

# Chapter 12

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## Production of Canadian rye whisky: the whisky of the prairies

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### Introduction

The establishment of rye grain distillery operations east of the Rocky Mountain foothills in Canada in the late 1940s was an attempt to utilize a potentially abundant and relatively cheap source of starch in the form of rye grain for the production of beverage spirits. The fertile sandy soil existing in many parts of the prairies was perfect for the fall planting of rye varieties such as Cougar, Musketeer, and Kodiak. The September planting and subsequent July/August harvesting of rye meshed well with the farmer's timetable; and with the establishment of distilleries at certain main centers, rye became a welcomed cash crop for many Western Canadian farmers.

The decision by the distilleries to use rye grain as the main starch source at that early date flew in the face of a considerable body of experimental knowledge about rye grain that promised high-viscosity mashing, foaming fermenters and poor quality distillers dried grains. Most of this experimental knowledge emanated from American corn grain distillers using small amounts of rye grain mainly for the production of rye

whisky flavoring used for blending. To these people, the problems of mashing rye grain were not worth solving and were handled by adjusting corn systems to inefficient operation levels.

This chapter presents the essential features of the process and quality control procedures developed over the years to understand, control and exploit the special attributes of rye grain in the manufacture of beverage spirits. This chapter includes flow charts and descriptions of the processes developed for rye grain spirits manufacture in the areas of grain handling, batch mashing and fermentation, dual-column continuous distillation and a multiple-effect- evaporator distillers dried-grain process.

### Rye grain reception and quality control

The supply of grain to a distillery, be it rye or any other grain, will involve several stages of activity. They are 1) submission of a sample for quality evaluation, 2) grain reception and quality checking, 3) initial storage, 4) cleaning, 5) clean grain storage, 6) milling, and 7) meal storage.

### Grain sample submission procedure

The rye grain used by distilleries in Western Canada is mainly of the Cougar or Musketeer variety and is supplied mainly from the 'rye crescent' of Alberta, located 50 to 70 miles (80 to 112 kilometres) south of Calgary, Alberta. In years of poor harvest, additional grain is usually available from the more eastern provinces of Saskatchewan and Southern Manitoba. Bushel weight, sprouts, appearance and dockage are combined with analytical data such as starch (reducing power removed by fermentation), protein (Kjeldahl), moisture (oven-dry) and sieve analysis (Tyler or US Standard); and the results are used by the grain department in their selection of the best grain inventories available.

It is important to note that the grain department's job is to select the best rye available and not just to make purchases according to a rigid preset specification similar to that used for corn purchases. While it is preferable to have minimum quality acceptance levels in mind, the conditions of cultivation and harvest in the region can vary greatly from year to year, causing wide swings in starch and protein content and in the degree of field sprouting.

Following selection of a particular supply of rye, a timetable for receiving it at the plant is established based on the current inventory and anticipated requirements. In a situation where grain is delivered by truck, and where weather can cause delays, an acceptable minimum inventory would be about a two week supply. Thus, in a distillery mashing 8,000 to 8,200 bushels per day (203-208 tonnes) on a seven-day-week basis, the inventory on hand at any time should be about 115,000 bushels (3,000 tonnes).

### Grain reception quality checks

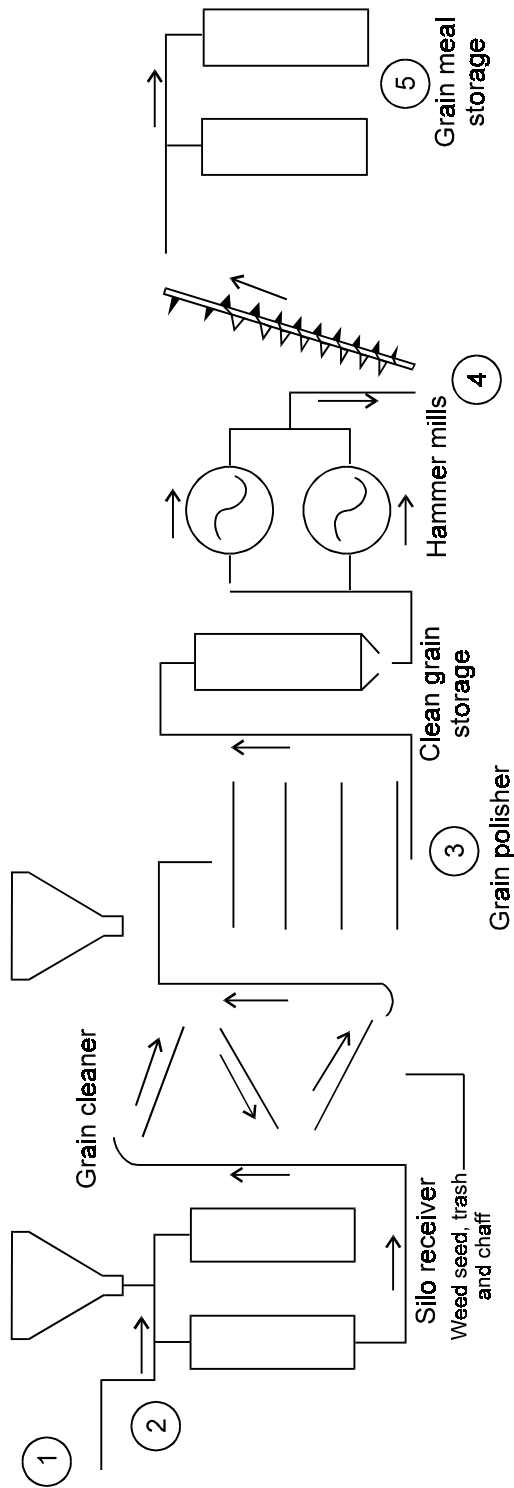
Upon arrival of a rye grain shipment, immediate on-site checks include dockage (trash), moisture (conductivity), ergot (caused by the organism *Claviceps purpurea*), sprouts and bushel weight. These features are graded against data received

from the sample submitted earlier to determine whether the quality expected is present in the delivered grain. As the grain makes its way through the system and approaches the milling stage, samples are taken for sieve analysis, moisture, starch levels, protein and cleanliness. These data affect hammer-mill performance, expected alcohol yield and quality, dried grain production and plant microbiology.

The presence of significant levels of ergot (>0.5%) in rye can cause a secondary problem in distillery operations by making the stillage unfit for animal feed production. The *Claviceps* fungus causes the rye spikelets to develop hard, slightly bent, blue-violet crescents instead of normal grains. The fungus crescents contain alkaloids that are toxic to animals and humans alike, and must not be allowed to pass through the distillery process (Starzycki, 1976). The blue-violet color of the ergot bodies (sclerotia) make it easy to spot contaminated grain and can be used as a guide in the rejection of rye shipments.

A process flow sheet for receiving and quality monitoring of rye grain up to the grain meal storage stage is shown in Figure 1. Five quality stations are recommended with appropriate physical and chemical checks needed for good manufacturing practice.

Previous distillery experience has shown a correlation between the degree of whole grain cleanliness and development of the undesirable congener acrolein and its intermediates in the fermenters, which may result in a multitude of reactions with normal fermentation congeners during the distillation stage. Rye distillers have noted that the frequency of acrolein formation tends to increase toward the end of a production year when silos and grain-handling vessels are cleaned of dust and grain debris. The grain dust was allowed to join the whole grain entering the cleaning cycle and it is likely that extraordinary amounts of the dust by-passed the screening to some degree and entered the production cycle. This condition at times led to higher delta acid titers in the finished fermenters followed by the production of spirits with an exceptionally irritating odor.



**QUALITY STATION**

- ① Preshipment sample
- ② Truck sample dockage
- ③ Polisher sample
- ④ Hammer mill sample
- ⑤ Grain meal storage

**REQUIRED PHYSICAL/CHEMICAL CHECKS**

- Starch, protein, fat, moisture, potential alcohol, dried grain protein
- Trash, chaff, weedseed, moisture, grain grade, ergot
- Size distribution
- Meal sieve analysis
- Starch, protein, fat, moisture, potential alcohol, plant theoretical yield

Figure 1. Receiving and monitoring rye grain (Morrison, 1992).

Acrolein is an acrid, lachrymatory compound formed when yeast glycerol is metabolized to  $\beta$ -hydroxypropionaldehyde by bacteria. It is this compound that is degraded to form the acrolein molecule in the rectification process. Acrolein has been accused of causing the phenomenon of peppery spirits, which may give considerable discomfort to operators. However, acrolein's propensity to react with other congeners in the spirits to form stable complexes places some doubt on whether acrolein *per se* is the real culprit (Kahn *et al.*, 1969).

Physical and chemical laboratory data for good quality western Canadian rye are presented in Table 1.

Depending upon rate of delivery, the incoming grain may be bucket-elevated into uncleaned grain silos for later cleaning or it may go directly to standard trash and weed seed separators and then on to a Crippen-style polisher for final dust and chaff removal. The polished rye is then stored in silos to feed the Praterstyle 100 HP hammer mills with selected screens that convert the grain to meal. The screens have a variety of sizes of geometrically shaped holes and are capable of producing several spectra of meal. Meal samples are monitored for final moisture and sieve analysis and the milling is adjusted accordingly.

The laboratory procedure for meal sieve analysis involves placing a sample on a set of US

**Table 1.** Physical and chemical data for western Canadian rye grain\*.

<b>Physical analysis</b>					
Grain size distribution (sieve analysis)					
Tyler screen No.	6	7	8	12	pan
Mesh opening, mm	3.35	2.80	2.38	1.68	
Grain found, %	0.10	15.70	40.30	41.80	2.1
<b>Chemical analysis (dry basis)</b>					
Amino acid content, g/100 g protein					
Glutamic acid	30.18		Glycine	3.98	
Proline	11.40		Threonine	3.67	
Aspartic acid	7.09		Ammonia	3.19	
Leucine	6.56		Lysine	3.18	
Valine	5.37		Cystine/Cysteine	2.51	
Phenylalanine	4.91		Histidine	2.30	
Serine	4.70		Tyrosine	2.07	
Arginine	4.60		Tryptophan	1.33	
Alanine	4.07		Methionine	1.28	
Isoleucine	4.00				
Kjeldahl protein (N x 6.25), %			10.29		
Starch (reducing power removal by yeast), %			64.05		
Nitrogen-free extract (total carbohydrate), %			80.10		
Lipids by ether extract, %			1.80		
Ash content, %			2.10		
<b>Mineral content, mg/100 g</b>					
Phosphorous	380	Potassium	520		
Iron	9	Manganese	7.5		
Calcium	70	Magnesium	130		
Copper	0.9				

\*Unpublished distillery data; Simmonds *et al.* (1976).

Standard or Tyler screens of varying mesh sizes that vibrate on a shaker for a determined time. Meal remaining on each screen is weighed and percentages calculated. The sample of meal tested should be a composite of samples taken over a period of time sufficient for all run conditions to be represented. On a set of Tyler screens with mesh ratings of 10, 20, 30, 40, 60, 80 and 100, it is recommended for rye that a total of 65% of the meal should rest on screens #30 to #60 and less than 10% should pass through screen #100 as flour. A typical sieve distribution analysis for rye meal is presented in Table 2.

**Table 2.** Typical meal sieve analysis for rye meal.

Tyler screen #	Mesh (mm)	Meal (%)
9	1.68	1
20	0.841	10
30	0.595	28
60	0.247	26
80	0.177	15
100	0.15	12
Through 100	-	8

### **Rye meal sieve analysis**

Meal size distribution (sieve analysis) will affect cooker performance in two ways, each creating opposite effects. The fermentable extract obtained during the mashing process will be maximized when the grain is ground as fine as possible thus creating the maximum surface area for contact with the cooking liquid. However, excessively fine meal will create problems in equipment such as batch cookers when large quantities of the powdered meal are added rapidly to the liquid phase of backset stillage and/or water. In the manner of most dry meal materials, the rye meal can form dough balls when a thin coat of wetted mash forms a very impervious covering around a quantity of dry meal. The tendency to form dough balls is temperature-sensitive and will become significant

at addition temperatures exceeding 55°C. This encapsulated meal will not be wetted and the starch will not be accessible to gelatinization processes and subsequent hydrolysis to lower molecular weight sugars.

Distillery experience has indicated that starch loss may exceed 5% when extensive dough ball formation occurs. Use of excessively fine meals in batch cookers can, however, be tolerated if mechanical distributors are employed in the meal addition system. The distributors rotate with the cooker agitator and add the meal in a spray pattern across the entire liquid surface.

In continuous mashing systems the extent of dough ball formation will depend on the degree of homogeneity of mixing in the slurry tanks and by the transfer pumps. The use of in-line mixers, which move the slurry through a high-shear rotor pump, will minimize the presence of the yield-reducing dough balls.

Detection of dough ball formation in cooker systems becomes somewhat difficult in that the balls may not survive the pumping system and thus may escape visual detection. The application of dilute iodine solution to a small amount of mash may indicate the presence of unhydrolyzed starch by producing an unusually pronounced blue coloration. A positive test result will be useful only if extensive experience of normal expectations is at hand against which the new test can be measured. The unusual-blue iodine coloration test should provide the first reason to suspect dough ball formation, especially if there is also an unexplained alcohol yield loss.

The next step in the investigation would be to alter the meal sieve analysis to a lower flour and fines content by changing hammer mill screens. If there is any control on the meal addition rate this control should be adjusted to slow the addition, and thus reduce the ball formation potential. If the meal addition temperature exceeds 55°C, then a stepwise reduction to 50°C should be tried over a sufficient period of time for proper evaluation. Each step of the change should be monitored using the dilute iodine test and by recording the alcohol yield.

Finally, the daily sieve analysis of the meal can be used for determining maintenance needs on hammers or screens. A sudden change in the meal grind would suggest a need to inspect screens for punctured areas or to inspect the hammer points for sharpness and mechanical integrity. Screen-tear problems may show up as quantities of whole grain on the upper screens. The hammer maintenance problems usually manifest themselves in higher percentages of meal on the upper screens.

## Rye mashing and fermentation

### The hydration and gelatinization of rye starch

The mashing or cooking of rye meal, like that of most other grains, is for the primary purpose of solubilizing carbohydrate components so they can be hydrolyzed to fermentable materials. The solubilization is a two-fold mechanism of hydration and gelatinization. The temperature range in which the processes take place most efficiently is specific for each grain starch used in a distillery (Table 3). Rye grain starch hydrates/gelatinizes over a range of 57 to 70°C (MacLeod, 1977). Carbohydrate-hydrolyzing agents such as amylolytic enzymes will act very slowly, if at all, on starch that has not undergone hydration and gelatinization.

The gelatinization temperature range for rye starch is determined microscopically by observing the temperatures at which birefringence or double refraction of the initially

crystalline starch granules begins to degrade and is then totally lost. Birefringence refers to the pattern of striations, or dark areas on starch granules, that appear when unheated crystalline starch solutions are viewed through a microscope using plane-polarized light. The birefringence pattern, in combination with the granular shape, is specific for each starch and can be used most effectively for the purpose of identifying starch origins. As the starch granule begins the process of gelatinization the granule swells, loses its crystal form and the birefringence gradually disappears.

Rye meal begins the hydration process immediately on dispersion in water with the exposed starch granules in the flour and fines chemically binding with the water molecules as the first step in solubilization of the starch. This means that the hydration of starch will be facilitated by the proper sieve analysis, which in turn will determine the dispersion efficiency of the particles.

Simultaneous to the chemical hydration process taking place, the physical phenomenon of gelatinization of the starch will begin. As the temperature of a grain meal slurry is raised to a critical level, the starch granules start to lose their crystalline structure. As water makes its way between the tightly-packed, hydrogen-bonded layers, granules swell and eventually the starch moieties disperse into the liquid phase. This swelling or gelatinizing of the granule and the subsequent starch dissipation will result in a substantial increase in the viscosity of the mash. If suitable liquefaction enzymes are not present, the viscosity will overload most mechanical mixing systems.

**Table 3.** Gelatinization temperature ranges of grain starches.

Grain starches	°Celsius	°Fahrenheit
Barley ( <i>Hordeum</i> )	52-59	126-138
Wheat ( <i>Triticum aestivum</i> )	58-64	136-147
Rye ( <i>Secale cereale</i> )	57-70	135-158
Maize/corn ( <i>Zea mays</i> )	62-72	144-162
Rice ( <i>Oryza sativa</i> )	68-77	154-171
Sorghum ( <i>Sorghum vulgare</i> )	68-77	154-171

### Rye starch structure

Rye starch, like most starches, consists of two main carbohydrate polymers called amylose and amylopectin (Figure 2). The basic unit in both starch forms is the monosaccharide D-glucose; and it is the method by which the units are joined that results in the two distinct entities. Amylose consists of unbranched chains of up to a thousand units of D-glucose (molecular weights up to one million) in which each glucose molecule is joined to the next by an oxygen bond between the No. 1 carbon atom of one unit and the No. 4 carbon atom of the next to form an  $\alpha$ -1,4 D-glucosidic link. Amylopectin starch contains the same linear chains of D-glucose units but the chains are much shorter, consisting of up to two dozen glucose units joined by  $\alpha$ -1,6 D-glucosidic linkages to give it a highly-branched structure with molecular weights up to six million. The ratio of amylopectin to amylose in rye starch is usually reported to be in the order of 4:1 (Headon, 1991).

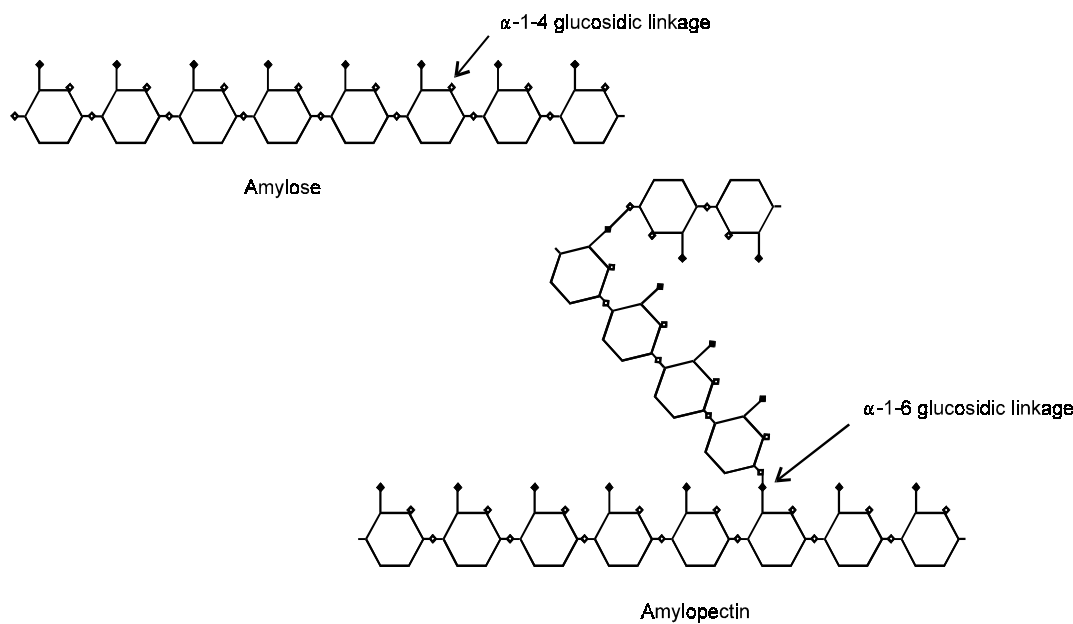
Owing to the differences in physical form, the two types of starch have numerous distinctive physical properties that have been described extensively in the literature (Noller, 1958; Whistler *et al.*, 1953). Of importance to the scope

of this chapter is the enzymatic degradation or hydrolysis of the two starch types by  $\alpha$ - and  $\beta$ -amylases.

### Sanitization of rye mash

The gelatinization temperature of rye grain starch is always exceeded in distillery cooking practice because the second purpose of mashing is sanitization of the mash as a means of microbial control during the fermentation stage. The higher mashing temperatures allow shorter exposure times to be effective for pasteurization and hence a more efficient mashing process is possible. A balance must be struck between using temperatures high enough to effect an efficient sanitization without destroying sensitive carbohydrates. The indigenous enzyme activity in rye meal may under certain cooker conditions produce significant quantities of low molecular weight sugars early in the mashing process. These will be susceptible to some degree of caramelization when exposed to high temperatures and will be lost as fermentables.

Similarly, the starch granules in the flour and fines of the rye grain meal may undergo irreversible damage from high temperatures,



**Figure 2.** Structure of amylose and amylopectin starch polymers (Morrison, 1992).

especially if gelatinization of the granules has not taken place due to dough balling. The damage may be in the form of granular distortions that prevent gelatinization. Hence the starch cannot be hydrolyzed to fermentable sugar.

### ***Hydrolysis of rye starch using malt enzymes***

The third purpose of mashing is to hydrolyze large carbohydrate polymers to intermediate-sized dextrans and/or fermentable sugars through the presence or addition of physiologically active proteins called enzymes. This physiological activity is indispensable to fermentation by yeast, because yeast are incapable of converting the large carbohydrate polymers directly to ethanol. The extent of hydrolysis and the type of fermentable carbohydrate formed will depend on the enzyme source (e.g. malt (essentially sprouted barley), commercial microbial enzyme products or indigenous enzymes) and on the cooking conditions.

The use of malt as an enzyme source in the cooker supplies a mixture of liquefying and saccharifying enzymes and will yield a mash with a high content of fermentable sugars and a low content of dextrans, which is ready for fermentation. When malt is used with rye meal, the indigenous enzyme activity is sufficient to significantly alter the sugar:dextrin ratio and affect both the mashing and fermentation processes. Further dextrin breakdown would take place, albeit under less than ideal conditions, in the fermenter (called secondary conversion) as the yeast deplete the fermentable carbohydrate supply.

The main physiological activity of malt meal is caused by the presence of two amylases (starch-splitting enzymes),  $\alpha$ -amylase and  $\beta$ -amylase. In the presence of gelatinized (cooked) starch, each enzyme carries out the hydrolysis in ways both specific and collaborative. The  $\alpha$ -amylase is called a liquefying (or dextrinizing) enzyme because of its ability to break down the large starch molecules into smaller, more soluble

dextrans (short chains of glucose units) and thereby to reduce the viscosity of gelatinized-starch solutions. The  $\beta$ -amylase is called a saccharifying (sugar-producing) enzyme because of its ability to hydrolyze starch solutions that have undergone gelatinization to yield the fermentable sugar maltose (a disaccharide, i.e., composed of two glucose units). Both  $\alpha$ - and  $\beta$ -amylases in malt meal are able to cleave  $\alpha$ -1,4 D-glucosidic linkages in both amylose and amylopectin starch, but do so in a different manner, to a different extent and to different sugar spectra. The  $\alpha$ -amylase hydrolyzes the  $\alpha$ -1,4 linkages in the amylose and amylopectin molecules in a random fashion to produce a carbohydrate spectrum consisting of glucose, maltose and various short-chain molecules known as dextrans (oligosaccharides), including some containing the  $\alpha$ -1,6 D-glucosidic bonds which the  $\alpha$ -enzyme cannot split. These amylopectin fragments have been given the name 'limit dextrin' as they cannot be reduced in size any further without the use of a  $\alpha$ -1,6 linkage-breaking enzyme.

The size of the initial limit dextrin from amylopectin will determine how close to the  $\alpha$ -1,6-branch linkages the enzyme can carry out the hydrolysis. The ultimate limit dextrin would be the trisaccharide panose with three units of glucose joined with  $\alpha$ -1,4 and  $\alpha$ -1,6 linkages. Of all the  $\alpha$ -amylases available to the distiller, the  $\alpha$ -amylase from malt is best able to approach the ultimate limit with the production of the panose (Whelan, 1964).

The  $\beta$ -amylase, starting from the non-reducing end, hydrolyzes every second  $\alpha$ -1,4 D-glucosidic linkage in amylose and in the straight chain portions of amylopectin up to the branch point containing the  $\alpha$ -1,6 linkage. Hence, the carbohydrate spectrum resulting from this activity will consist of maltose and a relatively high molecular weight limit dextrin. This limit dextrin will succumb to attack by the accompanying  $\alpha$ -amylase enzyme and will then be reduced to a true limit dextrin by the  $\beta$ -amylase.

### **Hydrolysis of rye starch using microbial amylases**

With the advent of thermostable bacterial  $\alpha$ -amylases (as from *Bacillus amyloliquefaciens* and *Bacillus licheniformis*), the ability to 'custom hydrolyze' in the mash cooker has become a reality. Whereas the malt enzymes produce a carbohydrate spectrum with the disaccharide maltose as the major component, bacterial  $\alpha$ -amylases tend preferentially to hydrolyze the starch molecule to maltohexose (with six glucose units) and other, larger dextrans. The higher molecular weight carbohydrates are less susceptible to heat damage (caramelization), less likely to retrograde to unusable forms and will not support bacterial growth to the same extent as maltose. Additionally, the bacterial amylases remain active for longer periods during the mashing process and may even survive into the fermenter. The conditions in the fermenter will be less than optimal for the bacterial amylases, but over a fermentation period of three days there is reason to believe that their contribution could be significant. In the fermenter the surviving amylases may be joined by the saccharifying fungal enzyme 'glucoamylase' (amyloglucosidase), which may be produced in the plant in mash form, or purchased as a commercial preparation. It will finish the conversion of the dextrans to fermentable sugars by hydrolyzing individual glucose units.

### **Rye distillery mashing procedure**

At a typical distillery in western Canada rye grain meal is mashed in vertical batch cookers of 11,500 US gallons (43,600 liters) equipped with two-speed 60 HP agitators, nine steam-injection spargers and internal and external cooling coils (Figure 3). The steam spargers, internal coils and the agitators work together during the mashing cycle to provide the necessary degree of mixing for rye mashes. Modernized distilleries now initiate and control the process steps for mashing and transfer to fermenters from the main console

of a microprocessor-style computer such as the Honeywell 2000.

Rye grain distilleries using batch cooking conduct the mashing in the following manner. Sufficient city water/recycled steam condensate is mixed with recycled backset (thin stillage) in a clean cooker to create a starting temperature of 30-35°C, a pH of 5.8-6.0 and a calcium concentration of 75 ppm. On full agitation the cooker receives 22,400 lbs (400 bushels = 10,160 tonnes) of rye meal from a microprocessor-controlled weighbelt or dropbin and the temperature is raised to 65°C by live steam. The microprocessor programs the addition of a thermostable  $\alpha$ -amylase liquefying enzyme (such as Allcoholase I) at a rate of 0.05% of the grain weight and allows a hold of 10-30 minutes as required. The length of hold at this point will be determined by whether the viscosity of the mash is causing any impediment to mixing. The viscosity of the mash at this stage will depend on the level of intrinsic enzymes in the rye capable of extensive hydrolysis of the large starch, protein and gum molecules in the mash, if given sufficient time. If intrinsic enzymes are in short supply, quantities of a  $\beta$ -glucanase enzyme (such as Allzyme) may be added at 65°C and a 30 minute hold put into effect. This is done to lower the molecular weight of the glucan gums (polymers of glucose with  $\beta$ -type linkages) to at least the five glucose unit size.

Steam injection continues until the mash temperature is 85°C and then a final gelatinization hold of 10 minutes is allowed. After cooling to 78°C, a further addition of liquefying  $\alpha$ -amylase enzyme is made at 0.05% of the grain weight. A 10-minute hold is taken and the mash is cooled to 70°C. It is then pumped to a holding vessel for transfer through a cooler to the fermenters.

If the production schedule permits a longer mashing period, a final gelatinization hold at 80°C for 20 minutes would suffice (instead of 85°C for 10 minutes) with a net saving of steam and possibly a decrease in caramelization of low molecular weight sugars. It is certainly worthwhile to stay flexible and to experiment with different mashing techniques now that energy is the major cost item.

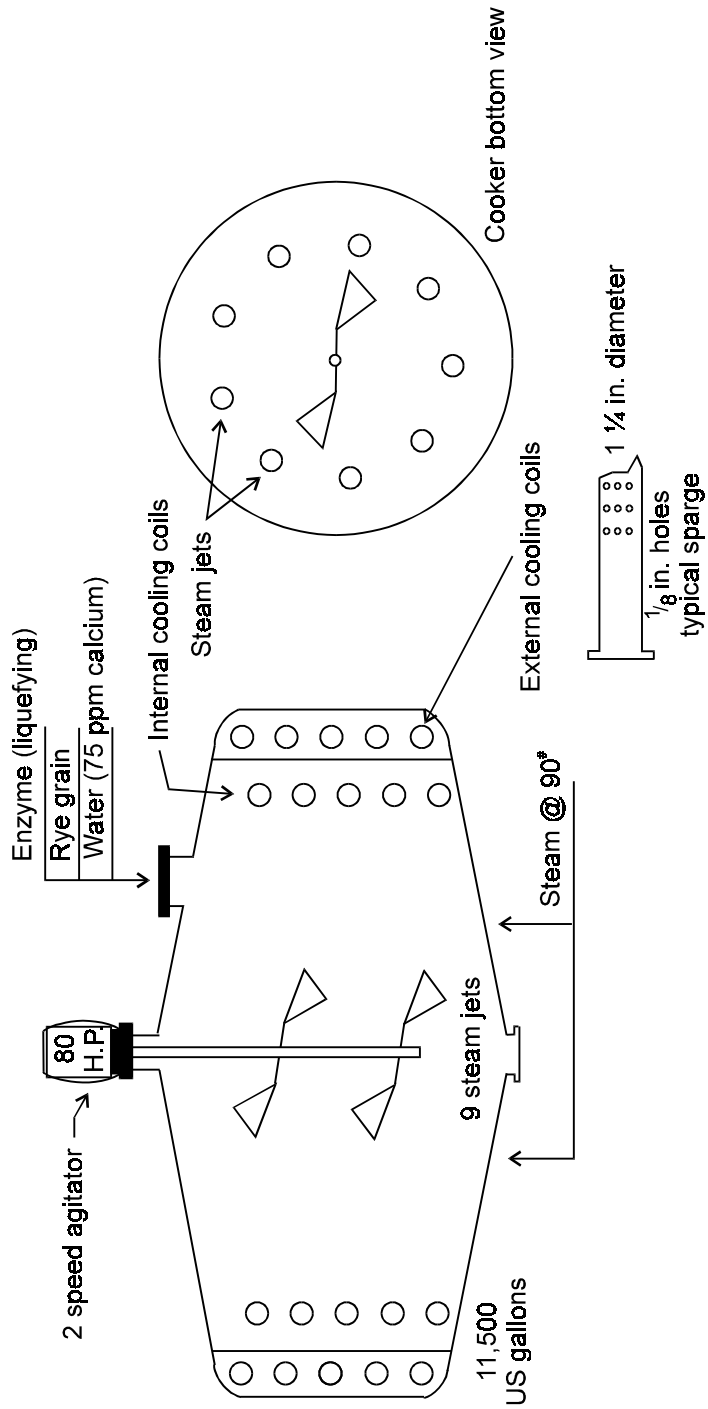


Figure 3. Vertical batch cooker for rye meal (Morrison, 1992).

It is important that the holding vessel be of sufficient capacity to be used for emergency storage of mash in case of maintenance problems or process delays that prevent the immediate transfer of mash to the fermenters. A surge volume in the holding vessel of at least twice the normal mash volume would be a good investment in plant flexibility. It is essential that the holding vessel be equipped with good agitation and a means of maintaining the mash temperature at 63-65°C.

The agitation and high temperature will maintain the mash in a pasteurized condition until delivery to the fermenter system takes place. Failure to maintain good microbial control at this stage may result in significant lactobacillus contamination with the loss of sugar to lactic acid production. It is also considered good manufacturing practice to have some steam sparged into the holding vessel when empty as a further retardant to bacterial growth. The final cooked mash analyzes at 21-21.5% solids, which calculates to about 264 US gallons liquid dilution per 56 lb distillers bushel (386 liters liquid dilution per 100 kg) of rye meal. Variations in the percent solids should be expected if cooker start temperatures, heat load in the steam and the proportions of hot backset and city water undergo variations from day to day.

### **Rye fermentation procedure**

The fermentation of rye mash in western Canada is typically carried out in a series of 45,600 US gallon (172,000 liters) fermenters equipped with internal horizontal cooling coils and side-mounted Lightnin mixers. Most fermenters are insulated and use live steam injection as a means of raising the internal temperature to pasteurization levels as part of the cleaning cycle. Temperature probes and cooling water flow recorder controllers complete the equipment package on these vessels. In some older plants the fermenters are open-topped, do not have pasteurization capabilities, and are fitted with a platform of internal cooling coils horizontally positioned halfway up the wall.

Into a clean fermenter (and thoroughly clean and constantly-steamed lines) sufficient backset stillage (usually 25% of total fermenter dilution) and wash water are combined with three or four cooker mashes (depending on fermenter size) and a 1.88% by volume yeast mash. The mash and backset are cooled to 35-38°C by passing through banks of counter-flow pipe coolers. For open-top fermenters with less efficient cleaning, the backset may be acidified as a bacterial control measure. Additionally, the stillage-acid mixture may be used at high temperatures for continuous circulation through coolers and mash lines as a means of bacterial control when production delays occur. The combination of the heat, acid and grain solids in the mixture has an excellent scouring action on the cooler and mash lines.

The yeast mash addition can be the liquid yeast mash produced as described later in this chapter, or it can be dried distillers yeast either in dry powder form or after reconstitution in a dilute mash for several hours. The equivalent of 50 lbs (22.6 kg) of the powdered yeast should be added to each fermenter. A yeast cell count of 250 million cells/ml is recommended for fermenter treatment for efficient conversion of the sugar to alcohol. The liquid dilution of the prepared yeast mash slurry is on the order of 70:1, so the fermenter should have an initial viable yeast count of approximately 3 million cells/ml.

For fermenters 'set' (filled) with mash that has been liquefied with commercial thermolabile  $\alpha$ -amylases only, a quantity of amyloglucosidase enzyme must be added. The enzyme can be added as either a prepared glucoamylase mash (described later) or an equivalent commercial amyloglucosidase (such as Allcoholase II). The dosage rate of 3,500 glucoamylase units (AMU) per 56 lb bushel of rye grain is needed for efficient saccharification. The target dilution in a rye fermenter is usually 36-39 US gallons liquid per 56 lb distillers bushel (526 liters liquid dilution per 100 kg) rye meal. This means that the total volume including grain meal, mashing water, backset stillage, yeast, enzyme mash and wash water will equal 36-39 US gallons for each 56

lbs of grain meal added. Set temperatures will vary according to cooling water temperatures, but generally are about 30°C with cooling water control settings at 33-34°C. Fermentation is continued for 72 hrs with slow, constant mixing. In cases where foaming becomes excessive, a cool water spray reinforced occasionally with antifoam is used as a means of control. This foam control is usually required when the plant receives large quantities of newly-harvested rye grain and/or when distillery-produced glucoamylase mash is not available.

### ***Rye fermenter process control***

Fermenter data are recorded at set times and then every 24 hrs throughout the 72 hr fermentation. Fermenter set data include mash sequence used, number of mashes, grain mash bill (percentages of grain types used if applicable), enzyme additions, yeast tub used and set temperature. Operator analyses include pH, titratable acidity, Balling hydrometer reading, temperature and time of fermenter activity. The rate at which pH and titratable acidity change in the fermenter can be an indication of cleaning operation efficiency and of the success in keeping the mash in a sanitized condition. The rate at which the Balling degrees change is an indication of the yeast cell population, propagation efficiency and the rate of consumption of sugars. The temperature recording will indicate whether the cooling water system is functioning properly in order to ensure that the yeast have an optimum working temperature.

At 72 hrs the fermenter is placed on full agitation for 1 hr and then sampled prior to transfer to the beer well to await distillation. A sample of each thoroughly mixed fermenter is sent to the laboratory for analyses, which include alcohol level and the resultant amount of absolute alcohol, the colloidal fraction, pH and final titratable acids. If the data indicate that the fermenter is still active, then it will be moved out of sequence for an additional half day and another fermenter may be transferred to the

beer well a few hours early. The beer alcohol percentage and total quantity of absolute alcohol are used to calculate the fermentation alcohol yield entering the distillation system. This in turn will allow calculation of the distillation efficiency. The colloidal percentage is used to predict viscosity levels in the last stages of the dryer plant evaporation system and to indicate the adequacy of the enzyme additions in the cooker and fermenter.

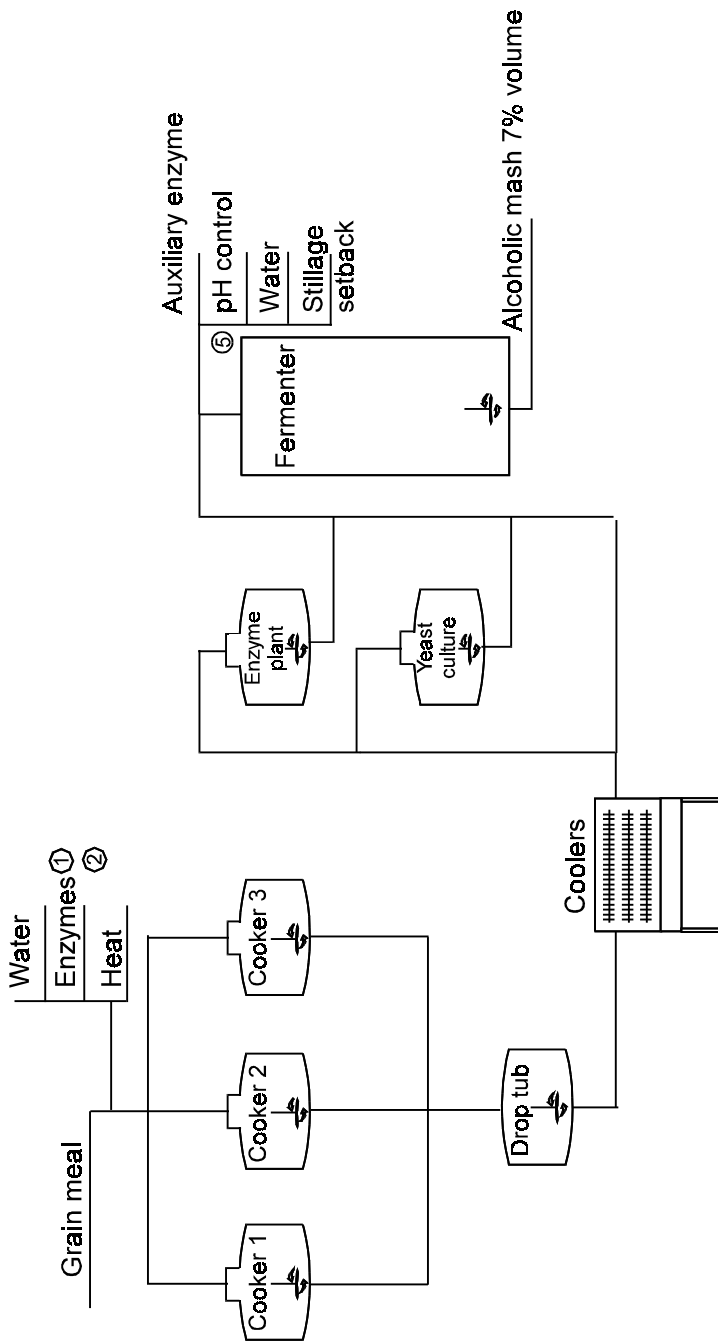
Figure 4 is a flow sheet of the mashing and fermenter-filling procedures and includes the positioning of the yeast and enzyme systems. Note the quality stations and the process checks recommended for good manufacturing practice.

## **Yeasting, lactic cultures and glucoamylase**

### **Yeast mash preparation - lactic souring**

The lactic-yeasting operation at rye grain distilleries uses an all-rye grain mash slurry, which is pre-cooked at 65.5°C for 30 minutes and then cooled to 53°C. A small amount of thermolabile  $\alpha$ -amylase enzyme is added for liquefaction and the mash is then inoculated with a *Lactobacillus delbrueckii* culture and maintained at 53°C for 9-11 hrs. Initially the lactobacillus inoculum may be grown from an American Type Culture slant incubated at 53°C in sterilized rye. However, when the first culture has been grown satisfactorily on a plant scale, pails of the culture can be used as described below for future inoculation.

Rye meal can be used as a yeast mash slurry (unfortified by malt or other nutrients) as it meets the nutritional needs of the yeast. The cooking process promotes the release of starches and proteins from the meal, which in turn undergo conversion by the indigenous rye enzymes to smaller-sized carbohydrates and amino acids. This resultant mash is further converted and altered by the action of the lactobacillus incubation as described below to produce additional nutrients required by the yeast.



**QUALITY STATION      REQUIRED PHYSICAL/CHEMICAL CHECKS**

- ① Mash water: calcium, potable condition
- ② Cooker enzymes: physiological enzymatic activity
- ③ Cooker: heat profile, mash solids, conversion pH, physical additions, mash designation
- ④ Microbiological: analytical profile for each area
- ⑤ Fermenter technology: set parameters, analytical control, date collection and processing

Figure 4. Mashing and fermentation flowsheet (Morrison, 1992).

During the lactic incubation period the lactobacilli multiply and produce lactic acid, which in turn lowers the pH to about 3.6. A healthy propagation of lactobacilli will generate up to 0.9% lactic acid in the 9-11 hrs and the mash viscosity will be lowered appreciably. Plant process quality control includes periodic pH checks and the titration of a 100 ml mash sample with standard sodium hydroxide to monitor acid development. As inoculum for future use, pails containing 6 US gallons (22 liters) of the completed lactic incubation (with a minimum of 0.9% lactic acid) are collected. Pails are cooled to room temperature, treated with powdered calcium carbonate to raise the pH to 5.5 and quick-frozen in upright freezers.

The deliberate cultivation of a lactobacillus culture in the preparation of yeast mash is good manufacturing practice with several benefits to the process. The selected temperature of incubation at 53°C encourages certain high acid-producing bacteria to predominate in the mash and to produce sufficient lactic acid in 11 hrs to drop the pH to 3.6. Deviations from the 53°C incubation temperature will decrease the purity of the population and the amount of lactic acid generated.

The time period and temperature of the lactobacillus incubation create an environment where bacterial spores are encouraged to metamorphose to a more easily-destroyed vegetative form prior to the final sterilization step. The lowered pH enhances sterility by creating an environment in which microbial proteins are made more susceptible to heat denaturation, which leads to the destruction of the organisms.

Finally, because of certain common biochemical pathways with yeast, the lactobacillus cultivation continues the conditioning of the rye mash by releasing certain growth factors that accelerate yeast growth. Presence of these growth factors, along with the nutrients in the rye mash, ensures healthy and prolific yeast cell growth.

### ***Yeast mash preparation - sulfuric acidification***

On occasion, timetable or maintenance problems make it difficult to allow the yeast mash sufficient time for a proper lactic acid hold. The mash is therefore prepared and precooked in the same manner as before; but the pH is adjusted to around 3.6 with sulfuric acid. This method allows no time for any bacterial spores to vegetate and subsequently be destroyed during sterilization; so an acid mash may undergo bacterial growth of considerable magnitude during the yeast incubation stage. Additionally, the intermediate nutrients mentioned above will not be produced and cannot contribute to healthy yeast growth. Thus, sulfuric acidification has the potential to produce inferior yeast inoculum for the fermenters. This method should be used only when all other options have been exhausted.

### ***Yeast mash sterilization and inoculation***

After acidification by lactic culture or sulfuric acid, the yeast mash is sterilized by heating to 121°C for 1 hr. It is then cooled to 30°C and treated with a small amount of thermolabile  $\alpha$ -amylase at 79°C and a saccharifying enzyme such as glucoamylase mash at 57°C, and inoculated with large cans of *Saccharomyces cerevisiae* yeast culture. These large cans of yeast culture are prepared by standard propagation techniques from working yeast slants using a concentrated malt syrup as the medium. The inoculated yeast tub is incubated under controlled temperature conditions for 16-18 hrs with automatic agitation for 1 minute every 7.5 minutes. At the end of the 16-18 hr time period the yeast tub is cooled to 16°C and allowed to remain at that temperature until needed in the fermenters. The yeast propagations are usually out of phase by about 12 hrs so that each yeast vessel may inoculate a series of fermenters with a minimum hold time.

### Yeast cell growth processes

Soon after the yeast culture is added to the sterile rye yeast mash, the yeast cells begin absorbing the available nutrients and will increase in size. Buds begin to appear at specific spots on the yeast cell, become larger and take on the appearance of the mother cell. If the budding is sufficiently rapid, the buds or daughter cells in turn form daughter cells and the yeast take on the appearance of chains of cells. Later in the incubation the chains will decrease in length and single or double budding cells will predominate. This budding process is the manner in which yeast cells replicate to produce a culture containing millions of cells in every milliliter of mash. Using phase contrast microscopy, the budding mechanism of yeast cells has been recorded on film and is presented in the literature (Stewart *et al.* 1981; Lyons, 1981).

The growth cycle described above is an aerobic process, meaning that oxygen (air) is essential. Oxygen becomes a limiting factor in

cell growth because of its role in providing cell membrane lipid materials in the growing yeast cell (Ingledew, 1991). Agitating the yeast mash periodically throughout the incubation facilitates the air incorporation into the mash. The agitator churns the mash and brings all parts of the mash into contact with air at the surface. Generally, the yeast cell count obtained during the incubation will be a function of the amount of oxygen made available.

After the 16-18 hrs incubation, when the viable cell count should be approximately 200 million cells/ml, contents of the yeast vessel are cooled to 16°C. The cooling is done in such a way that the rapid cell growth potential is retained and may be recovered after the yeast are pumped to the fermenter as inoculum. The logic of slowing yeast cell development through cooling becomes clear by viewing the growth curve for yeast presented in Figure 5. Cooling is carried out at a point when the growth curve has maximum slope and the cells have the greatest potential for budding. The fermenter

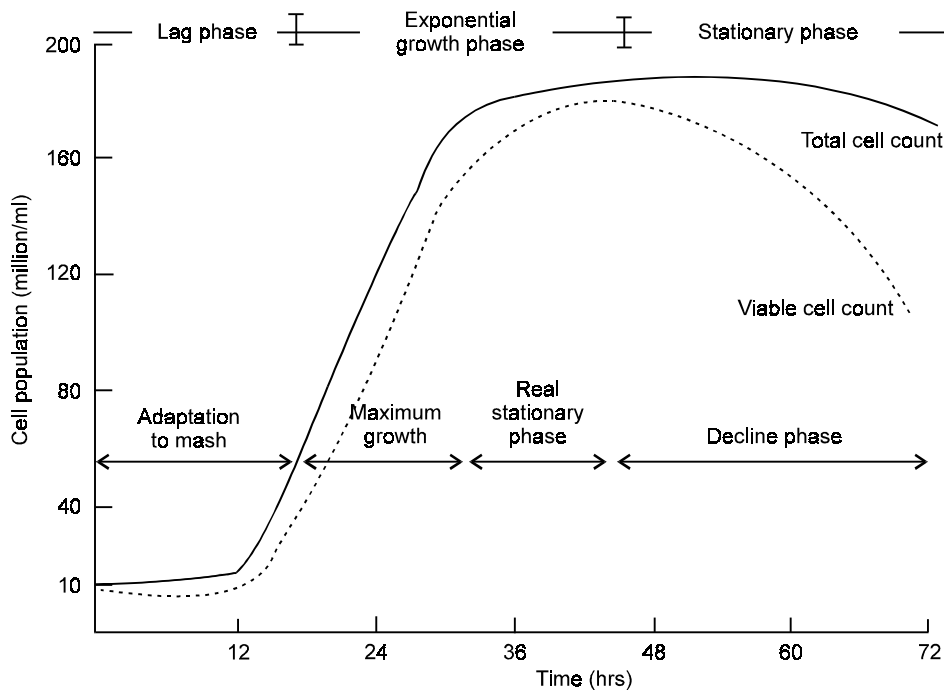


Figure 5. Typical yeast growth curve in rye grain mash (Lyons, 1981).

mash will in turn supply nutrients and oxygen to the yeast inoculum so that a new cell growth curve takes over and appropriate yeast counts are achieved in the fermenter.

### **Production of glucoamylase by submerged culture**

Fungal amylases such as amyloglucosidase (glucoamylase) are produced by a submerged culture process in which a sterilized grain mash substrate (usually corn) is cooled to 37.7°C, subjected to a steady flow of sterile air (usually 0.5 volumes of air per volume of mash per minute), agitated rapidly (to assist air dispersion), inoculated with a strain of *Aspergillus* or *Rhizopus* and allowed to incubate for 4-6 days. Fungal inoculum may take the form of spores suspended in sterile water that is pressure-jetted into the reactor via a sterilizable transfer valve arrangement. Alternatively, a standard aseptic propagation procedure of fungal slant to broth flask to media flask to dona tank to reactor can be used. Both systems work equally well with the larger dona inoculum having the possible advantage of a shorter incubation period. The fungal spores will vegetate into individual cells and begin propagating new cells end to end to form filaments called hyphae. The hyphae branch and intertwine to form a mycelium (Pelczar, 1958). In about 40 hrs the mycelium will propagate throughout the grain medium mash and the mash will take on a thick, lumpy texture. A very characteristic mold odor will become apparent; and microscopic examination reveals extensive networks of elongated cells with very distinct walls dividing the cells.

Incubation is continued at full agitation and aeration for four days, and then a sample is removed for enzyme analysis, pH, citric acid level and microscopic examination. These data are compared to standard reactor data for a high enzyme-yielding incubation and adjustments in operating parameters may be made if necessary. Usually by the sixth day of incubation the fungus has developed its maximum level of enzyme, and

the reactor contents are analyzed again for enzyme level and a calculation is made of the amount required for addition to fermenters.

Submerged culture glucoamylase mash is used as a source of saccharifying enzyme in the fermenter in conjunction with the use of thermostable bacterial  $\alpha$ -amylases in the cooker. The enzyme combination is able to carry out the quantitative hydrolysis of starch to glucose owing to ability of the glucoamylase mash to hydrolyze the dextrans left in the cooker.

A typical glucoamylase production facility in a rye distillery will consist of 2-4 reactors of 3,400-3,900 US gallons (13,000 to 15,000 liters) and one or two scale-up reactors of 450 US gallons (1,700 liters). Operation is coordinated with the mashing and fermenting schedules in order to cultivate sufficient glucoamylase in mash form for use as the saccharifying enzyme in the fermenters. The mash additions to the fermenters are determined by the enzyme concentration of the mash and the need to supply 3,500 glucoamylase units for each 56 lb bushel of grain in the fermenter.

## **Distillation of rye whisky spirits**

### **Beer still operation**

At the end of the fermentation cycle, usually after 72 hrs, the contents of the fermenter are pumped to a holding tank known as the beer well. The beer well, essentially a surge tank, provides a continuous supply of beer to the first stage of distillation in the beer still. The beer still separates the volatile organic components of the beer from the grain solids and the carbon dioxide gas.

Rye distillers commonly use a 45 tray beer still to convert a fermented beer with 8% alcohol by volume into a solids-free high wine of about 120° US proof (60% alcohol by volume or 60° GL). The beer is fed through a preheater onto the top sieve tray of the stripping section of the beer still. The beer travels across the tray in a shallow layer and is prevented from flowing

through the holes by an upward flow of steam. It then descends through the downpipes and across the trays repeatedly. The steam drives off the volatiles, which proceed up the column undergoing alcohol enrichment to become the high wine product at 120° US proof. At the same time, the beer moving down the stripping section becomes increasingly depleted of alcohol and arrives at the bottom tray as stillage. A stillage containing less than 50 ppm alcohol is normally considered acceptable for process control.

Figure 6 is an integrated flow sheet for the production of high wine from rye grain with nine stations for quality control analyses. Such flow sheets are invaluable in illustrating the total quality management necessary for good manufacturing practice. It is important to see the quality stations as loops in the process where the quality decisions at any stage affect the expected result at the following stage.

### **Rye whisky spirits manufacture**

Rye whisky spirits are distilled from high wine using a two-column rectification system consisting of a 36-40 tray extractive distillation column followed by a 65 tray rectifying column. The system may also be equipped with two additional columns of smaller size, one for fusel alcohol separation and the other for concentration of the aldehydes and other heads congeners. The two column system is able to convert 34,500 US proof gallons of high wine at 120° US proof (108,000 liters at 60° GL) into rye whisky spirits at 192° US proof (96° GL).

### ***Extractive distillation***

An extractive distillation column is used to concentrate and remove the major portion of unwanted congeners before the alcohol stream enters the rectification column to be concentrated as whisky spirits. The whisky spirits will be essentially free of all objectionable impurities, but will contain controlled amounts of the congeners that create the proper balance

of whisky character and flavor during the subsequent aging in oak barrels.

An extractive distillation column operates on the theory that higher (fusel) alcohols, aldehydes and esters are less miscible in water. Controlled water addition allows these compounds to go up the column and be drawn off as a heads stream while the diluted alcohol (containing controlled amounts of congeners) emerges from the bottom of the column and is fed into the rectifying column. Thus, an extractive distillation system can control the amounts of alcohol and heavy fusels that leave the top of the column and control the amount of congeners that exit out the bottom of the column with the diluted alcohol.

On a continuous basis, high wine at approximately 120° US proof (60° GL) is passed into a surge tank and diluted 1:1 with recycled rectifier bottoms. The 60° proof (30° GL) mixture is fed into the 36-40 tray extraction column about two-thirds of the way up the column. A 20° proof (10° GL) alcohol stream containing small levels of congeners emerges from the bottom of the column. This stream may be further diluted if live steam is injected at the base of the column. At the same time higher alcohols, aldehydes and some esters make their way to the top of the column and are taken off from the condensate-reflux loop and fed either to a fusel oil column or to a decanter. In the fusel oil column there is a bottom take-off containing appreciable quantities of recoverable alcohol. The decanter does a similar job by allowing a phase separation to take place, forming an oil layer with a higher concentration of the unwanted congeners and a water layer containing small amounts of congeners and a considerable amount of recoverable alcohol. The water layer is fed back into the extraction distillation column for alcohol recovery and the oil layer is removed for later processing and disposal.

### ***Rectification***

A rectifying column operates on the principle that an alcohol/water vapor leaving a boiling

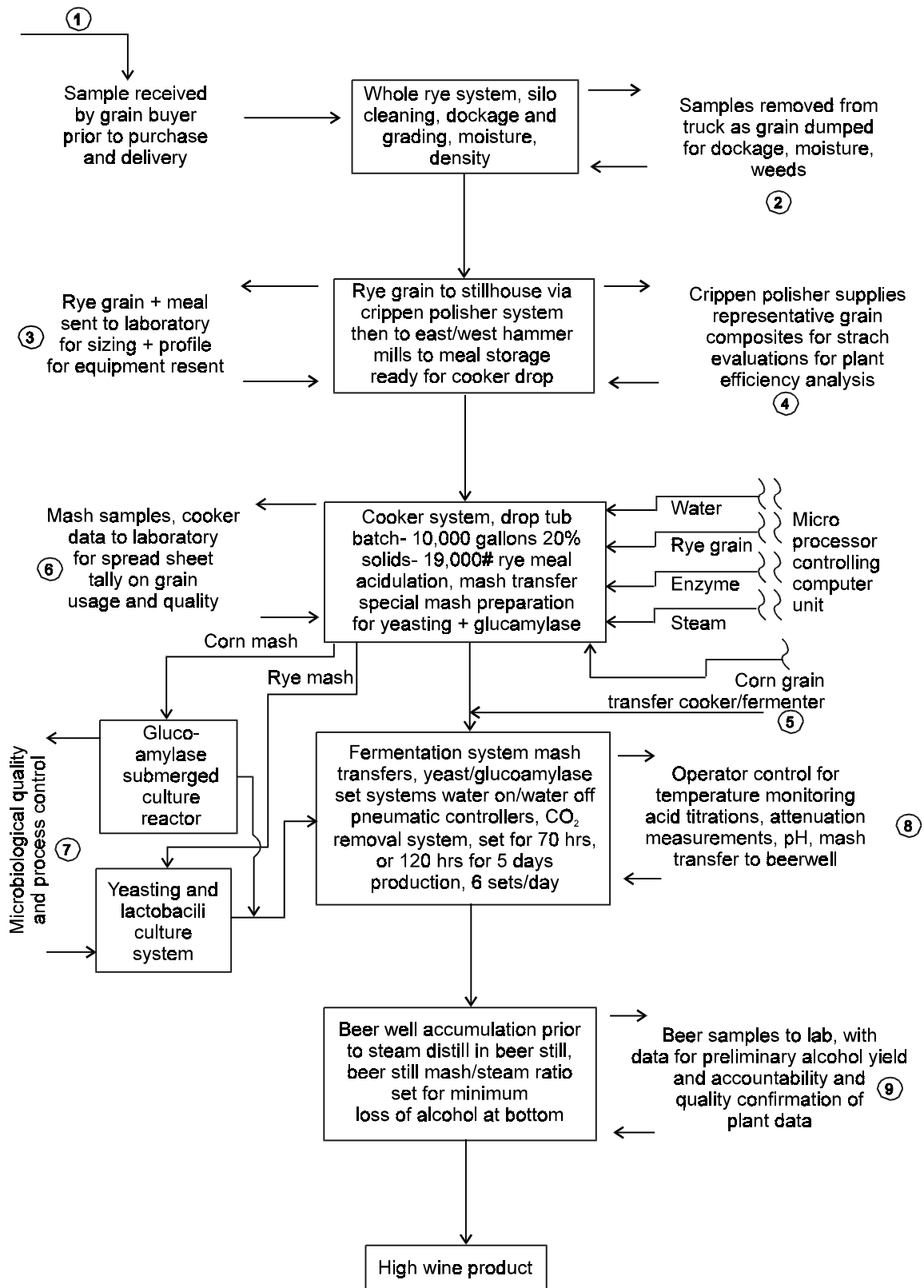


Figure 6. Quality assurance/processing control routes (Morrison, 1992).

layer of liquid on a tray passes up through a liquid layer on the tray above via the bubble caps, condenses and transfers its heat to the liquid. In turn, a portion of this enriched alcohol-water liquid is vaporized and rises to the next tray to repeat the process. Since the alcohol has a higher vapor pressure and evaporates more readily than water, the alcohol concentration of the rising vapor continually increases from plate to plate. Depending on the operation of the column, some portion of the condensed liquid on each tray will flow back down the column to replenish the liquid level on the lower trays. So, as the alcohol-enriched vapor passes up the column, the alcohol-depleted liquid passes down the column. At the top of the column total-vapor condensation occurs and the condensate is fed back into the column to assist liquid replenishment on the trays while the product is drawn off just below the top of the column. The amount of condensate flowing back compared to the product take-off from the column is known as the reflux ratio. The reflux ratio will determine the alcohol concentration on each tray and the alcohol concentration achieved at the product take-off point.

Thus, under proper operation, a rectifying column will move alcohol up the column increasing in strength from tray to tray until the required strength is achieved. Equally important, the tower will move water down the column decreasing the strength from tray to tray until the minimum-detectable alcohol level exists on the bottom tray.

The extractive distillation column bottoms flow at 20° proof (10° GL) passes into the rectifier about one-third way up the column (at tray 26) and proceeds to enrich to 192° proof (96° GL) on trays 58-60, which are the product take-off trays. Alcohol product strength, tray temperatures, base pressures and high wine feed rate are used by the distillation operators in their process control of the column. Gas-chromatographic monitoring of congener presence and magnitude on a number of control trays can follow this column operation. These data are fed back to the operator center on a daily basis to

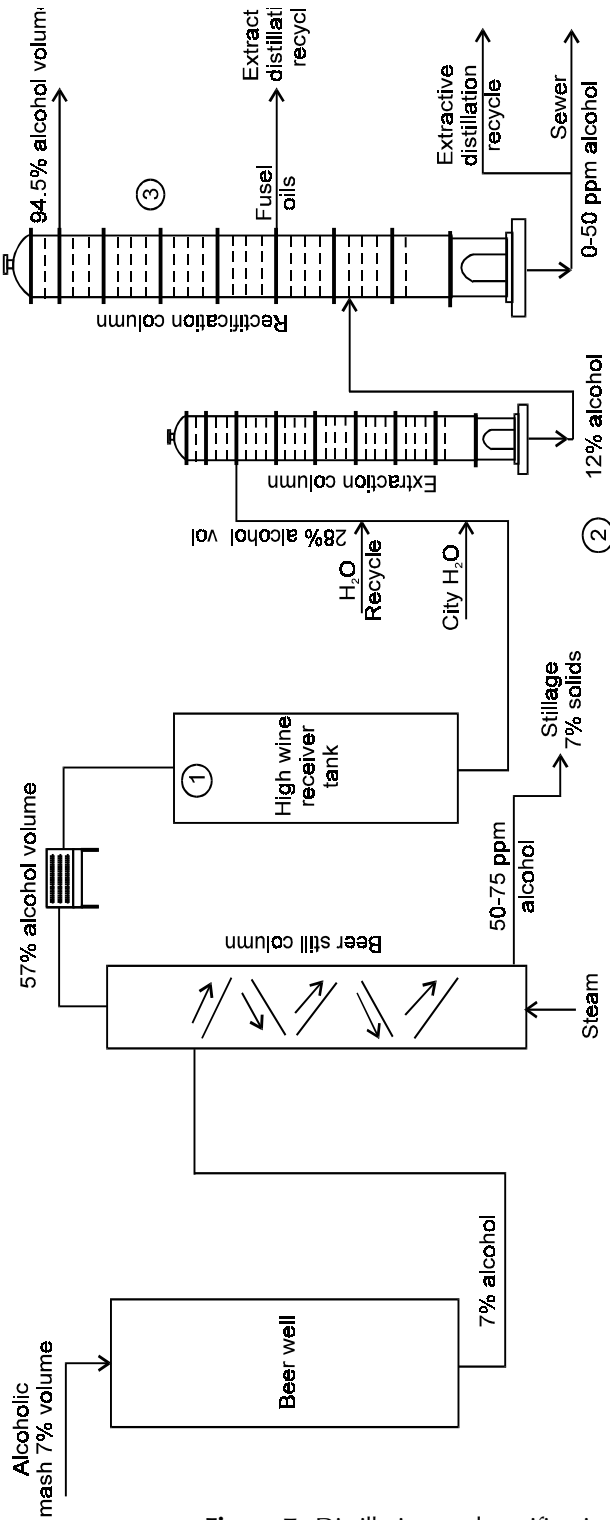
form an ongoing record of column conditions and are used for daily adjustment of column parameters.

Figure 7 is a flow sheet of the beer still and two-stage rectifier for the production of high wine and its processing into rye whisky spirits, respectively. Note the quality stations and the process control checks recommended for good manufacturing practice. The gas chromatographic monitoring of tray components and the use to which the data are put are explained below.

Samples for gas chromatographic analysis from the various trays in the rectification column are taken via sample valves. The sample is passed through a cooler (or simply a long take-off line) exposed to the lower room temperature and collected. For sample lines without proper coolers it will be necessary to take precautionary steps to avoid alcohol 'flashing' into the air when taking samples from the trays at high temperature. Safety equipment must be worn to protect against scalding with the boiling alcohol.

Various monitoring programs may be established. Samples can be analyzed either from all of the selected trays several times daily or certain tray samples can be analyzed only once daily, with additional emphasis on product take-off and perhaps on the congener buildup on fusel oil take-off trays. The analysis of one or more congener substances present on various trays can be used in column process control. When tray analyses are available over a significant period of operation, it then becomes possible to make certain predictions about how the column will react if current run conditions are allowed to continue.

The congeners found and monitored on selected column trays are listed in Table 4. It is important to realize that the analytical monitoring is carried out to determine concentration trends from one production shift to another. For example, if the methanol concentration on the top tray of the column remains reasonably constant at 1,100 ppm; it probably means that the take-off rate of the heads is sufficient for good column operation. Removal of the methanol also means that other undesirable heads fractions are



**QUALITY STATION**

- ① High wine product
- ② Stillage/spent grain
- ③ Rectification column

**REQUIRED PHYSICAL/CHEMICAL CHECKS**

- Organoleptic acceptance, percent alcohol by volume  
acrolein check, daily dip yield calculations,  
clearance for flavoring manufacture
- Alcohol analysis, total solids, solids separation test
- Samples from trays 65, 60, 40, 30, 0 for congener profile  
and operational data. Organoleptic acceptance for vodka,  
barrelling off and gin manufacture, vodka analytical profile

Figure 7. Distillation and rectification flowsheet (Morrison, 1992).

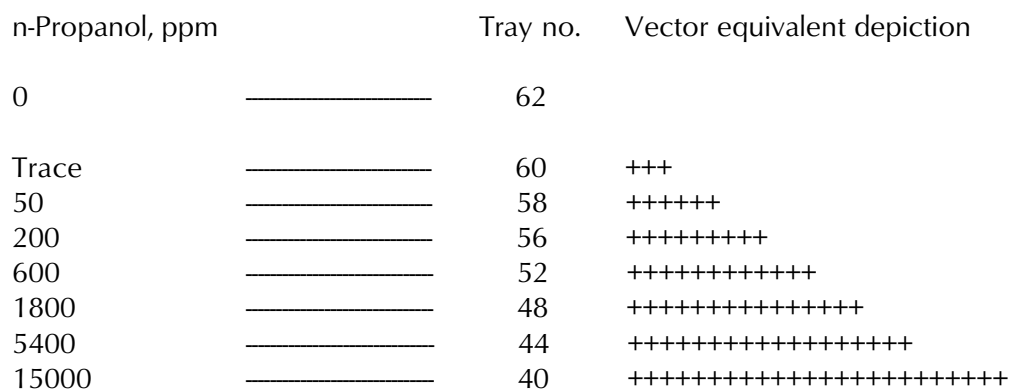
**Table 4.** Congeners found and monitored on rectifier trays.

Congener	Tray number				
	T65	T60	T40	T30	T0
Methanol (methyl alcohol)	X		X		
Ethanol (ethyl alcohol)			x		x
n-Propanol (propyl alcohol)		X	X		
Isobutanol (isobutyl alcohol)			X	X	X
n-Pentanol (n-Amyl alcohol)					X
Isopentanol (isoamyl alcohol)					X
Propenol (allyl alcohol)				X	

being removed and will not eventually spread down the column to appear in high concentrations on the product take-off tray. In this manner the methanol becomes an indicator of column conditions and with a little interpretation can lead to proper adjustments. The methanol analysis along with other chemical testing can also assist in column adjustment for the manufacture of high quality neutral spirit for vodka production.

The rectifier feed from the bottom of the extractive distillation column will contain small amounts of higher alcohols that tend to accumulate in the upper part of the rectifier. Propanol can be used as an indicator of this accumulation because of its ability to exist on certain trays without isobutanol or amyl alcohols

being present. This becomes very important if fusel oil-free spirits or spirits with controlled amounts of congeners are desired. At a position in the column several trays down from the product tray, the n-propanol will accumulate significantly and decrease in magnitude as one moves up the column. If a vector is drawn to represent the n-propanol magnitude on each tray, then the bank of vectors form a triangle with the base on tray 40 (for example) and the apex on a tray somewhere above (Figure 8). As the n-propanol concentration increases in the rectifier, the apex of the triangle will rise higher and higher until n-propanol appears in the product tray. Using historical analysis it is then possible to predict when n-propanol will appear in the product by monitoring propanol



**Figure 8.** n-Propanol concentration as vectors on distillation trays (Unpublished distillery data).

concentration on tray 40. A level of n-propanol on tray 40 will become an operational specification or process control point below which propanol will never appear in the product spirits. Tray 40 then becomes an indicator tray as it indicates what will happen in the near future if the column conditions are unchanged. This information allows a decision about the take-off rate at the fusel oil tray (at tray 30 for example) to be made, thus reducing the total fusel alcohols in the column.

### ***Rye distillation fusel oils***

Fusel oil is the term applied to a group of aliphatic alcohols whose formation is related to the amino acid metabolism of yeast cells. The creation of these alcohols appears to be the result of transamination reactions in which amino groups are transferred from a supplied amino acid to an oxo acid molecule thus leading to the synthesis of a different essential amino acid. The original (amino acid) carbon skeleton, now an  $\alpha$ -oxo acid, then undergoes decarboxylation and subsequent reduction to form the appropriate fusel alcohol. The creation of oxo acids also appears to be possible from carbohydrate metabolism by the yeast, so that fusel alcohol levels continue to rise even after amino acid requirements have been met (MacLeod, 1977; Ingledew, 1991). Total concentration and diversity of the fusel alcohols are significantly related to the amount and form of free amino acid nitrogen available in the grain mash.

The fusel oil alcohols consist of n-propanol, isobutanol and two pentanols (amyls), optically active amyl alcohol and isoamyl alcohol. The combined amyls may constitute the major portion of the fusel oil at a margin of 2:1 over the isobutanol and 10:1 over the n-propanol. Table 5 presents an analysis of fusel oil removed from a two-column distillation system for rye whisky spirits.

The rectifying column trays below the feed tray (about tray 26) are considered stripping trays; and their function is to decrease the alcohol concentration on each lower tray such that the

column bottoms contain essentially zero alcohol. In reality, alcohol concentration will probably remain in the 30 to 50 ppm range with the column running efficiently.

The bottom take-off liquid is usually divided between a recycle stream to the extractive distillation column and excess liquid going to the sewer. Knowledge of the discharge rate and the alcohol content allows the calculation of total alcohol lost. Alcohol concentration in the rectifier bottoms is a function of the column temperature profile and the balance between the rate of alcohol fed into the column and the rate of alcohol removal. Increasing the amount of alcohol fed into the rectifier at any time will cause the alcohol to move to lower and lower trays. This is shown on the control panel as the increased alcohol concentration lowers boiling point temperature. If the imbalance persists, significant quantities of alcohol will appear in the bottoms and be detected by temperature reduction and gas chromatography. The alcohol content of the bottoms then becomes a means of monitoring the process and of suggesting alteration in run parameters. If the bottom effluent is a part of the waste stream from the plant, then the alcohol level will have additional significance, as it will be contributing to biological oxygen demand (BOD) of the waste stream.

### **Stillage and feed grain recovery systems**

Stillage is removed from the bottom of the beer still at about 90°C after passing through a heat exchanger and is pumped to a 240,000 US gallon hold tank equipped with a steam sparger and side-mounted agitator. Running continuously at about 13,000 US gallons (49,000 liters) per hour, hot stillage containing around 8% solids is passed through three Sharples centrifuges to produce a liquid and wet cake containing about 2.5% and 25% total solids, respectively.

The centrifugate containing 2.5% solids is passed into a surge tank and subsequently fed to the first stage of a four-stage falling film evaporator that raises solids content to 30%. The falling film evaporator is followed by a two stage, rising-film, finisher evaporator that increases the solids to a final 38 to 40%. The evaporation system is basically atmospheric and operates in the 109°C range and uses thermal recompression to enhance the heat efficiency.

The wet centrifuge cake moves to a first stage mixer where it is mixed with low moisture recycle and fed to the drying ring of the first stage flash dryer with a cake product take-off for further processing at 90% solids. The 90% solids cake then moves to a second mixer and is homogenized with second stage low moisture recycle and the 40% solids evaporator syrup to produce an 80% solids material which is fed to the drying ring of the second stage flash dryer. At a 90% solids (10% moisture) the process yields about

65 tonnes of distillers dried grains (DDG) per production day.

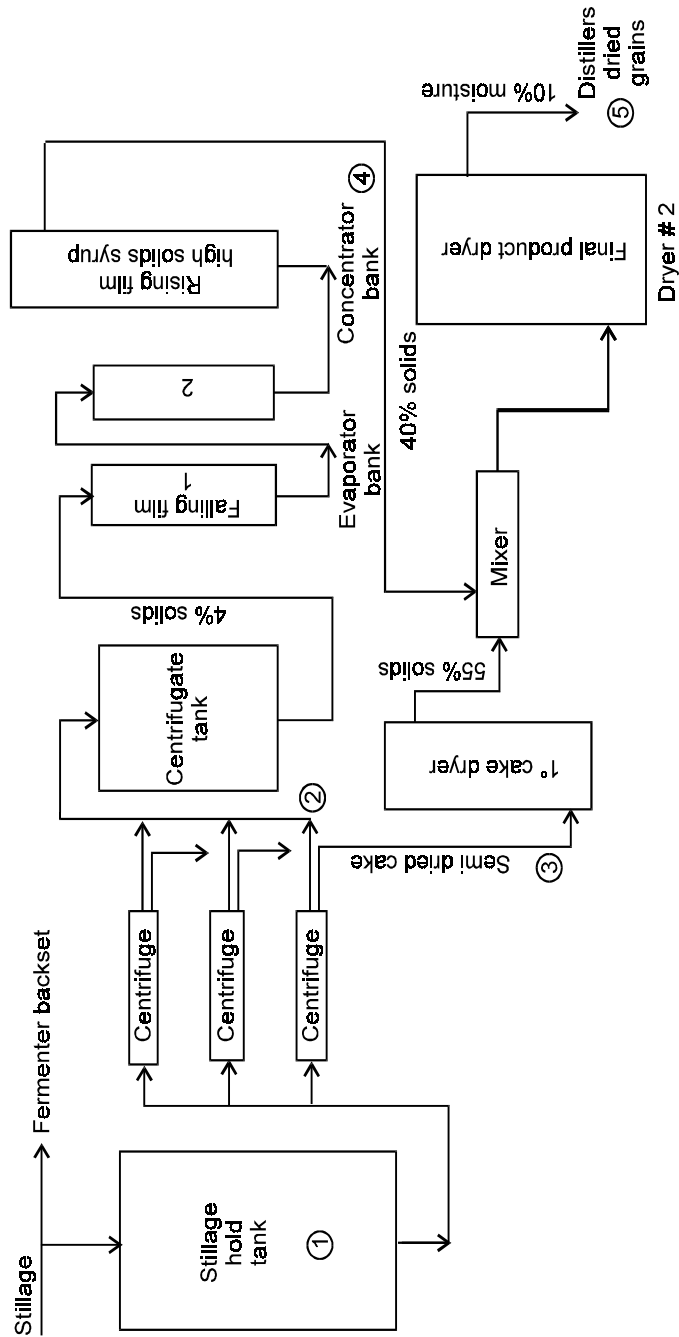
Process control of the drying unit centers on monitoring flow rates, temperatures and pressures at the various stages. Of key importance is the moisture content, which is checked frequently by operators and laboratory staff. While each stage has a certain range of parameters for proper operation, it is essential to ensure that the product does not overdry and become a fire or explosion hazard. Equally, the product must not be stored at too high a moisture level or it may become susceptible to spontaneous combustion in the silos or during transportation.

Figure 9 is a flow sheet of the stillage-handling and drying procedures. Note the quality stations and the process checks needed for good manufacturing practice. Data collected on centrifuge solids ratios are used to determine operating conditions of the centrifuges and to

**Table 5.** Composition of a typical fusel oil from rye spirits distillation.\*

Physical appearance	Clear, slightly yellow liquid	
Gross composition, %	Water	13.87
	Alcohols	86.13
		100.00
Alcohol composition, % of total alcohols		
Methanol (1 carbon atom)		0.14
Ethanol (2 carbon atoms)		11.92
Propyl (3 carbon atoms)		4.82
Isobutyl (4 carbon atoms)		27.21
Amyls (5 carbon atoms)		54.17
Others		1.49
		100.00
Composite analysis, %		
Water		13.87
Methanol		0.12
Ethanol		10.27
Propanol		4.15
Isobutanol		23.44
Amyl alcohols		46.66
Others		1.49
		100.00

\*Unpublished distillery data.



**QUALITY STATION      REQUIRED PHYSICAL/CHEMICAL CHECKS**

- ① Stillage solids, centrifuge solids ratio, efficiency of centrifuge
- ② Centrate solids, pH, acids
- ③ Cake moisture level
- ④ Syrup analysis profile
- ⑤ Distillers dried grains analysis profile, shipments

**Figure 9.** Stillage handling and rye distillers dried grain manufacture (Morrison, 1992).

determine syrup/cake ratios that will affect the recombining of cake with syrup in the second-stage mixer. The syrup analysis profile includes viscosity checks at the fourth and sixth stages of the evaporator system so that product take-off rates and recycling can be programmed.

The DDG analysis profile is a set of specifications for shipment. The profile includes crude protein (Kjeldahl), fat (ether extract), density (loose fall), sieve analysis, color, moisture (oven), ash content (calcium and phosphorus), total dissolved nutrients and total solids. Table 6 contains a typical analysis of DDG (with solubles) and a comparison with DDG produced from Canada grade #1 corn. The darker color usually associated with rye distillers dried grain is the result of high temperature effects on nonfermentable pentoses present in the rye. The pentoses will caramelize as well as combine with protein in the typical browning reaction, causing significant changes in the color of the DDG. Note also the difference in yield of DDG from each grain type. This difference in degree of concentration is the reason for the relatively high protein values present in the corn DDG. The protein content of the grain multiplied by the factor 56, divided by lbs of DDG produced from

one distillers bushel of grain will give the protein content of the DDG.

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**Table 6.** Comparative composition of distillers dried grains with solubles from western Canada rye and no. 1 Canadian corn.

Ingredients	Rye grain #2	Canada grade #1
	Western Canada*	Corn grain**
	%	
Calcium	0.16	0.27
Phosphorus	0.70	0.89
Crude protein (Kjeldahl N x 6.25)	25.30	29.30
Crude fat (ether extract)	2.40	11.30
Ash content	3.80	5.80
Crude fiber	5.20	6.90
Total dissolved nutrients	65.0+	80.0+
Moisture (oven dry)	10.10	10.00
Yield, lb/bushel	23.00	17.00
Color of product	Dark tan	Light tan

\*Unpublished distillery data.

\*\*Preston, 1987.

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