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SEPARATION OF CORN ETHANOL STILLAGE INTO
ITS SOLID AND LIQUID PORTIONS

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Bruce W. Wilkinson
Major professor

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SEPARATION OF CORN ETHANOL STILLAGE INTO ITS SOLID AND LIQUID PORTIONS

By

Barbara Elizabeth Goodrich

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ABSTRACT

SEPARATION OF CORN ETHANOL STILLAGE INTO
ITS SOLID AND LIQUID PORTIONS

By

Barbara Elizabeth Goodrich

Farm scale ethanol production can only be economically feasible if the by-product, corn ethanol stillage, can be utilized. Some fractions of stillage can be used as livestock feed, while others have use as fertilizer. The limiting criteria is the moisture content, which must be kept low if the material is to be used as feed. The protein content of the feed should be maximized if possible. The unsuitable material can be used as fertilizer because of its mineral content.

Four simple separation devices were investigated for separation devices were investigated for separation performance. The separated material was analyzed for protein and moisture content. It was then categorized as to whether it should be used as livestock feed or fertilizer. Finally, these products were analyzed in terms of their market value.

Stillage made from coarser ground corn and separated on the gyratory device appears to be the best choice.

ACKNOWLEDGMENTS

I wish to thank my advisor, Dr. Bruce W. Wilkinson, for all of his support during this project and also a very special thanks to Mr. Gary M. Webber of Animal Science for all the help and knowledge he so willingly provided.

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CHAPTER I

INTRODUCTION

Over the past decade, beginning with the 1973 Arab Oil Embargo, the energy market has experienced gross fluctuations which have produced cost escalations and temporary shortages. The fear of future shortages has stimulated the search for alternative sources of liquid fuels. These instabilities have been of particular concern to the farmer whose livelihood depends on the availability and affordability of liquid fuels to operate his farm machinery. The possible on-site production of ethanol from corn poses a potentially attractive source of liquid fuel. Since the typical farmer produces more corn than is required to feed his livestock, this surplus corn could either be sold, assuming that there is a buyer, or converted to fuel via fermentation. If the conversion process could be accomplished on the farm, it would eliminate certain transportation and handling costs plus give an additional sense of security to the farmer.

The fermentation of corn results in a "beer" containing 8-10% ethanol. Recovery of ethanol through distillation produces a stillage (distillation bottoms) stream composed mainly of water, but which also contains unfermented grain parts in both solid and liquid form. This material is disposed of or utilized in the farm operation. The protein content of the stillage is adequate to make it attractive as

a feed supplement for cattle. However, its high moisture content makes it unusable as a direct feed supplement. Cattle will only tolerate so much moisture in their diet or their dry matter intake will go down. This results in a reduced yield which translates into less meat on their carcasses at market time. Excessive moisture also causes manure handling problems.

Some fractions of separated stillage are unsuitable for use as livestock feed because of a high moisture content. These fractions may have use as a fertilizer because of their nutrient content. The important nutrients contained in this material are nitrogen, potassium, and phosphorus.

Commercial ethanol plants further process the stillage to recover the protein content in a dry concentrated form. The solid content of stillage is separated out by screening and passed through a hot gas drier. The drying process requires a significant capital and energy investment. The liquid portion (containing some soluble proteins) is evaporated to remove the water and the resultant concentrated soluble protein is added to the solids during the drying process. This operation produces a dry concentrated protein-rich "Distillers Dried Grains with Solubles (DDGS)" product which is suitable for long-term storage and transportation to markets. The equipment requirements and the energy consumption incurred make this process attractive only in large commercial scale plants.

Prospective owners of farm scale ethanol plants have limited funds available for investments and expenditures. The distillation

column in itself is a major investment.[1] Finding funds for an extensive stillage drying process is another matter. The potential exists, however, for direct feeding of separated distillers grain to cattle that reside on the farm. In this case, it would be unnecessary to dry the grain if storage can be minimized. The separation process should be as simple as possible to enable the farmer to minimize his investment in terms of capital and time. Furthermore, the protein content of the separated grain (which is known as "Wet Distillers Grain (WDG)") should be maximized. This is complicated by the fact that the insoluble protein associated with small particle sizes and the soluble protein will probably not be recovered due to the expense associated with their separation from the water phase.

A simple screening process which separates the wet grains from the water phase has the potential of arranging WDG into a form suitable for feeding cattle. Furthermore, if the protein content can be established as a function of particle size, selective preparation of a particular particle range might be used to maximize the protein content.

The purpose of this study was to demonstrate and evaluate several separation processes which do not require the drying of grains. The variance of the protein and moisture content in different fractions of a corn-fermented stillage stream was also determined. This study was part of a joint undertaking by the Departments of

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[1]Joseph W. Geiger, "Design, Energy, and Economic Analysis of Small Scale Ethanol Fermentation Facilities," (M.S. Thesis, Michigan State University, 1981).

Chemical Engineering and Animal Science. Chemical Engineering was responsible for the separation of the stillage into WDG. Animal Science had the responsibility of investigating different methods of storing WDG on a long-term basis. The funding for this project came from the U.S. Department of Energy Grant DE-FG07-81ID-12334.

The feed stream used for this work was prepared at the M.S.U. Beef Cattle Research Center Ethanol Pilot Plant. This facility was built during the previous joint effort of the Departments of Chemical Engineering and Animal Science with a grant from the Michigan Department of Agriculture.[2] Separation tests were conducted using three pieces of equipment:

1. CRIPPEN Mfg. Co. Inclinced Screen, Model KV 1236
2. SWECO, Inc. Circular Vibro-Energy Screen, Model LS18C3333
3. EIMCO Rotary Vacuum Filter

In addition, further tests using a buchner funnel with Whatman filter paper were run on the liquid residue of separations 1 and 2 above. The funnel employed a vacuum pulled by a water aspirator. A series of tests using the CRIPPEN unit was also conducted on commercial stillage supplied by a local ethanol plant.

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[2]Ibid., p. 3.

CHAPTER II

NUTRITIONAL VALUE OF "WET" DISTILLERS GRAIN

Fermentation and distillation processes remove only the starch or sugar in the feedstock, thus concentrating the remaining nutrients.[3] When corn is used for feedstock, the concentration of nutrients is increased approximately 300% (dry weight basis) when compared with the original corn feedstock.[4]

A number of studies have been conducted concerning the suitability of WDG as a livestock feed source and supplement. In his article, "Wet Distillers' Grains, An Excellent Substitute for Corn in Cattle Finishing Rations," Stanley D. Farlin[5] cites testing done at the University of Nebraska during the summer of 1980. Three different percentages of WDG were substituted for corn in the feeding of three groups of cattle. A fourth group served as the control group. The control rations consisted of 85% corn, 5% dry supplement, and 10% hay. In addition, urea supplement was employed so that the total ration protein was 11%. The percentages substituted for corn were

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[3]National Research Council, Feeding Value of Ethanol Production By-Products (Washington, D.C.: National Academy Press, 1981), p. 15.

[4]Ibid.

[5]Stanley D. Farlin, "Wet Distillers' Grains: An Excellent Substitute for Corn in Cattle Finishing Rations," Animal Nutrition and Health (April 1981): 35.

25%, 50%, and 75%. The WDG contained 75% moisture and from 27-29% protein on a dry basis. It was found that the carcasses of cattle fed at 50% were 23 pounds heavier than the groups fed at 25% and 75%. The carcasses of cattle fed at those latter percentages were similar in weight to that of the control group. Carcass characteristics such as quality grade, ribeye area, fat thickness, and dressing percentage were not affected by the use of WDG.[6]

It has been documented that the nutrient composition of WDG is comparable to DDGS when they are compared on a dry matter basis.[7] Animals have been found to perform equal to or better on wet by-products than on the same by-product in a dried form.[8] There is evidence that DDG, when used as a feed supplement, increases milk yield and percent milk fat in lactating cows.[9]

The amount of extra moisture that can be incorporated in a cattle's diet is limited by the amount which will not cause a reduction in weight gain rate.[10] If the animal becomes waterlogged, their dry matter intake will go down.

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[6]Ibid.

[7]J. C. Waller, et al., "Use of Fuel Ethanol By-Products in Livestock and Poultry Diets" (paper prepared for Michigan State University), p. 3.

[8]Ibid.

[9]Distillers Feed Research Council, Distillers Feeds (Cincinnati, Ohio), pp. 48-53.

[10]National Research Council, Feeding Value of Ethanol, p. 40.

High moisture by-products such as WDG are subject to microbial contamination.[11] Therefore, it is important that they are stored properly. Lake (1976)[12] said that the feeding of high moisture feeds reduced the bunker life of the feedlot diets. Because of the spoilage that might occur in the bunker, it is necessary to feed the cattle again within 12 hours before the feeds spoil.[13] If WDG becomes spoiled, it loses its palatability. Cattle are likely to eat less of it and possibly refuse it altogether. This could result in a reduction of dry matter intake.

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[11]J. C. Waller, et al., "Separation and Storage of High Moisture Distillers Feeds" (paper prepared for DOE, Michigan State University), p. 20.

[12]National Research Council, Feeding Value of Ethanol, p. 40.

[13]Ibid.

CHAPTER III

STILLAGE PREPARATION

The M.S.U. Ethanol Pilot Plant operates on a 500 gallon batch fermentaion scale. Dry corn was ground to pass a U.S. No. 8 (0.093 inch) screen and mixed with water to form a fluid mash. Takatherm alpha-amylose enzyme was added to the mash following a pH adjustment to 6.5. (A 50% sodium hydroxide solution was used if pH needed to be raised; a 50% sulfuric acid solution was used if the pH needed to be lowered.) The mixture was boiled for at least an hour to ensure the hydration of starch to the hexose sugars, maltose and glucose. The batch was cooled to 194°F and a final pH adjustment to 6.5 was made. Additional enzyme was added and the mixture was vigorously agitated to promote enzymatic activity. The solution was held at 194°F until an iodine test showed no starch intact in either the solid or liquid phases. Cool water was then added to lower the temperature to 135-140°F. The pH was adjusted to 4.2 and diazyme glucoamylose enzyme was added to produce at least 10% glucose. (If the corn starch is not completely hydrolyzed to glucose and maltose, the yeast will only be able to produce ethanol equal to the amounts of sugars present.) M.S.U.-produced stillage was overtreated with enzyme to guarantee complete hydrolysis. The resultant material was cooled to 90°F by the addition of cold water. Brewers yeast was then

added. Fermentation proceeded for two to two-and-a-half days until carbon dioxide was no longer evolved or until glucose could no longer be detected in the mash.

The resultant beer was stored in an agitated tank until distillation. At that time it was pumped to the top of a 12-inch glass sieve tray stripping column (see Figure 1). Reboiler heat was supplied by a steam coil (as contrasted to steam sparaging which is often done.) An ethanol stream was drawn off the condenser at about 120 proof. The stillage from the reboiler was essentially ethanol free and 90-93% water. See Figure 2 for a flowchart of the process.

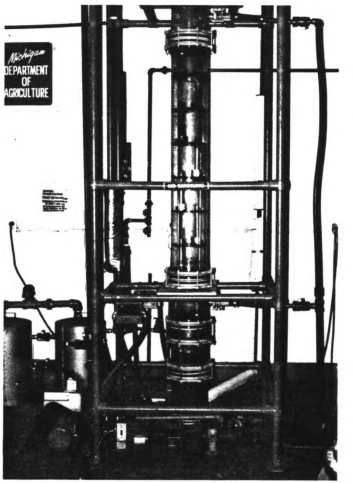

Figure 1.--Distillation Column.

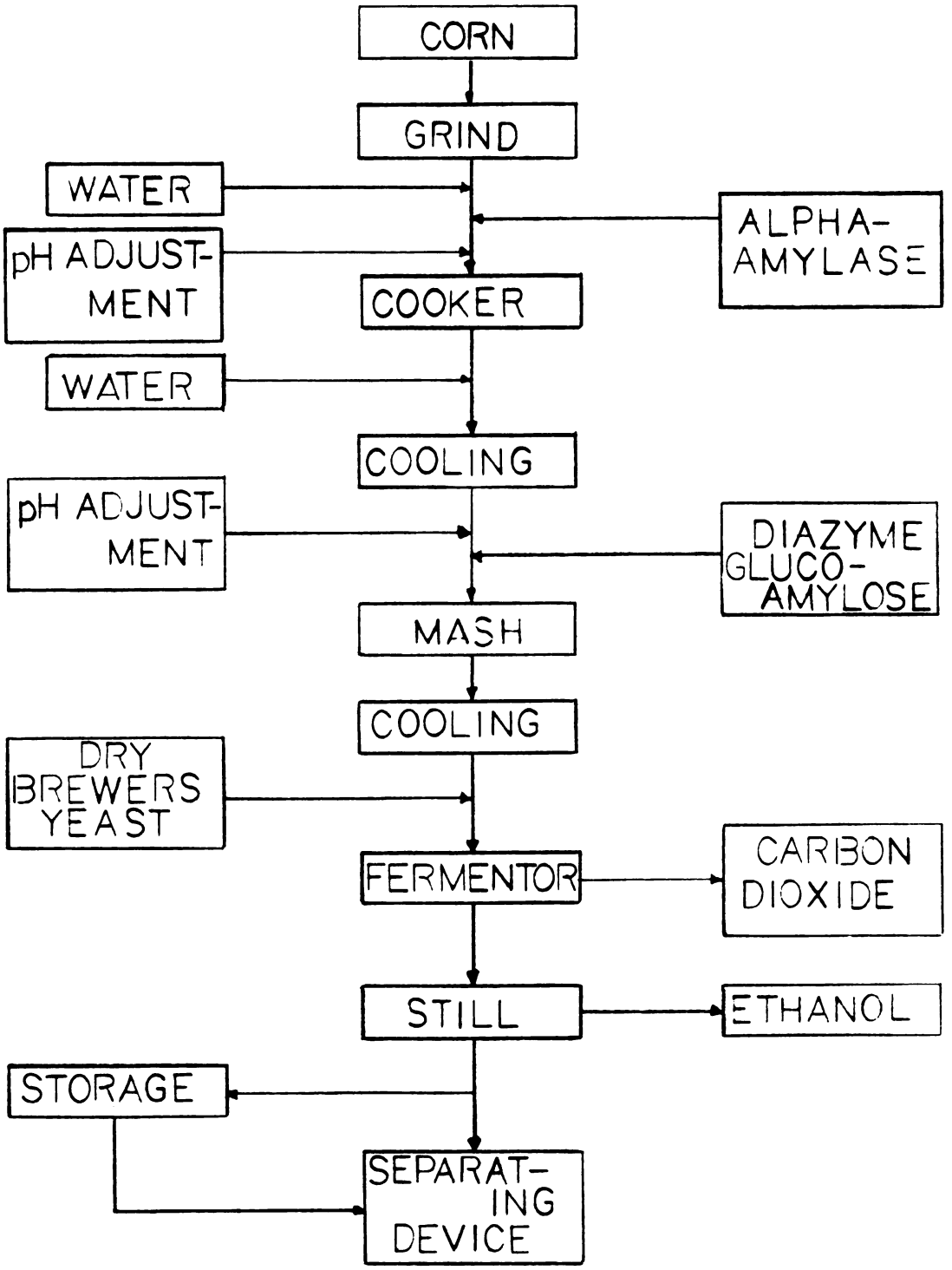

Figure 2.--Process Flowchart.

CHAPTER IV

SEPARATION TESTS

SWECO and CRIPPEN Separators

For most tests, the stillage was pumped directly from the reboiler to a test separation unit. The pumping and separation rates were determined by the still operation.

Screens were set up on these separators with two mesh sizes mounted such that three fractions of particle sizes were collected. These fractions were designated "Dry," "Intermediate," and "Wet" in reference to their relative moisture content. The "Dry" fraction was material that stayed on the top screen; the "Wet" was that which passed through both screens. Each fraction was collected over a timed interval so that a flow rate could be assigned to it. The collected fractions were analyzed for protein via a modified Kjeldahl method (see Appendix C) for moisture content by oven drying at 60°C for 24 hours.

SWECO Circular Vibro-Energy Screen (See Figure 3)

This device consists of circular screens (device used in experimental work had two screens and three discharges) with an 18-inch diameter working surface and is powered by a 0.5 HP electric motor. It vibrates about its own center of mass. This vibration is

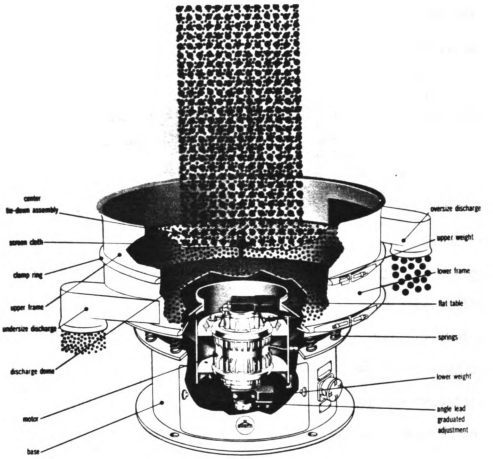

Figure 3.--SWECO Circular Vibro-Energy Screen.

caused by eccentric weights on the upper and lower ends of the motor shaft.[14] The upper weight acts to create vibration in the horizontal plane which moves the material across the screen to the periphery.[15] The lower weight tilts the machine creating vibration in the vertical and tangential planes.[16] The SWECO separator is commercially used to separate stillage. Larger units are used to separate high moisture waste streams in vegetable processing plants on a commercial basis. The unit was supplied by a regional commercial supplier.

Samples were collected on a timed basis by placing a container under the appropriate discharge.

CRIPPEN Inclined Screen
(See Figure 4)

This unit consists of a rectangular screen with a 32" x 10¼" working area powered by a 0.5 HP electric motor. It was designed to separate dry particles rather than wet sludge such as stillage; thus its mechanical motion was not optimized for handling wet material. Many CRIPPEN units today are used in a certified seed business to separate weed or off-type seeds from certified seedstock.[17]

The screen angle of the device was varied to see if an optimum existed. If the screen angle was too near horizontal, material on

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[14]SWECO, Inc., SWECO Vibro-Energy Separator (Los Angles, California), p. 8.

[15]Ibid.

[16]Ibid.

[17]Waller, "Use of Fuel Ethanol By-Products in Livestock and Poultry Diets," p. 6.

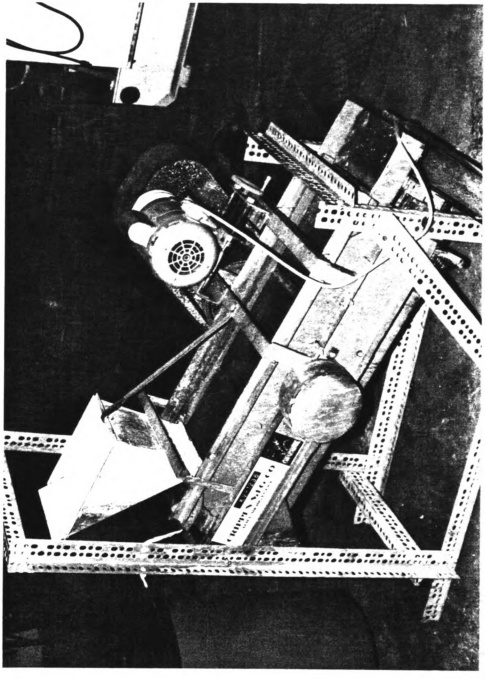

Figure 4.--CRIPPEN Inclined Screen.

the screen would clump together. On the other hand, if the angle was too steep, the residence time would be too short to permit an adequate separation.

The tests that were conducted on the commercial product using this device were carried out on "cold" product which had been transported in barrels to M.S.U. from the commercial ethanol plant. The material was fed manually semi-continuously (small frequent batches) to the separator.

Samples were collected on a timed basis by placing aluminum containers underneath the device to catch the various fractions. Losses occurred frequently due to the difficulty of catching all the material.

EIMCO Rotary Vacuum Filter (See Figure 5)

This device consists of a cylindrical drum supported in an open-top vat which allows the drum to rotate about its own axis in the horizontal plane.[18] The lower portion of the drum is confined within the tank walls and the upper portion is exposed.[19] The drum shell contains a number of shallow compartments which are covered with a drainage grid and a filter cloth.[20] A vacuum is applied to those compartments of the drum which pass through the material to be filtered. This, in turn, creates a vacuum within the compartments,

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[18] R. H. Perry, and C. H. Chilton, Chemical Engineers' Handbook, 5th ed. (New York: McGraw Hill, 1973), pp. 19-76.

[19] Ibid.

[20] Ibid.

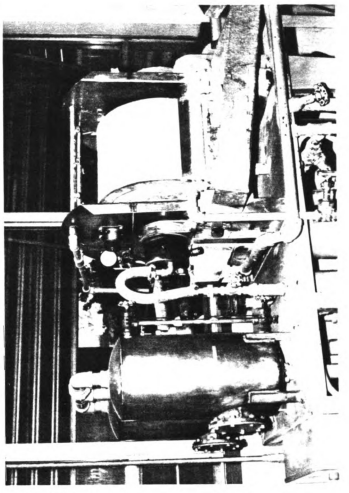

Figure 5.--EIMCO Rotary Vacuum Filter

causing a flow through the filter medium, conduits, and automatic valve.[21] A layer of cake solids is deposited upon the filter, covering the submerged particles of the drum.[22]

Stillage from the M.S.U. Ethanol Pilot Plant was transported in barrels to the Chemical Engineering Laboratory for separation using the EIMCO device. Consequently, the tests were conducted on cold stillage.

Vacuum Filter (See Figure 6)

The liquid and solid portions in the fraction previously designated as "Wet" were further separated by vacuum filtration. A buchner funnel with an 11-centimeter diameter was used with various Whatman filter paper for the filtration. The funnel employed a vacuum pulled by a water aspirator. The filter paper ranged from "slow" to "fast" in filter speed depending on porosity. Protein tests via a modified Kjeldahl method were run on the filtrate to see how much protein remained in the liquid phase.

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[21] Ibid., pp. 19-77.

[22] Ibid., pp. 19-78.

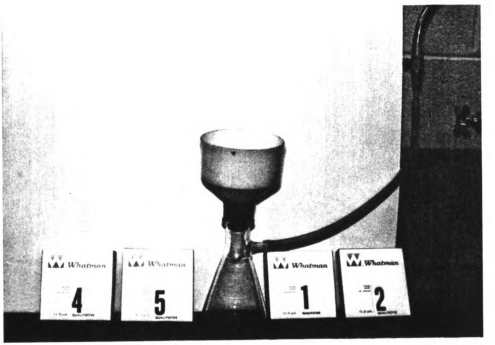


Figure 6.--Vacuum Filter (Buchner Funnel)

CHAPTER V

RESULTS AND DISCUSSION

M.S.U. Stillage

Tests were conducted on the SWECO separator, the CRIPPEN separator, a buchner funnel, and the EIMCO filter. Samples were collected in plastic containers (see Figure 7). Both the SWECO and CRIPPEN devices gave reasonable separations, whereas the EIMCO device would not achieve a separation. For this latter system, the presence of a significant quantity of fines plugged the filter cloth and halted the separation of the product. Diatomaceous earth filter aid was added to the stillage at 10% of the stillage solids content to enhance filtration. Greater amounts were felt to make the material unusable as animal feed. Even with filter aid, the filtration process could not be carried out.

The buchner funnel used in conjunction with Whatman paper allowed for further separation of the "Wet" fraction.

SWECO Separator

The location of the feed point (see Figure 8 for location of feed points) was varied to see if an optimum existed. The results are listed in Tables 1, 2, and 3.

The feed point location seemed to impact the moisture content and percent of the total flow in the particle size range $x \geq 995\mu$

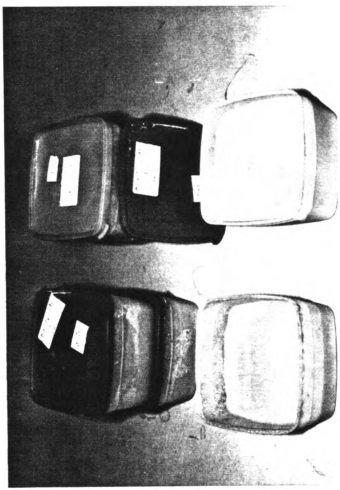
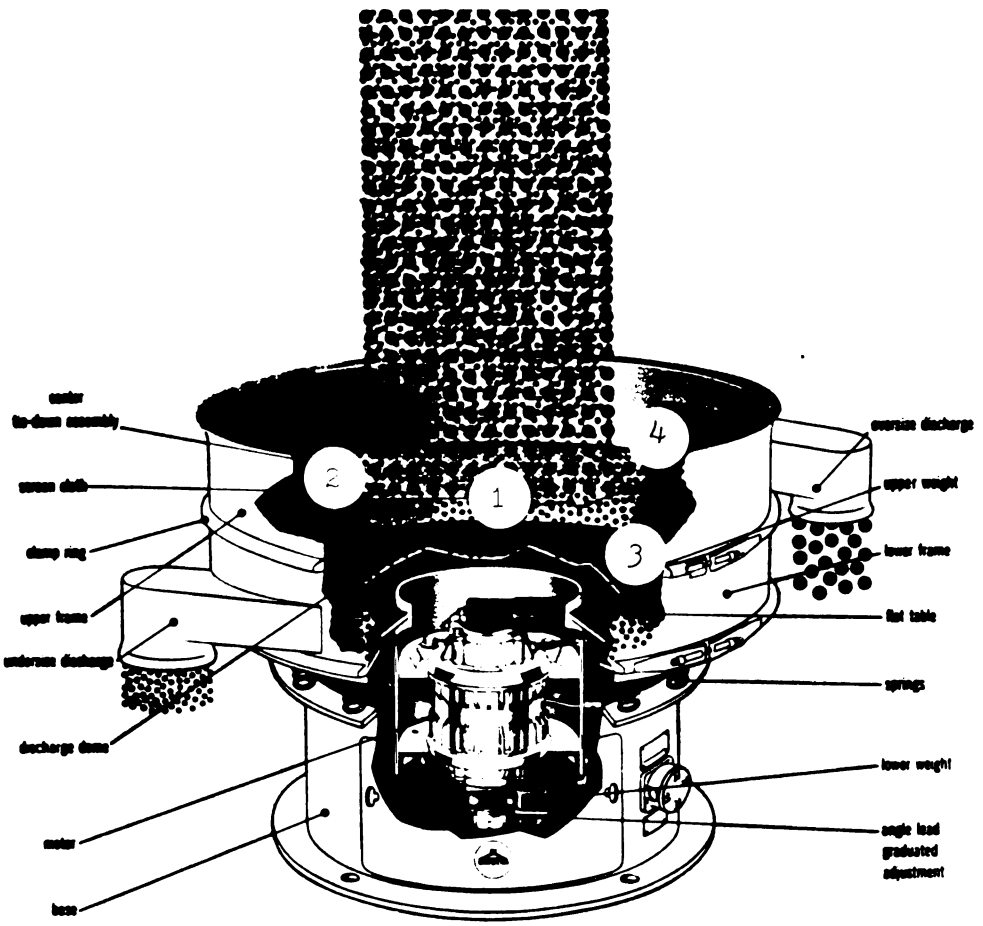


Figure 7.--Collected Samples.



CUTAWAY...SWECO VIBRO-ENERGY SEPARATOR

Figure 8.--Feed Point Locations.

TABLE 1.--Effect of varying feed location for particle size $x \geq 999\mu$

Position	% of Total Flow	% Moisture	% Protein (dry basis)
1	14.5	82.8	27.9
2	16.7	82.9	27.0
3	20.9	87.0	27.4
4	16.5	82.0	28.2

TABLE 2.--Effect of varying feed location for particle size $995\mu > x \geq 438\mu$

Position	% of Total Flow	% Moisture	% Protein (dry basis)
1	9.2	87.8	50.6
2	10.6	88.8	52.1
3	7.4	86.8	49.4
4	7.7	87.4	45.3

TABLE 3.--Effect of varying feed location for particle size $x < 438$

Position	% of Total FLow	% Moisture	% Protein (dry basis)
1	76.3	96.4	33.5
2	72.8	96.5	38.2
3	71.7	96.4	35.5
4	75.9	96.4	31.6

more than it affected the particle size ranges $995\mu > x \geq 438\mu$ and $x < 438\mu$. In the particle size range $x \geq 995\mu$, the moisture content is highest when the feed point position is in position 3. This is probably due to a short residence time on the top screen resulting in a poor separation. The manufacturer recommends that the feed point be located in position 1 which is in the middle of the screen. Overall, the variation caused by feed point location was slight and the results were, in general, quite consistent.

The moisture and protein content of the separated stillage varied with particle size. Fraction yields were also dependent on particle size. See Tables 4 and 5.

TABLE 4.--The moisture and protein content of SWECO separated M.S.U. stillage

Particle Size (μ)	Moisture %			Protein % (dry basis)		
	Average	High	Low	Average	High	Low
$995 \leq x$	82.9	87.0	80.0	27.7	29.1	26.8
$995 > x \geq 438$	87.7	88.9	86.7	49.6	52.3	39.0
$438 > x$	96.4	96.6	95.9	34.3	39.3	27.1

TABLE 5.--The fraction yields of SWECO separated M.S.U. stillage (per 100 lbs of feed)

Particle Size (μ)	Average Yield lbs	lbs Water	lbs Dry Solids	lbs Protein (dry basis)
$995 \leq x$	17.0	14.0	3.0	0.83
$995 > x > 438$	9.3	8.2	1.1	0.58
$438 > x$	73.7	71.0	2.7	0.91

By examining Table 4, several trends can be seen. The moisture content increases as particle size decreases. This is to be expected since most of the solids' weight is concentrated in the large particles as seen in Table 5. Furthermore, the dewatering of the large particles is more easily accomplished than with the fines.

The protein content of the intermediate particle size is significantly higher than either the fine or coarse fractions. The solids' fractions produced are still extremely wet with more than 80% moisture. This is to be expected in light of the gravity separation process utilized and the relative difficulty of water removal from fine solids. The average mass yield is greatest for the fine particles and lowest for the intermediate particles. The intermediate particles may have the highest protein content, but they have the lowest contained protein.

The main advantage of screening the product is the reduction of the moisture content. The feed is calculated to have an overall moisture content of 93.2%, whereas the solid fractions collected have a moisture content which ranged from 80 to 87%. This reduction, although not great, represents a reduction of over a factor of two in the amount of water ingested by the animal for an equal weight of solids consumed. However, the separation process used to prepare this feed resulted in the loss of about one-third of the solids (and contained protein) into the liquid stream. Some portion of this material is in soluble form and cannot be recovered, except by evaporative concentration. Another portion, finely divided suspended

solids, cannot be easily recovered due to the very fine particle size. See Appendix A for further data.

CRIPPEN Separator

The results obtained with this device were less reproducible since it was not meant to separate wet sludges such as stillage. Because of its design, it was also difficult to pull consistent samples and losses occurred frequently. While varying the screen mesh size, tests were conducted by altering the screen angle in an attempt to improve the separation. The best angle for the tests appeared to be about 17° with respect to the horizontal.

Separation results for the CRIPPEN separator using M.S.U. stillage are summarized in Tables 6 and 7. (Details are given in Appendix A under Moisture and Proteins and under Fraction Yields.)

TABLE 6.--The moisture and protein content of CRIPPEN separated M.S.U. stillage

Particle Size (μ)	Moisture %			Protein % (dry basis)		
	Average	High	Low	Average	High	Low
$940 \leq x$	82.4	82.6	81.8	29.1	32.0	24.2
$940 > x \geq 622$	94.1	95.1	93.0	30.7	32.1	29.3
$622 > x$	95.2	95.6	94.5	31.3	33.1	27.7

TABLE 7.--The fraction yields of CRIPPEN separated M.S.U. stillage

Particle Size (μ)	Average Yield lbs	lbs Water	lbs Dry Solids	lbs Protein (dry basis)
940 $\leq$ x	14.7	12.1	2.6	0.76
940 > x > 622	2.7	2.5	0.2	0.06
622 > x	82.6	78.6	4.0	1.24

The higher protein composition occurs in the fine particle size. However, due to the poorer separation with the CRIPPEN unit, the variation in protein by particle size is less pronounced. Most of the flow is in the fine particle fraction.

Commercial Stillage

CRIPPEN Separator

The stillage produced by a commercial ethanol plant was separated using the CRIPPEN unit. The quantity of material was somewhat limited but the results are given in Tables 8 and 9. (See Appendix A for further data.)

TABLE 8.--The moisture and protein content of CRIPPEN separated commercial stillage

Particle Size (μ)	Moisture %			Protein % (dry basis)		
	Average	High	Low	Average	High	Low
940 $\leq$ x	86.9	87.1	86.6	25.3	26.2	24.3
940 > x $\geq$ 622	93.6	93.6	93.6	23.6	23.6	23.6
622 > x	94.5	94.8	94.3	23.2	26.8	20.4

TABLE 9.--The fraction yields of CRIPPEN separated commercial stillage

Particle Size (μ)	Average Yield lbs	lbs Water	lbs Dry Solids	lbs Protein (dry basis)
$940 \leq x$	44.9	39.0	5.9	1.49
$940 > x \geq 622$	9.2	8.6	0.6	0.14
$622 > x$	45.9	43.4	2.5	0.58

Little variation occurred in protein composition between fraction sizes with this material. Due to an initially higher water content, it was more difficult to remove water from this material (as indicated by its higher moisture content) than the M.S.U. stillage.

A calculation of the percent solids and percent protein in the commercial feed shows 9.0% and 2.21% (wet basis), respectively. It is apparent, then, that this material had a higher solids content than the M.S.U. product. On the other hand, a much larger fraction exists as large particles. This is due to a coarser grind of the corn. It is noted that the moisture content of the coarsest fraction was decreased from 91.0% (feed) to only about 86.9% so that the amount of moisture ingested by cattle would still be quite high.

A large fraction of protein can be recovered easily by coarse screen separation. The fine fraction, however, will contain a significant protein value.

The variance in yield (see Appendix A, Fraction Yields) is due to the manual batch method used to transport the stillage to the separating device.

SWECO Separator

The results using the SWECO device to separate commercial stillage would have been very helpful. However, at the time commercial stillage was available for testing, the SWECO device was not. Based on the M.S.U. results, SWECO separated commercial stillage would exhibit higher solids content and lower labor requirements.

Comparison of SWECO Separated M.S.U. Stillage, CRIPPEN Separated M.S.U. Stillage, and CRIPPEN Separated Commercial Stillage

The total protein content of the commercial stillage is lower than the M.S.U. stillage. This could have occurred for several reasons. Either the corn used for the commercial product had a lower protein content to begin with and/or the starch was not completely hydrolyzed during fermentation. The two M.S.U. stillages had similar protein contents; the variation is probably due to a difference in the degree of hydrolyzation. Of the intermediate fractions, the SWECO separated M.S.U. stillage had the highest contained protein.

In Figures 9, 10, and 11 the top line in each bar represents the maximum value, the middle line is the average value, and the bottom line represents the minimum value. If only one line appears, then there was only one piece of data for that particular bar.

In the large particle fraction range, the CRIPPEN separated M.S.U. stillage had the highest protein composition. The SWECO separated M.S.U. intermediate fraction was much higher in protein composition than either the CRIPPEN M.S.U. or CRIPPEN commercial separated intermediate fraction. It is quite possible that for the

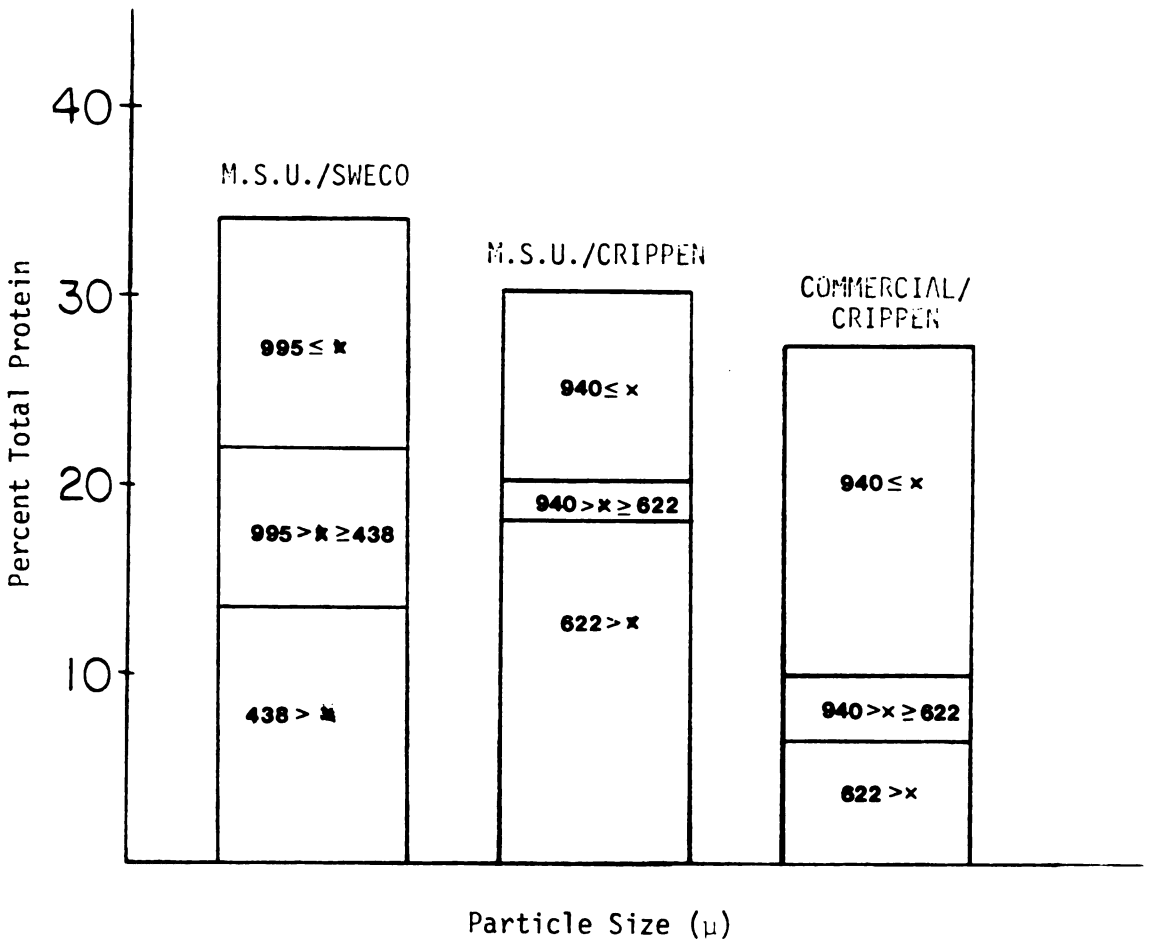


Figure 9.--Comparison of Protein Content in Separated Corn Ethanol Stillage

Figure 10.--Comparison of Percent Protein by Particle Size in Separated Corn Ethanol Stillage.

Percent Protein (Dry Basis)
52
48
44
40
36
32
28
24
20
M.S.U./SWECO
995≤x
M.S.U./CRIPPEN
940≤x
COMMERCIAL/CRIPPEN
940≤x
M.S.U./SWECO
995>x≥438
M.S.U./CRIPPEN
940>x≥622
COMMERCIAL/CRIPPEN
940>x≥622
M.S.U./SWECO
438>x
M.S.U./CRIPPEN
622>x
COMMERCIAL/CRIPPEN
622>x
Particle Size (μ)

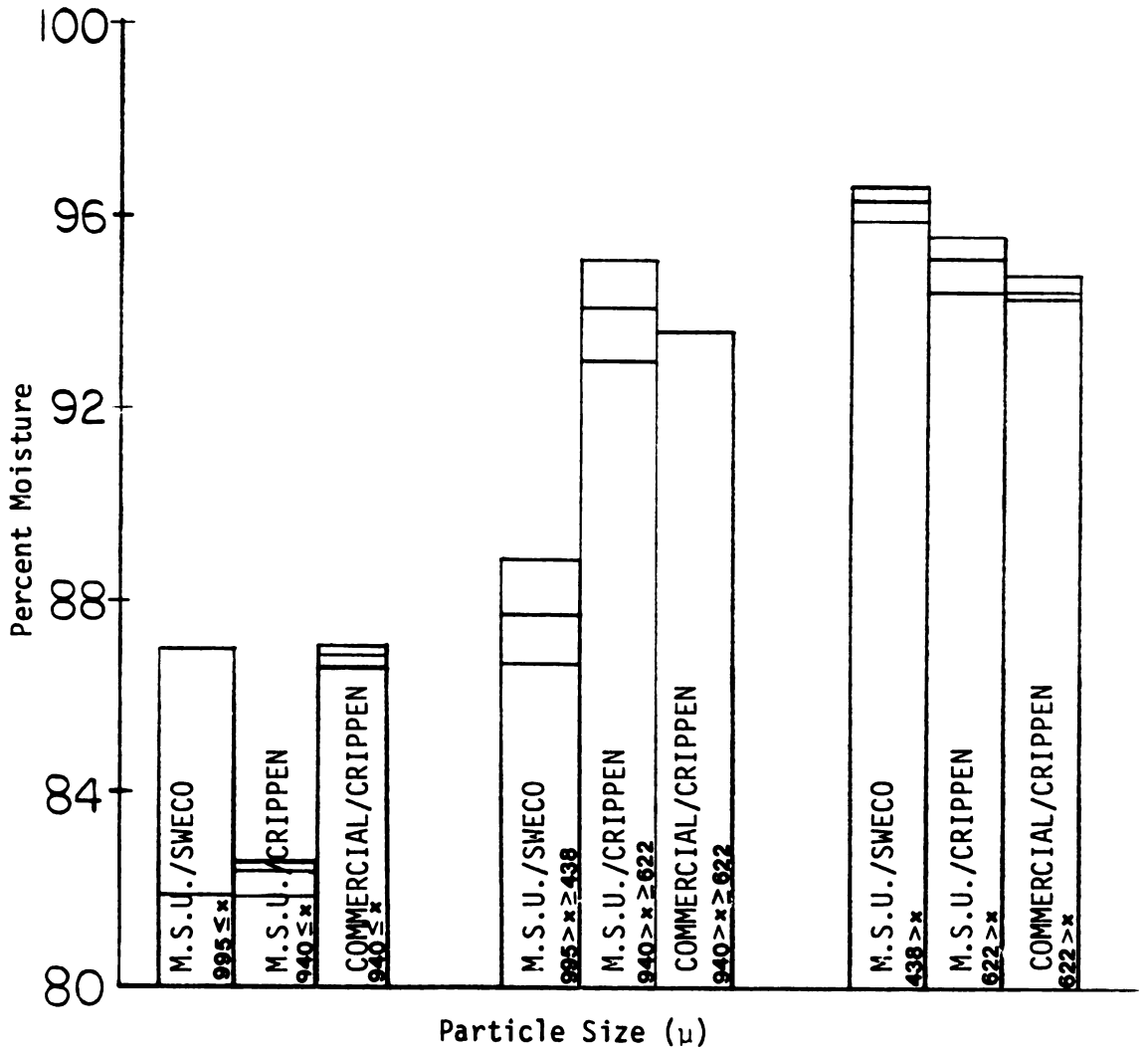


Figure 11.--Comparison of Moisture Content by Particle Size in Separated Corn Ethanol Stillage.

SWECO separation, this fraction contained the corn gluten protein which has a 60 to 70% protein content.[23] Having the gluten present in this fraction could have come about from the way the corn was ground. Most of the gluten may have ended up in a particular particle size. Upon examination of the M.S.U. stillage, it can be concluded that a high protein content seems to be associated with the particle size range $622\mu > x \geq 438\mu$. Note that the intermediate fraction in all three groups has the lowest mass of contained protein of all the fractions.

Upon examination of the total moisture content (Figure 12), it can be seen that the CRIPPEN commercial large particle size has a higher moisture content than either the M.S.U. CRIPPEN or SWECO fraction. The commercial product also had a greater amount of material in the large particle fraction than either of the M.S.U. materials. Because of the poor separation using the CRIPPEN device, water would sit on the large particles. Since the commercial product had such a large yield in this fraction, dewatering was more difficult. A longer residence time would be needed in order to dewater the commercial product to a dryness comparable to the M.S.U. product.

The moisture content of the commercial CRIPPEN and the M.S.U. CRIPPEN intermediate fractions are similar, but the M.S.U. SWECO is much drier. In the fine particle range, all the separations have similar moisture contents.

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[23]Corn Industries Research Foundation, Corn Gluten Feed and Corn Gluten Meal (Washington, D.C., 1959), p. 12.

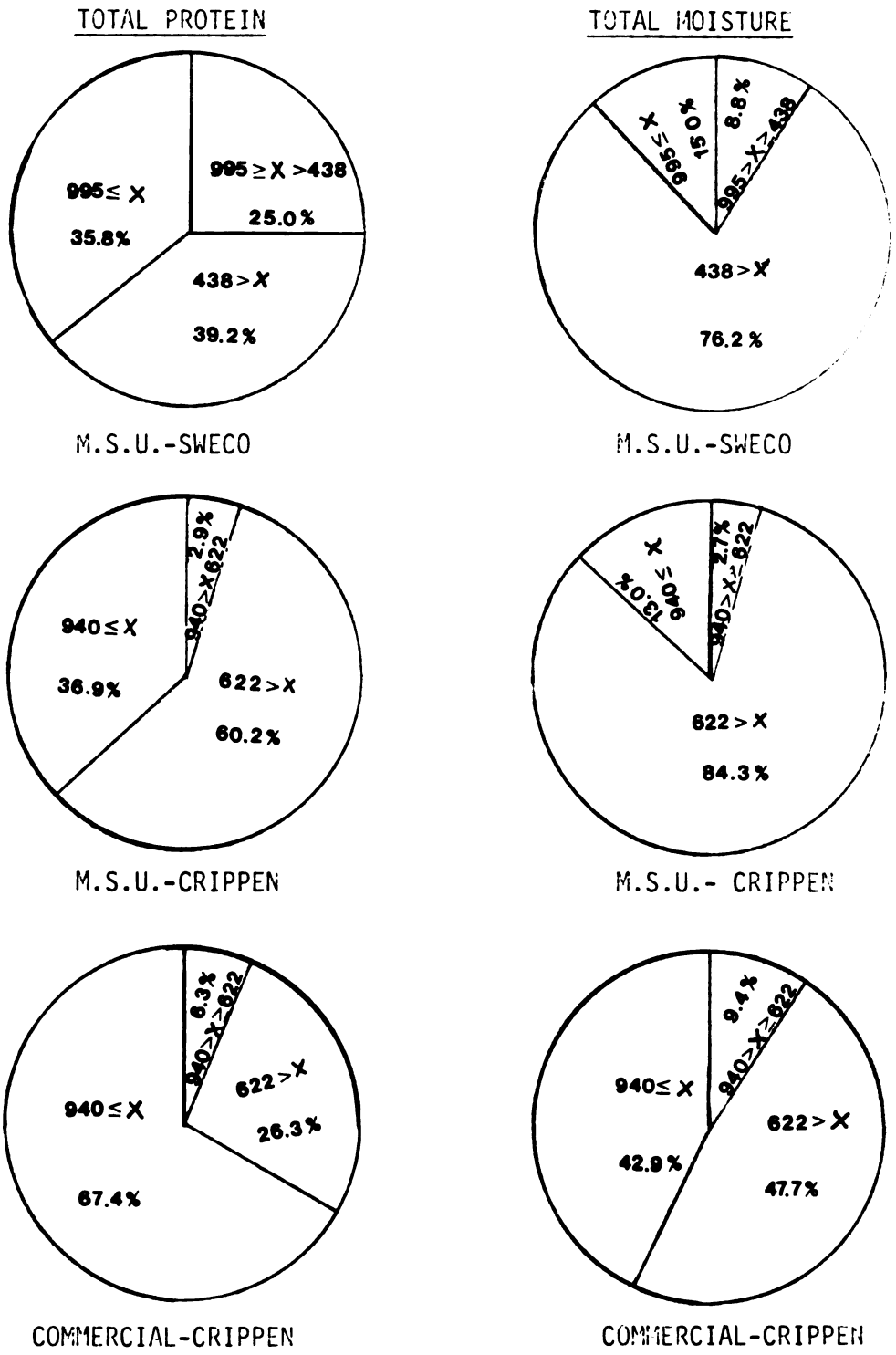

Figure 12.--Distribution of Total Protein and Total Moisture in Feed by Particle Size (μ)

The division of total protein mass was more evenly distributed in the M.S.U. SWECO stillage than in either of the other two stillages. The commercial CRIPPEN separated stillage had its highest contained protein in its large particle fraction, whereas the M.S.U. CRIPPEN separated stillage's appeared in its fine particle fraction.

The distribution of the total moisture in the commercial CRIPPEN separated stillage is about the same for the large and fine particle fractions. In the other two separations, the fine particle fraction contains most of the moisture.

Buchner Funnel with Whatman Paper

The fine particle fraction, previously designated as "Wet," was further separated using a buchner funnel. Tests were conducted both on SWECO and CRIPPEN separated stillage fine particle fractions. Four different types of Whatman filter paper were used. The results are summarized in Table 10.

This separation did an excellent job capturing much of the remaining protein. The captured protein remained on the filter paper. Overall, there was little variation whether or not a higher or lower porosity filter paper was used in the capture of the protein.

After the filtration, the filtrate obtained was clear, but mold growth did occur despite refrigeration during the time lag between the filtration run and the protein analysis. This spoilage is probably responsible for the variation in protein content.

TABLE 10.--Filtration results using Whatman paper to further separate M.S.U. stillage fine particle fractions

Paper No.	Filter Speed of Paper	Porosity[a] of paper (μ)	N Content[b] in feed	N Content[b] in Filtrate	% Protein Passing Filter
	Particle Size x < 438μ and moisture content = 96.4%				
4	Fast	20-25	1.8	0.20	11.1
1	Medium Fast	11	1.8	0.25	13.9
2	Medium	8	1.8	0.25	13.9
5	Slow	2.5	1.8	0.20	11.1
	Particle Size x < 724μ and Moisture Content = 95.8%				
1	Medium Fast	11	2.7	0.319	11.8
2	Medium	8	2.7	0.288	10.7
	Particle Size x < 724μ and Moisture Content = 96.6%				
4	Fast	20-25	2.5	0.238	9.5
2	Medium Fast	8	2.5	0.331	13.2
5	Slow	2.5	2.5	0.219	8.8

[a]Whatman Paper Division, 1979 Laboratory Catalog Paper Products, Publication 800, Clifton, New Jersey.

[b]N stands for nitrogen. Unit is mg N/ml.

Although the mold does not affect protein content, it does produce a tough rubbery growth. If this growth is not broken down sufficiently, the sample will not be representative. This procedure was very difficult and often unsuccessful.

Since the filter paper is made of a cellulose material, there is no problem in feeding it along with the captured protein to cattle. Furthermore, they would be difficult to separate. However, on a large scale basis, this method of capture is unfeasible due to the expense of the materials involved and the labor and time required to separate large volumes of material. The filtration rate is very slow.

EIMCO Filter

No separation was possible.

Analysis of Alternative Uses of the "Wet" Fraction

The "Wet" fraction could be utilized as fertilizer because of the certain minerals contained in the solids portion of the stillage. The solids portion has the following mineral content: 1.68% phosphorus, 2.2% potassium, and 4.78% nitrogen.[24] The remainder of the solids material is organic matter such as carbohydrates and very small amounts of other minerals such as calcium. An economic analysis can be performed on all the products basing it on a bushel of corn.

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[24]Personal conversation with Gary M. Webber, Research Assistant, Animal Science, Michigan State University, April 1983.

The April, 1983,[25] commodity prices are: corn @ $3.09/bu, anhydrous ethanol @ $1.70/gal, DDGS @ 6.5¢/lb, phosphorus @ 22.8¢/lb, potassium @ 11.4¢/lb, nitrogen @ 30.0¢/lb, and lime @ 1.675¢/lb. Assume that 7.19 lbs of dry matter (DDGS) are produced per gallon of ethanol and 2.5 gallons of ethanol are produced per bushel of corn.[26] There is an additional cost for neutralizing the acidity of the material to be used as fertilizer, but it is negligible. (See Appendix B). The material has a pH of 4.0. If it were applied directly to the soil without neutralization, it would be detrimental to plant life. In Scotland, this material has been used successfully as a fertilizer after it was neutralized with lime.[27]

If this fine material is not utilized as fertilizer, it must be disposed of. The hauling expense would vary, depending on the number of miles the material is transported and the size of the hauling operation. Cost estimates range from 1.5¢/gal up to 6.0¢/gal.[28] There might also be a septic tank charge once the material is at the disposal site. An upper bound on this cost is

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[25]Chemical Marketing Reporter, April 1983, Schnell Pub. Co., Inc. Also The Drovers Journal, Shawnee Mission, KN, April 1983.

[26]J. Waller and Gary M. Webber, "Development of a 'Controllable' Farm Scale Research Still and Assoc. Research Package" (Paper prepared for the Department of Animal Science, Michigan State University).

[27]Personal conversation with Gary M. Webber, Research Assistant, Animal Science, Michigan State University, April 1983.

[28]Personal conversation with Dr. Mackenzie L. Davis, professor of Civil and Sanitary Engineering, Michigan State University, July, 1983.

1.0¢/gal.[29] Therefore, on the high end, disposal costs should run 7.0¢/gal.

Table 11 tabulates the market value breakdown of the products produced under two headings. Under the first heading, the "Wet" fraction is utilized as fertilizer, while under the second heading it is sent to a disposal. For the CRIPPEN commercial stillage, the "Intermediate" fraction is added to the "Wet" fraction since its moisture content is so high. All figures are based on one bushel of corn and maximum disposal costs were used. (See Appendix B.)

For a true economic assessment, investment and operational costs must be included. The SWECO device costs more than the CRIPPEN device, but operationally it is less labor intensive. Since labor costs constitute a major expense in any operation, the number of man-hours required is important.

Excluding investment and operational costs, it can be seen from Table 11 that the Crippen separated commercial product has a slightly better market value than the others, when the "Wet" fraction is used as fertilizer. However, it has a significantly better market value than the others when the "Wet" fraction is disposed of. It should be noted though that the DDGS of the CRIPPEN separated commercial product is slightly wetter than the other two. It is assumed that percent protein (dry basis) is not a limiting criteria. The CRIPPEN commercial product had the highest contained protein, but the lowest percent protein on a "dry" basis.

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[29]Personal conversation with Dr. Mackenzie L. Davis, professor of Civil and Sanitary Engineering, Michigan State University, July 1983.

TABLE 11.--A market value breakdown of products per bushel of corn

"Wet" Fraction Utilized as Fertilizer		"Wet" Fraction Sent to Disposal	
SWECO--M.S.U. Stillage			
Corn	$3.09 (-)	Corn	$3.09 (-)
Anhydrous Ethanol	4.25	Anhydrous Ethanol	4.25
Fertilizer Value	0.1475	DDGS (coarse + int.)*	0.7045
DDGS (coarse + int.)*	0.7045	Disposal Cost	1.64 (-)
TOTAL Market Value	$2.012	TOTAL Market Value	$0.224
CRIPPEN--Commercial Stillage			
Corn	$3.09 (-)	Corn	$3.09 (-)
Anhydrous Ethanol	4.25	Anhydrous Ethanol	4.25
Fertilizer Value	0.1280	DDGS (coarse)*	0.8438
DDGS (coarse)	0.7659	Disposal Cost	0.92 (-)
TOTAL Market Value	$2.054	TOTAL Market Value	$1.084
CRIPPEN--M.S.U. Stillage			
Corn	$3.09 (-)	Corn	$3.09 (-)
Anhydrous Ethanol	4.25	Anhydrous Ethanol	4.25
Fertilizer Value	0.2189	DDGS (coarse + int.)*	0.4811
DDGS (coarse + int.)*	0.4811	Disposal Cost	1.83 (-)
TOTAL Market Value	$1.860	TOTAL Market Value	$0.189 (-)

*Each DDGS figure assumes that all the material not contained in the "Wet" fraction can be used as DDGS. (Recall that the CRIPPEN commercial stillage's "Intermediate" fraction is added to the "Wet" fraction for the tabulation of Table 11. Its moisture content is comparable.) This material contains water, so it is not truely DDGS, but its market value is based on contained dry solids. If a portion of this material is deemed too wet to be used as feed, it must either be used as fertilizer or disposed of.

Using the "Wet" fraction material as a fertilizer is more viable. Disposal costs are high relative to the raw material[30] cost and the products' market value.

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[30]The only raw material cost that is included in Table 11 is corn. There are also some chemical costs which vary, depending on the fermentation recipe.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

The fractionation of M.S.U. produced stillage into various particle sizes allows for the preparation of enriched (protein-wise) product. The quantity of this material is relatively small, however, and it would best be produced as a speciality item if at all.

A significant amount of the protein content remains in the "liquid" phase and simple screening would not permit the recovery of this material, although ultra fine filtration recovered most of it. Filtration has its limitations due to the expense and limited throughput handling capability. The screening operation reduces the moisture content of the stillage such that animal feeding is more practical. However, something must be done with the material which contains the fines. As far as recovering the solids in this material for use as animal feed, drying by evaporation seems to be the only solution. Conventional methods of evaporation are energy intensive and the value of the material recovered probably is not worth the time and the expense for a farm scale ethanol plant. However, this material can be utilized as fertilizer because of the minerals contained in it.

Of the tests run, the CRIPPEN separated commercial product gave the best product market value. It is assumed its slightly higher

moisture content is not a detriment. However, the SWECO device requires less labor than the CRIPPEN device, even though it costs more to purchase. A separation using the SWECO device with the commerical product (which has a high solids content) would probably give the best economics. The coarser grind is responsible for the higher solids content in the larger particle size. An actual separation of this nature should be run for verification. If moisture is a large factor, then the SWECO separated M.S.U. product is the best choice. It had the lowest moisture content and the next highest product market value.

Further work needs to be done on protein recovery in the "Wet" fraction if an enriched (protein-wise) product is ever going to be produced in notable amounts. A significant amount of protein is contained in this fraction. Possibly, some type of membrane could be used for such a separation.

APPENDICES

APPENDIX A

SEPARATION DATA TABLES

TABLE A-1.--SWECO Separator-M.S.U. Stillage

Particle Size (μ)	% of Total Flow	% Moisture	% Protein (Dry Basis)
995 ≤ x	16.0	82.8	27.5
995 ≤ x	16.6	81.7	28.3
995 ≤ x	12.6	82.3	27.9
995 ≤ x	12.9	82.3	27.9
995 ≤ x	20.9	87.0	27.4
995 ≤ x	15.1	80.0	27.3
995 ≤ x	17.8	83.9	29.1
995 ≤ x	18.0	82.1	26.8
995 ≤ x	15.3	83.7	27.1
995 > x ≥ 438	12.0	87.8	52.3
995 > x ≥ 438	8.7	87.9	48.8
995 > x ≥ 438	7.9	87.8	50.6
995 > x ≥ 438	8.1	87.8	50.6
995 > x ≥ 438	7.4	86.8	49.4
995 > x ≥ 438	8.1	86.7	51.6
995 > x ≥ 438	7.3	88.0	39.0
995 > x ≥ 438	9.8	88.7	52.1
995 > x ≥ 438	11.3	88.9	52.1
438 > x	72.0	95.9	27.1
438 > x	74.7	96.5	36.0
438 > x	79.5	96.6	37.0
438 > x	79.0	96.4	34.0
438 > x	71.7	96.4	35.5
438 > x	76.8	96.4	32.2
438 > x	74.9	96.4	31.0
438 > x	72.2	96.5	39.3
438 > x	73.4	96.5	37.0

TABLE A-2.--CRIPPEN Separator-M.S.U. Stillage

Particle Size (μ)	% of Total Flow	% Moisture	% Protein (Dry Basis)
940 $\leq$ x	16.1	81.8	24.2
940 $\leq$ x	16.1	82.6	28.3
940 $\leq$ x	11.8	82.6	31.7
940 $\leq$ x	13.7	82.6	32.0
843 $\leq$ x	11.6	81.2	32.3
843 $\leq$ x	33.1	81.6	34.2
814 $\leq$ x	23.4	81.6	29.1
814 $\leq$ x	19.4	82.4	29.8
814 $\leq$ x	11.9	82.1	29.4
814 $\leq$ x	15.1	82.9	30.5
940 > x $\geq$ 843	0.4	94.3	29.5
940 > x $\geq$ 724	14.6	92.8	32.8
940 > x $\geq$ 724	5.3	89.5	37.1
940 > x $\geq$ 622	2.6	95.1	32.1
940 > x $\geq$ 622	2.6	93.0	29.3
843 > x $\geq$ 724	4.4	93.5	32.2
843 > x $\geq$ 724	7.6	92.0	33.2
814 > x $\geq$ 724	2.4	90.7	37.8
814 > x $\geq$ 686	6.8	90.1	37.0
814 > x $\geq$ 622	2.0	90.0	37.5
814 > x $\geq$ 622	1.1	89.6	40.3
724 > x	83.9	95.0	31.2
724 > x	62.5	93.6	25.3
724 > x	80.8	94.6	32.0
724 > x	71.7	95.3	34.7
724 > x	82.7	94.1	29.4
686 > x	81.3	95.7	35.5
686 > x	83.8	95.2	31.5
622 > x	83.4	95.6	33.3
622 > x	76.5	95.4	32.3
622 > x	74.6	94.5	27.7

TABLE A-3.--CRIPPEN Separator-Commercial Stillage

Particle Size (μ)	% of Total Flow	% Moisture	% Protein (Dry Basis)
940 $\leq$ x	57.7	86.6	24.3
940 $\leq$ x	54.4	87.1	26.2
814 $\leq$ x	53.9	85.6	23.5
814 $\leq$ x	33.3	83.9	20.6
724 $\leq$ x	10.0	85.5	26.4
686 $\leq$ x	26.0	84.6	20.2
686 $\leq$ x	26.0	85.4	22.6
940 > x $\geq$ 843	3.8	94.2	25.4
940 > x $\geq$ 814	6.4	94.7	25.7
940 > x $\geq$ 622	11.5	93.6	23.6
814 > x $\geq$ 724	8.2	92.1	24.6
814 > x $\geq$ 724	8.2	94.8	24.8
814 > x $\geq$ 686	5.0	95.0	26.2
814 > x $\geq$ 622	4.8	94.5	25.6
814 > x $\geq$ 622	4.8	94.4	25.8
724 > x $\geq$ 686	10.1	95.2	28.9
724 > x $\geq$ 686	10.1	94.7	22.2
724 > x $\geq$ 622	3.9	93.8	25.8
724 > x $\geq$ 622	3.9	94.0	24.8
686 > x $\geq$ 622	7.5	95.5	30.3
686 > x $\geq$ 622	7.5	95.0	28.3
843 > x	36.8	94.8	25.5
814 > x	35.9	94.3	22.5
724 > x	58.5	94.6	24.8
686 > x	76.0	94.6	23.5
686 > x	41.1	94.5	24.4
622 > x	34.2	94.4	22.4
622 > x	51.6	94.3	20.4
622 > x	86.1	94.8	26.8

TABLE A-4.--Fraction Yields

Particle Size (μ)	Avg Yield, lbs	lbs H_2O	lbs Solids	lbs Protein (Dry Basis)
SWECO Separator-M.S.U. Stillage				
$995 < x$	17.0	14.2	3.0	0.83
$995 \geq x \geq 438$	9.3	8.2	1.1	0.58
$438 > x$	73.7	71.0	2.7	0.91
CRIPPEN Separator-M.S.U. Stillage				
$940 < x$	13.9	11.5	2.4	0.70
$940 \geq x \geq 724$	10.0	9.1	0.9	0.31
$724 > x$	76.1	71.9	4.2	1.28
$940 < x$	14.7	12.1	2.6	0.76
$940 \geq x \geq 622$	2.7	2.5	0.2	0.06
$622 > x$	82.6	78.6	4.0	1.24
$843 < x$	21.4	17.4	4.0	1.33
$843 \geq x \geq 724$	5.7	5.3	0.4	0.13
$724 > x$	72.9	68.9	4.0	1.22
$814 < x$	18.2	15.0	3.2	0.95
$814 \geq x \geq 724$	2.5	2.3	0.2	0.18
$724 > x$	79.3	74.9	4.4	1.34
$814 < x$	16.4	13.5	2.9	0.86
$814 \geq x \geq 686$	6.4	5.8	0.6	0.22
$686 > x$	77.2	73.7	3.5	1.17
$814 < x$	18.0	14.8	3.2	0.95
$814 \geq x \geq 622$	1.6	1.4	0.2	0.08
$622 > x$	80.4	76.5	3.9	1.21
CRIPPEN Separator-Commercial Stillage				
$940 < x$	58.0	50.4	7.6	1.91
$940 \geq x \geq 843$	3.9	3.7	0.2	0.05
$843 > x$	38.1	36.1	2.0	0.51
$940 < x$	57.0	49.5	6.5	1.89
$940 \geq x \geq 814$	6.5	6.2	0.3	0.08
$814 > x$	36.5	34.4	2.1	0.47
$940 < x$	44.9	39.0	5.9	1.49
$940 \geq x \geq 622$	9.2	8.6	0.6	0.14
$622 > x$	45.9	43.4	2.5	0.58
$814 < x$	39.5	33.5	6.0	1.32
$814 \geq x \geq 724$	7.5	7.0	0.5	0.12
$724 > x$	53.0	50.1	2.9	0.72

TABLE A.4.--Continued

Particle Size (μ)	Avg Yield, lbs	lbs H_2O	lbs Solids	lbs Protein (Dry Basis)
814 < x	40.7	34.5	6.2	1.37
814 ≥ x ≥ 686	4.6	4.4	0.2	0.05
868 > x	54.7	51.7	3.0	0.72
814 < x	41.3	35.0	6.3	1.39
814 ≥ x ≥ 622	4.5	4.3	0.2	0.05
622 > x	54.2	51.2	3.0	0.70
724 < x	12.7	10.9	1.8	0.48
724 ≥ x ≥ 686	12.8	12.2	0.6	0.15
686 > x	74.5	70.4	4.5	0.98
724 < x	14.0	12.0	2.0	0.53
724 ≥ x ≥ 622	5.5	5.2	0.3	0.08
622 > x	80.5	76.1	4.4	1.02
686 < x	28.6	24.3	4.3	0.92
686 ≥ x ≥ 622	8.3	7.9	0.4	0.12
622 > x	63.1	59.6	3.5	0.81

TABLE A.5.--Moistures and proteins

Particle Size (μ)	Moisture, %			Protein, %		
	Avg.	High	Low	Avg.	High	Low
SWECO Separator-M.S.U. Stillage						
995 < x	82.9	87.0	80.0	27.7	29.1	26.8
995 ≥ x ≥ 438	87.7	88.9	86.7	49.6	52.3	39.0
438 > x	96.4	96.6	95.9	34.3	39.3	27.1
CRIPPEN Separator-M.S.U. Stillage						
940 < x	82.4	82.6	81.8	29.1	32.0	24.2
940 ≥ x ≥ 843	94.3	94.3	94.3	29.5	29.5	29.5
940 > x ≥ 724	91.2	92.8	89.5	35.0	37.1	32.8
940 > x ≥ 622	94.1	95.1	93.0	30.7	32.1	29.3
843 > x	81.4	81.6	81.2	33.3	34.3	32.2
843 > x ≥ 724	92.8	93.5	92.0	32.7	33.2	32.2
814 > x	82.4	82.9	82.1	29.7	30.5	29.1
814 > x ≥ 724	90.7	90.7	90.7	37.8	37.8	37.8
814 > x ≥ 686	90.1	90.1	90.1	37.0	37.0	37.0
814 > x ≥ 622	89.8	90.0	89.6	38.9	40.3	37.5
724 > x	94.5	95.3	94.1	30.5	37.4	25.3
686 > x	95.5	95.7	95.2	33.5	35.5	31.5
622 > x	95.2	95.6	94.5	31.1	33.3	27.7
CRIPPEN Separator-Commercial Stillage						
940 < x	86.9	87.1	86.6	25.3	26.2	24.3
940 ≥ x ≥ 843	94.2	94.2	94.2	25.4	25.4	25.4
940 > x ≥ 814	94.7	94.7	94.7	25.7	25.7	25.7
940 > x ≥ 622	93.6	93.6	93.6	23.6	23.6	23.6
814 > x	84.8	85.6	83.9	22.1	23.5	20.6
814 > x ≥ 724	93.5	94.8	92.1	24.7	24.8	24.6
814 > x ≥ 686	95.0	95.0	95.0	26.2	26.2	26.2
814 > x ≥ 622	94.5	94.5	94.4	25.7	25.8	25.6
724 > x	85.5	85.5	85.5	26.4	26.4	26.4
724 > x ≥ 686	95.0	95.2	94.7	25.6	28.9	22.2
724 > x ≥ 622	93.9	94.0	93.8	25.3	25.8	24.8
686 > x	85.0	85.4	84.6	21.4	22.6	20.2
686 > x ≥ 622	95.3	95.5	95.0	29.3	30.3	28.3
843 > x	94.8	94.8	94.8	25.5	25.5	25.5
814 > x	94.3	94.3	94.3	22.5	22.5	22.5
724 > x	94.6	94.6	94.6	24.8	24.8	24.8
686 > x	94.6	94.6	94.6	24.0	24.4	23.5
622 > x	94.5	94.8	94.3	32.2	26.8	20.4

APPENDIX B

SAMPLE CALCULATIONS

ETHANOL AND DDGS MARKET VALUE SAMPLE CALCULATIONS

I. Ethanol

Basis: 1 bushel of corn

2.5 gal ethanol/bu corn x \$1.70/gal ethanol = \$4.25/bu corn

II. DDGS

Basis: 1 bushel corn

5,000,000 gal ethanol

18,000 tons DDGS

This gives 7.19 lb of DDGS per gallon of ethanol

a.

SWECO-M.S.U. Stillage (μ)	lbs, dry matter
$995 < x$	3.0
$995 \geq x \geq 438$	1.1
$438 > x$	2.7
	6.8

Adding the "Dry" and "Intermediate" fractions together and then dividing by the total gives

(3.0 + 1.1)/6.8 = 0.603 or 60.3%

b. $\frac{0.603}{} \times \frac{7.19 \text{ lb DDGS}}{\text{gal ethanol}} \times \frac{2.5 \text{ gal. ethanol}}{\text{bu corn}} \times \frac{\$0.065}{\text{lb DDGS}} =$

\$0.7045/bu corn

FERTILIZER VALUE SAMPLE CALCULATIONS

I. Basis: 5,000,000 gal ethanol
18,000 tons DDGS

This gives 7.19 lb of DDGS per gallon of ethanol.

II.

SWECO-M.S.U. Stillage (μ)	lbs, dry matter
$995 \leq x$	3.0
$995 \geq x \geq 438$	1.1
$438 < x$	2.7
	6.8

In "Wet" fraction or 438 > x, 2.7/6.8 = 0.397 or 39.7%

III. Basis: 7.19 lb dry matter/gal ethanol

SWECO-M.S.U. Stillage

$$7.10 \text{ lb dry matter/gal ethanol} \times 0.397 = \frac{2.85 \text{ lb dry matter}}{\text{gal ethanol}}$$

438 > x contains 2.85 lb dry matter/gal ethanol

IV. Basis: 2.5 gal ethanol/bu corn

SWECO-M.S.U. Stillage:

$$\frac{2.85 \text{ lb dry matter}}{\text{gal ethanol}} \times \frac{2.5 \text{ gal ethanol}}{\text{bu corn}} = \frac{7.13 \text{ lb dry matter}}{\text{bu corn}}$$

V. The "Wet" Fraction material is 1.68% phosphorus, 2.2% potassium, and 4.78% nitrogen on a dry basis.

SWECO-M.S.U. Stillage

$$\frac{7.13 \text{ lb dry matter}}{\text{bu corn}} \times 0.0168 = \frac{0.12 \text{ lb phosphorus}}{\text{bu corn}}$$

$$\frac{7.13 \text{ lb dry matter}}{\text{bu corn}} \times 0.022 = \frac{0.157 \text{ lb potassium}}{\text{bu corn}}$$

$$\frac{7.13 \text{ lb dry matter}}{\text{bu corn}} \times 0.0478 = \frac{0.341 \text{ lb nitrogen}}{\text{bu corn}}$$

a. Molecular Weight of $Ca(OH)_2$ = 74

$$\frac{88.41\ l}{bu\ corn} \times \frac{4.995 \times 10^{-5}\ gmole}{2} \times \frac{74\ g}{g\ mole} \times \frac{0.0022\ lb}{g}$$

$$= \frac{7.189 \times 10^{-4}\ lb}{bu\ corn}$$

$$\frac{7.189 \times 10^{-4}}{bu\ corn} \times \frac{\$0.01675}{lb} = \frac{\$1.204 \times 10^{-5}}{bu\ corn}$$

DISPOSAL COST OF "WET" FRACTION SAMPLE CALCULATION

I. Hauling cost range (depends on the number of miles)

= 1.5¢/gal to 6.0¢/gal.

Septic tank charges (may not be applicable)

= 1.0¢/gal (high estimate)

Assuming maximum costs, cost of disposal = 7.0¢/gal.

II. Basis: 1 bushel of corn
density of "Wet" fraction = 8.34 lb/gal

a.

SWECO-M.S.U. Stillage (μ)	lbs, dry matter	avg yield, lbs
$995 \leq x$	3.0	17.0
$995 \geq x \geq 438$	1.1	9.3
$438 > x$	2.7	73.7
	6.8	

In "Wet" fraction, 2.7/6.8 = 0.397 or 39.7%

From previous sample calculations, it is known that there are 7.19 lb of DDGS or dry matter per gallon of ethanol.

b. $$0.397 \times \frac{7.19 \text{ lb DDGS}}{\text{gal ethanol}} \times \frac{73.7 \text{ lb}}{2.7 \text{ lb DDGS}} \times \frac{\text{gal}}{8.34 \text{ lb}} \times$$

$$\frac{2.5 \text{ gal ethanol}}{\text{bu corn}} \times \frac{\$0.07}{\text{gal}} = \$1.64/\text{bu corn}$$

APPENDIX C

KJELDAHL ANALYSIS

MODIFIED KJELDAHL METHOD - M.S.U. DEPARTMENT

OF ANIMAL SCIENCE

Digestion of Samples for Auto Kjeldahl

1. General. Weigh a sample of an amount sufficient enough so that 50-200 ppm may be obtained in a minimum of 25 ml. For each 1% crude protein (CP), figure 0.2 g in 250 ml final volume.

For example:	10% $CuSO_4$	K_2SO_4	Conc. H_2SO_4
10% CP use 1-2 g (dry matter) in 250 ml	2 ml	2 g	15 ml
0.8 g (dry matter) in 100 ml	1 ml	1 g	12 ml
0.6 g (dry matter) in 50 ml	1 ml	1 g	10 ml
0.2 g (dry matter) in 25 ml	1 ml	1 g	4 ml

2. To calculate the approximate ppm for your sample:

   a. 1 ppm = 1g/1,000,000 g or 1 g/1,000,000 ml (using the approximation that 1 ml or 1 cc of H_2O weights 1 g)

      Divide by 1,000,000. Then 1 ppm = 1 µg/g or 1 µg/ml

   b. CP% ÷ 100 = CP/g feed and CP/g ÷ 6.25 = g N/g feed

      g N x 1,000,000 = µg and µg/size of flask = µg/ml or ppm

      CP% x 1,000,000/(100 x 6.25 x (250 ml flask))

      $$= \frac{CP \times 1{,}000{,}000}{625 \times \text{flask size}}$$

   For example:

   For a sample estimated to be 10% CP in a 250 ml flask:

   $$\frac{10\% \times 1{,}000{,}000}{100 \times 6.25 \times 250} = 64 \text{ g/ml or } 64 \text{ ppm}$$

   1 g in a 250 ml flash would be between 50 - 200 ppm.

For a sample estimated to be 6% CP in a 250 ml flask:

$$\frac{6\% \text{ CP} \times 1{,}000{,}000}{100 \times 6.25 \times 250} = 38.4 \text{ ppm}$$

This amount is not within the 50-200 ppm range, so 2 g must be used to obtain 76.8 ppm which is within the range.

3. Weigh about 1.0 g of feed mix on a weighing paper. Record the exact weight. Do not touch the sample. Transfer this sample to a special 250 ml volumetric flask. Add 2 g of K_2SO_4 and then carefully add 2 ml of $CuSO_4$. Add some concentrated H_2SO_4. To speed up digestion, carefully add 4-5 ml of 30% H_2O_2 to the above mixture and let it sit overnight if desired (see 4).

4. Oxidation digestion. All nitrogen compounds (except NO_3^- (nitrate)) are oxidized to NH_3 and dissolved in the acid solution as $(NH_4)_2SO_4$. Allowing the sample to sit overnight reduces frothing, but this is unnecessary. Digestion may begin immediately. During the charring stage, if frothing occurs, shake well or swirl the sample. The frothing doesn't harm the analysis, it simply increases the odds the sample will puff itself right out of the flask.

   a. Set the heaters at #3-4 to begin. Gradually increase the heat. Turn to HI when the sample starts fuming with white fumes of SO_2 which comes from the decomposition of the H_2SO_4. These fumes are poisonous!! Shake down the charred material occasionally.

   b. Boil until most of the solid material is in solution. The mixture will be a dark reddish brown.

   c. Remove the two flasks and cool them. Add 30% or 50% H_2O_2 from a pasteur pipet. Try to wash down the bits of charred material. Return mixture to digester.

H_2O_2 addition should not cause the mixture to sizzle and steam. It should not spit out of the flask. Cool longer if spitting occurs. Continue adding H_2O_2 until mixture is blue-green.

d. If the mixture is still somewhat yellow-green, repeat the peroxide procedure twice.

e. Boil 15 minutes past the last peroxide addition.

f. Cool to room temperture.

5. Standards. Make 5000 ppm. Weigh on an analytical balance 23.6 g $(NH_4)_2SO_4$ into a one-liter volumetric flask. Add some dionized water and mix. Make the solution up to the mark on the flask. Mix well and transfer to a glass stoppered bottle. Label and store in a cold room with parafilm or plastic wrap around the stopper.

   a. For each set of 10 samples, digest one 10 ml aliquot of the above standard in a 250 ml volumetric flash and one 5 ml aliquot in another 250 ml flask.

   b. 10 ml of 1000 = 1/100 or 0.236 g/250 ml flask. 200 N ppm
      5 ml of 1000 = 1/100 or 0.118 g/250 ml flask. 100 N ppm

6. Dilution:

   a. Dilute samples and standard to the 250 ml mark while swirling with deionized distilled water.

   b. Mix well.

   c. Pour some of the solution into two screw cap bottles-- 50 cc is plenty. Label correctly and cap tightly.

   d. Run samples on an Auto Kjeldahl unit.

7. Calculations and traces. Two aliquots from each bottle will go into the instrument. Sometimes there is a slight drift. The double injection will average the drift. Count and average the two peaks for each aliquot.

a. Average count x 2 = ppm of N or μg/ml

b. μg/ml x 250 ml = total μg in the flask

c. Total μg/ml ÷ sample weight = μg N/g sample

d. μg x 1,000,000 = g

e. g N/g sample x 100 = g% N

f. g% N x 6.25 = % CP

$$\% \text{ CP} = \frac{\text{avg count} \times 2 \times 250 \times 100 \times 6.25}{\text{sample weight} \times 1{,}000{,}000 \times (\text{dry matter})} =$$

$$\frac{\text{avg count} \times 0.3125}{\text{s.w.} \times \text{d.m.}}$$

g. (1) Run a linear regression on the average transmittance percentage versus the concentration such as:

x	y
0	0
50 ppm	24.6
100 ppm	49.5
150 ppm	73.0
200 ppm	99.5

(2) Ratio of actual slope to the theoretical value

$$= \frac{2.021}{2.000} = 1.01$$

(3) Divide the factor in f above by 1.01 and complete the calculations.

h. Average the % CP and report if the match is 90% or better.

8. mg protein/g sample = chart reading (ppm) x 2 ÷ $\frac{1000}{\text{ml of dilution}}$

$$\times \frac{6.25}{\text{sample weight in g}}$$

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