
D = weight of material after drying in grams.
DM = percent dry matter.

4) Crude protein (CP) : AOAC section 2.049 (see Appendix).

Refers to the section of reagents, apparatus and procedure (by improved Kjeldahl method for nitrate-free samples).

The determination by the Kjeldahl method (1883) is based on the fact that most nitrogen containing macromaterials in most feeds are proteins, and protein on the average is approximately 16 percent nitrogen.

The Kjeldahl analysis is done in two steps : H_2SO_4 digestion and NH_3 distillation (Figure 7).

Basically, the samples are heated in sulfuric acid and digested until the carbon and hydrogen are oxidized, and the protein nitrogen is reduced and transformed into ammonium sulfate. To shorten the reaction, catalysts, such as mercuric oxide and potassium sulfate are used.

Then concentrated sodium hydroxide is added to remove the ammonium by making the solution alkaline.

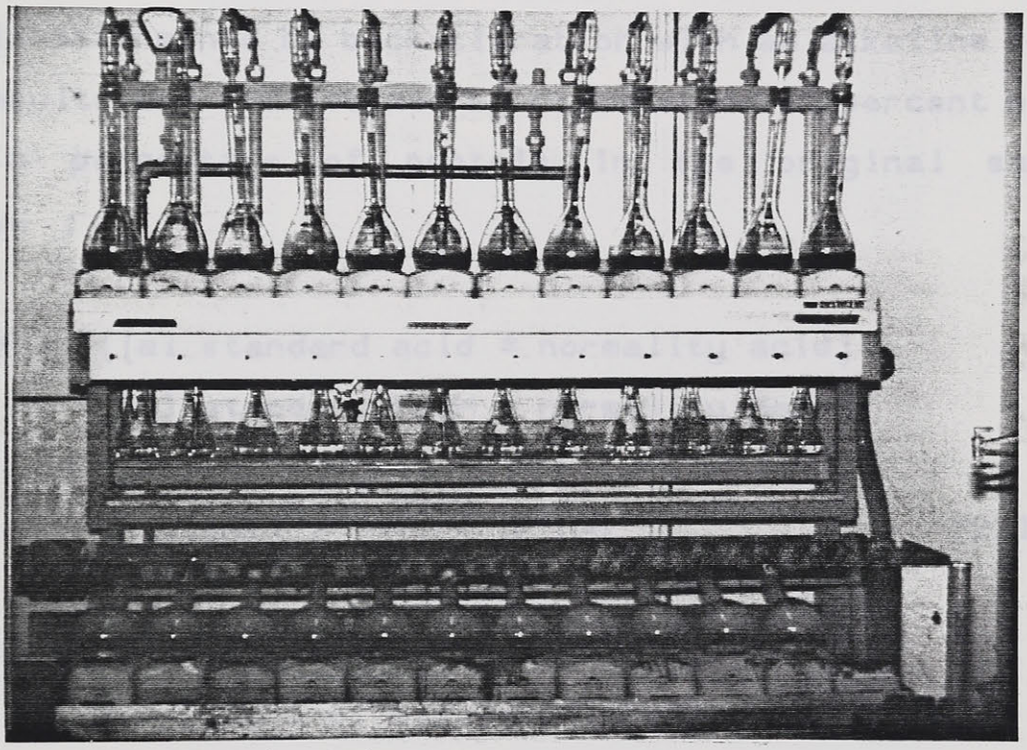


Figure 7. Kjeldahl rack for nitrogen determination in full operation.

During digestion, mercury salts formed a complex with the ammonium. Sodium thiosulfate is therefore added to convert mercury salts into mercury and prevented the ammonium from reacting with salts.

After adding the two solutions mentioned above, the mixture is heated to drive off the liberated ammonia into a known volume of a standard acid solution during distillation. The unreacted acid is determined by back-titration with an alkaline solution. The results are transformed by calculation of percent nitrogen, into a percentage of protein in the original sample as follows :

$$N = (((\text{ml standard acid} * \text{normality acid}) - (\text{ml standard NaOH} * \text{normality NaOH})) - N\ \text{blank}) * \frac{1.4007}{W} * 100 \qquad (2.1)$$

where N = percent nitrogen.

W = weight of sample in grams.

N blank is a control since reagents may contain nitrogen.

$$CP = N * \frac{100}{16} \qquad (2.2)$$

This percent crude protein is a wet basis or a fresh weight basis. The percent crude protein by dry weight basis (100% DM) was found as follows :

$$\frac{\%CP * 100}{\%DM \text{ of sample}} \qquad (2.3)$$

This crude protein is a mixture of true protein and non protein nitrogen.

CHEMICAL REACTIONS :

- digestion

$$N \text{ (in the sample)} + H_2SO_4 \xrightarrow[\text{catalysts}]{} (NH_4)_2SO_4$$

- distillation

$$(NH_4)_2SO_4 \xrightarrow[\text{heat}]{} NH_3 \uparrow$$

$$NH_3 + H_2O + HCl \longrightarrow NH_4Cl + H_2O$$

5) Acid detergent fiber (ADF) : AOAC section 7.055 (see Appendix)

An aqueous solution of 1N H_2SO_4 and 2 percent of cethyltrimethyl ammonium bromide is used to extract proteins, lipids, some type of carbohydrates (starches, soluble sucrose) and some structural carbohydrates (hemicellulose). After refluxing for one hour, the solution is filtrated and the

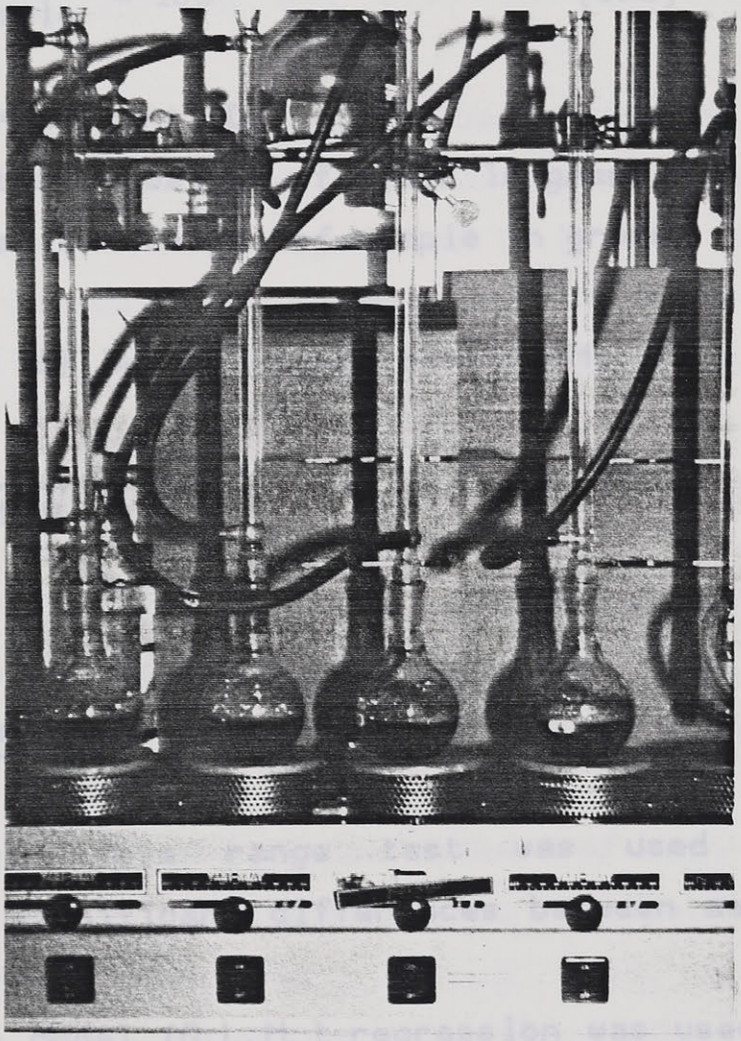


Figure 8. Acid detergent fiber refluxing apparatus.

residue left, consisted of lignin and cellulose, and materials chemically bound to both (Figure 8).
The procedure was found by Van Soest (1964).

$$ADF = \frac{W\ fiber}{W\ sample} * 100 \qquad (3.1)$$

where ADF = percent ADF
W fiber = weight of fiber in grams.
W sample = weight of sample in grams.

The percent of ADF by dry weight basis (DM) :

$$\frac{\%ADF}{\%DM\ of\ sample} * 100 \qquad (3.2)$$

C. Statistical Analysis

Duncan's multiple range test was used to identify statistically significant differences between means at the 5 percent level.
General Linear Model (G.L.M.) regression was used to find the correlation between protein and acid detergent fiber.

VI. RESULTS AND DISCUSSION

Bales for each test were taken from the field, representing one cutting and with an equivalent stage of maturity.

After the visual examination of the bales, it was found to have no apparent difference. However, with the samples taken from the field curing test, the hay seemed to be more moist and less colored. With the bales taken from the two artificial methods of drying, the hay had a bright green color and a good aroma.

1) Test # 1

The results of the first test, which incorporated samples from the three methods, are shown in TABLE 1.

To bring the moisture content of alfalfa down to 12 percent took six days of drying in each method.

It was found that the dry matter content of hay, obtained from the two forced ventilation systems, to be significantly higher than that of field cured hay.

As seen from TABLE 1, an increase in crude protein and a decrease in acid detergent fiber contents are noted for the solar heated air drying method over the other two methods. However, this increase is not statistically significant at a 95 percent confidence interval.

Since a high proportion of protein is present in the leafy region of the alfalfa plant and a high proportion of fiber

present in the stem, a linear relationship is feasible. Therefore, the amount of protein and fiber present in the leaves and the stems respectively can be predicted.

2) Test # 2

From the second test, as shown in TABLE 2, statistical significant difference between the drying methods, is noted in dry matter, crude protein and acid detergent fiber. The dry matter is higher with heated air drying, whereas crude protein is lower and fiber higher as compared with ambient air drying. In terms of quality, the best result was found in the samples of ambient air drying, which had a slightly higher protein content and a lower fiber content than that of samples from the heated air drying. To reach a moisture content of approximately 11 percent, it took 15 days of drying.

3) Test # 3

From the third test, as shown in TABLE 3, no significant difference between the drying methods is noted in the crude protein content. However, a statistical difference is seen between the two drying methods, in acid detergent fiber and in dry matter. The ADF content is higher and the DM lower for the method using ambient air drying than that of the heated air drying. After nine days of drying, the final moisture content reached approximately nine percent.

TABLE 1. Summary of Results from the First Test.
Drying process from 2th to 8th August 1983.

Early bloom alfalfa hay	Forced ambient air	Forced heated air	Field curing
Drying time (day)	7	7	7
Ave. air flow rate (m^3/sec)	0.07	0.07	
Ave. initial MC (% wet basis)	28	28	28
Water removal (kg)	5	5	
Final MC (% wb)	9.9	10.0	12.3
Dry matter (%)	90.1	90.0	87.7ab*
Crude protein (% DM)	17.3	19.2	16.5
Acid detergent fiber (% DM)	40.0	39.3	45.0

* ab : letters denote significance at 5 percent level using Duncan's new multiple-range test. Means followed by the two letters differ significantly.

TABLE 2. Summary of Results from the Second Test.
Drying process from 15th August to 1st Sept. 1983.

Early bloom alfalfa hay	Forced ambient air	Forced heated air
Drying time (day)	15	15
Ave. air flow rate (m^3/sec)	0.07	0.07
Ave. initial MC (% wet basis)	23	23
Water removal (kg)	10.9	15.4
Final MC (% wb)	10.6	8.9
Dry matter (%)	89.4ab*	91.1ab*
Crude protein (% DM)	15.5ab*	14.3ab*
Acid detergent fiber (%DM)	39.9ab*	43.9ab*

* ab : letters denote significance at 5 percent level using Duncan's new multiple-range test. Means followed by the two letters differ significantly.

TABLE 3. Summary of Results from the Third Test.
Drying process from 6th to 15th Sept. 1983

Early bloom alfalfa hay	Forced ambient air	Forced heated air
Drying time (day)	9	9
Ave. air flow rate (m^3/sec)	0.07	0.07
Ave. initial MC (% wet basis)	34	34
Water removal (kg)	26.3	33.6
Final MC (% wb)	8.6	7.0
Dry matter (%)	91.4ab*	93.0ab*
Crude protein (% DM)	19.1	20.8
Acid detergent fiber (%DM)	33.1ab*	28.3ab*

* ab : letters denote significance at 5 percent level using Duncan's new multiple-range test. Means followed by the two letters differ significantly.

4) Comparison with previous experiments

According to previous research done on the use of artificial drying as opposed to field curing, the benefits gained in terms of quality, are not questionable.

In this project, only one test with field curing was done. This was not sufficient however, since it did not corroborate with previous results done on this topic. The test, comparing hay quality from the field curing and artificial drying methods, did not give any indication of an advantage in using artificial drying. This could be due to favorable conditions in the field curing test. Mechanical handling of the samples, from each of the three drying methods, was done similarly (care was taken when collecting the sample). This reduced the problem of leaves shattering, which usually occurs under normal conditions when the hay is collected from the field.

Research done by Strait (1944) and other researchers have found that drying with the aid of heated air by fuel created an evident increase in the quality of hay. This advantage would be magnified by periods of high humidity in the air.

Air heated by solar energy can not be used during times of bad weather. The system would operate like the one of ambient temperature drying. The effect of the forced air, in this case, would only be used to reduce the respiration of the hay and the enzyme action.

Whatever the atmospheric conditions, if the bales are handled similarly before drying, any additional heat in the forced ventilation system would have minimal effect on protein and

fiber contents. This is substantiated by the fact that protein and fiber are affected by the proportion of leaves and stems, rather than by the chemical reaction of the hay alone.

On the aspect of using a particular drying method to achieve the best quality of alfalfa hay, no conclusions can be drawn from the results of the three tests performed in this study. Since these tests were carried out in the month of August 1983, few cuttings of alfalfa were possible. In this experiment only three cuttings were performed. They are not representative of a full growing season as hay is primarly affected by the atmospheric conditions. This can be shown by the results obtained in the three tests. For this reason, it would be best to suggest a number of ideas to help with future research on this subject. This will enable reseachers to achieve the given objective of comparing hay quality as affected by different drying methods.

VII. CONCLUSION

The results from the first test did not show any difference in the quality of alfalfa hay between the three drying methods. Only the dry matter was found to be lower in the field cured hay as compared to the hay obtained from the two artificial driers. The second test showed a better quality in using the unheated drier compare to that of a heated air drier. In the third test, no difference in crude protein between the driers was noted, but the use of solar heated air in the drying process gave better results in terms of acid detergent fiber and dry matter (low ADF, high DM).

It is difficult to conclude, with the given results, which drying method will produce the best quality hay. Further research is required in order to achieve the objective. Supplemental tests must be conducted on other nutrients and over a number of haying seasons.

VIII. RECOMMENDATIONS

1) Sampling

The first recommendation would be to have more than three replicates of samples for each of the three drying methods.

2) Other nutrients determination

Some other nutrients, which also play an important role in determining the quality of hay, should be analyzed to see the effect of forced heated air in the drying process.

- Vitamin A and carotene

Vitamin A or carotene, is one of the nutrients which is a good index in determining hay quality. The amount present is also affected by drying. In this study, no evaluation of vitamin A was done. This was not done since its determination requires sophisticated apparatus and numerous reagents. Due to time restrictions, it was not possible to conduct these tests. As well, a lack of financial assistance restricted the possibility of sending samples to a commercial laboratory for testing. Costs for testing vitamin A are $ 40 and for carotene $ 30 per sample, as stated by the department of Animal Science of Macdonald College. A high carotene content usually means that the hay is a leafy legume hay, subjected to a minimum loss

of leaves. It has been cured to a dryness that has effectively stopped or greatly reduced respiration, enzyme action and fermentation. As a result, not only the carotene but also the other vitamins and the proteins, carbohydrates, fat and ash are well preserved.

The plant enzyme, carotenase, destroys carotene under conditions of sufficient moisture and temperature, during storage.

Thus, artificially dried alfalfa hay contains a minimal amount of enzymes. Due to the destruction of most of the enzymes, only about one fourth of the initial carotene, during a six months storage period, is lost. In comparison to sun cured alfalfa hay, losses of carotene frequently amount to half of the initial carotene value, during the same storage period (Bohstedt, 1944).

- Nitrogen free extract

The nitrogen free extract (NFE) measurements are an indication of the amount of carbohydrates present in the samples. The loss in NFE is probably due to continued respiration by the plant cells and fermentation by the bacteria, yeasts or mold (Wood and Parker, 1971).

- Minerals and other vitamins

The effect of drying on calcium, phosphorous and other vitamins can also be evaluated. However, if these nutrients are

not present in sufficient amounts, they can be provided by other sources in animal ration.

3) Feeding trials and test for milk production

The last interesting analysis would be to see the effectiveness of drying methods from feeding trials with animals. These would evaluate the digestibility of alfalfa hay using different drying methods. The quantity of milk production from cows should also be recorded after feeding a ration of hay dried by different methods.

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X. APPENDICES

A. Results of statistical analysis

1) Test #1

TABLE 4.1 Analysis of Variance for DM.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	2	10.254	5.13	246.88	0.0001	0.988	0.161
Error	6	0.125	0.02		Std.Dev.		Mean
Cor.tot.	8	10.378			0.144		89.29

Source	DF	Anova SS	F	PR > F
Class	2	10.25	246.88	0.0001

Test #1 continued

TABLE 4.2 Analysis of Variance for CP.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	2	12.043	6.02	1.57	0.2838	0.343	11.105
Error	6	23.086	3.85		Std.Dev.		Mean
Cor.tot.	8	35.129			1.961		17.66

Source	DF	Anova SS	F	PR > F
Class	2	12.04	1.57	0.2838

Test #1 continued

TABLE 4.3 Analysis of Variance for ADF.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	2	52.188	26.09	1.86	0.2491	0.426	8.959
Error	5	70.182	14.04		Std. Dev.		Mean
Cor. tot.	7	122.370			3.747		41.82

Source	DF	Anova SS	F	PR > F
Class	2	52.19	1.86	0.2491

Test #1 continued

TABLE 4.4 GLM Procedure for CP vs ADF.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Regres.	1	25.684	25.68	194.52	0.0001	0.970	2.018
Error	6	0.792	0.13		Std. Dev.		Mean
Cor. tot.	7	26.476			0.363		18.01

Parameter	Estimate	PR > T	Std. error
Intercept	37.17	0.0001	1.38
ADF	-0.46	0.0001	0.03

2) Test #2

TABLE 5.1 Analysis of Variance for DM.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	1	4.301	4.30	7590.12	0.0001	0.999	0.026
Error	4	0.002	0.00		Std. Dev.		Mean
Cor. tot.	5	4.303			0.024		90.27

Source	DF	Anova SS	F	PR > F
Class	1	4.30	7590.12	0.0001

Test #2 continued

TABLE 5.2 Analysis of Variance for CP.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	1	2.124	2.12	11.12	0.0290	0.736	2.932
Error	4	0.764	0.19		Std. Dev.		Mean
Cor. tot.	5	2.888			0.437		19.90

Source	DF	Anova SS	F	PR > F
Class	1	2.12	11.12	0.0290

Test #2 continued

TABLE 5.3 Analysis of Variance for ADF.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	1	20.758	20.76	25.04	0.0075	0.862	2.181
Error	4	3.316	0.83		Std.Dev.		Mean
Cor.tot.	5	24.074			0.911		41.75

Source	DF	Anova SS	F	PR > F
Class	1	20.76	25.04	0.0075

Test #2 continued

TABLE 5.4 GLM Procedure for CP vs ADF.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Regres.	1	2.513	2.51	26.78	0.0066	0.870	2.055
Error	4	0.375	0.09		Std. Dev.		Mean
Cor. tot.	5	2.888			0.306		14.90

Parameter	Estimate	PR > T	Std. error
Intercept	28.39	0.0004	2.61
ADF	-0.32	0.0066	0.06

3) Test #3

TABLE 6.1 Analysis of Variance for DM.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	1	3.330	3.33	45.23	0.0025	0.919	0.295
Error	4	0.294	0.07		Std.Dev.		Mean
Cor.tot.	5	3.625			0.271		92.12

Source	DF	Anova SS	F	PR > F
Class	1	3.33	45.23	0.0025

Test #3 continued

TABLE 6.2 Analysis of Variance for CP.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	1	4.420	4.42	4.81	0.0935	0.546	4.806
Error	4	3.679	0.92		Std. Dev.		Mean
Cor. tot.	5	8.099			0.959		19.96

Source	DF	Anova SS	F	PR > F
Class	1	4.42	4.81	0.0935

Test #3 continued

TABLE 6.3 Analysis of Variance for ADF.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Model	1	34.082	34.08	10.97	0.0296	0.733	5.743
Error	4	12.431	3.12		Std. Dev.		Mean
Cor. tot.	5	46.513			1.763		30.69

Source	DF	Anova SS	F	PR > F
Class	1	34.08	10.97	0.0296

Test #3 continued

TABLE 6.4 GLM Procedure for CP vs ADF.

Source	DF	Sum of sq.	Mean sq.	F	PR > F	R^2	CV
Regres.	1	7.244	7.24	33.87	0.0043	0.894	2.318
Error	4	0.856	0.21		Std. Dev.		Mean
Cor. tot.	5	8.099			0.462		19.95

Parameter	Estimate	PR > T	Std. error
Intercept	32.07	0.0001	2.09
ADF	-0.39	0.0043	0.07

B. Sections from AOAC

(b) *Nonacidulated samples.*—Place 1 g sample (ground to pass No. 40 sieve in case of Ca metaphosphate) on dry 9 cm paper. Without previous washing with H_2O, proceed as in (a)(*1*) or (*2*). If (*2*) is used, wash residue until vol. soln is ca 350 mL. Cool, dil. to 500 mL, and mix.

2.051 *Alkalimetric Quinolinium Molybdophosphate Method (16)—Official Final Action*

Treat 1 g sample by appropriate modification of 2.050. Transfer aliquot contg ≤30 mg P_2O_5 and ≤10 mL NH_4 citrate soln, 2.044(a), to 500 mL erlenmeyer. Dil., if necessary, to 50 mL, add 10 mL HNO_3 (1+1), and boil gently 10 min. Cool, dil. to 100 mL, and continue as in 2.031(a), beginning "Add 60 mL quimociac reagent, . . ."

2.052 *Spectrophotometric Molybdovanadophosphate Method (18)—Official Final Action*

(Not applicable to materials yielding colored solns or solns contg ions other than orthophosphate which form colored complexes with molybdovanadate. Not recommended for basic slag.)

Prep. std curve as in 2.023, using photometer, 2.021.

Pipet, into 100 mL vol. flasks, 5 mL aliquots std phosphate solns contg 2 and 3.5 mg P_2O_5/aliquot, 2.022(b), resp., add 2 mL 70% $HClO_4$, and develop color as in 2.023. Adjust instrument to zero *A* for 2 mg std and det. *A* of 3.5 mg std. (*A* of latter must be practically identical with corresponding value on std curve.)

Prep. sample as in 2.050.

(a) *Samples containing up to 5% P_2O_5.*—Pipet 10 mL sample soln into 125 mL erlenmeyer, and treat by one of following methods (*Caution: See* 51.019, 51.026, and 51.028):

(*1*) Add 5 mL 20% $NaClO_3$ soln and 10 mL HNO_3-$HClO_4$ mixt., 2.049(a). Boil gently until greenish-yellow color disappears (ca 20 min), cool, and add 2 mL HCl. After vigorous reaction subsides, evap. to fumes of $HClO_4$, and fume 2 min.

(*2*) Add 5 mL ternary acid mixt., 2.049(b), swirl, boil gently 15 min, and digest at 150–200° until clear white salt or colorless soln remains. Evap. to white fumes and continue heating 5 min.

Cool, add 15 mL H_2O, and boil 5 min. Transfer to 100 mL vol. flask, dil. to 50 ml, swirl, and cool to room temp. Add 5 mL std phosphate soln contg 2 mg P_2O_5 and 20 mL modified molybdovanadate soln, 2.049(c). Dil. to 100 mL, and continue as in 2.025(a).

(b) *Samples containing more than 5% P_2O_5.*—Dil. soln to such vol. that 5–10 mL aliquot contains 2–5 mg P_2O_5. Digest as in (a)(*1*) or (*2*). Without adding std phosphate soln, continue as in (a).

2.053 *Gravimetric Quinolinium Molybdophosphate Method (19)—Official Final Action*

(a) *Solns containing no organic phosphorus.*—Prep. sample as in 2.050. Pipet, into 500 mL erlenmeyer, aliquot contg ≤25 mg P_2O_5 and ≤10 mL original NH_4 citrate soln. Dil., if necessary, to ca 50 mL, add 10 mL HNO_3 (1+1), and boil gently 10 min. Cool, dil. to 150 mL, and proceed as in 2.028(a) or (b).

(b) *Solns containing organic phosphorus.*—(*Caution: See* 51.019, 51.026, and 51.028.) Select aliquot as in (a). Add 10 mL 20% $NaClO_3$ and 10 mL HNO_3-$HClO_4$ mixt., 2.049(a). Boil vigorously until greenish-yellow color disappears (usually ca 30 min), cool, and add 2 mL HCl. After vigorous reaction subsides, evap. to white fumes, and continue heating 5 min. Cool, and proceed as in 2.028(a) or (b).

NITROGEN

2.054 Detection of Nitrates—Official Final Action

Mix 5 g sample with 25 mL hot H_2O, and filter. To 1 vol. of this soln add 2 vols H_2SO_4, free from HNO_3 and oxides of N, and let cool. Add few drops *concd $FeSO_4$ soln* in such manner that fluids do not mix. If nitrates are present, junction at first shows purple, afterwards brown, or if only minute amt is present, reddish color. To another portion of soln add 1 mL *1% $NaNO_3$ soln* and test as before to det. whether enough H_2SO_4 was added in first test.

Total Nitrogen

(Provide adequate ventilation in laboratory and do not permit accumulation of exposed Hg.)

2.055 *Reagents—Official Final Action*

(a) *Sulfuric acid.*—93–98% H_2SO_4, N-free.

(b) *Mercuric oxide or metallic mercury.*—HgO or Hg, reagent grade, N-free.

(c) *Potassium sulfate (or anhydrous sodium sulfate).*—Reagent grade, N-free.

(d) *Salicylic acid.*—Reagent grade, N-free.

(e) *Sulfide or thiosulfate soln.*—Dissolve 40 g com. K_2S in 1 L H_2O. (Soln of 40 g Na_2S or 80 g $Na_2S_2O_3.5H_2O$ in 1 L may be used.)

(f) *Sodium hydroxide.*—Pellets or soln, nitrate-free. For soln, dissolve ca 450 g solid NaOH in H_2O, cool, and dil. to 1 L. (Sp gr of soln should be ≥1.36.)

(g) *Zinc granules.*—Reagent grade.

(h) *Zinc dust.*—Impalpable powder.

(i) *Methyl red indicator.*—Dissolve 1 g Me red in 200 mL alcohol.

(j) *Hydrochloric or sulfuric acid std soln.*—0.5*N*, or 0.1*N* when amt of N is small. Prep. as in 50.011 or 50.039.

(k) *Sodium hydroxide std soln.*—0.1*N* (or other specified concn). Prep. as in 50.032–50.034.

Stdze each std soln with primary std, Chap. 50, and check one against the other. Test reagents before use by blank detn with 2 g sugar, which ensures partial reduction of any nitrates present.

Caution: Use freshly opened H_2SO_4 or add dry P_2O_5 to avoid hydrolysis of nitriles and cyanates. Ratio of salt to acid (wt:vol.) should be ca 1:1 at end of digestion for proper temp. control. Digestion may be incomplete at lower ratio; N may be lost at higher ratio. Each g fat consumes 10 mL H_2SO_4, and each g carbohydrate 4 mL H_2SO_4 during digestion.

2.056 *Apparatus—Official Final Action*

(a) *For digestion.*—Use Kjeldahl flasks of hard, moderately thick, well-annealed glass with total capacity ca 500–800 mL. Conduct digestion over heating device adjusted to bring 250 mL H_2O at 25° to rolling boil in ca 5 min or other time as specified in method. To test heaters, preheat 10 min if gas or 30 min if elec. Add 3–4 boiling chips to prevent superheating.

(b) *For distillation.*—Use 500–800 mL Kjeldahl or other suitable flask, fitted with rubber stopper thru which passes lower end of efficient scrubber bulb or trap to prevent mech. carryover of NaOH during distn. Connect upper end of bulb tube to condenser tube by rubber tubing. Trap outlet of condenser in such way as to ensure complete absorption of NH_3 distd over into acid in receiver.

057 ***Improved Kjeldahl Method for Nitrate-Free Samples (20)—Official Final Action***

(*Caution: See* **51.030** and **51.065.**)

Place weighed sample (0.7–2.2 g) in digestion flask. Add 0.7 HgO or 0.65 g metallic Hg, 15 g powd K_2SO_4 or anhyd. Na_2SO_4, nd 25 mL H_2SO_4. If sample >2.2 g is used, increase H_2SO_4 by 10 L for each g sample. Place flask in inclined position and heat ently until frothing ceases (if necessary, add small amt of araffin to reduce frothing); boil briskly until soln clears and en ≥30 min longer (2 hr for samples contg org. material).

Cool, add ca 200 mL H_2O, cool <25°, add 25 mL of the sulfide thiosulfate soln, and mix to ppt Hg. Add few Zn granules to revent bumping, tilt flask, and add layer of NaOH without gitation. (For each 10 mL H_2SO_4 used, or its equiv. in dild H_2SO_4, dd 15 g solid NaOH or enough soln to make contents strongly k.) (Thiosulfate or sulfide soln may be mixed with the NaOH oln before addn to flask.) Immediately connect flask to distg ulb on condenser, and, with tip of condenser immersed in std cid and 5–7 drops indicator in receiver, rotate flask to mix ontents thoroly; then heat until all NH_3 has distd (≥150 mL istillate). Remove receiver, wash tip of condenser, and titr. xcess std acid in distillate with std NaOH soln. Correct for blank etn on reagents.

$$\% N = [(mL\ std\ acid \times normality\ acid) - (mL\ std\ NaOH \times normality\ NaOH)] \times 1.4007/g\ sample$$

058 ***Improved Kjeldahl Method for Nitrate-Containing Samples—Official Final Action***

(Not applicable to liqs or to materials with high $Cl:NO_3$ ratio. *Caution: See* **51.030** and **51.065.**)

Place weighed sample (0.7–2.2 g) in digestion flask. Add 40 L H_2SO_4 contg 2 g salicylic acid. Shake until thoroly mixed and t stand, with occasional shaking, ≥30 min; then add (*1*) 5 g $a_2S_2O_3.5H_2O$ or (*2*) 2 g Zn dust (as impalpable powder, not anulated Zn or filings). Shake and let stand 5 min; then heat ver low flame until frothing ceases. Turn off heat, add 0.7 g gO (or 0.65 g metallic Hg) and 15 g powd K_2SO_4 (or anhyd. a_2SO_4), and boil briskly until soln clears, then ≥30 min longer hr for samples contg org. material).

Proceed as in second par. of **2.057.**

Comprehensive Nitrogen Method (21)
Official Final Action

(Applicable to all fertilizer samples. *Caution: See* **51.030** and **51.079.**)

059 ***Reagents***

(a) *Chromium metal.*—100 mesh, low N (Fisher Scientific Co.). C-318 or Sargent-Welch Scientific Co. No. SC11432 is satis- tory).

(b) *Alundum.*—Boiling stones. 8–14 mesh (Arthur H. Thomas . No. 1590–D18, or equiv.).

(c) *Dilute sulfuric acid.*—Slowly add 625 mL H_2SO_4 to 300 mL O. Dil. to ca 1 L and mix. After cooling, dil. to 1 L with H_2O and x. Avoid absorption of NH_3 from air during prepn, particularly tream of air is used for mixing.

(d) *Sodium thiosulfate or potassium sulfide soln.*—160 g $_2S_2O_3.5H_2O$/L or 80 g K_2S/L.

or other reagents, *see* **2.055.**

060 ***Determination***

lace 0.2–2.0 g sample contg ≤60 mg nitrate N in 500–800 mL dahl flask and add 1.2 g Cr powder. Add 35 mL H_2O or, with , amt to make total vol. 35 ml. Let stand 10 min with occasional gentle swirling to dissolve all nitrate salts. Add 7 mL HCl and let stand ≥30 sec but ≤10 min.

Place flask on preheated burner with heat input set at 7.0–7.5 min boil test, **2.056**(a). After heating 3.5 min, remove from heat and let cool.

Add 22 g K_2SO_4, 1.0 g HgO, and few granules Alundum. Add 40 mL dil. H_2SO_4, (c). (If adequate ventilation is available, 25 mL H_2SO_4 may be added instead of dil. H_2SO_4. If org. matter which consumes large amt of acid exceeds 1.0 g, add addnl 1.0 mL H_2SO_4 for each 0.1 g org. matter in excess of 1.0 g.)

Place flask on burners set at 5 min boil test. (Pre-heated burners reduce foaming with most samples. Reduce heat input if foam fills ≥⅔ of bulb of flask. Use variable heat input until this phase is past.) Heat at 5 min boil test until dense white fumes of H_2SO_4 clear bulb of flask. Digestion is now complete for samples contg ammoniacal, nitrate, and urea N. For other samples, swirl flask gently and continue digestion 60 min more.

Proceed as in **2.057**, second par., substituting **2.059**(d) for **2.055**(e).

Modified Comprehensive Nitrogen Method (22)
Official First Action

(Applicable to all fertilizer samples)

2.061 ***Reagents***

See **2.055**(a), (c), (f), (i), (j), (k), **2.059**(a), (b), and in addn:

Copper sulfate pentahydrate (or anhydrous copper sulfate).—Reagent grade, N-free.

2.062 ***Determination***

(*Caution: See* **51.019** and **51.030.**)

Proceed as in **2.060**, par. 1 and 2, using 0.2–1.6 g sample. For samples contg orgs other than urea or urea-form, use ≥0.5 g sample.

Add 15 g K_2SO_4 or 12 g anhyd. Na_2SO_4, 0.4 g anhyd. $CuSO_4$ or 0.6 g $CuSO_4.5H_2O$, and ca 0.8 g Alundum granules. Add 37 mL H_2SO_4 (1+1). (If adequate ventilation is available, 20 mL H_2SO_4 may be added instead of H_2SO_4 (1+1). If org. matter other than urea exceeds 1.0 g, add addnl 1.0 mL H_2SO_4 for each 0.1 g fat or 0.2 g other org. matter in excess of 1.0 g.)

Proceed as in **2.060**, par. 4, substituting 75 min for 60 min in last sentence.

Cool flask until it can be handled without gloves, and add ca 250 mL H_2O. Swirl to dissolve contents, and cool <25°. Add ca 0.8 g Alundum granules to minimize bumping, tilt flask, and add layer of NaOH without agitation. (For each 10 ml H_2SO_4 used, or its equiv. in H_2SO_4 (1+1), add 15 g solid NaOH or enough soln to make contents strongly alk.) Proceed as in **2.057**, par. 2, beginning "Immediately connect flask to distg bulb . . ."

Raney Powder Method (21)
Official Final Action

(Applicable to all fertilizer samples except "nitric phosphates" contg nonsulfate S. *Caution: See* **51.030** and **51.079.**)

2.063 ***Reagents***

(a) *Raney catalyst powder No. 2813.*—50% Ni, 50% Al (W. R. Grace & Co., Davison Chemical Division, 10 E Baltimore St, Baltimore, MD 21203). *Caution:* Raney catalyst powders react slowly in H_2O or moist air to form alumina; avoid prolonged contact with air or moisture during storage or use.

(b) *Sulfuric acid-potassium sulfate soln.*—Slowly add 200 mL H_2SO_4 to 625 mL H_2O and mix. Without cooling, add 106.7 g

repeat with three 50 mL washings. (Work rapidly to keep mat from becoming dry.) Remove filter from beaker and drain all H_2O from line by raising above trap level. Return mat and residue to beaker by breaking suction and blowing back. Add 200 mL boiling 1.25% NaOH and boil exactly 30 min. Remove beaker, and filter as above. Without breaking suction, wash with 25 mL boiling 1.25% H_2SO_4 and three 50 mL portions boiling H_2O. Drain free of excess H_2O by raising filter. Lower filter into beaker and wash with 25 mL alcohol. Drain line, break suction, and remove mat by blowing back thru filter screen into ashing dish. Proceed as in (c).

(b) *Using California buchner.*—Filter contents of beaker thru buchner (precoated with asbestos if extremely fine materials are being analyzed), rinse beaker with 50–75 mL boiling H_2O, and wash thru buchner. Repeat with three 50 mL portions H_2O, and suck dry. Remove mat and residue by snapping bottom of buchner against top while covering stem with thumb or forefinger and replace in beaker. Add 200 mL boiling 1.25% NaOH and boil exactly 30 min. Remove beaker, and filter as above. Wash with 25 mL boiling 1.25% H_2SO_4, three 50 mL portions H_2O, and 25 mL alcohol. Remove mat and residue; transfer to ashing dish.

(c) *Treatment of residue.*—Dry mat and residue 2 hr at 130±2°. Cool in desiccator and weigh. Ignite 30 min at 600±15°. Cool in desiccator and reweigh.

% Crude fiber in ground sample = C = (Loss in wt on ignition − loss in wt of asbestos blank) × 100/wt sample.

% Crude fiber on desired moisture basis = C × (100 − % moisture desired)/(100 − % moisture in ground sample).

Report results to 0.1%.

Asbestos-Free (AF) Method (20)
Official Final Action

7.066 ***Principle***

Principle is same as in **7.061**, except sample is exposed to min. vac. needed to regulate filtration, and heating of sample solns prevents gelling or pptn of possible satd solns.

7.067 ***Apparatus and Reagents***

See reagents **7.062**(a), (b), and (f); app. **7.063**(a), (c), (d), and (f), and **14.088**; and in addn:

(a) *Filtration apparatus.*—System to permit application of min. vac. necessary for filtration and washing of each sample within 3–5 min. Each unit consists of reservoir manifold connected to (*1*) H_2O aspirator thru 120° stopcock, (*2*) atm. thru second stopcock with metering device, and (*3*) receptacle contg cone-shaped hard rubber gasket which provides vac. seal with crucible. Vac. gage attached to manifold indicates vac. applied to crucible. Crucible can be heated before and during filtration by flow of hot H_2O in surrounding jacket. (For photograph of app., *see* JAOAC **56**, 1353(1973). Filtration unit is available as Model 150 from Analytical BioChemistry Laboratories, Inc., PO Box 1097, Columbia, MO 65201.)

(b) *Crucible.*—Fritted glass, 50 mL, coarse porosity. Clean as follows: Brush, and flow hot tap H_2O into crucible to remove as much ash as possible. Submerge crucible in base soln, (c)(*2*), ≥5 min, remove, and rinse with hot tap H_2O. Submerge in HCl (1+1), (c) (*1*), ≥5 min, remove, and rinse thoroly with hot tap H_2O followed by distd H_2O. After 3–4 uses, back wash by inverting crucible on hard rubber gasket in filtration app., and flowing near-boiling H_2O thru crucible under partial vac.

(c) *Cleaning solns.*—(*1*) *Acid soln.*—HCl (1+1). (*2*) *Base soln.*—Dissolve 5 g Na_2H_2EDTA, 50 g Na_2HPO_4 (tech. grade), and 200 g KOH in H_2O, and dil. to 1 L. Storage in sep. wide mouth containers holding 2–3 L soln into which crucibles can be placed is convenient.

7.068 ***Determination***

(*Caution: See* **51.011** and **51.073**.)

Ext 2 g ground material with ether or pet ether, **14.088**. If fat is <1%, extn may be omitted. Transfer to 600 mL reflux beaker, avoiding fiber contamination from paper or brush. Add 0.25–0.5 g bumping granules, followed by 200 mL near-boiling 1.25% H_2SO_4 soln in small stream directly on sample to aid in complete wetting of sample. Place beakers on digestion app. at 5 min intervals and boil exactly 30 min, rotating beakers periodically to keep solids from adhering to sides. Near end of refluxing place California buchner, **7.063**(d), previously fitted with No. 9 rubber stopper to provide vac. seal, into filtration app., and adjust vac. to ca 25 mm Hg (735 mm pressure). At end of refluxing, flow near-boiling H_2O thru funnel to warm it; then decant liq. thru funnel, washing solids into funnel with min. of near-boiling H_2O. Filter to dryness, using 25 mm vac., and wash residue with four 40–50 mL portions near-boiling H_2O, filtering after each washing. Do not add wash to funnel under vac.; lift funnel from app. when adding wash.

Wash residue from funnel into reflux beaker with near-boiling 1.25% NaOH soln. Place beakers on reflux app. at 5 min intervals and reflux 30 min. Near end of refluxing, turn on filtration app., place crucible, (b), in app., and adjust vac. to ca 25 mm. Flow near-boiling H_2O thru crucible to warm it. (Keep near-boiling H_2O flowing thru jacket during filtration and washing.) At end of refluxing, decant liq. thru crucible and wash solids into crucible with min. of near-boiling H_2O. Increase vac. as needed to maintain filtration rate. Wash residue once with 25–30 mL near-boiling 1.25% H_2SO_4 soln, and then with two 25–30 mL portions near-boiling H_2O, filtering after each washing. (Filtering and washing takes ca 3–5 min/sample.) Do not add wash to crucible under vac.

Dry crucible with residue 2 hr at 130±2° or overnight at 110°, cool in desiccator, and weigh. Ash 2 hr at 550±10°, cool in desiccator, and weigh. Do not remove crucibles from furnace until temp. is ≤250°, as fritted disk may be damaged if cooled too rapidly.

% Crude fiber = Loss in wt on ignition × 100/wt sample.

Acid-Detergent Fiber and Lignin (*21*)
Official Final Action

(*Caution: See* **51.086**.)

7.069 ***Reagents***

(a) *Sulfuric acid.*—72% by wt. Stdze reagent grade H_2SO_4 to sp gr 1.634 at 20° or 24.00*N*: Add 1200 g H_2SO_4 to 440 mL H_2O in 1 L MCA vol. flask with cooling. Stdze to 1634 g/L at 20° by removing soln and adding H_2O or H_2SO_4 as required. (*Caution: See* **51.030**.)

(b) *Acid-detergent soln.*—Add 20 g cetyl trimethylammonium bromide (tech. grade) to 1 L 1.00*N* H_2SO_4, previously stdzd. Agitate to aid soln.

(c) *Asbestos.*—Place 100 g asbestos in 3 L flask contg 850 mL H_2O. Add 1.4 L H_2SO_4 (tech. grade), mix, and let cool 2 hr at room temp. Filter on large buchner and wash with H_2O. Resuspend mat in H_2O and pour into bag sewn from rectangle of fiberglass window screening, 14 × 18 mesh (bag should be ≥45 cm wide × 30 cm deep). Wash by immersion and agitation in partly filled sink to remove fine particles. Ash recovered asbestos 16 hr in 800° furnace. Store in dry form until use. Used asbestos may be rewashed, reashed, and reused. Com. prepd acid-washed asbestos is unsatisfactory unless treated with 72% H_2SO_4 and ashed at 800°.

7.070 ***Apparatus***

(a) *Refluxing apparatus.*—Any conventional app. suitable for crude fiber detns. Berzelius beakers (600 mL) and condensers made from 500 mL r-b flasks are also satisfactory.

(b) *Fritted glass crucibles.*—Use coarse porosity, 40–50 mL Pyrex crucible. Wash new crucibles and ash at 500°. Remove while still hot and place in 100° forced-draft oven ≥1 hr. Cool 15 min in desiccator over P_2O_5 or $Mg(ClO_4)_2$ and weigh in same order samples are to be weighed. Check balance 0 after each weighing if crucibles are still warm. Hold length of time from oven to balance pan as const as possible and always weigh crucibles in same order.

7.071 ***Determination of Acid-Detergent Fiber***

Weigh 1 g air-dried sample ground to pass 1 mm screen, or approx. equiv. amt wet material, into refluxing container. Add 100 mL acid-detergent soln at room temp.

Heat to boiling in 5–10 min; reduce heat to avoid foaming as boiling begins. Reflux 60 min from onset of boiling, adjusting boiling to slow, even level. Remove container, swirl, and filter thru weighed (W_1) fritted glass crucible, using min. suction. Increase vac. only as needed. Shut off vac. Break up filtered mat with rod and fill crucible ⅔ full with hot (90–100°) H_2O. Stir and let soak 15–30 sec. Dry with vac. and repeat H_2O washing, rinsing sides of crucible. Wash twice similarly with acetone.

Repeat acetone washings until no more color is removed, breaking up all lumps so that solv. wets all particles of fiber. Remove residual acetone with vac. Dry 3 hr or overnight in 100° forced-draft oven and weigh (W_2). Calc. % acid-detergent fiber = $100\ (W_2 - W_1)/S$, where S = g sample × g oven-dried matter/g air-dried or wet matter, detd on sep. sample.

7.072 ***Determination of Lignin***

To crucible contg fiber, **7.071**, add 1 g asbestos. Place crucible in 50 mL beaker for support or arrange crucibles in shallow enamel pan. Cover contents of crucible with cooled (15°) 72% H_2SO_4 and stir with glass rod to smooth paste, breaking all lumps. Fill crucible about half-way with acid and stir. Leave glass rod in crucible; refill with 72% H_2SO_4 and stir hourly as acid drains, keeping crucible at 20–23° (cool if necessary). After 3 hr, filter as completely as possible with vac., and wash with hot H_2O until acid-free to pH paper. Rinse sides of crucible and remove stirring rod. Dry crucible in 100° forced-draft oven, cool in desiccator over P_2O_5 or $Mg(ClO_4)_2$, and weigh (W_3). Ignite crucible in 500° furnace 2 hr or until C-free. Place crucible while still hot into 100° forced-draft oven 1 hr. Transfer to desiccator, cool, and weigh (W_4).

Det. asbestos blank by weighing 1 g asbestos into tared crucible. Proceed as above, beginning "Cover contents of crucible . . ." Record any loss in wt on ashing (W_5). Discontinue detn of blank if asbestos blank is <0.0020 g/g asbestos. Calc. % acid-insol. lignin = $(W_3 - W_4 - W_5)/S$.

Total Sugars (22)—Official Final Action

7.073 ***Reagents***

(a) *Soxhlet modification of Fehling soln.*—Prep. as in **31.034(a)** and **(b)**.

(b) *Invert sugar std soln.*—1.0%. Prep. as in **31.034(c)**, but do not neutze. Dil. to 0.5% just before use for analysis of most products.

(c) *Lactose std soln.*—1.0%. Dissolve 5.000 g lactose in H_2O and dil. to 500 mL. Prep. daily.

7.074 ***Apparatus***

(a) *Lamp.*—Fluorescent desk lamp or 150 watt reflector spot lamp, to illuminate boiling soln.

(b) *Heater.*—Glas-Col mantle, 250 mL, placed over mag. stirrer. Adjust heat so that 50 mL H_2O contg stirring bar will boil in 3 min. Mag. stirring hot plate is also satisfactory.

7.075 ***Preparation of Sample and Inversion***

(a) *Feeds containing molasses.*—Weigh appropriate size sample, prepd as in **7.002** but not ground, to provide final soln ca 0.5% invert sugar but ≥5 g, into 250 mL P flask (Corning Glass Works No. 5840, or equiv.). Add 150 mL H_2O, swirl to wet and mix, and heat just to bp. Let stand to cool, dil. to vol., mix, and let stand to settle coarse particles. Transfer 50 mL supernate to 100 mL vol. flask and add 2.5 mL HCl (sp gr 1.18 at 20/4°). Let stand overnight at ≥25°, dil. to vol., and mix. (If aliquot to be used in detn is >25 mL, it is necessary to neutze inverted soln.)

(b) *Feeds containing milk products.*—Weigh appropriate size sample to provide final soln ca 1% lactose into 250 mL vol. flask. Thoroly moisten sample with H_2O, swirl to dissolve lactose, dil. to vol., mix, and let stand to settle coarse particles. Proceed as in **7.077(b)**.

7.076 ***Standardization***

Fill 50 mL buret, with offset tip, with std sugar soln (invert sugar for use with **7.077(a)** and lactose with **7.077(b)**). Proceed as in **31.080**, par. 2, except use same type flask as used in **7.077**, do not add H_2O, and start stirring after addn of indicator.

7.077 ***Determination***

(a) *Difference method.*—Add reagents and stirring bar to 250 mL extn flask (Corning Glass Works No. 5160, or equiv.) or to erlenmeyer, as in **7.076**. Transfer aliquot inverted soln, (a), to flask so that >1 but <5 mL std soln will be required to reach end point, place on preheated mantle or hot plate, heat to bp, boil 2 min, add ca 1 mL indicator, and begin stirring. Complete detn by titrg with std sugar soln to same end point used in stdzn. Color change is not so sharp as in stdzn, but under suitable light it is definite, discernible, and repeatable.

(b) *Alternative method.*—Fill buret with sample soln., (b), or inverted sample soln, (a). As in **7.076**, place reagents in flask, place on heater, add sample soln to within 2 mL of final titrn (detd by trial), bring to bp, boil 2 min, and complete titrn as in (a).

7.078 ***Calculations***

% Total sugar (as invert or lactose)

$$= [(F - M) \times I \times 100]/[V \times (W/250) \times D)],$$

where F = mL std sugar required to reduce mixed Soxhlet reagent in stdzn; M = mL std soln required to complete detn (omit in alternative method); I = concn std soln; V = mL sample soln in aliquot used; W = g sample; and D = diln factor.

Report total sugars, expressed as invert or as lactose.

7.079 Sucrose (23)—Official Final Action

Place 10 g sample in 250 mL vol. flask. If material is acid, neutze by adding 1–3 g $CaCO_3$. Add 125 mL 50% alcohol by vol., mix thoroly, and boil on steam bath or by partially immersing flask in H_2O bath 1 hr at 83–87°, using small funnel in neck of flask to condense vapor. Cool and let mixt. stand several hr, preferably overnight. Dil. to vol. with neut. 95% alcohol, mix thoroly, let settle or centrf. 15 min at 1500 rpm, and decant closely. Pipet 200 mL supernate into beaker and evap. on steam