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Operation and Testing Experience With the DOE Small-Scale Fuel Alcohol Plant

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EG&G Idaho

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OPERATION AND TESTING EXPERIENCE WITH THE DOE SMALL-SCALE FUEL ALCOHOL PLANT

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ABSTRACT

This document contains information and data pertaining to the operation and testing of the Small-Scale Fuel Alcohol Plant (SSFAP), built and tested by EG&G Idaho, Inc., for the Department of Energy. The information found in this report supplements the facility design documents, which are available as identified in the text.

Operation and testing with this plant occurred mostly during FY-81 under limited budget conditions. The information produced in the SSFAP about operations, necessary equipment modifications, yeast and enzyme optimization, process quality assurance, laboratory versus plant results, plant performance with alternative feedstocks, safety, and operating procedures should be of value throughout the industry. Laboratory experimentation and subsequent trial runs of a number of starch-containing cereal grains conclusively demonstrated the flexibility and effectiveness of

this facility. Once shakedown and startup was completed, the plant routinely achieved yields of 2.6 gal of ethanol per bushel of corn. Further trial runs were conducted to reduce the cost of materials used in alcohol production by using smaller amounts of enzyme and yeast. After plant operating parameters and conversion efficiency were optimized, barley, corn dust, and dried potato waste were tested as potential feedstocks, and all performed admirably, with more than 90% conversion of the available starch to alcohol.

The authors of this report recognize that there was much more that could and should have been done with the facility. Some of these additional activities are identified in the report. Budget limitations resulted in the plant being mothballed at the start of FY-82, and construction of a lignocellulose conversion front-end unit and cheese whey studies were terminated.

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OPERATION AND TESTING EXPERIENCE WITH THE DOE SMALL-SCALE FUEL ALCOHOL PLANT

INTRODUCTION

This report contains the results of testing and operating experience with the Department of Energy's Small-Scale Fuel Alcohol Plant (SSFAP). Some of the principal features sought after in the design of this plant were minimum labor requirements, semiautomatic control, high yields, low process energy requirements, feedstock versatility, and reliability. The degree to which all of these features could be verified was limited by budget restrictions. Nevertheless, very valuable information for parties interested in small plants was produced. The reader should find the areas discussing yields, feedstock versatility, energy requirements, yeast and enzyme optimization, process quality assurance, and hardware and equipment problems all of interest. One fact that becomes increasingly apparent is that alcohol facilities of this type are really small chemical processing plants.

Plant History

In response to widespread public requests, the Department of Energy (DOE) through its Alcohol Fuels Office (AFO) committed to prepare and publish a design for a small-scale ethanol production plant suitable for use by farmers or farm cooperatives. The AFO assigned this task to EG&G Idaho, Inc., the primary contractor at the Idaho National Engineering Laboratory.

The design task was initiated in March 1980. A limited-edition "Preliminary Design Report" was published in early May and was reviewed at an open public meeting in Denver on May 15. Construction design was completed in July, and construction of a prototype plant in Idaho Falls, Idaho, started in August. A design report was prepared and limited distribution made in December 1980. The design report at that time described the plant as it was in November 1980. Operational testing started at this time resulted in minor changes to the original design. A design report revision was published in August 1981 reflecting the plant configuration as it existed in June 1981. Plant testing continued with a sharply

reduced budget through September of 1981, at which time operations with the plant were terminated.

The revised SSFAP Design Report published in August of 1981 is entitled *Design Report: Small-Scale Fuel Alcohol Plant*. The document numbers for the three-volume report are:

IDO 10088, Volume One
IDO 10088, Volume Two
IDO 10088, Volume Three.

The revised report is available through the U.S. National Technical Information Service:

U. S. Department of Commerce
National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
(703) 557-4650

Most of the operational and testing data that evolved from the SSFAP testing program were not included in the Design Report identified above. The objective of this report is to publish the testing information and make it available in the public domain.

Test Program

The test program with the SSFAP consisted of three separate phases. The first phase was a checkout of components and systems, or system operational (SO) tests, and included the first production runs along with a period of system modifications. The second phase consisted of optimization tests using corn as a feedstock and included an extended run with the plant. The third phase was an evaluation of the SSFAP with other feedstocks. These separate phases are discussed briefly below and in more detail in the body of the report or the appendices.

Phase 1 Tests—Shakedown. The Phase 1 tests started with a plant component and systems

checkout (SO tests). This was initiated just prior to the completion of plant construction and continued for a short time after construction. All electrical, electronic, and mechanical components were checked to determine if they were functioning properly. The microprocessor control system was programmed. Metering pumps, motor valves, and the vibrator feeder were calibrated. Flow, pH, pressure, and temperature measuring equipment was also calibrated. The grain handling, cooking, saccharification, fermentation, and distillation systems were checked for operational readiness. The final startup test was a series of four continuous plant runs in which all systems were operated at design levels when possible. Corn was the feedstock, and enzymes, yeast, and other required chemicals were used in this test. The information gathered was used to determine and correct deficiencies through equipment repair or design modifications, thus bringing the plant up to design operating requirements.

Phase 2 Tests—Optimization for Corn. The second phase of the testing program for the SSFAP consisted of a series of operational tests using corn as a feedstock. The purpose of the tests was to develop operational procedures and techniques so that the labor requirements for the plant

operation could be determined. Also during this period, plant energy needs were evaluated, and yeast and enzyme requirements were optimized. The final test in this series was a five-batch continuous run. The purpose of this test was to operate the plant at design levels where the automated systems would reduce the need for operator participation.

Phase 3 Tests—Alternative Feedstocks. The final period of the SSFAP testing program was used to test feedstocks other than corn. Those selected for testing were barley, dry potato waste, grain dust, and cheese whey. The objective of these tests was to determine the plant yields, enzyme and yeast requirements, and what plant modifications were necessary to process a particular feedstock. The cheese whey tests were not completed because the program was terminated. Other information added during this final period of the testing program contributed to record-keeping procedures, the practice and writing of good operating procedures, setting guidelines for the SSFAP safety program, establishing plant sterilization procedures, and determining what plant modifications would improve the overall operation.

SSFAP SYSTEM DESCRIPTION

The information necessary to understand the SSFAP system is presented in this section. A complete system design description is to be found in the three-volume design report identified in the Introduction above. To aid the reader, however, the schematic layout and the schematic diagram of the plant from the Design Report are included in this report as Figures 1 and 2, respectively. Also, the plant process P&ID drawings (Drawing 414342, Sheets 1 through 6) are shown as Figure 3. These figures and drawings are also pertinent to Appendix A.

The original design requirements for the SSFAP were:

- Continuous production of 25 gph of anhydrous (approximately 200 proof) ethanol.
- Operator time, excluding administrative tasks, to be 4 hr or less per 24 hr day.
- Existing technology and off-the-shelf equipment to be used wherever practical.
- Corn to be the primary feedstock.
- Plant lifetime to be a minimum of 10 years.
- Plant products to include dewatered stillage suitable for animal feed.

There were some modifications to these design criteria as the project progressed. A device for dewatering the byproduct stillage was included, but byproduct drying was not, because systems evaluated were too expensive for a plant of this size. Alcohol dehydration was not included for similar reasons, and the product throughput requirement was altered to 26.4 gph of approximately 190 proof ethanol (equivalent to 25 gph of anhydrous ethanol). Subsequently, commercial molecular sieve units have become available at costs that could allow economical drying of ethanol for a plant of this size. Such units could easily be added to the SSFAP. Other dehydration systems that are promising for a plant of this size are also becoming available.

The plant that evolved is reasonably complete, having the capability for feedstock preparation, cooking, saccharification, fermentation, distillation, byproduct dewatering, and process steam generation. An interesting feature is an instrumentation and control system (microprocessor) designed to allow the plant to run 24 hr/day with only 4 hr of operator attention. The design capacity of the plant is 26.4 gph of 190 proof ethanol. Most of the processes and equipment used in the plant represent conventional ethanol production technology. Two slight deviations are the control system, which is common in larger plants, and the continuous cooker, a plug flow system, which was adapted from the food industry. A discussion of operating procedures for the SSFAP plant and of plant safety are given in Appendix A.

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Figure 1. Schematic layout of the Small-Scale Fuel Alcohol Plant.

Figure 2. Schematic diagram of the Small-Scale Fuel Alcohol Plant.

Figure 3. Small-Scale Fuel Alcohol Plant P&I Diagram.

Figure 3. (continued)

REVISIONS			
ZONE	DESCRIPTION	DATE	APPROVED
A			

SIZE	AREA	DATE	TYPE	CL	DRG	414342
E	1000	10/20/80	20			
SCALE	NONE	RELEASE DATE	7/16/80	CL		

Figure 3. (continued)

Figure 3. (continued)

TEST PROGRAM RESULTS

As described in the Introduction, the SSFAP operations and testing program consisted of three phases, all leading to a set of results on overall plant performance. The results of these phases are presented in the following sections.

Phase 1 Tests

System Operation Tests. The purpose of the SO testing was to check, repair, or modify plant equipment and to bring the plant operation to design level. This testing was accomplished in two parts. First, the component checkout was completed, and then the system tests were carried out.

Component testing began at the completion of plant construction. A preliminary program for the plant computer control system was prepared for the component testing. This program was upgraded during the testing period. The preliminary program was necessary so that computer-controlled circuits could be activated for test purposes. Component testing began with a general inspection of all plant hardware and equipment for visible damage. Then, electrical circuits were closed to all electric motors and motor valves to determine if the motors were operational and rotating correctly. All drive belts and chains were checked for proper tension and alignment. All motors, couplings, and the equipment they drive were checked for alignment. All equipment requiring lubrication was lubricated to the manufacturers' specifications. All bolts, clamps, and fasteners were checked for tightness. Electrical circuits were closed to all solenoid valves to determine if they were operational. All pumps were tested to determine if they were operational. Finally, all tanks, heat exchangers, reactors, columns, and associated piping were hydrotested for leaks. In general, this was an overall check of plant hardware and equipment to determine operational readiness.

The systems testing began as soon as the component tests were complete. For this series of tests the plant was divided into the following systems: front-end (grain handling equipment), cooking, saccharification and fermentation, distillation, product storage, and byproduct handling. To test these systems, the following was accomplished:

- The program for computer control of these systems was completed.
- All temperature, pressure, and pH sensors; motor valves; metering pumps; and the vibrator feeder were calibrated.

Once the systems were tested and found to be operational, the plant was ready for the first production test.

First Production Test. The first production test was performed during the last week of January 1981. The objectives of the test were to complete operational shakedown of the plant and to identify any remaining operational problems that could prevent meeting plant design specifications.

The objectives for this test were successfully completed. One excellent result was that the plant was brought under automatic control after only four trial batches. The information necessary to upgrade the plant for operation at design levels was obtained. A detailed description of the problems encountered in this test and the solution of these problems are reported in Appendix B—System Improvements.

Phase 2 Tests

Optimization Tests. The objective of the optimization tests was to operate the plant in the automated mode at design levels: 20 bu/hr of corn, a water-to-corn mixture of approximately 20 gal/bu, complete fermentation within 54 hr, and distillation of 190 proof ethanol at 26.4 gph. The amount of enzymes and yeast necessary for the dextrinization, saccharification, and fermentation processes was to be determined. Also during this series of tests, the plant energy and operational manpower requirements were to be established.

The optimum amount of alpha-amylase for dextrinization was determined by a step-by-step reduction of the amount used from 0.19 lb/bu of corn to 0.06 lb/bu. When 0.06 lb/bu of corn was used, the mash would not liquify adequately. The amount was then increased to 0.09 lb/bu of corn, at which point adequate liquifaction took place.

During this testing period, laboratory and plant tests determined that 0.05 lb of glucoamylase/bu of corn was adequate for saccharification.

The plant is equipped with three fermentation tanks. This requires the cooking, dextrinization, saccharification, and fermentation to be completed in 72 hr, which allows approximately 54 hr of fermentation. To accomplish this, 0.12 lb of dry yeast/bu of corn or 0.07 lb of yeast conditioned in a 3.0% solution of corn syrup was required. The cost of the extra yeast was about the same as the cost of labor for conditioning yeast.

The energy requirements for the plant operation were determined from the actual steam used. The results of these determinations are shown in Table 1.

Operational manpower requirements were determined by logging the time for each operational task. This study did not include any time for administrative tasks. The results of this study are shown in Table 2.

The list in Table 2 includes tasks that are necessary when operating in a testing and a continuous mode. Task Numbers 1, 2, 5, 7, 8, 9, 10, 11, 12, 13, 14, 16, 17, and 18 would be necessary when operating in a continuous or production mode. With hourly monitoring, it would require about 9 hr and 37 min of operator time. If the monitoring of the vibrator-feeder was eliminated and the plant systems monitored every 4 hr, the operator time would be 5 hr and 25 min. There are several other tasks that should be automated to meet design criteria. This would reduce operator time further.

Extended Test. The primary objective of the extended test was to operate the plant in a con-

tinuous, automated mode while processing five full batches (approximately 240 bu of corn per batch).

The test was highly successful with respect to automated plant operation and minimum operator participation. The front-end, cooking, saccharification, and fermentation cycles required only monitoring the corn and mix pump feed rates, adjusting the pH, and adding glucoamylase and yeast. After startup, the distillation system required no operator participation. The test also confirmed that the hammer mill required a 15 hp motor in order to process corn at the design rate of 20 bu/hr. The maximum amount of corn that could be processed with the 10 hp motor was 17 to 18 bu/hr. Thus, 1017 bu of corn were processed during this test instead of the projected 1200; 2615 gal of ethanol were produced at the design proof. Yield data are reported in the SSFAP Data Summary, Table 3. Table 3 includes data results for all the testing of the SSFAP.

Phase 3 Tests

The primary objective of the Phase 3 tests was to examine plant changes that would be needed to process several alternative feedstocks. The principal result was the verification that most feedstocks in this category could be processed with relatively few changes, primarily associated with plant front-end processing sections, as might be expected.

Barley. Barley was the first alternative feedstock tested at the SSFAP. The barley tests were successful in proving that the grain could be processed in the SSFAP with only a few minor modifications. Additional testing is required to optimize the process. Plant yield data are given in Table 3.

Table 1. Energy requirements

Energy Requirements	Operation (Btu/gal)	Design (Btu/gal)
Feedstock preparation (including sterilization, etc.) through fermentation	7,000 to 9,000	9,940
Distillation	21,000 to 27,000	21,260
Total (190-proof)	30,000 to 35,000	31,200

Barley, which is a softer grain than corn with a tough hull, required approximately 50% more power to mill than corn. For this reason, a screen with larger holes, 5/64 in. ID, and rated for 180 lb/hr/hp was installed in the hammer mill. With this screen, the hammer mill could process barley at a maximum rate of 18 to 19 bu/hr, and at this processing rate, the plant could be operated with computer control. However, the particle size of the barley after milling was too large. The mash was more viscous than corn mash, and consequently, the combination of large particles and high viscosity caused plugging in the cooking system, the mash cooler, and the beer still bottoms heat exchanger. Agitation was not adequate to keep the solids suspended in the fermentation and beer tanks.

A second test, using a screen with 3/64 in. ID holes and rated for 100 lb/hr/hp, was more successful. The maximum milling rate was approximately 12 bu/hr. A reduction in the feed pump rate to approximately 6 gpm was required to maintain a water-to-barley ratio of 25 gal/bu. The

reduced particle size of the milled barley eliminated the plugging problems, and the agitation in the fermentation and beer tanks was adequate to keep the solids suspended. Cooking time was increased and required either a partial manual operation of the plant or modification of the computer program.

Potato Waste (Dry). The objective of processing dry potato waste in the SSFAP was to confirm parameters extrapolated from bench scale tests of potato waste conversion to ethanol.

The potato waste used in this test was made up of slices, floor sweepings, and subquality flakes. This nonhomogeneous material caused some variation in the feed rate through the vibrator feeder. The only modification to the plant system was the installation of the screen with 5/64 in. ID holes, rated for 180 lb/hr/hp, in the hammer mill. There were no other modifications and no processing problems. The water to feedstock mixture was 1.0 gal of water to 1.4 lb of potato waste. Fermentation produced 5.1% v/v ethanol in the

Table 2. Plant operations manpower requirements

Task Number	Operation	Time (min)
1	Transfer corn (~240 bu)	30
2	Clean and sterilize fermentation tank	20
3	Start up boiler	10
4	Start computer and turn on knife switches	5
5	Prepare alpha-amylase	15
6	Start distillation	45
7	Calibrate vibrator feeder per startup	15
8	Line up valves and start cooking cycle	15
9	Calibrate feed pump (two to three times)	40
10	Check vibrator-feeder rate (every 30 min)	72
11	Adjust pH (calibration check and sampling)	15
12	Set up valves to circulate through the fermentation cooler	5
13	Weigh and add glucoamylase	10
14	Weigh and add yeast	10
15	Wash down beer tank and flush distillation column lines	20
16	Load distillers grains	30
17	General plant cleanup	60
18	Monitor plant systems (hourly)	240

Table 3. SSFAP data summary

Material Requirements and Plant Yields	Plant Tests					
	Enzyme Optimization	Extended Tests (five runs)	Yeast Optimization	Barley	Potato Waste	Corn Dust
Feedstock (lb)	26,100	56,960	12,428	8,072	4,300	4,520
(bu)	457	1,017	222	176		
Alpha-amylase (lb)	58	106	25.5	25.7	12.4	20
Glucosylase (lb)	24	60	12	7	5.5	9
Yeast, type	DADY ^a	DADY ^a	DADY ^a	DADY ^a	DADY ^a	DADY ^a
unconditioned (lb)	50		14		6.0	12
conditioned (lb)		60	8	4		
Sulfuric acid (N)	35	35	35	35	35	35
(lb)	72	9	27	18	11	18
Beer, volume (gal)	14,445	26,660	6,655	5,580	3,735	3,090
% v/v ethanol	8.7	9.8	8.7	4.9	5.1	6.2
Mash pH, initial	6.2	5.2 ^b	6.4	6.0	6.3	5.6
after acid addition	5.2	5.2	5.2	4.8	5.3	4.9
Ethanol proof	190	190	190	190	190	190
Ethanol yield (gal/bu)	2.68	2.57	2.60	2.20		
Laboratory yield (gal/bu)	2.70 to 2.75	2.70 to 2.75	2.70 to 2.75	2.45		
Ethanol yield (gal/100 lb)					4.4	4.3
Laboratory yield (gal/100 lb)					4.8	4.4 to 4.5

a. Distillers active dried yeast (Universal Foods).

b. Unverified pH reading.

beer, which was distilled to produce 137 gal of 190 proof ethanol. A large sample of the stillage was collected for evaluation (see discussion under Laboratory Support Program, below). Additional data are given in Table 3.

Corn Dust. The objective of this test was to study the cooking, fermentation, and distillation processes when using corn dust as a feedstock. Information obtained from this test was used to determine what modifications to the SSFAP would be required so that grain dust could be used as the primary feedstock.

The corn dust used in this test was purchased from a large grain handling facility in the midwest. The waste material results from the stringent dust control requirements for these facilities. The starch content of the dust was almost equal to the starch content of corn. The material is like a flour and quickly forms bridges in flow areas. This particular property caused problems in the front end (grain handling equipment) of the plant.

The fermentation and distillation processes proceeded without any serious problems in this test. About 4000 lbs of corn dust were fermented to produce a beer with a 6.2% v/v ethanol content. The overall plant yield was within 7.0% of the laboratory projected yield. Problems in the front-end processing, in the mix tank, and with the feed pump resulted from the bridging, nonwetting, and abrasive properties of the corn dust.

The first of these problems, bridging, increased the time for filling the day bin by a factor of 10 over the time required for the transfer of an equal volume of corn. Bridging caused the flow from the day bin to the vibrator feeder to be erratic. This problem could be corrected by agitating the dust in the day bin and/or using a bin with a vibrating outlet cone.

Another problem area occurred in the mix tank. The corn dust did not wet easily and would float on the water in the mix tank, eventually backing up into the hammer mill. Increased agitation properly located in the mix tank would solve this problem.

The most serious problem was caused by the abrasiveness of the corn dust. The feed pump failed after approximately 11 hr of operation. To determine the cause of failure, the pump was disassembled. Considerable amounts of fine sand particles were found on the working parts of the pump. This sand presumably was introduced to the corn dust from floor sweepings or during transportation, etc., and may not be typical of all grain dusts. The pump was a piston-type positive displacement pump. The moving parts in the pump were machined to very close tolerances and case hardened. These parts showed excessive wear. To overcome this problem, a centrifugal or a tubing pump could be used for processing corn dust containing sand. An improved feed pumping system is described in Appendix B as part of the recommendations for equipment improvements. Additional data are given in Table 3.

SSFAP LABORATORY SUPPORT AND PROCESS QUALITY CONTROL PROCEDURES AND RESULTS

A laboratory support program and process quality control program accompanied the SSFAP tests. These programs and procedures are expected to be of interest to entities engaged in alcohol production, particularly at the small-scale production level. The programs, procedures, and results are described in separate sections below.

Laboratory Support Program

Before startup and during actual processing in any pilot-scale or commercial test facility, laboratory analysis is always done, not only to verify the concept but to determine the processing parameters to be used on the larger scale. During the course of operating the SSFAP, biochemists at EG&G Idaho conducted bench-scale conversions of all potential feedstocks. Determinations were made on the water-to-grain ratio, the minimum amounts of alpha-amylase and glucoamylase required to maintain optimum conversion efficiencies, the most effective means of adding yeast to initiate fermentation, and yield. A number of analytical techniques were used in each of the above determinations. The methodology in these analyses is described in Reference 1.

Corn. Because of its availability in the U.S., corn was chosen as the primary feedstock. Laboratory investigations showed that when a concentration of 0.09% w/w of alpha-amylase to ground corn was used, and glucoamylase was added at a ratio of 2500 units per bushel (0.05 lb/bu), 2.70 gal of ethanol could be produced. This was found to be the lowest concentration of enzymes that could be used while still maintaining good conversion efficiencies in laboratory experimentation. Plant trials were started using an initial concentration of 0.3% w/w of enzyme (alpha-amylase) to ground corn, and this concentration was steadily decreased until 0.125% w/w was determined to be the lowest concentration of enzyme that still maintained good conversion. Throughout these runs, the glucoamylase concentration was held at 2500 units (0.05 lb/bu). Using these quantities of enzymes in the fuel alcohol production process contributed \$0.07 to the operating costs incurred per gallon of ethanol manufactured.

Yeast is also an expense in the production of alcohol. Two different practices of adding yeast to

the fermenters were explored. The first method was simply the direct addition of distiller's active dry yeast to the mash. The target time of 54 hr of fermentation was achieved with 3.5 lb of yeast per 1000 gal of mash. The second method employed a conditioning step in which a 5.0% glucose solution was prepared, adjusted to a pH of 4.8, and brought to a boil for 15 min. The solution was then cooled to about 35°C, and 2 lb of yeast per 1000 gal of mash to be fermented were added. The suspension was agitated for 5 min to mix the yeast and then allowed to set for 4 to 5 hr before being added to the fermenter. Once again, this procedure allowed the target of a 54 hr fermentation to be achieved. The two procedures are essentially equal in cost. Depending on the availability of operator time and the sequence of events going on while the fermenter is reaching the proper temperature, the choice of technique for yeast preparation is best decided by the operations manager. The use of yeast in the alcohol production process contributes \$0.055 to \$0.06 to the cost per gallon of ethanol manufactured.

Throughout the runs to optimize the plant performance, samples were taken at the plug flow reactors (before and after entering the cooking cycle), and from the fermenters before yeasting and every 16 to 20 hr thereafter. Depending on the analysis performed, we were able to determine the water-to-grain ratio, the extent of starch liquefaction (dextrose equivalent), the theoretical yield, and the actual plant yield.

Barley. Following the use of corn as the principal feedstock, trial runs at the SSFAP were made using barley. Before testing the new feedstock in the plant, processing parameters were determined by laboratory experiments. The results showed that a finely ground sample of barley required 0.15% w/w alpha-amylase for optimum starch liquefaction and 3000 units of glucoamylase/bu for the saccharification process. Yield in the laboratory was 2.45 gal/bu of alcohol. The actual plant runs yielded a satisfactory result, 2.23 gal/bu.

Corn Dust. One of the more unique feedstocks evaluated at the alcohol plant was corn dust. This material is generated in the conveying of corn from grain elevator to truck or barge and poses a

waste disposal problem to those facilities located in grain producing and coastal regions. The most interesting features of the potential feedstock are that it has almost the same starch content as corn, can usually be purchased at a much lower price, and does not require the usual milling process. The corn dust was processed in the same manner as corn with respect to yeast and enzyme quantities, but because of the presence of some chaff (corn cob flakes), a lower yield was obtained than with corn, 2.45 gal per 56 lb of dust. Analysis of the distiller's dried grains (DDG) showed a protein and fiber content almost identical to that reported for DDG when corn is used as the feedstock.

Potato Waste. The final feedstock evaluated at the SSFAP came from the potato processing industry and consisted primarily of dried raw potatoes in flake and powder form. Laboratory fermentations showed that 4.8 gal of alcohol could be produced from 100 lb of this material. The amounts of enzyme and yeast were maintained at the concentrations used in the grain fermentations. Saccharification and fermentation proceeded in the usual manner, and 4.4 gal of alcohol were obtained per 100 lb of potato waste.

Evaluation of Potato Waste Residuals. As with most industrial or chemical processes, there is always an effluent stream that requires some form of waste treatment; alcohol production is no different. Thin stillage, the liquid stream obtained from filtering the undegraded solids (DDG's), is the biggest waste disposal problem to the ethanol plant owner/operator. When using potato waste as the feedstock, the material remaining after fermentation is usually in the form of dissolved solids. This product has high biological oxygen demand (BOD) and chemical oxygen demand (COD) levels and thus poses a problem to the sewage treatment facility. EG&G conducted an ultrafiltration application test and found that for every 1000 gal of potato stillage, 50 gal of concentrate could be obtained. It's our opinion that the protein-containing material was retained by the device, thus lowering the levels of BOD and COD to a processable concentration. Animal feeding studies are warranted to test the palatability of this byproduct stream.

Quality Control Program

Introduction. Almost all industries have a quality control or assurance program to some degree.

The quality program is oriented toward meeting requirements or standards that either have been established by the market for the product or are the goals of the management. The alcohol production industry is no exception to this philosophy. The marketplace has established requirements for the alcohol as well as the byproduct. Management goals are to achieve the greatest yield per unit of feedstock in the shortest time frame for the lowest cost.

A small quality laboratory is the heart of the quality program in a fuel-grade alcohol plant, just as it is in beverage alcohol facilities; such a laboratory is essential in even the smallest plant. The individual analyses are usually wet chemical tests, e.g., pH, titrations, hydrometer readings, and glucose determinations. The technician performing these assays requires only minimum training. The cost of outfitting such a laboratory is very reasonable, and the payback is guaranteed in a year or less.

Many small plants that have not adopted a step-by-step approach to quality have suffered from poor yields for prolonged periods. Some plants have been forced to shut down because they never achieved the productivity necessary.

An example of the loss that can and does occur can be demonstrated by reviewing the output of a plant that has a capacity of 1 million gal at a yield of 2.6 gal/bu but only achieves a routine yield of 1.9 to 2.0. This results in a production of only 750,000 gal. The loss of revenue from 250,000 gal of alcohol, without considering the feedstock costs, represents \$425,000. If one adds to this the cost of the feedstock for the 250,000 gal that was not produced, on a corn base at 2.6 gal/bu, the cost would be \$264,000 with corn priced at \$2.75/bu. The total loss is approximately \$690,000. Some of this loss would be offset by an increase in byproduct. Nevertheless, a loss of this size cannot be tolerated, not even during part of the year. A quality control lab with the minimum apparatus needed to handle a million-gallon plant would cost about \$30,000 to \$35,000, which is less than 10% of the loss just described, with a payback in a month or two.

Since the SSFAP was designed to be a functional prototype of plants used by small-scale producers, a decision was made to examine each phase of the process to determine the critical areas relating to yield and to loss of alcohol. Procedures

for the assays mentioned in the following quality program can be obtained directly from EG&G Idaho, Inc.; they will also be published in a report now in preparation.¹

Feedstock. The condition of the feedstock is very important. Yields are quoted on feedstocks of good quality. If distressed, immature, spoiled, or treated feedstocks are obtained, starch or sugar concentration should be determined and a laboratory fermentation and yield study should be performed. Although the price of such a feedstock may be favorable, the yield may be drastically reduced. A little experimentation in the lab might produce a better yield and permit the use of specific feedstocks. For example, washing might remove fermentation-inhibiting chemicals from chemically treated feedstock. For feedstock that is spoiling, heat treating could stop further spoilage and also prevent bacterial contamination during fermentation. These are some examples of how to improve a distressed feedstock. However, if starch or sugar are not present in sufficient quantity, alcohol production will never achieve satisfactory yields. In addition to the specific chemical assays that determine the sugar and starch content, which are described later, visual and odor checks are a good indication of feedstock quality. Any spoilage indicates that the sugar or starch content is decreasing because the microorganisms causing the degradation are living off the carbohydrate substances contained in the feedstock.

Proper storage of feedstock on the premises is also critical. The moisture and temperature content of the feedstock must be kept in the proper ranges to prevent the start of spoilage.

Milling. The actual processing starts at this point. Most feedstocks require some size reduction. Tuberous ones should be thoroughly washed to remove the soil. Known particle size distributions should be achieved and yields at these known distributions compared. Different distributions are obtained by varying the grind size. Different feedstocks require a different particle size for optimum yield. With corn, the SSFAP achieved optimum yields by passing it through a 20 mesh screen on the hammer mill. This gave a profile of 10% retained on a No. 12 screen, 35% on No. 20, 40% on No. 60, and the rest passed through the No. 60 screen. Barley, however, had to be milled finer than corn to achieve optimum yields.

Cooking and Conversion. This is the most important phase of the process. Starch is a large

molecule packed very tightly in the endosperm of cereal grains. Before the yeast can metabolize it to alcohol, the molecule must be released from the endosperm and completely broken down to its simplest subunits, glucose and maltose. This is accomplished by first solubilizing it in a heated aqueous slurry and then, in two steps, enzymatically breaking up the molecule.

Each plant should follow the conversion phase to achieve the optimum parameters for the facility. Since there are several cooking schemes now in use—pressure cooking, atmospheric, and low temperature—and also several different amylase enzymes available, the optimum conditions for each plant must be verified on an individual basis.

The alpha-amylase enzyme breaks (hydrolyzes) the large starch molecules into many smaller molecules called dextrins. This prevents the grain slurry from gelatinizing when cooled and allows the second enzyme to completely convert the starch. The pH and temperature are critical parameters when enzymes are used. The grain-to-enzyme ratio is also important. There are several ways of checking for proper liquefaction, e.g., viscosity changes, iodine color test, and dextrose equivalent test. The dextrose equivalent assay was used for the SSFAP. The test requires only titrations and weight measurements. The time per test is about 20 to 30 min, and the dextrose equivalent (D.E.) range indicating good liquefaction is 10 to 15 D.E. units. This type of assay should be run at startup and any time the feedstock or other parameters are changed. The cooking step should be monitored from start to finish when this assay is being used. The equipment required consists only of general laboratory apparatus—glassware, balance, and volumetric glassware.

The second enzymatic conversion further hydrolyzes the dextrins to units of glucose and maltose, both of which are metabolizable by yeast cells. The enzyme that accomplishes this complete breakdown is called glucoamylase, and the conditions for optimum reaction rates are different than the conditions for the alpha-amylase. Many facilities simultaneously saccharify and ferment, and therefore no test is required to follow this reaction. However, after fermentation has stopped, the remaining starch, dextrins, and glucose should be checked. This can be accomplished with the same test as was used for liquefaction. The test is a reducing sugar assay developed by Schoorl.² There is an instrument on

the market that will analyze only glucose, and which could be used for determining the total sugar or starch present before fermentation is started and after fermentation has stopped. The starch and dextrins are first hydrolyzed with acid, and then the glucose is determined with the glucose analyzer. This test is rapid, requiring only a minute or two. The instrument costs approximately \$4000.

The information obtained by determining the total glucose present prior to fermentation permits calculation of a theoretical yield. That data plus the determination of the alcohol content in the beer permits one to calculate the actual yield and plant efficiency percentages. These parameters provide information on how well the plant is performing. The yields should be 90% or better.

The amount of alcohol in the beer can be determined by using a hydrometer on filtered beer. Other alcohol assay methods include instrumental techniques or a refractometer assay. Both of these methods are more precise but require more time and are more costly than the hydrometer.

A fermentation or process sheet should be developed for your facility on which to record batch-by-batch information. This provides information on how well the plant is producing alcohol. A sample is shown in Figure 4. Usually, any decline in production is a gradual one caused by insufficient cooking, poor enzyme conversion, fermentation contamination, or distillation losses. A record provides a way of accurately assessing the conditions in the plant.

Distillation. There have been very few quality limitations placed on fuel grade alcohol except for the complete removal of water. Therefore, the testing required before marketing the product is minimal. A good hydrometer is adequate to determine the alcohol concentration after rectification and azeotropic distillation or molecular sieve dehydration.

The other area of concern during distillation is the base loss from the stripping column. This should be checked periodically, after startup or anytime a column has been readjusted.

The level of alcohol lost in the stillage at the base of the column is too small to detect with a hydrometer. The measurement must be made either directly, with an instrument or in the

laboratory with a refractometer, using a concentrated sample.

Byproducts. The whole grain mash yields a very valuable byproduct. This high protein commodity is used mainly as a dairy animal feed supplement. If it is dried to 10% water or less, its shelf life is dramatically increased. Otherwise, the material quickly spoils.

One concern about the quality of this product arises if the feedstock had been treated with chemicals or has been partially spoiled prior to processing. The chemicals or the toxin produced by specific fungi, called aflatoxin, can be carried through the entire process and out in the stillage, ending up in the feed. Feed thus contaminated can cause problems if fed to animals in sufficient quantities. Aflatoxin can be detected with a simple assay kit that is on the market.

Sanitation and Sterilization. This parameter is very important in obtaining optimum alcohol production. Although contamination may originate anywhere in the plant, it usually shows up and causes problems during fermentation. The best way to prevent severe contamination is to develop rigid procedures for the cleaning and sterilizing of all equipment upstream from the beer well.

In the SSFAP testing, all the pipes and tanks were thoroughly rinsed as soon as they were out of the process stream. Then the equipment was steam sterilized for approximately 30 min before it was reused. If contamination occurs, steaming for longer periods and more frequently will usually help. Another technique is to perform a hot caustic flush before sterilization.

Contamination can be detected during fermentation in several ways, depending on the contaminating organism. A decrease in fermentation activity, a lengthening of fermentation time, unusual tastes and odors, acid rise, and yield reduction can all be indicators that contamination is present.

Several simple tests exist to verify that contamination is actually present. Several of the common contaminating organisms of fermentation produce organic acids as the byproducts of metabolism, e.g., lactic, acetic, citric acids, etc. The rise in the amount of acid during fermentation was measured at the SSFAP as an indicator of contamination. This test requires only a titration of a filtered fermentation sample with a standard solution of base.

DAILY PLANT RECORDS							
Date	Grind Size	Cook Time	Alpha-Amylase	Fermenter No.	Grain Wt/Ferm.	Volume	Yeast Added

Date	Fermenter No.	Set Time	pH	Temperature	Glucoamylase Added	Drop Time	Total Acid	% Alcohol

Date	Fermenter No.	Actual Yield	Plant Yield	Price/Gal	Tons of Distillers Grains	Price/Ton

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Figure 4. Sample process sheet.

CONCLUSIONS AND RECOMMENDATIONS FOR SYSTEMS, EQUIPMENT, AND OPERATIONS

At completion of the testing program, the SSFAP was operating very near the general guidelines used in the design of the plant. For example, it produced a maximum of 28 to 30 gph of 190 proof ethanol. The plant was shown to be capable of greater than 2.6 gal of ethanol per bushel of corn. It was also shown to be capable, with minor modifications, of processing a wide range of feedstocks. The plug flow cookers were shown to be a very viable cooker concept for alcohol plants. Operational manpower requirements appeared to be about 5.5 hr/day, but the 4 hr/day design requirement appeared to be achievable with more plant operating experience. The energy requirements for the operation were within the design projections. The plant was constructed of off-the-shelf equipment using existing technology. One notable achievement was the microprocessor control, with which the plant was brought under automation after only four checkout runs. However, three major components

in the plant system would be improved by modification or replacement. They are the vibrator feeder, the feed pump, and the beer metering pump. A discussion of the problems with these components and recommended solutions are found in Appendix B, System Improvements.

Finally, with regard to equipment performance, a general conclusion of the SSFAP operations was that the plant performance would be notably more successful during periods of harsh weather conditions if part of the plant were housed in protective buildings. This plant was designed to operate without building protection under the seasonal conditions prevailing in Idaho. The plant was operated successfully under many periods of severe weather, but operations were considerably complicated during these periods. Housing of the front-end feed preparation, fermentation, and cooking areas of the facility is recommended.

FOLLOW-ON VALIDATION AND NEW DEVELOPMENT NEEDS

In addition to the equipment changes that would improve the performance of the SSFAP, other follow-on validation and development needs have been identified and are presented in the following sections.

Validation Needs

The fact that the fuel alcohol plant routinely achieved an alcohol yield in excess of 2.6 gal/bu of corn plus a conversion efficiency of 90% or better with barley, potato waste, and corn dust makes this a unique facility. Although these yields were respectable milestones, other tasks need to be performed to facilitate fuel alcohol development. The plant performance should be optimized for such feedstocks as wheat, rice, milo, barley, potatoes, and cheese whey. Most of these materials may require a different grind (particle size distribution) in order to achieve optimum starch conversion to alcohol. In some cases, distillation requirements and conditions will vary from the usual case when corn is used as the feedstock.

There are a number of different enzyme preparations available commercially for accomplishing the liquefaction and saccharification steps, each with differing temperature optimums. Data published has concentrated on the use of alpha-amylase and glucoamylase in the corn wet milling industry, not on the dry milling technique used most often in small-scale fuel alcohol facilities. A study should be conducted to determine which preparations are the most cost effective, the amounts of each enzyme to be used, and their respective pH and temperature options.

In those plants where a separate saccharification process is performed in which glucoamylase is allowed to hydrolyze the maltodextrins to glucose prior to fermentation, a series of experiments should be conducted to determine the cost effectiveness of high yeast loading during fermentation. It has been demonstrated that, under the foregoing conditions, the rate of fermentation is increased and alcohol formation may be accomplished within 12 hr. Recognizing that the fermenter capacity will be turned over faster, it stands to reason that higher yeast populations should be added to the fermenter so that alcohol productivity will be improved.

New Development Needs

Fuel alcohol production on both the large scale and the small scale will benefit considerably by any improvement that will yield a greater alcohol productivity per dollar invested. These improvements can be either mechanical, chemical, or biological. The most significant advances will probably be made in improved enzyme systems that aid the starch liquefaction and saccharification process, and through the use of improved microbes for increasing the rate of alcohol formation.

Before the 1960's, starch liquefaction and saccharification were accomplished with mineral acids at dilute concentrations. This process necessitated extensive cleanup of the hydrolyzate, employing such separation technology as charcoal treatment and ion exchange resins. Although the acid hydrolysis process was inexpensive to operate, its ability to produce a high yield of glucose (greater than 96%) was disappointing. Due to degradations of the carbohydrates in the hot, acid environment, yields rarely exceeded 88% conversion of starch to glucose.

With the advent of commercial production of alpha-amylase and glucoamylase, the corn wet milling industry, brewing companies, and other grain processors began to alter their processes to accommodate the more efficient enzyme hydrolysis systems. Conversion efficiencies of starch to glucose now approach 97%, with essentially no side products formed. There are disadvantages to using enzymes. Hydrolysis times approach 1-1/2 to 2 days with enzymes, whereas acids can accomplish the conversion of starch to glucose in a matter of hours. Furthermore, because enzymes are biological materials, temperature and pH must be controlled or their action will be impeded.

Present-day technology for converting maltodextrins to glucose involves the use of the enzyme, glucoamylase. Optimum conditions for the hydrolytic step are a temperature of 60°C, a pH of 4.5 to 4.8, and a concentration of approximately 0.15% w/w of enzyme to substrate. Two methods of using the enzyme are employed in fuel alcohol production. The first employs a separate

saccharification step in which glucoamylase is added to the liquefied starch and held at 60°C for 12 to 24 hr, producing glucose for fermentation. In the second method, enzyme and yeast are added concurrently, allowing simultaneous saccharification and fermentation (SSF) at 30 to 32°C. Although the second step appears to allow much easier plant operation, the first step will enable greater turnaround of the fermenters when identical quantities of yeast and enzyme are used. This is primarily because the sugar (glucose) is present at the initiation of fermentation and is readily metabolized by the yeast. On the other hand, the SSF requires that the enzymatic reactions produce glucose continuously so that the conversion to alcohol takes place. At 30°C, the rate of glucose production is not as rapid as it is during conventional saccharification at 60°C. If the field of genetic engineering could develop organisms expressing a glucoamylase system with optimum activity at 30 to 35°C, the rate of alcohol production using the SSF methodology would be equivalent to the rate obtained by the sequential process of alcohol production. Furthermore, because of a higher turnover of the fermentation capacity, plant productivity would be increased.

Starch hydrolysis currently requires a two-step enzymatic process at different pH's and temperatures to yield fermentable sugars. Liquefaction, the breakdown of the sugar polymer to soluble compounds called maltodextrins, requires an enzyme called alpha-amylase. This enzyme requires a temperature of 90 to 95°C and a pH of 5.2 to 6.6 for optimum action. The second process, referred to as saccharification, requires the enzyme glucoamylase, which has temperature and pH optimums for glucose production of 60°C and 4.5 to 4.8, respectively. The liquefaction and saccharification steps could be consolidated into a single process if an organism, such as an *Aspergillus* species, could be engineered to pro-

duce both alpha-amylase and glucoamylase in an extracellular, thermostable form. Fermentation times could be decreased, plant capital costs would be lower since the saccharification vessels could be omitted, and operating costs would also be reduced.

The use of glucoamylase in fuel alcohol production adds approximately 3.0 to 3.5¢ to the cost of each gallon produced. This expense could be reduced through use of a yeast that has glucoamylase producing ability. Several species, particularly *Saccharomyces diastaticus*, have already demonstrated this characteristic. However, the amounts of enzyme expressed require a long fermentation period to reach the final concentration of alcohol expected. With the recent advances in genetic engineering and developments in methods for amplifying certain characteristics within an organism, it is not unrealistic to speculate that an organism may be developed that would express high levels of glucoamylase so that the enzyme would not be required exogenously for the conventional saccharification process.

Developments in yeast genetics will also benefit the overall alcohol production process. Improvements in alcohol tolerance, temperature stability, and increased rate of fermentation are all possible goals for trait enhancement. The yeast, *Saccharomyces sake*, has already shown that beer concentrations of 16 to 20% v/v alcohol can be realized. The bacterium, *Zymomonas mobilis*, has shown that alcohol can be produced at four times the rate obtained with conventional yeast, while a bacterium of the genus *Clostridium* has shown that alcohol fermentation can be accomplished at temperatures as high as 60°C. Each of these organisms is outstanding in what it achieves during the fermentation process, but substantial improvements could be made in fuel alcohol production if two of the traits could be combined.

REFERENCES

1. J. H. Wolfram, *Quality Procedures in Alcohol Production*, EGG-2180, in preparation.
2. *Official Methods of Analysis*, 9th edition, Association of Official Agricultural Chemists, 1960, p. 427.

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APPENDIX A OPERATING PROCEDURES

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APPENDIX A OPERATING PROCEDURES

This appendix briefly describes the operating procedures used with the SSFAP. A section on the safety aspects of small plant operations is also included. The step-by-step description of the many operational tasks has been simplified by using tables to show daily chemical and material needs and the manually performed valve alignments that are necessary for operating the plant. Computer controlled operations are not included in these procedures. Only the program entry into the computer, the entry of set points, computerized startup of the plant, and the plant alarm systems are described. Since many of the tasks required in plant startup are not repeated in the continuous operation of the plant, efforts were made to separately identify daily tasks and those required only in plant startup. Safety practices are discussed in the areas that apply to small-scale fuel alcohol plants in general and, where appropriate, to the SSFAP in particular.

Prestart Procedures

Cooking, Saccharification, and Fermentation

1. Fill the 300 bu storage bin (day bin) with 240 bu of corn.

2. Fill all chemical tanks as described in Table A-1.
3. Transfer the fermented beer from the fermentation tank to the beer tank by setting the manual valves involved as described in Table A-2, closing the knife switch to Pump P-SF-13 located on the fermentation pad, closing Switch P-SF-13 on the manual switch control panel of the computer, and pushing the start button on the switch located next to Pump P-SF-13. Monitor the beer level in the fermentation tank so that the pump can be shut off as soon as the tank is empty. Close Manual Valve V-SF-37 immediately after the pump is shut off so that the beer will not siphon back into the fermentation tank.
4. Wash the empty tank thoroughly with water to make sure that all solids left behind when the beer was transferred are removed. Sterilize the tank by setting the manual valves as described in Table A-2, allowing the tank temperature to rise to $\sim 200^{\circ}\text{F}$, and keeping it at that temperature for ~ 10 min. After 10 min, close Manual Valve V-SG-26 and allow the tank to cool.

Table A-1. Feedstock and chemical amounts used daily

<u>Feedstock and Chemicals</u>	<u>Volume (bu)</u>	<u>Weight (lb)</u>	<u>Water Added (gal)</u>	<u>Concentration or Type Used</u>
Corn	240			No. 2 yellow <15% H ₂ O
Alpha-amylase		22.5	23	Termamyl
Glucoamylase		12	14	AMG-15-0
Yeast		28	30	Distillers dry yeast
Sulfuric acid		18	13	Concentrated
NaOH		10	15	50% solution

Table A-2. Manual valve lineup

Operation	Tank 1		Tank 2		Tank 3	
	Open	Closed	Open	Closed	Open	Closed
Transfer fermented beer to beer tank	V-SF-2*	V-SF-81	V-SF-4*	V-SF-2*	V-SF-6*	V-SF-4*
	V-SF-12*	V-SF-85	V-SF-12*	V-SF-82	V-SF-12*	V-SF-83
	V-SF-37*	V-SF-4*	V-SF-37*	V-SF-85	V-SF-37*	V-SF-85
		V-SF-6*		V-SF-6*		V-SF-2*
		V-SF-44		V-SF-44		V-SF-44
		V-SF-65		V-SF-65		V-SF-65
		V-SF-47		V-SF-47		V-SF-47
		V-SF-58		V-SF-58		V-SF-58
Sterilize tank	V-SF-26	V-SF-3	V-SF-26	V-SF-1	V-SF-26	V-SF-1
	V-SF-1	V-SF-5	V-SF-3	V-SF-5	V-SF-5	V-SF-3
	V-SF-81		V-SF-82		V-SF-83	
Day bin	VFP-1 to vibrator feeder					
Bucket elevator	VFP-2 to vibrating screen					
Mash feed to: (start at mix tank)	V-SF-48	V-SF-61	V-SF-48	V-SF-61	V-SF-48	V-SF-61
		V-SF-62		V-SF-62		V-SF-62
	V-SF-43	V-SF-13	V-SF-43	V-SF-13	V-SF-43	V-SF-13
	V-SF-66	V-SF-21	V-SF-66	V-SF-21	V-SF-66	V-SF-21
	V-SF-72	V-SF-73	V-SF-72	V-SF-73	V-SF-72	V-SF-73
	V-SF-68	V-SF-18	V-SF-68	V-SF-18	V-SF-68	V-SF-18
	V-SF-74	V-SF-64	V-SF-74	V-SF-64	V-SF-74	V-SF-64
	V-SF-75	V-SF-71	V-SF-75	V-SF-71	V-SF-75	V-SF-71
	V-SF-76	V-SF-36	V-SF-76	V-SF-36	V-SF-76	V-SF-36
	V-SF-70	V-SF-37	V-SF-70	V-SF-37	V-SF-70	V-SF-37
	V-SF-1*	V-SF-25	V-SF-3*	V-SF-25	V-SF-5*	V-SF-25
		V-SF-60		V-SF-60		V-SF-60
		V-SF-15		V-SF-15		V-SF-15
		V-S-27		V-S-27		V-S-27
		V-SF-3		V-SF-1*		V-SF-1*
		V-SF-5		V-SF-5*		V-SF-3*
		V-SF-9		V-SF-9		V-SF-9
Mash circulation through saccharification cooler, no cooling water	V-SF-12	V-SF-37	V-SF-12	V-SF-37	V-SF-12	V-SF-37
	V-SF-47	V-SF-4	V-SF-47	V-SF-2	V-SF-47	V-SF-2
	V-SF-2	V-SF-6	V-SF-4	V-SF-6	V-SF-6	V-SF-4
	V-SF-40	V-SF-85	V-SF-39	V-SF-85	V-SF-38	V-SF-85
		V-SF-44		V-SF-44		V-SF-44
		V-SF-81		V-SF-82		V-SF-83
		V-SF-88		V-SF-89		V-SF-90
		V-SF-45		V-SF-45		V-SF-45
Cooling water saccharification cooler agitation to and (same valves as above plus valves in water line)	V-W-22*	V-W-27	Same as Tank 1		Same as Tank 1	
		V-W-34				
		V-W-33				

* Valve is changed daily.

Be sure that the tank hatch cover is open so that a vacuum will not be formed that could collapse the tank.

5. Position all the valves in the mash line from the mix tank to the cleaned and sterilized fermentation tank as shown in Table A-2. Many of these valves will not have to be changed once the plant is in continuous operation. The asterisk beside the valve number in Table A-2 identifies those valves that are changed daily.
6. Line up manual valves for mash recirculation as described in Table A-2. Mash circulation starts 90 min after the mash starts flowing into the fermentation tank.

Startup Procedure

Computer Startup. The computer operates continuously when the plant is running. The following procedures apply only to plant startup or in the case of power failure.

The computer controls the manual control panel and two cathode-ray tube (CRT) displays. The manual control panel contains the manual control switches for controlled components in the plant operation sequence and enables manual override of the computer control when necessary. One CRT display shows the following information for each component controlled by or furnishing information to the computer:

- Pump P&ID numbers, 0 for off and 1 for on
- Variable speed pump P&ID numbers, percent of maximum speed
- Solenoid valve P&ID numbers, 0 for closed and 1 for open
- Motor valve P&ID numbers, percentage of maximum opening
- Thermocouple and RTD P&ID numbers, temperature readings in °F
- Pressure gauge P&ID numbers and pressures
- Level control P&ID numbers, 0 when not activated and 4.9 V when activated.

The second CRT display shows the following information:

- Time
- Date
- The sequence to Vats 1, 2, or 3
- Feedstock control parameters
- Distillation control parameters.

Perform the following steps to start up the computer:

1. Close the power switches to both CRT displays.
2. Close the power switch to computer.
3. Insert the computer tape, 64K Basic Revision 3.47.
4. Close the following switches on the computer in the order listed:
 - Halt (HLT)
 - Reset (OVR)
 - Load.
5. The CRT display on the right will then display the following:

Multi-Task Basic Ru 3.47
Date (m/d/y)? (cursor is located here).
6. Press the return key on the keyboard of the CRT display.
7. Remove the 64K Basic Revision 3.47 tape from the computer.
8. Insert the computer tape, MTO: Master Program Tape 1, 12-Hour Run.
9. Type

ENTER MTO:0

on the right hand CRT display keyboard.
NOTE: The 0 is a zero, not the letter O.

10. Press the return key on the CRT display keyboard. Watch for the cursor to travel one line feed down. The display will show the following:

ENTER MTO:0

the cursor will be located here.

NOTE: This takes several minutes.

11. Enter date and time by typing the following on the right hand CRT display keyboard:

SDATE—enter 2 digits each for month, day, and year here, and press the return key

STIME—enter 2 digits each for hours, minutes, seconds, and press the return key.

12. Type

RUN

on the keyboard and press the return key.

13. At this time, both CRT displays should display and update plant components and plant parameter status.
14. To start the printer, close the power switch located on the back of the printer. To initiate a printout of the information shown on the two CRT displays, press the Printer Engage switch located on the manual control panel upward. To have a printout of the two CRT display information at 30-min intervals, press the Automatic Printer Engage switch located on the manual control panel upward.

Cooking, Saccharification, and Fermentation

1. At this time, the computer is assumed to be on. (Computer startup was described above.)
2. Open Control Valve CV-SF-10 by pushing the switch on the manual control panel upward. This valve will remain open until the plug flow reactors are filled with mash. When the reactors are full, the mash will flow through the overflow pipe. At this time, close Valve CV-SF-10.

Opening Control Valve CV-SF-10 is necessary only during the initial plant startup. In a continuous operation, this valve will remain closed.

3. Table A-3 shows the switches to be set in the automatic mode and the set point temperatures. These switches should be set and the set point temperatures entered in the computer before computer control of the plant is initiated.
4. Table A-4 shows the relationship between the switch that initiates the cooking, saccharification, and fermentation cycles and the tank into which the mash will be pumped. Select the Initiate Fermentation Sequence switch for the fermentation tank to be used. For example, if Tank 1 is to be used, push the switch Initiate Fermentation Sequence No. 1 up.
5. As soon as the vibrator feeder starts, check the calibration of the corn feed rate. Adjustments to the feed rate can be made at the switch mounted next to the vibrator feeder.
6. When the corn-water mixture in the mix tank becomes consistent, check the flow rate for the feed pump. This can be measured by opening Valve V-SF-62 and closing Valve V-SF-43 at the same time. As soon as the measurement is made, return the valves to their original positions.
7. When the cooking cycle is complete, adjust the pH of the mash between 4.8 and 5.0 by operating the acid pump from the manual control panel to add H_2SO_4 from the acid tank. If too much acid is used, adjust the end point by operating the NaOH pump from the manual control panel to add NaOH from the NaOH tank. To determine pH, take samples at Valve V-SF-44 and analyze with a pH meter.
8. As soon as the pH is adjusted to 4.8 to 5.0, add the glucoamylase by starting the glucoamylase pump at the control panel. Monitor the glucoamylase level in the tank, and shut off the pump when the tank is empty.
9. Start cooling the mash by setting the valves as shown in Table A-2.

Table A-3. Set points for cooking, saccharification, and fermentation control

<u>Operation</u>	<u>Automatic/Manual</u>	<u>Set Point (°F)</u>
Mash heater	Automatic	200
Feed/mix tank level control	Automatic	
Feed/mix tank steam sparger	Automatic	140
Alpha-amylase flow rate control	Automatic	
Mash cooler	Automatic	140
Saccharification cooler	Automatic	85
Automatic printout	Enable	
Manual printout	Enable (when needed)	

NOTE: All the above switches are pushed up for AUTOMATIC CONTROL and ENABLE and down for MANUAL CONTROL and OFF. Set point switches are pushed up to raise temperature and down to lower temperature.

Table A-4. Determination of mash flow

<u>Operation</u>	<u>Mash is Pumped to</u>
Initiate Fermentation Sequence 1	Fermentation Tank 1
Initiate Fermentation Sequence 2	Fermentation Tank 2
Initiate Fermentation Sequence 3	Fermentation Tank 3

10. When the mash temperature reaches 85°F, shut off the fermentation cooler and the cooling water valve, V-W-22. Add the yeast by starting the yeast pump at the manual control panel. Monitor the yeast level in the yeast tank, and shut off the yeast pump when the tank is empty.

11. If the beer temperature exceeds 92°F during fermentation, cool the beer to ~85°F by starting the fermentation cooler and opening Valve V-W-22. When the desired

temperature is reached, shut off the fermentation cooler and close Valve C-W-22.

Distillation Startup. (When plant is in continuous operation, the distillation process is a continuous operation.)

1. Start the beer agitation pump, P-SF-5, by closing the knife switch located on the saccharification-fermentation pad. Next, close the switch for P-SF-5 located on the manual control panel.

2. Close the knife switches to the following pumps:
 - Beer metering pump, P-DG-1
 - Column 1 bottoms pump, P-DC-2
 - Column 2 bottoms pump, P-DC-3
 - Reflux pump, P-DC-4
 - Product pump, P-DC-5.
3. Table A-5 shows the manual valve line up for the distillation, product, and byproduct systems. Check all manual valves from the beer metering pump through the feed lines

Table A-5. Manual valve lineup for distillation

<u>Opened</u>	<u>Closed</u>
V-DC-1	V-SF-84
V-DC-26	V-SF-46
V-DC-5	V-SF-59
V-DC-2	V-DC-6
V-DC-50	V-DC-36
V-DC-3	V-DC-19
V-DC-4	V-DC-22
V-DC-18	V-DC-30
V-DC-12	V-DC-51
V-DC-10	V-DC-34
V-DC-13	V-DC-31
V-DC-14	V-DC-39
V-DC-7	V-DC-40
V-DC-25	V-DC-41
V-DC-27	V-DC-42
V-W-17	V-DC-43
V-W-18	V-DC-44
V-S-5	V-DC-45
	V-DC-46
	V-DC-47
	V-DC-28
	V-DC-37
	V-DC-38
	V-DC-35
	V-DC-20
	V-DC-49
	V-W-31
	V-W-28
	V-S-25
	V-S-22

- and distillation columns for proper alignment.
4. Check the control panel for the operational mode of all variable speed motors—on-off, manual-automatic, etc.
5. Turn on Column 1 Automatic Level Control at the control panel and wait for the Column 1 bottoms pump to clear the column.
6. Turn on Column 2 Automatic Level Control at the control panel and wait for the Column 2 bottoms pump to clear the column.
7. Turn on the automatic steam control. The computer automatically sets the control temperature at 200°F. This is the temperature at the top of Column 1. This temperature can be raised or lowered by pushing the switch on the manual control panel for the Column 1 set point up or down.
8. With the Column 1 set point temperature at 200°F, the steam control valve will open. When the Column 2 top temperature reaches 178°F, start the beer metering pump by pushing the switch on the manual control panel up and holding it there until the reading on the main control panel for P-DC-1 reaches 35% of maximum pumping speed. Measure the flow rate of the beer by using a 5-gal graduated container and a stopwatch. The flow can be determined by opening Valve V-DC-30 and closing Valve V-DC-2. The flow should be between 4.5 to 5.0 gpm. Pressure Gauge PI-DC-7 should indicate between 10 to 12 psi, depending on the alcohol content and the viscosity of the beer.
9. Turn on the dewatering screen motor DS-SR-1 at the manual control panel if the distillers grains are to be dewatered, and close the bypass valve to the sewer.
10. After 10 to 20 min of operation, check Accumulator Level LT-DC-5D on the control panel. If the reading is 0, the ethanol level in the accumulator is below

Level LT-DC-5D. If the reading is ~4.9 volts, the ethanol level is above Level LT-DC-5D. If the ethanol level is above Level LT-DC-5D, turn on Reflux Pump P-DC-4 at the manual control panel. Push the switch upward and hold it until the percentage of maximum speed reaches about 40%. After about 30 min, check the proof of the ethanol. If possible, reduce the reflux pump speed but maintain the proof of the ethanol at 190.

11. The alcohol is denatured by adding 1 gal of lead-free regular gasoline to 99 gal of ethanol, producing a 1.0% solution of gasoline in ethanol. Add the gasoline by using Hand Pump TP-DC-1, which is mounted on a 55-gal storage tank, to pump the gasoline into the produce tank. Calibrate the hand pump by determining the number of pump strokes required to pump 1 gal of gasoline.
12. To remove the ethanol from the product tank, open Manual Valve V-DC-23 and turn on Product Pump P-DC-6 at the manual control panel. Also turn on a manual switch located close to the product pump.
13. The dewatered distillers grains can be collected on the storage pad designed for this plant. It was designed to hold distillers grains from 48 hr of plant operation. This requires removal of the distillers grains from the pad at least every 48 hr. An end loading tractor can be used to load them onto a truck.

Plant Alarm System

The alarm system for the SSFAP is designed to warn the operator of unacceptable variations in certain plant control systems. If the operator does not respond quickly enough to correct the problem causing these variations, the computer will shut the system down. The alarm system alerts the operator in three ways:

- It sets off an audible alarm in the control trailer.
- It displays the cause of the alarm on one of the CRT displays.

- It instructs the printer to print out the cause of the alarm.

Table A-6 shows the operating parameters and the degree of variation required to set off the alarm in the control trailer. As shown in the table, the distillation system is the only one that will shut down due to lack of response or inability to correct the cause for the alarm. Distillation system shutdown consists of shutting off all pumps and closing the steam control valve, CV-5-7.

Safety

The purpose of this subsection is to give a brief overview of the major safety problems encountered in the operation of a small-scale fuel alcohol plant. The SSFAP incorporates many safety features that are fully discussed in the design manual. Some of these are explosion-proof motors, separation of cooking and distillation modules, hatches, and pressure relief systems.

In general, an alcohol plant of this type is a small chemical processing plant requiring the use of a few hazardous chemicals. A few hazardous chemicals are also produced. It requires operators to work around motor-driven equipment, hot liquids and steam, hot pipes and tanks, and in many other potentially hazardous situations. Operating personnel should be trained to identify major hazards and to protect themselves from injury.

First consideration should be given to preventive safety. This means that any safety hazard that can be eliminated should be. For example, motor-driven equipment should have drive shaft coupling and belt guards, hot pipes should be insulated, and working areas located above the plant floor should be equipped with platforms with railings and constructed of materials that will assure good footing. Permanently constructed ladders or stairways should provide access to these platforms. These are but a few of the areas within the plant system that should be considered for preventive safety. Many hazardous areas will be specific to the particular alcohol plant.

Next, the operating personnel should have available and be trained in the use of protective equipment such as hard hats, safety shoes, safety glasses or goggles, protective clothing, and other safety equipment necessary for safe operation of the plant.

Table A-6. Plant alarm system

<u>Plant System</u>	<u>Alarm Parameter</u>	<u>Alarm</u>	<u>System Shutdown</u>
Cooking, Saccharification, and Fermentation			
Mash heater	Temperature	< 160°F, > 220°F	
Steam control valve, MH-SF-1	% of valve opening	< 1%, > 99%	
Feed mix tank	Ta ^a —Ts ^b , Ts—Ta	> 25°F, > 15°F	
Distillation			
Reflux temperature	Temperature	> 140°F	> 160°F
Column 2, top temperature	Temperature	< 160°F	> 195°F
Column 1, control temperature	Ta—Ts, Ts—Ta	> 6°F, > 12°F	Ta—Ts > 10°F for 10 min
Steam flow	ACFM ^c	< 20 ACFM	< 20 ACFM for 5 min
Column 1, bottoms pump	Pump motor on	> 10 min	> 15 min
Reflux pump	% of maximum speed	> 99%	> 99% for 5 min

a. Ta—actual temperature.

b. Ts—set point temperature.

c. ACFM—actual cubic feet per minute.

Finally, and in conjunction with the above, operating procedures for the small-scale fuel alcohol plant should be written so that hazards within the plant are identified for each task. Each task procedure should recommend required safety procedures or reference available sources of information for that particular problem. These operating procedures can then be used as a valuable training tool for new operators and an important reference for trained operators.

The only safety problem of consequence that occurred with the SSFAP was the imploding of the beer well top cover. This happened after a steam cleaning operation. A very low pressure vapor mixture still existed in the beer well, and the transfer of cold beer from a fermentation tank created a small vacuum that the beer well vacuum breaker system could not accommodate. A procedural change requiring the hatch cover to be open during such transfers eliminated any potential recurrence of such an incident.

APPENDIX B SYSTEM IMPROVEMENTS

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APPENDIX B

SYSTEM IMPROVEMENTS

After the first production test with the SSFAP was completed, work began on repair, equipment upgrading, and design modifications to correct the deficiencies identified in the SSFAP Phase 1 tests. These changes and repairs are described below under each plant section, i.e., the front-end (grain handling) section, the cooking section, the distillation section, and the byproduct handling section.

Front End (Grain Handling Equipment)

Day Bin (ST-FP-1). All leaks were caulked to reduce moisture in the grain, which had caused bridging and plugging at the bin outlet (V-FP-1).

Vibrator Feeder (VF-FP-1). The grain feed rate was erratic. To remedy this, the angle of the feed tray was increased, and tape bridges were placed in the tray. This improved the control of the vibrator feeder, but it did not solve the problem completely. The changing ambient humidity appeared to be the major factor affecting the feed rate.

The vibrator feeder should feed corn into the plant system at a rate of 20 bu/hr. However, this rate varied considerably during the 12-hr cooking cycle because of changes in humidity. In the current design, the vibrator feeder is located at the base or outlet of the 300-bu storage bin. The corn flows from the bin onto the sloped tray of the vibrator feeder. The feed rate is determined by setting a rheostat control switch that controls the amplitude of the vibrations to the tray. If the unit were located in a building with controlled humidity, it would probably perform adequately. However, the SSFAP is located in the open and is subject to the changing outside humidity. A feeder system to remedy this problem would use an auger or screw driven by a adjustable-speed motor to feed the corn at a constant rate. Also, the 300-bu storage bin could be equipped with a vibrating base to assure a constant flow of corn into the auger feed. Manufacturers of this type of feeder claim metering accuracies at levels that are very acceptable for this application.

Hammer Mill (SRM-FP-1). The screen used during the first production test had

5/64-in.-diameter holes and was rated at 180 lb/hr/hp. The particle size of the milled corn was too large, causing plugging in heat exchangers and restrictions in the piping system. Also, the solids settled out in the fermentation and beer tanks, trapping enough beer to cause unacceptable losses in product yield. By using a smaller screen that had 3/64 in. diameter holes and was rated at 100 lb/hr/hp and by increasing agitation in the beer tank, the plugging and settling problems were solved. However, the hammer mill motor size had to be increased from 10 hp to 15 hp to mill at the 20 bu/hr rate.

Cooking

Mix Tank (ST-SF-7). The problems encountered here were as follows:

- When the mash that was pumped through the mash heater was not preheated, the steam supplied to the mash heater was not adequate to raise the mash temperature to the 200°F required for cooking. To remedy this, steam was piped into the feed mix tank to preheat the mash. The steam to the mix tank was controlled by the computer using a thermocouple in the mix tank and a solenoid valve in the steam line to the mix tank. The mix tank temperature was normally set at 140°F.
- The mix tank originally was constructed of polypropylene. The 140°F mash temperature caused the tank to deform. It was replaced with a stainless steel tank.
- Sprayers were installed on the inlet water line to reduce the amount of corn dust lost through the agitator opening and the viewport in the lid.

Alpha-amylase System (ST-SF-1 and CV-SF-14). The alpha-amylase metering pump was found to be inadequate. When other metering pumps were used and did not perform adequately, the system was modified. The modification included placing the alpha-amylase tank on the top deck above the mix tank. A solenoid valve that was opened and closed by a computer time sequence was used to control the alpha-amylase flow rate.

Feed Metering Pump (P-SF-17). The feed metering pump is a variable-volume, rotary-piston, positive-displacement pump. The original pump was constructed of brass, with a nylon shuttle. Close tolerances in the moving parts in the pump were necessary for good performance. The abrasive action of the corn mash on these parts caused the pump to fail after approximately 24 hr of operation. All of the moving parts, the pump body, and back plate were replaced with case hardened steel parts. This extended the pump life to approximately 360 hr of operation, but this is still not an acceptable performance record. It appears that any positive displacement pump, because of the close tolerances required for the internal moving parts, would not perform adequately in this application. Figure B-1 shows a schematic drawing of a feed pumping system that could be used. The system uses a centrifugal pump plumbed into a bypass line. The bypass line would carry any excess mash flow back to the mix tank where it could be used for additional agitation. The mash flow to the cookers would be controlled by a motor valve and a magnetic flowmeter. The output signal of the flowmeter would be used by the computer to regulate the motor valve opening. The desired flow rate of the mash to the cookers would be entered into the computer as a set point.

Mash Heater Pump (P-SF-17). The original heat pump type steam injector for heating the

mash to the cooking temperature caused a serious plugging problem. The injector had a 1/4-in. opening for the mash to pass through. The mash would plug almost immediately on entry into the injector. A new injector was constructed from a 2-in. pipe Y-screen, using a 1 in. stainless steel pipe and pipe cap for the steam nozzle. Figure B-2 is a schematic drawing of the new injector.

Plug Flow Reactors (YS-SF-4 & 5). The original piping of the plug flow reactors was designed for mash parallel flow through to the top of the reactors. In this configuration, the solids in the mash appeared to be settling in the bottom of the reactors and causing plugging. A pipe manifold was constructed that allowed the mash to travel through the reactors in either a parallel or a series pathway and enter the reactors from either the bottom or the top. The mash flow path that appeared to minimize the settling and plugging problem was a series configuration in which the mash entered through the bottom of the reactors. Steam and water lines were piped into the manifold for flushing and sterilizing the reactors.

Saccharification and Fermentation

In general, there were few problems with this system, none serious. The procedure for adjusting

Figure B-1. Feed pump modification.

the pH and adding the glucoamylase was modified. Instead of adjusting pH and adding glucoamylase in the feed line going into the fermentation tank, this process was completed in the line between the saccharification cooler (HX-SF-4) and the fermentation tank. The pH was adjusted while the tank was filling, and the glucoamylase was added after the tank had filled.

Acid Tank (ST-SF-3). This system was designed with a 15-gal tank. It takes approximately 2 gal of concentrated sulfuric acid to adjust the pH of the mash. Two gal of concentrated sulfuric acid diluted with water to 15 gal would result in an acid normality of approximately 5. If a 50-gal tank were used, the acid normality would be approximately 1.3. This would provide a safer working environment, and the metal components in the acid system would not be affected as much.

Distillation

Beer Agitation Pump (P-SF-5). The original beer agitation pump was a 1 hp centrifugal pump. This pump did not keep the solids in the beer suspended for transport through the still. It was replaced with a 10 hp centrifugal pump, which solved the problem.

Beer Metering Pump (P-DC-1). The motor for the beer metering pump did not have enough starting torque to start the pump. The first consideration was to get a larger motor. However, when the larger beer agitation pump was installed, it was determined that part of the flow from this pump could be used in conjunction with a flowmeter and motor valve to replace the beer metering pump.

Flow Indicator for Beer Metering Pump (FI-DC-1). The flow indicator for the beer meter-

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Figure B-2. Mash heater modification.

ing pump is a turbine flowmeter. Because of the high solids content of the beer and the resulting blockage of the rotor, the flowmeter was inadequate for this application. Currently, the beer flow is determined by the motor speed of the beer metering pump. The beer metering pump was calibrated by determining the beer flow in the line, using a stopwatch and a small graduated plastic tank. However, when the beer metering pump is replaced by using part of the flow from the beer agitation pump as described in the previous paragraph, this flowmeter would have to be replaced with a flowmeter that would not become fouled from the solids in the beer.

Still Bottoms—Beer Heat Exchanger (HX-DC-3). Most heat exchanger problems result from the beer or mash flowing through the shell side of the heat exchanger. In most cases, this can be resolved by having the mash or beer flow

through the tubes. However, in this particular heat exchanger, fluids with high solid content are flowing on both sides of the heat exchanger. To resolve this problem, a heat exchanger with widely spaced tubes was used. A 1 hp centrifugal pump replaced the positive displacement pump used for pumping the still bottoms out of Column 1. The pump circulates the beer through a bypass line and back into the heat exchanger. A solenoid valve controls the flow to the dewatering screen. The flow from the bypass line into the heat exchanger keeps the solids in the still bottoms suspended when the solenoid valve is closed. Figure B-3 is a flow schematic of the system.

Totalizing Flowmeter (EFI-DC-8). The totalizing flowmeter was designed for petroleum products. Liquids such as fuel oil, gasoline, and diesel would lubricate the moving parts in the flowmeter. Ethanol is not a good lubricant, and

Figure B-3. Beer still bottoms heat exchanger modification.

consequently the flowmeter would not work properly. Product tank measurements were used to determine yields and the dilution rates for the plant.

Fusel Oils. After approximately 27 hr of distillation, it was noted that fusel oils in Column 1 were interfering with the distillation process. The only method that effectively removed the fusel oils was to shut off the beer and steam feed and drain Column 1.

Byproducts

Dewatering Screen (DS-SR-1). The only problem encountered with the dewatering screen occurred when the screen was not tightened properly. This caused the screen to break along the screen support frame. When the screen was tightened according to manufacturer's recommendations, that problem was eliminated.

Byproduct Storage Pad. The SSFAP Design Report did not include a drawing for a byproduct

storage pad. Figure B-4 shows the sketch of the pad.

Product Storage. There were no notable problems within this system.

Instrumentation and Controls. The only problem with the instrumentation and controls occurred because of power failure. This required placing the program cassette in the computer and reentering the program. This would interfere with some time sequenced controls in the front-end, cooking, and saccharification systems. These controls would require manual operation until the cooking and saccharification cycles were complete. If the program was interrupted, the vibrator feeder, bucket elevator, cleaning screen, hammer mill, alpha-amylase agitator and solenoid valve, mix tank agitator, and feed pump would have to be manually shut down at the end of the cooking cycle. The beer pump would have to be started and stopped manually at the proper time. Manual operation in all of the above cases refers to use of the program override switches on the control panel.

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Figure B-4. Distillers grains storage pad.

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