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An investigation of distillery losses.

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AN INVESTIGATION OF DISTILLERY LOSSES

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Ephram I. Brendlen

1945

AN INVESTIGATION OF DISTILLERY LOSSES

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ABSTRACT

This investigation concentrated primarily on an overall plant survey of the Joseph E. Seagram & Sons, Inc. Distillery, Louisville, Kentucky, for the location and evaluation of plant losses.

The results obtained indicated that the major portion of the losses were purely mechanical losses. It was found that 0.835 pounds of bone dry solids per bushel of grain of 12% moisture mashed were lost. The spirits beer still was the chief source of solids loss, making up 84.3% of the total solids lost throughout the distillery.

The secondary related phase of this investigation was the determination of a practical "theoretical" dried grain yield from a bushel of grain mashed. Laboratory fermentations were carried out under plant simulated conditions. A correlation of per cent starch to the fermenter, fermentation efficiency, and per cent dried solids recovered were established. Under the conditions obtained in the determination, the yield of dried grain at 9% moisture was 20.3 pounds per 56 pound bushel of grain with 12% moisture.

INTRODUCTION

In processing grain for alcohol production, the residual materials remaining after the volatile products of fermentation have been removed should contain all the minerals, protein, fat, fiber of the entering grain, residual starch, sugar and dextrins not converted into alcohol, together with such non-volatile products of fermentation as glycerol and succinic acid. However an accounting of the residual material reveals a definite discrepency between matter entering and that leaving.

It has been accepted that "When possible to save the entire production, it is possible to recover from 17 to 18 pounds of distiller's dried grains from each bushel of grain mashed." (1)

To this end, the Joseph E. Seagram & Sons, Inc., instituted a program of research in the Institute of Industrial Research at the University of Louisville. This research has concerned itself with the investigation of the possible sources where losses may occur, the nature of the loss, and where possible an indicated trend of the magnitude of loss versus operating conditions. Also, it became necessary to determine a practical "theoretical" dried grain recovery yield using plant processed materials and simulating plant operations in order that a useful index of dried grain recovery be established.

HISTORICAL

It is estimated that the cost of raw material entering into the production of ethyl alcohol from grain is approximately seventy-five per cent of the total cost of alcohol produced. The complete recovery of residual solids is of paramount importance, for its value is used to reduce considerably the cost of manufacturing a proof-gallon of whiskey or grain neutral spirits. It became apparent to Joseph E. Seagram & Sons, Inc. in 1939 that a program of dried grain research and development was necessary in order that a broader viewpoint be obtained on the material processed from stillage to dried grains. The program resolved itself into (A) a fundamental investigation with the view of producing new types of products from this material, such as vitamin concentrates, recovery of corn oil, glycerine, lactic acid and protein derivatives plus a means of producing a food for human consumption by increasing food value and palatibility and (B) an engineering investigation with the view of solving problems encountered in production and improving present methods from the standpoint of economics in both initial and operating cost, with respect to yield on material processed and to the quality of the final products.

Of the work done in the program of dried grain research, only that pertinent to the problem "An Investigation of Distillery Losses" was reviewed. A paper prepared by Morrison (2) showed that the protein analysis of dried grain produced from bourbon mash remained unchanged during

THE LABORATORY COOKING AND MASHING TO THE FINAL DRYING OPERATION. A REPORT PRESENTED BY DAVIS (3) SEEMED TO VERIFY MORRISON'S FINDINGS. THE REPORT COVERED A STUDY OF THE PROTEIN CONTENT OF THE GRAIN AT VARIOUS STAGES OF THE PROCESS THROUGH THE DISTILLERY AND DRYER HOUSE. DAVIS CONCLUDED THAT NO PROTEIN IS LOST DURING ANY OF THE PLANT PROCESSING STEPS. DAVIS INVESTIGATED FURTHER AND REPORTED ON A PROTEIN INVESTIGATION THAT THE THEORETICAL YIELD IN THE DRYER HOUSE IS 18.7 POUNDS OF DRIED GRAIN RECOVERED PER BUSHEL OF GRAIN MASHED.(4) IN A REPORT ON THE DISTRIBUTION OF MATERIALS IN THE RELAY FOODS AND FEEDS DEPARTMENT, RANN (5) GIVES DATA AND QUANTITIES AT VARIOUS PROCESSING POINTS IN THE RELAY DRYER HOUSE. HE CONCLUDED THAT WHEN MASHING 2/3 CORN SPIRITS AND 1/3 RYE THE THEORETICAL YIELD IN THE DRYER HOUSE SHOULD BE 18.18 POUNDS PER BUSHEL. ATTENTION WAS BROUGHT TO THE ACCOUNTING OF MATERIALS PROCESSED AT THE SEAGRAM LAWRENCEBURG PLANT FOR THE MONTH OF DECEMBER, 1939. DURING THIS MONTH, A REPRESENTATIVE PERIOD, THE PLANT MASHED A SPIRITS BILL CONTAINING 88.5% CORN, 1.5% RYE, AND 10% BARLEY MALT. THE OVERALL PLANT FERMENTATION EFFICIENCY FOR THAT MONTH WAS 92%. WITH THESE CONDITIONS, CAMPBELL(6) REPORTED THE ACTUAL DRIED GRAIN YIELD TO BE 16.91 POUNDS PER BUSHEL. HE ATTEMPTED TO SHOW THAT WITH A 92% EFFICIENCY, 3% OF STARCH TO DRIED GRAIN AND REMAINDER, 5% TO YEAST, GLYCERINE, SUCCINIC ACID, ACETIC ACID, ETC., THE EXPECTED YIELD SHOULD BE IN THE NEIGHBORHOOD OF 19.0 POUNDS PER BUSHEL. IN THE DETERMINATION OF THE DRIED GRAIN

YIELD FROM A PURE SPIRITS BILL, UNGER (7) CALCULATED A THEORETICAL YIELD FROM A RELATIONSHIP BETWEEN PER CENT SOLIDS IN GRAIN BILL, FERMENTATION EFFICIENCY AND PER CENT SOLIDS IN DRIED GRAIN, A YIELD OF 20.25 POUNDS PER BUSHEL. THUS AS COMPARED TO HIS LABORATORY YIELD, UNGER WAS ABLE TO OBTAIN 95% OF CALCULATED THEORETICAL YIELD. FURTHER WORK WAS CARRIED OUT BY UNGER TO DETERMINE THE ACTUAL YIELD AND RECOVERY EFFICIENCY OF SEVERAL TYPES OF MASH BILLS USED AT THE VARIOUS PLANTS. IN HIS REPORT (8) THE CONCLUSION DRAWN WAS THAT THE AVERAGE RECOVERY WAS 94.7% OF THE THEORETICAL AS BASED ON BLANKMEYER'S EQUATION.

THE EQUATION DEVELOPED BY BLANKMEYER (8) DEFINES $Y = \frac{56(A - BC)}{D}$ WHERE Y IS THE YIELD OF DRIED GRAIN IN POUNDS PER BUSHEL, A = (100 - % H_2O) IN GRAIN BEFORE MASHING, B = PER CENT STARCH IN GRAIN, C IS THE FERMENTATION EFFICIENCY OF STARCH TO ALCOHOL, AND D = (100 - % H_2O) IN DRIED GRAIN.

SCHMITT (8) REVISED BLANKMEYER'S EQUATION SO THAT THE ONLY DATA NECESSARY ARE ALCOHOL YIELD, GRAIN MOISTURE AS RECEIVED, AND DRIED GRAIN MOISTURE AS SHIPPED. THE STARCH ANALYSIS AND FERMENTATION EFFICIENCY FACTORS HAVE BEEN ELIMINATED.

$$Y = \frac{56a - a/0.17185}{b}$$

WHERE Y IS THE CALCULATED DRIED GRAIN YIELD, POUNDS PER BUSHEL, a IS THE PER CENT SOLIDS IN GRAIN RECEIVED, A IS THE ANALYTICAL YIELD IN PROOF-GALLONS PER BUSHEL, b IS THE PER CENT SOLIDS IN DRIED GRAIN SHIPPED.

BERESFORD AND CHRISTENSEN (9) REPORTED THAT IN THE

FERMENTATION OF CORN MASH LOSSES FROM 11.8% TO 15.9% WERE FOUND.

CHRISTENSEN (10) REPORTED THAT IN THE GRAIN PROCESS FOR ETHANOL PRODUCTION A LOSS OF 14.5% OF THE DRY MATTER CHARGED TO THE PROCESS WAS FOUND. THIS LOSS IS ATTRIBUTED TO THE NITROGEN-FREE EXTRACT PORTION OF THE GRAIN.

THEORETICAL

The processes in a grain distillery, although mechanically simple, are nevertheless chemically and biologically complex. The flow diagram presented in Figure 1 represents the Louisville Plant of Joseph E. Seagram & Sons, Inc. The processes depicted are typical of a modern grain distillery with major developments in mashing, fermentation, and low temperature distillation.

Briefly, the milled grain is added to the precooker together with water to adjust the water to grain ratio and backset in order to adjust the pH to optimum conversion value. The grain slurry is kept in suspension by mixing pumps. The mixed grain slurry is then pumped at 130°F. from the precooker to a steam jet heater by means of a triplex pump. The slurry is immediately heated in the jet heater to a cooking temperature of 360°F. and held at that temperature during its passage through the "U" tube, approximately 60 seconds. The cooked mash is then instantaneously flashed into a flash chamber operating normally at 20 inches Hg vacuum, where it is cooled by flashing to a temperature approximating 150°F. The cooled mash falls into the cone of the flash chamber where it is met by a cool stream of malt infusion which has been made up in the malt infusion vessel. The converted mash, at approximately 145°F., is pumped through Paraflow water jacketed coolers, cooled to 75 - 80°F., depending on operating conditions, and then into the fermenters.

The vapors flashed from the superheated mash passes

THROUGH AN ENTRAINMENT SEPARATOR (FLASH CHAMBER No.2) AND IS USED TO OPERATE THE LOW VACUUM, 26 INCHES Hg, SPIRITS BEER STILL. THE FERMENTED MASH OR BEER IS SPRAYED INTO THE SPIRITS BEER STILL FIGURE 2, WHICH STRIPS THE BEER OF ITS RELATIVELY VOLATILE COMPONENTS. THE VAPORS FROM THE BEER STILL ARE CONDENSED IN THE DEPHLEGMATOR, THE DISTILLATE BEING PUMPED TO THE PURIFYING COLUMN AND ON TO THE OTHER STILLS FOR FURTHER TREATMENT AND RECTIFICATION. THE SPENT GRAIN OR STILLAGE FROM THE BEER STILL IS PUMPED OVER VIBRATING SCREENS WHERE SEPARATION BETWEEN WHOLE STILLAGE AND THIN STILLAGE IS EFFECTED. THE THICK STILLAGE RETAINED ON THE SCREEN IS CONVEYED TO PRESSES FOR FURTHER DEWATERING AND IS FINALLY CONVEYED TO ROTARY STEAM TUBE DRYERS WHERE IT IS DRIED TO A PRODUCT, DISTILLER'S LIGHT GRAIN, APPROXIMATELY 8-10% MOISTURE. THE THIN STILLAGE IS SENT TO A SIX-BODY QUADRUPLE EFFECT EVAPORATOR WHERE IT IS CONCENTRATED TO A THICK SYRUP OF 25% SOLIDS. THE THICK SYRUP IS THEN DRIED OVER THE REVOLVING DRUMS OF A DRUM DRYER TO PRODUCE DISTILLER'S DRIED SOLUBLES.

A REVIEW OF THE LOUISVILLE PLANT FLOW DIAGRAM, FIGURE 1, INDICATES THAT THE SYSTEM IS A COMPARATIVELY CLOSED ONE. FROM PLANT PRODUCTION RECORDS, IT IS OBSERVED THAT PLANT PRODUCTION OF DRIED GRAIN LAGS APPROXIMATELY 2-21/2 POUNDS PER BUSHEL BEHIND LABORATORY YIELDS. THIS LOSS CAN BE DUE EITHER (A) TO PURELY MECHANICAL LOSS, (B) TO PURELY CHEMICAL LOSS OR (C) TO COMBINATION OF MECHANICAL AND CHEMICAL. MECHANICAL LOSS CAN BE IN THE FORM OF DUST FROM DRIER STACKS, ENTRAINMENT LOSSES IN EVAPORATORS, STILLS AND WHEN EVER VAPOR VELOCITIES

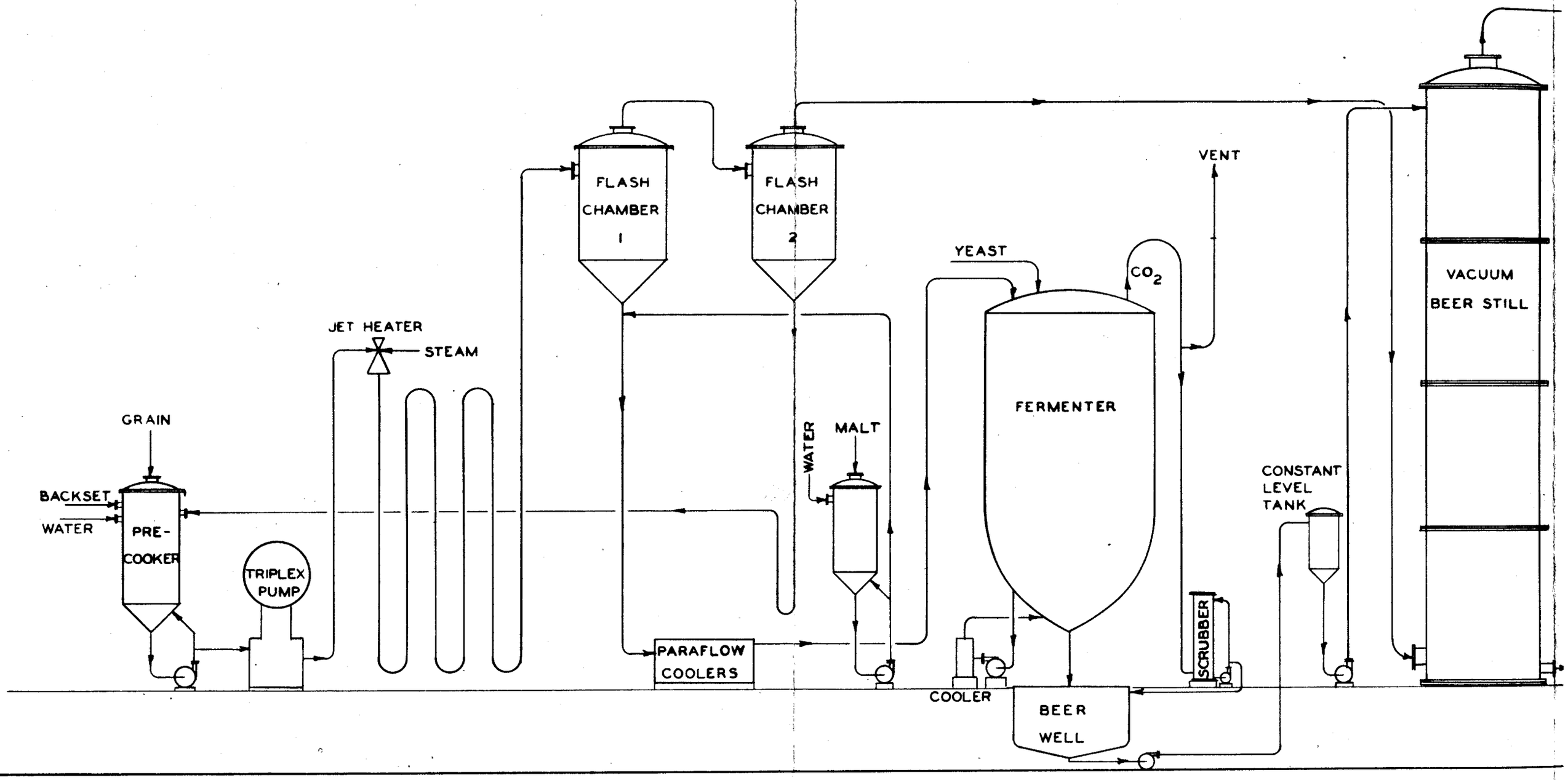


FIGURE 1 - FLOW DIA

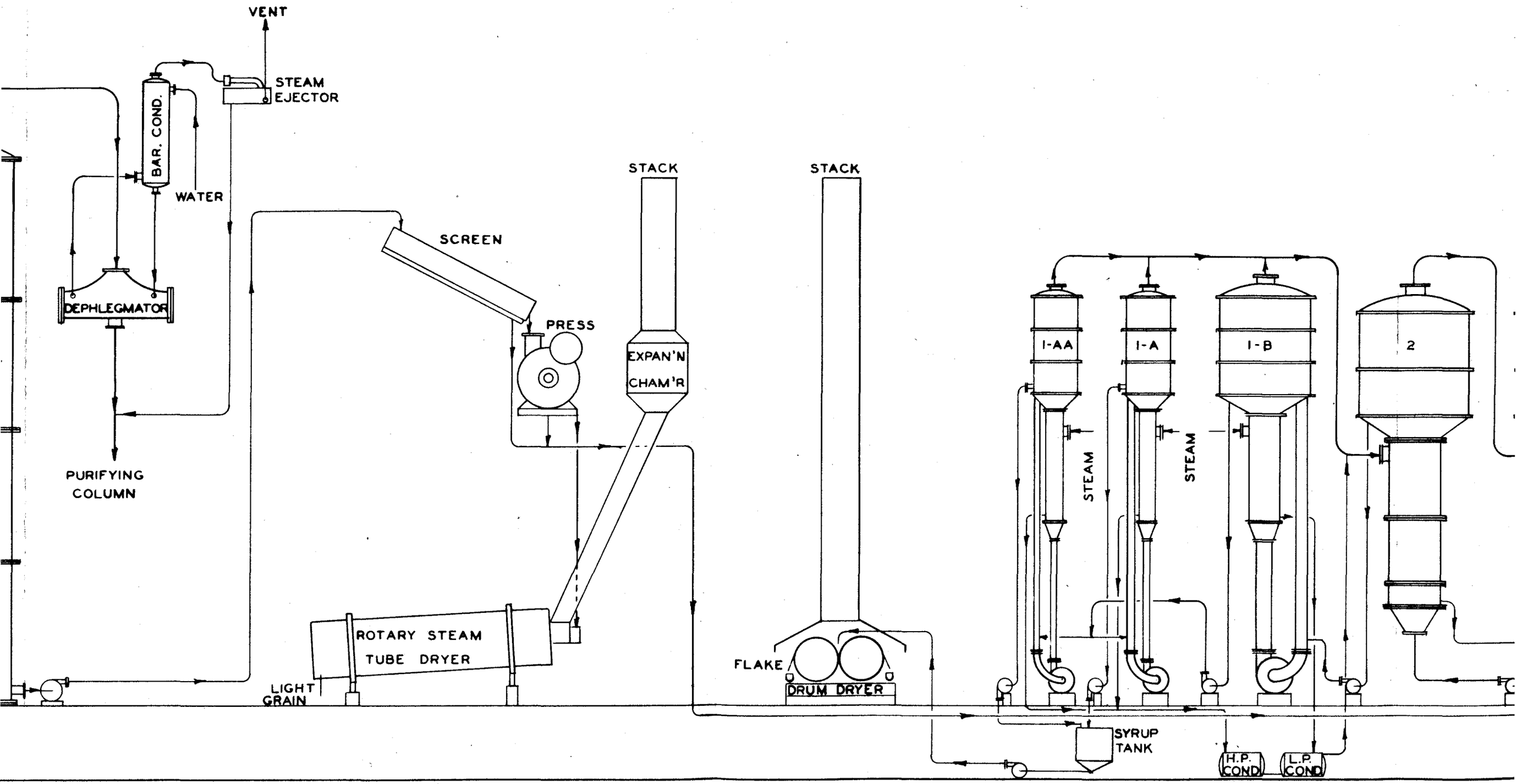
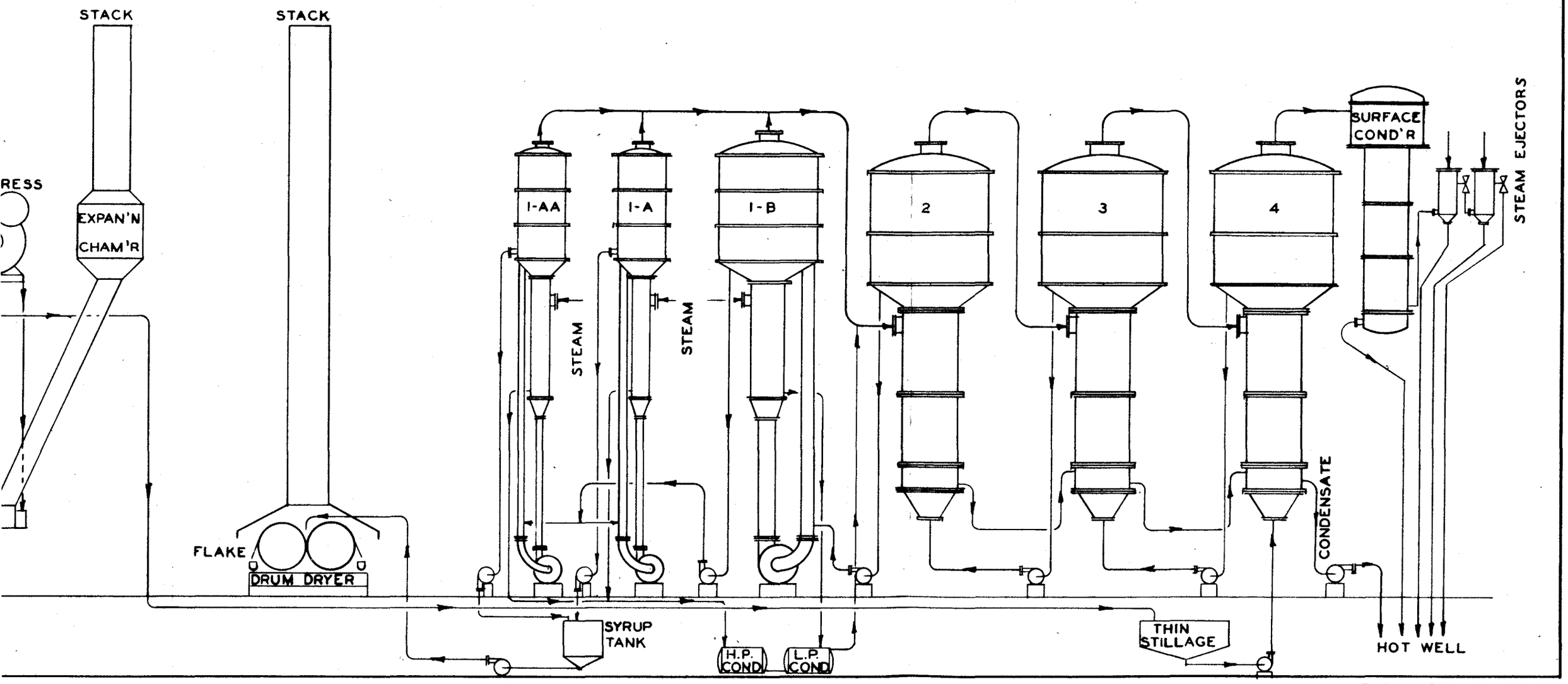


DIAGRAM OF LOUISVILLE PLANT - JOSEPH E. SEAGRAM & SONS, INC.



BRENSILBER E. 12-1-44

PH E. SEAGRAM & SONS, INC.

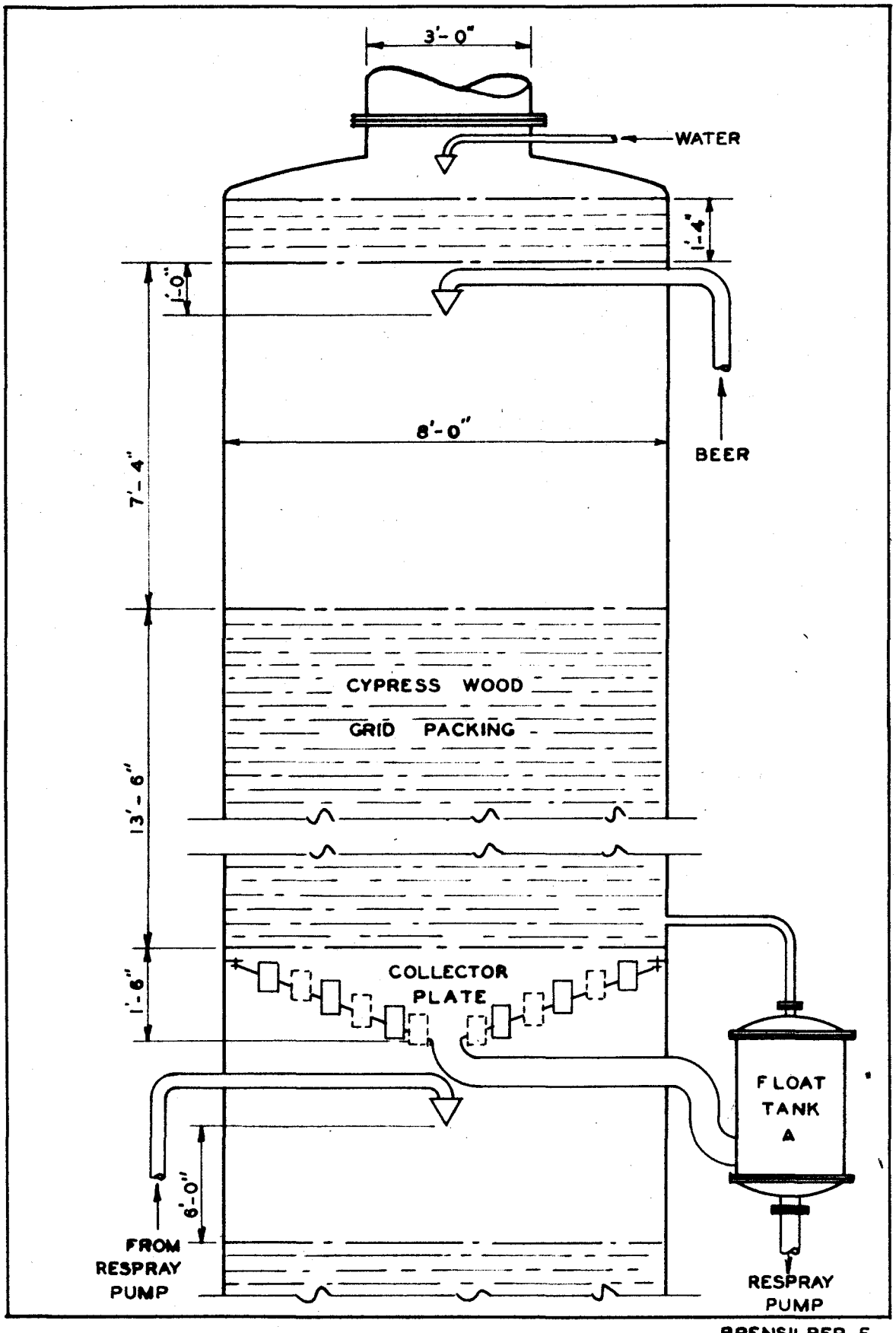


FIGURE 2

TOP SECTION OF SPIRITS BEER STILL

become excessive. Chemical loss may be material decomposition such as might be expected during drying of the grain when nitrogen in the form of proteins may decompose to a volatile nitrogenous product.

A loss is to be considered as such only when material other than as a recoverable product leaves the processing system. Whole stillage going down the sewer during emergency breakdowns or the undercapacity of equipment is not to be considered a "distillery loss".

Ten overall spot test points were selected over the whole system. At each of these points material was suspected of leaving the process either going down the sewer or being vented into the air, in every case the material was not recoverable under the present set-up. The overall spot test sampling points included:

(1) Vapor from spirits beer still to dephlegmator
(2) Vapor from spirits beer still steam ejector
(3) Vapors from rotary steam tube dryer stack
(4) Vapors from drum dryer stack
(5) Liquid from condensate leg of evaporator surface condenser
(6) Liquid from intercondenser No.1 tailpipe
(7) Liquid from intercondenser No.2 tailpipe
(8) Vapors from steam ejector tailpipe
(9) Condensate from No.4 effect
(10) Flashed vapors from flash chamber No.2 to spirits beer still

The low vacuum spirits beer still is of radical construction in comparison with other vacuum beer stills in that the still is divided into several sections and each section is filled with wood packing composed of cypress wood grids measuring 4 inches by 1/4 inch and spaced 1 inch apart. The entering beer feed is sprayed across the top surface of the packing. The beer, in process of stripping, falls to the bottom of one section onto a collector plate, is withdrawn, and is then resprayed in the section below. The vapor coming from the section below is transferred by pipes or risers through the collector plate into the section above. The risers are of sufficient length that the flow of vapor is not impeded by any liquid which may accumulate on the collector plate. Material leaves the spirits beer still either as volatile material composed of ethyl alcohol and other volatile by-products of fermentation at the top of the still or as stillage at the base of the beer still. The stillage is pumped to the Dryer House and recovered. Because of the inherent construction of the spirits beer still, the respraying of liquid across the packing, and excessive vapor velocities, the vapor leaving the top of the still to the dephlegmator was suspected of containing undistilled material. The distillate from the dephlegmator together with the entrained solids is pumped to the purifying column then rectified in the rectifying column. The purifying column separates the ethyl alcohol from the "heads". The dilute solution of

ETHYL ALCOHOL PLUS THE ENTRAINED SOLIDS CARRIED OVER FROM THE DEPHLEGMATOR IS REMOVED FROM THE BASE OF THE PURIFYING COLUMN AND PUMPED TO THE RECTIFYING COLUMN. THE ENTRAINED SOLIDS, BEING NON-VOLATILE, LEAVE TOGETHER WITH THE DISCHARGED WATER AT THE BASE OF THE RECTIFYING COLUMN, WHICH IN TURN, EMPTIES INTO THE SEWER.

NON-CONDENSED VAPORS LEAVING THE DEPHLEGMATOR ARE DRAWN IN SERIES THROUGH A BAROMETRIC CONDENSER, TWO STAGE STEAM EJECTOR AND OUT INTO THE ATMOSPHERE. IT WAS BELIEVED THAT ANY ENTRAINED SOLIDS LEAVING THE DEPHLEGMATOR WITH THE NON-CONDENSED VAPORS WOULD BE WASHED BACK INTO THE SYSTEM AS IT PASSED THROUGH THE BAROMETRIC CONDENSER AND THE TORTUOUS PATH THROUGH THE STEAM EJECTORS AND SURFACE CONDENSERS. HOWEVER, A SAMPLING TUBE WAS INSERTED IN THE VENT LEAVING THE LAST STAGE STEAM EJECTOR IN ORDER TO VERIFY THIS BELIEF.

IT WAS DECIDED TO INVESTIGATE THE FLASHED VAPOR FROM THE FLASH CHAMBER FOR (A) THE EFFICIENCY OF THE ENTRAINMENT CHAMBER WAS UNKNOWN (B) WHEN THE LOW VACUUM SPIRITS BEER STILL WAS "DOWN", THE FLASHED VAPORS WERE SENT TO A BAROMETRIC CONDENSER WHICH EMPTIED INTO THE SEWER (C) THE POSSIBILITY OF CHEMICAL DECOMPOSITION DURING HIGH TEMPERATURE (360°F.) COOKING AND IMMEDIATE FLASHING EXISTED.

IN THE PAST, THE CONTENTION HAS BEEN THAT THE GREATEST SOURCES OF, IF NOT ALL, DISTILLERY LOSSES WERE IN THE DRYER HOUSE. FIGURE 1 INDICATES THAT ONLY SEVEN POSSIBLE EXITS WHERE SOLIDS OR VOLATILE MATERIALS NOT BEING RECOVERED

MAY ESCAPE THE SYSTEM IN THE DRYER HOUSE. THEY ARE (1) ROTARY STEAM TUBE DRYER STACK, (2) DRUM DRYER STACK, (3) CONDENSATE LEG FROM SURFACE CONDENSER, (4) INTERCONDENSER No.1 TAILPIPE, (5) INTERCONDENSER No.2 TAILPIPE, (6) STEAM EJECTOR TAILPIPE, AND (7) EFFECT No.4 CONDENSATE PUMP. THE FEED TO THE ROTARY STEAM TUBE DRYER IS WET PRESS CAKE CONTAINING APPROXIMATELY 70% MOISTURE. THE ROTARY DRYER IS HEATED BY TUBES WHICH EXTEND THE FULL LENGTH OF THE DRYER. AT THE DISCHARGE END THE TUBES ARE CONNECTED TO A HEADER RING AND TRUNNION, THROUGH WHICH THE STEAM ENTERS AND THE CONDENSATE IS REMOVED. THE GRAIN IS CARRIED THROUGH THE DRYER BY THE ROTATING ACTION AND THE INCLINATION OF THE UNIT. THE VAPORS, NEARLY SATURATED AIR, ARE REMOVED BY NATURAL OR INDUCED DRAFT THROUGH A STACK AT THE FEED END OF THE DRYER. THE DRIED LIGHT GRAIN CONTAINING 8-10% MOISTURE IS RELATIVELY LIGHT IN WEIGHT. THIS PROPERTY MAKES IT NECESSARY TO PREVENT THE DRAFT THROUGH THE DRYER FROM BECOMING EXCESSIVE IN ORDER TO AVOID LOSSES OUT THROUGH THE STACK. THE ROTARY DRYERS ARE EQUIPPED WITH EXPANSION CHAMBERS WHICH ORIGINALLY WERE INSTALLED TO REDUCE THE VAPOR VELOCITIES OF THE LEAVING VAPOR PLUS ENTRAINED SOLIDS SO THAT THE GRAIN PARTICLES WOULD DROP OUT AND RETURN TO THE DRYER. THE CONSTRUCTION OF THE EXPANSION CHAMBERS ARE SUCH THAT THE SIDES SOON BECOME CAKED WITH THE DEPOSITED GRAIN AND ULTIMATELY BUILT UP TO AN EXTENT THAT THE EFFECTIVE DIAMETER OF THE EXPANSION CHAMBER WAS ALMOST EQUAL TO THE DIAMETER OF THE STACK ITSELF. IT WAS ALSO BELIEVED THAT SOME PROTEIN

BREAKDOWN OR DECOMPOSITION MAY TAKE PLACE AS THE GRAIN CAME IN CONTACT WITH THE STEAM HEATED TUBES AT 330°F.

THE DRUM DRYER CONSISTS OF TWO LARGE CAST IRON DRUMS WITH A CLEARANCE OF ABOUT 0.03 INCH BETWEEN THEM. THE DRUMS ROTATE TOWARDS EACH OTHER AT A SPEED OF 2 TO 4 REVOLUTIONS PER MINUTE. THE CONCENTRATED SYRUP PRODUCED IN THE EVAPORATORS IS FED INTO THE "V" TROUGH FORMED BY THE DRUMS. THE HOLLOW DRUMS RECEIVE STEAM THROUGH TRUNNIONS AT ONE END, AND THE CONDENSATE FORMED IS REMOVED THROUGH THE SAME TRUNNION. AS THE HOT DRUMS ROTATE, THE THIN FILM OF SYRUP IS DRIED AND REMOVED BY A STEEL KNIFE. THE CONTINUOUS SHEET SO FORMED IS FURTHER COOLED BY A BLAST OF AIR BLOWN ACROSS THE SURFACE OF THE SHEET; THEN THE SHEET IS BROKEN UP BY A CUT FLIGHT CONVEYOR. THE VAPORS FROM THE DRUM DRYER ARE REMOVED BY NATURAL DRAFT THROUGH THE STACK. THE CONSTANT BLAST OF AIR ACROSS THE SHEET AND THE SUBSEQUENT PULVERIZATION INDICATED A POSSIBLE SOURCE OF SOLIDS ENTRAINMENT WAS PRESENT. SINCE THE EVAPORATOR SYRUP WAS MORE INTIMATELY IN CONTACT WITH THE HEATED SURFACE OF 330°F. THAN WAS THE DRIED GRAIN IN THE ROTARY STEAM TUBE DRYER, GREATER BREAKDOWN AND DECOMPOSITION WAS EXPECTED.

THE SIX-BODY QUADRUPLE EFFECT EVAPORATOR OPERATED AT THE LOUISVILLE PLANT CONSISTS OF TWO BODIES, 1A, 1AA, WHICH RECEIVE 20 POUNDS PER SQUARE INCH GAGE STEAM AS THEIR HEATING MEDIUM, ONE BODY, 1B WHICH IS HEATED WITH 10 POUND STEAM AND THREE REMAINING BODIES OPERATING UNDER SUCCESSIVELY LOWER PRESSURES AND RECEIVING VAPOR FROM THE PRECEDING EFFECT AS THE

HEATING MEDIUM. THE EVAPORATOR IS MADE UP OF A VERTICAL BODY ENCLOSING A TUBE BUNDLE 15 TO 25 FEET LONG, SURMOUNTED BY A VAPOR SPACE. IMMEDIATELY ABOVE THE TUBE BUNDLE IS A DEFLECTOR PLATE TO REDUCE ENTRAINMENT AS THE BOILING LIQUID RISES FROM THE TUBES. EACH EVAPORATOR IS PROVIDED WITH AN EXTERNAL DOWN-TAKE SO ARRANGED THAT EACH BODY MAY BE OPERATED WITH A DEFINITE LIQUID LEVEL OR AS A SINGLE PASS EVAPORATOR. EFFECTS No.2, No.3, AND No.4 HAVE NATURAL CIRCULATION OF LIQUID THROUGH THE TUBES. THE THREE BODIES OF THE FIRST EFFECT HAVE PUMPS WHICH PROVIDE FORCED CIRCULATION THROUGH THE TUBES. THE FINISHED PRODUCT, CONSISTING OF A SYRUP CONTAINING 25% SOLIDS, IS RE-MOVED FROM EITHER 1A OR 1AA. THE CONDENSATE FROM THE THREE BODIES OF THE FIRST EFFECT IS RETURNED AFTER FLASHING TO THE STEAM CHEST OF No.2 EFFECT, TO THE POWER HOUSE. THE CONDENSATE FROM EFFECT No.2 IS SENT TO THE STEAM CHEST OF No.3 WHERE PART FLASHES, THE CONDENSATE FROM No.3 IS SENT TO EFFECT No.4 WHERE PART FLASHES, AND THE ACCUMULATIVE CONDENSATE IN EFFECT No.4 IS PUMPED FROM THE STEAM CHEST OF No.4 AND SENT DOWN THE SEWER. THE COMPOUNDED VAPORS FROM THE FIRST THREE BODIES OF THE FIRST EFFECT ARE SENT TO THE STEAM CHEST OF EFFECT No.2; THE VAPORS FROM THE VAPOR SPACE OF No.2 ARE SENT TO Effect No.3 STEAM CHEST; THE VAPORS COMING FROM EFFECT No.4 ARE SENT DIRECTLY TO A SURFACE CONDENSER. THE NON-CONDENSIBLES ARE DRAWN IN SERIES THROUGH INTERCONDENSER No.1, STEAM EJECTOR STAGE No.1, INTER-CONDENSER No.2, AND FINALLY STEAM EJECTOR STAGE No.2. THE DISCHARGE FROM THE SURFACE CONDENSER, INTERCONDENSERS No.2,No.3,

AND SECOND STAGE STEAM EJECTOR EMPTY INTO THE SEWER.

WHEN CLEAN, THE EVAPORATOR IS OPERATED WITH A BACKWARD FEED, THAT IS, 4-3-2-1B-1A-1AA. WITH THIS CONDITION OF OPERATION, THE FEED, CONSISTING OF THIN STILLAGE ENTERS THE FOURTH EFFECT WHICH OPERATES UNDER THE GREATEST VACUUM. THE TENDENCY TOWARDS ENTRAINMENT IS GREATER IN THIS LAST EFFECT BECAUSE OF THE LESSER CONSISTENCY OF THE LIQUID AND GREATER VAPOR VELOCITY RESULTING FROM DECREASED PRESSURE. AFTER A PERIOD OF OPERATION, EFFECT No.2 BECOMES FOULED BECAUSE OF THE CONCENTRATION OF SOLIDS AND RELATIVELY SLOW CIRCULATION OF THE LIQUID THROUGH THE TUBES BECAUSE OF THE INCREASE OF VISCOSITY AS A RESULT OF INCREASE IN SOLIDS CONTENT. AT THIS STAGE, THE FEED PATTERN BECOMES A MIXED FEED, 2-3-4-1B-1A-1AA.

EFFECTS No.1AA, No.1A, AND No.1B HAVE PUMPS WHICH PROVIDE FORCED CIRCULATION OF THE LIQUOR THROUGH THE TUBES. IT IS BELIEVED THAT THE HIGH VELOCITY OF THE LIQUOR AS IT HITS AGAINST THE DEFLECTOR PLATE RESULTS IN THE FORMATION OF A MIST OF VAPOR PLUS ENTRAINED LIQUID DROPLETS. THESE DROPLETS PASS ON INTO THE STEAM CHESTS OF THE FOLLOWING EFFECTS AND ULTIMATELY ARE REMOVED BY THE CONDENSATE PUMP.

THE SECOND PHASE OF THIS RESEARCH CONCERNED ITSELF WITH THE DETERMINATION OF A PRACTICAL "THEORETICAL" DRIED GRAIN YIELD. THE RAW MATERIALS WHICH ENTER A DISTILLERY IN THE FORM OF SUCH GRAINS AS CORN, WHEAT, RYE, MILO MAIZE, RYE MALT, BARLEY MALT OR OTHER STARCHY SUBSTRATES ARE IN THEMSELVES VARIABLE, NOT ONLY FROM ONE MATERIAL TO ANOTHER BUT EVEN IN THE

single material. The structure of the grain is chemically complex. To convert the starch contained in the grain to fermentable sugars, the grain, in the presence of water, is heated to sufficient temperatures in order to burst the starch cell and bring the starch into a colloidal suspension. The starch suspension is converted through the enzymatic action of diastase, present in barley malt, into a fermentable sugar. It is known that this initial conversion does not exceed 70-80%. With the addition of yeast, a secondary conversion with the malt enzyme still present, resumes conversion of the unconverted starch. Upon this secondary conversion depends largely the alcohol yield. The hydrolysis of starch yields chiefly glucose together with some unfermentable sugars. This reaction may be represented by the following equation:

$$C_6H_{10}O_5 + H_2O \qquad C_6H_{12}O_6$$

Pasteur had shown that the weight relationships set forth by Gay-Lussac on the fermentation of glucose to ethanol was in error. Gay-Lussac believed the reaction could be represented by

$$C_6H_{12}O_6 \qquad 2C_2H_5OH + 2CO_2$$

However, in actual plant practice, ethyl alcohol and carbon dioxide are not the only products produced during the fermentation of the hydrolyzed grain. The by-products of fermentation produced at the expense of the grain include fusel oil, which is produced in appreciable quantities, acetaldehyde, ethyl acetate, and glycerine, which are produced only in traces.

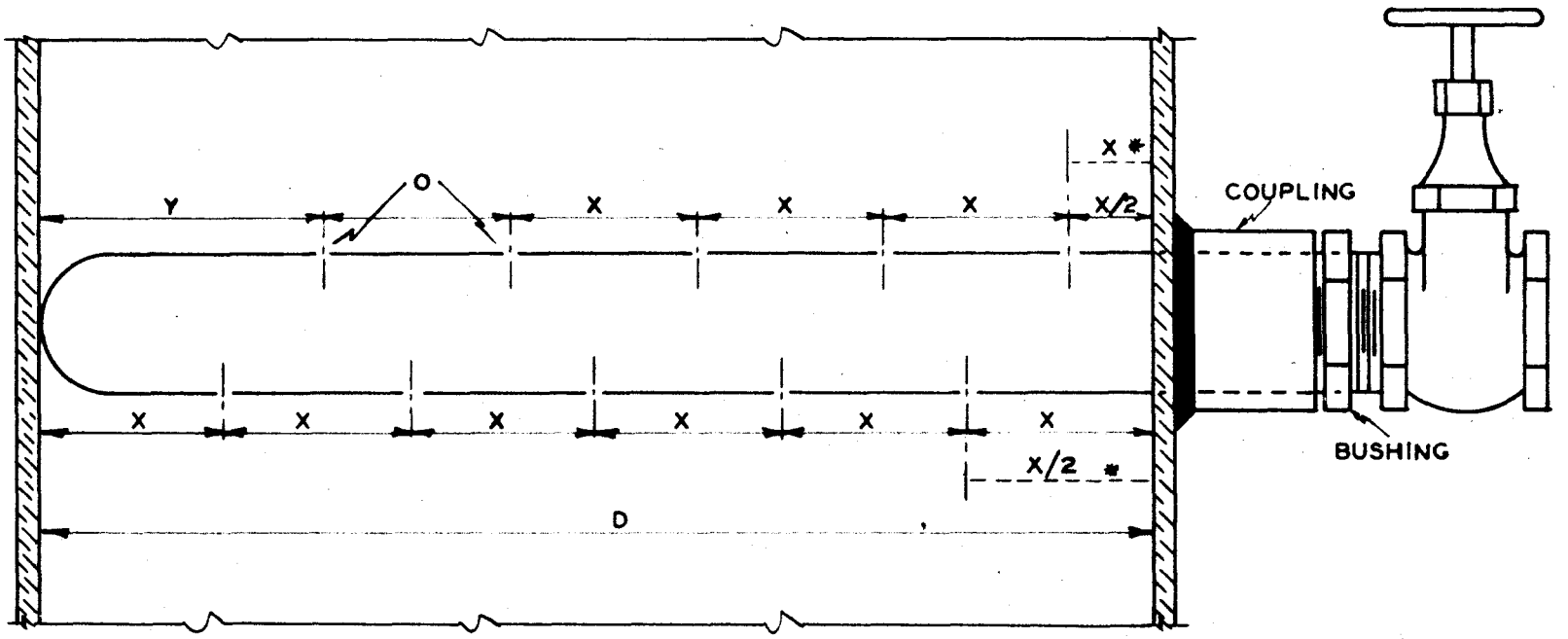
Fusel oil and succinic acid are both formed by the yeast during the breakdown of the amino acids from the protein in the grain. Some furfural is also produced.

Since the products obtained from a bushel of grain are dependent upon the chemical composition of the grain, the degree of conversion of the cooked grain, and the mechanisms of fermentation, it is difficult to present a theoretical relationship between entering and leaving materials. However, a relationship between the percent starch entering the fermenter, fermentation efficiency, and per cent solids recovered could predict the practical yield or recovery in the Foods and Feeds Department or Dryer House. The per cent starch is an index to the ethanol which may be produced. The fermentation efficiency defined as the actual alcohol produced to the ethyl alcohol which may have been produced theoretically, is a measure of the combined effect of the degree of conversion, time of fermentation, degree of fermentation, and factors affecting fermentation. The per cent solids recovered is defined as the solids recovered after the volatile products have been removed by distillation to the solids entering the fermenter. The solids remaining should contain all the proteins, minerals, fat, fiber, unconverted starch, dextrins, and sugar, plus the yeast, glycerol, and acids not removed during distillation.

EXPERIMENTAL

At each of the selected sampling points a perforated sampling tube was installed to obtain for each run a single sample which would be representative of the entire stream of gases or liquids flowing past the particular point in the system. Ten individual sampling tubes were prepared. Each tube varied in either the size of openings, number of perforations, distance between centers of perforations, overall length, or pipe diameter. Figure 3 describes in detail the dimensions of each tube. In general, the sampling tube, made of brass pipe, was closed completely at one end and valved off at the other. A number of holes, 1/8 inch or 3/32 inch in diameter, were drilled a definite distance apart in opposite sides of the pipe, each hole midway between two on the opposite side, as shown in the Figure 3. The sampling tube was securely fastened and supported at each of the sampling points by a combination bushing and coupling arrangement, as shown in Figure 3, which also seals the sampling tube in the system. In each installation, the sampling tube was placed diametrically across the stream, with the perforations in line with the flow of materials. At the valved-off end of the tube, suitable reducers were attached to accomodate 1/4 inch diameter rubber pressure tubing.

Glass wool filter traps were prepared to filter any entrained solids or dust from the vapor streams. The filter traps were fabricated from 20 x 150 mm pyrex test tubes. A hole was made at the bottom of the tube and a short length of 6 mm glass tubing was then fused to the bottom. The filter



SAMPLING POINT	D-INCHES	PIPE SIZE	X-INCHES	Y-INCHES	O-INCH
SPIRITS BEER STILL	36	1	4	6	1/8
STILL EJECTOR VENT	4	1	1	1 1/2	3/32
ROTARY STEAM TUBE DRYER STACK	30	1	4 *	6 *	1/8
DRUM DRYER STACK	32	1	4	6	1/8
COOKER FLASH STEAM LINE	16	1	2	4	1/8
EVAPORATOR SURFACE CONDENSER	4	1/2	1/2	3/4	3/32
INTERCONDENSER TAILPIPE 1	3	1/2	1/2	3/4	3/32
INTERCONDENSER TAILPIPE 2	3	1/2	1/2	3/4	3/32
STEAM EJECTOR TAILPIPE	2 1/2	1/2	1/2	3/4	3/32
EVAPORATOR CONDENSATE PUMP	3	1/4	1/2	3/4	3/32

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FIGURE 3 - SAMPLING TUBE DETAIL

trap was packed approximately two-thirds full with Fiberglas No. 800 glass wool. Protecting tubes were prepared for each of the filter traps in order to permit the necessary handling and preparations for each sampling run. The protecting tubes were fabricated from 25 x 200 mm test tubes from which the bottoms were removed. The complete assembly is shown in Figure 4.

A volume measuring apparatus was fabricated from six inch standard iron pipe primarily to prevent breakage during sampling and transit from one sampling point to another; secondly, to permit relatively easy and few operations in the measuring of the gas volume. The volume measuring apparatus consisted of a 16 inch length of six inch pipe closed at the bottom and topped by an eight inch cone. The cone was fitted with three openings to accomodate a thermometer, pressure connection to a manometer, and receiving inlet for the entering gas. The body of the six inch pipe was fitted with a 13 inch length of standard $\frac{1}{2}$ inch gage glass and cocks. The bottom of the closed pipe, supported one foot from the floor by pipe legs, was fitted with a $\frac{1}{2}$ inch diameter nipple and quick-opening valve. A tee connection from the quick-opening valve carried two 1/4 inch diameter valved nipples. One of the 1/4 inch nipples permitted water to fill up the apparatus, the other 1/4 inch nipple allowed water from the volume measuring apparatus to be removed to provide volume for the entering gas. The apparatus was calibrated to displace a volume of eight liters

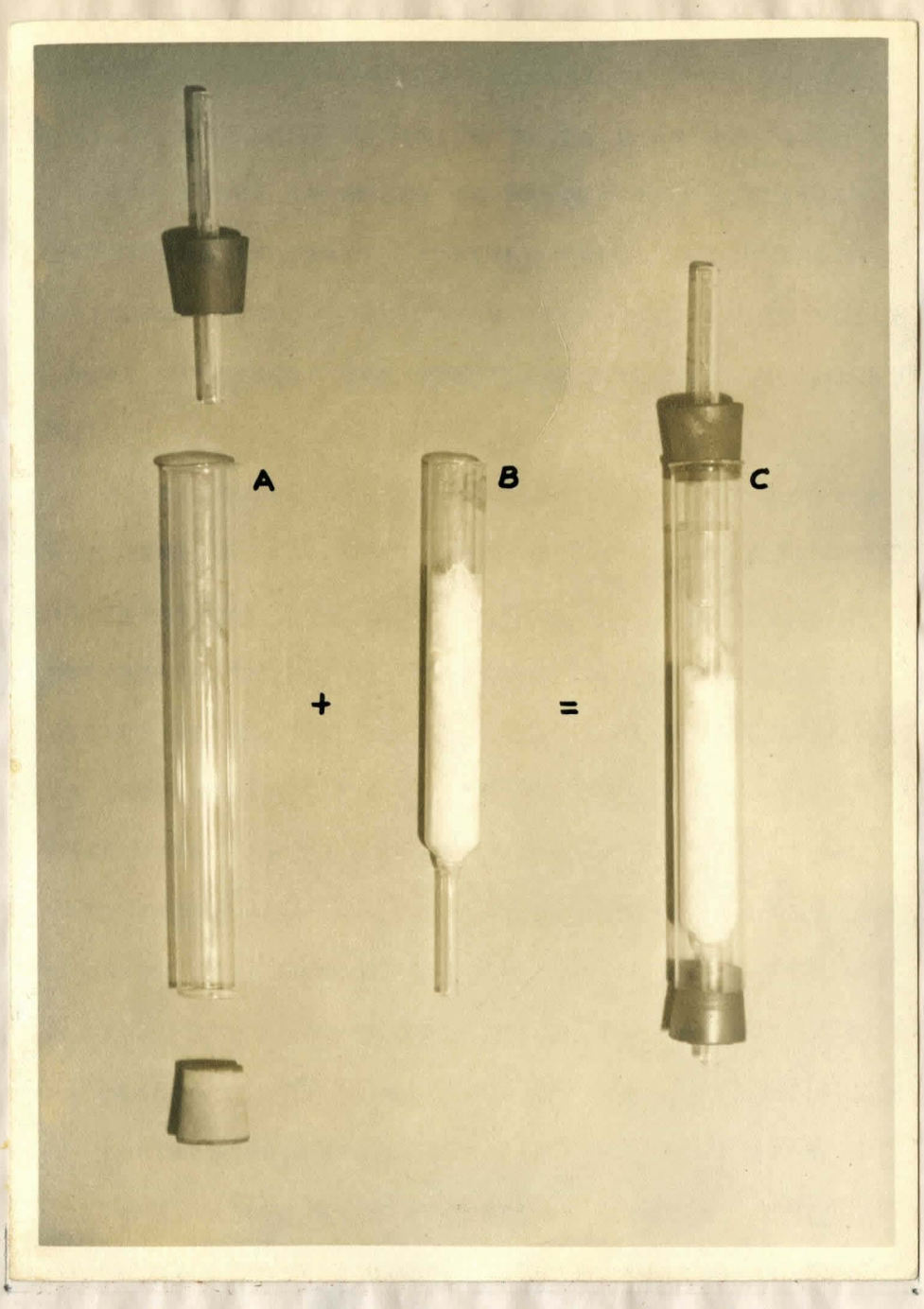

FIGURE 4 GLASS WOOL FILTER TRAP ASSEMBLY

A. PROTECTING TUBE
B. GLASS WOOL FILTER TRAP
C. COMPLETE GLASS WOOL FILTER TRAP ASSEMBLY

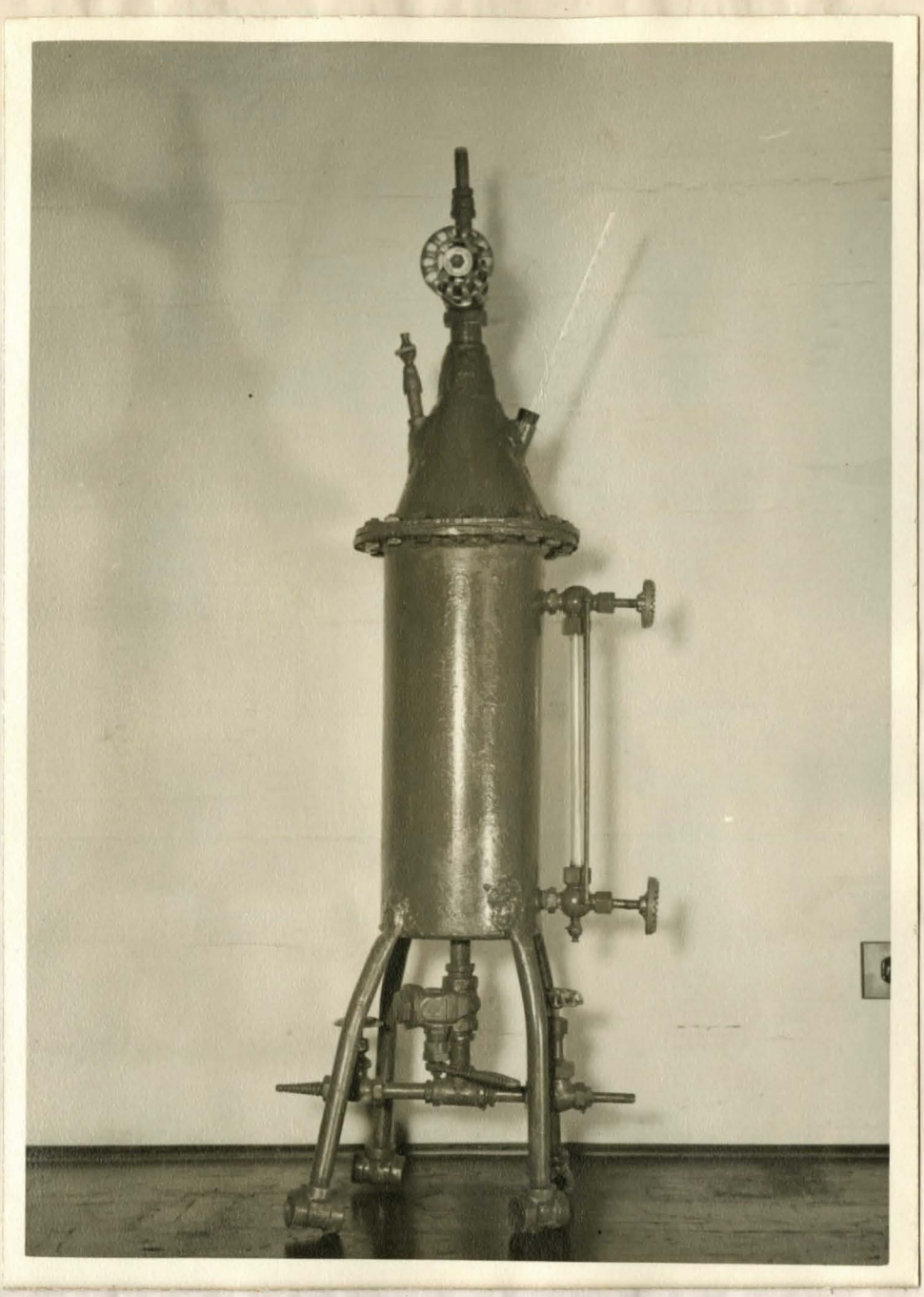

FIGURE 5 GAS MEASURING APPARATUS

of water. Figure 3 shows the complete apparatus.

Liquid samples were removed from the discharge of the condensate pump, surface condenser leg discharging to the hot well, tailpipes from intercondensers No.1 and No.2 and water supply to intercondensers. All the samples but the last had to be removed by suction. The vapors from the steam ejector tailpipe were condensed and collected. Each liquid sample measured approximately one liter. Sampling procedure for the liquids consisted (a) opening valve to sampling tube and allowing air to be drawn in thus flushing and clearing the sampling tube and perforations (b) removal of first liter of sample and discarding (c) collecting one liter of liquid sample.

It was realized that because of the organic nature of the material contained in the liquid samples and of the tendency to decompose at elevated temperatures, a standard drying method was necessary. Three methods of drying were tried; (1) natural draft electric oven at 105°C. (2) vacuum drying oven at 50°C., and (3) forced convection oven at 105°C. Of the three, the forced convection oven at 105°C. with a drying period of three hours, (approximately two hours for evaporation and one for drying) for a 50 ml sample, yielded consistent comparable results without danger of bumping or splattering and in the minimum drying time.

Liquid samples from the two intercondensers and the condensed vapor from the steam ejector indicated that there was no detectable increase in solids content of the water

ENTERING THE INTERCONDENSERS AND NO SOLIDS CONTENT IN THE CONDENSED STEAM. PH MEASUREMENTS MADE ON THE WATER TO THE INTERCONDENSERS AND FROM THE INTERCONDENSERS WERE:

(A) WATER TO INTERCONDENSERS ---------- 7.40

(B) WATER FROM INTERCONDENSER NO.1 ---------- 7.65

(C) WATER FROM INTERCONDENSER NO.2 ---------- 7.50

THE LIQUID SAMPLES FROM THE CONDENSATE PUMP AND SURFACE CONDENSER LEG WERE ANALYZED FOR SOLIDS CONTENT, PH, AND TITRATABLE ACIDITY EXPRESSED AS MILLIGRAMS OF ACETIC ACID. THE RESULTS ARE SHOWN IN TABLE I.

TABLE 1

EVAPORATOR SOLIDS LOSSES

Run No.	Operating Conditions: Feed gals/hr	Method of Feed	Vacuum in No.4 Effect inches Hg	Condensate Pump: Solids gm/50ml	pH	Acids	Surface Condenser: Solids gm/50ml	pH	Acids
1	8300	Backward	26	0.0073	3.75	37.85	0.0189	3.85	32.82
2	8000	Mixed	23	0.0073	3.85	24.15	0.0375	3.80	39.01
3	10600	Backward	26.5	0.0113	3.80	20.43	0.0381	3.85	36.53
4	9000	Backward	26	0.0160	3.65		0.0329	3.65	

Backward feed indicates 4-3-2-1B-1A-1AA

Mixed feed indicates 2-3-4-1B-1A-1AA

Acids are tabulated as titratable acidity, milligrams of acetic acid per 100 ml

The vapors from the rotary steam tube dryer were removed by the perforated sampler described in Figure 3. The vapor sample was passed in series first through a weighed glass wool filter to trap any entrained dust, then bubbled by means of a No.2 gooch crucible through a known volume of distilled water to absorb any volatile constituents, and finally to the volume measuring vessel. The arrangement of the apparatus is shown in Figure 6. The procedure for sampling the vapors consisted of (a) flushing the perforated sampling tube with water under pressure (b) withdrawal of approximately 8 liters of vapor from the stack in order to prepare the system for representative sampling (c) removal of a total of 24 liters for each sampling run. The rate of sampling was one liter per minute. Pressure and temperature measurements were made on the volume measuring apparatus. The vapor velocities of the rotary steam tube dryer vapors were made with an anemometer and temperatures of the vapors were determined with the aid of a copper-constantan thermocouple.

On each of the sampling runs, determinations were made for entrained solids (in the form of dust), protein, fat, PH_3, and titratable acidity of the absorbent. The protein determinations were made following the modified A.O.A.C. method used at the Seagran-Calvert laboratories. The reagents are:

100gms P_2O_5 per 200 ml conc. H_2SO_4

Mixture of 5 gms red HgO and 100 gms K_2SO_4

5% K_2S solution

45% NaOH solution

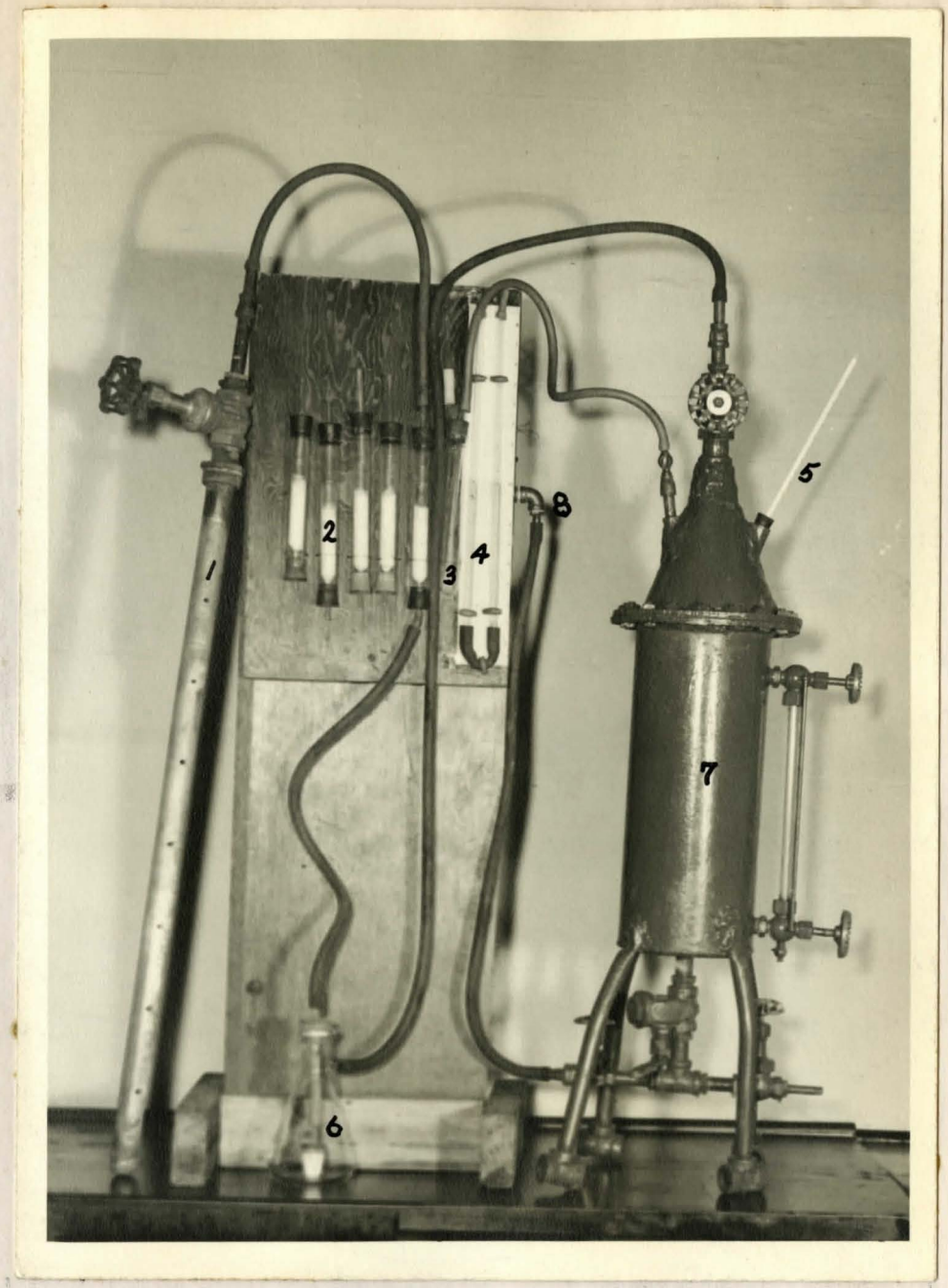

FIGURE 6 ROTARY STEAM TUBE DRYER STACK SAMPLING APPARATUS

(1) PERFORATED SAMPLING TUBE (2) FILTER (3) WATER TRAP TO MANOMETER (4) MANOMETER (5) THERMOMETER (6) ABSORBER (7) GAS MEASURING APPARATUS (8) WATER ASPIRATOR

0.1N H_2SO_4

0.1N NaOH

THE PROCEDURE FOLLOWED WAS:

1. ADD 100ML OF ABSORBENT SAMPLE
2. ADD 10ML OF 30% H_2O_2
3. AGITATE AND ADD CAUTIOUSLY 10ML OF P_2O_5 REAGENT
4. ROTATE FLASK IN DIGESTION APPARATUS UNTIL INITIAL REACTION IS COMPLETE
5. ADD 1GM OF MgO-K_2SO_4 REAGENT
6. DIGEST FOR 10 MINUTES
7. COOL, ADD 200ML OF DISTILLED WATER
8. ADD 5ML OF K_2S REAGENT AND AGITATE
9. NEUTRALIZE AND MAKE ALKALINE WITH 45% NaOH SOLUTION
10. DISTILL 100ML INTO RECEIVING FLASK CONTAINING 20ML OF 0.1N H_2SO_4 PLUS 100ML DISTILLED WATER
11. TITRATE WITH 0.1N NaOH USING METHYL RED AS INDICATOR
12. USE FACTOR OF 6.25 TO MULTIPLY MILLIGRAM-EQUIVALENTS OF ACID USED IN ABSORPTION OF NH_3 FROM SAMPLE TO OBTAIN PROTEIN FROM DRIED GRAIN

FAT DETERMINATIONS WERE MADE USING PETROLEUM ETHER AS THE EXTRACTION SOLVENT. A 100ML SAMPLE OF THE ABSORBENT WAS MIXED WITH AN EQUAL VOLUME OF PETROLEUM ETHER AND AGITATED IN A SEPARATORY FUNNEL. ABOUT 70% OF THE PETROLEUM ETHER LAYER WAS REMOVED IN ORDER TO INSURE A GOOD CUT AND EVAPORATED OVER A STEAM BATH. THE RESIDUE REMAINING IN THE EVAPORATING DISH WAS DRIED AND WEIGHED.

TITRATABLE ACID DETERMINATIONS WERE MADE ON 100ML ALIQUOTS OF THE ABSORBENT WITH 0.1N NaOH SOLUTION.

THE ANALYSIS OF THE ABSORBENTS INDICATED NO TRACE OF PROTEIN AND FAT. ALTHOUGH THE AVERAGE PH OF THE ABSORBENTS WAS 5.0, NO TITRATABLE ACID WAS DETECTED.

ENTRAINED SOLIDS IN THE STACK VAPORS WERE FOUND TO VARY OVER EACH DAY'S SAMPLING RUNS. THE RANGE OF VALUES FOR

THE SERIES OF RUNS ARE TABULATED IN TABLE II.

TABLE II

ROTARY STEAM TUBE DRYER STACK LOSSES

Run No.	Solids Loss gm/cu.ft (20°C., 1ATM)
1	0.0014
2	0.0019
3	0.0013
4	0.0010
5	0.0026
7*	0.0015
8*	0.0022
9*	0.0009
10*	0.0004
11	0.0060

*RUNS MADE WITH STEAM JET OPEN TO STACK

TABLE III

ANEMOMETER AND TEMPERATURE READINGS IN DRYER STACK

Condition	Velocity Feet/min	Temp. °C.	Stack Gas Velocity (20°C, 1ATM)
Steam jet open	867	76	3560
Steam jet closed	636	81	2595

The vapors generated by the drum drying of the syrup produced by the evaporators were sampled following the procedure outlined for the rotary steam tube dryer vapors. In place of the gas measuring apparatus shown in Figure 6, a wet gas meter

was used. It was found during the sampling of the rotary dryer stack vapors that at the end of eight minutes sampling time the run had to be interrupted in order to refill the gas measuring apparatus. Since approximately 24 liters were removed for each run, this necessitated three refills per run. A wet gas meter was calibrated by drawing air through the meter and displacing a known volume of water. The meter was calibrated for air at 22°C. and a differential of 5.2 inches of water. In place of a water aspirator a vacuum pump was used to withdraw the vapor samples from the stack. Figure 7 illustrates the arrangement of the apparatus. The sampling rate with the gas meter was 0.085 cubic feet per minute. The initial series of runs were made with the wet gas meter. An increase in sampling rate in the final series of runs necessitated the substitution of an orifice flow meter in place of the wet gas meter. The flow meter was calibrated against the wet gas meter. The calibration curve is shown in Figure 8.

The analysis of the absorbents indicated no trace of protein and fat. Although the pH of the absorbents ranged between 4.5 to 5.9, no titratable acids were detected. The entrained solids in the drum dryer stack were found to be much finer than the solids from the rotary steam tube dryer stack. This was indicated by the fact that the solids particles trapped from the drum dryer penetrated much deeper in the glass wool filter trap in contrast to the relatively localized deposit obtained from the rotary dryer. The drum dryer dust is darker

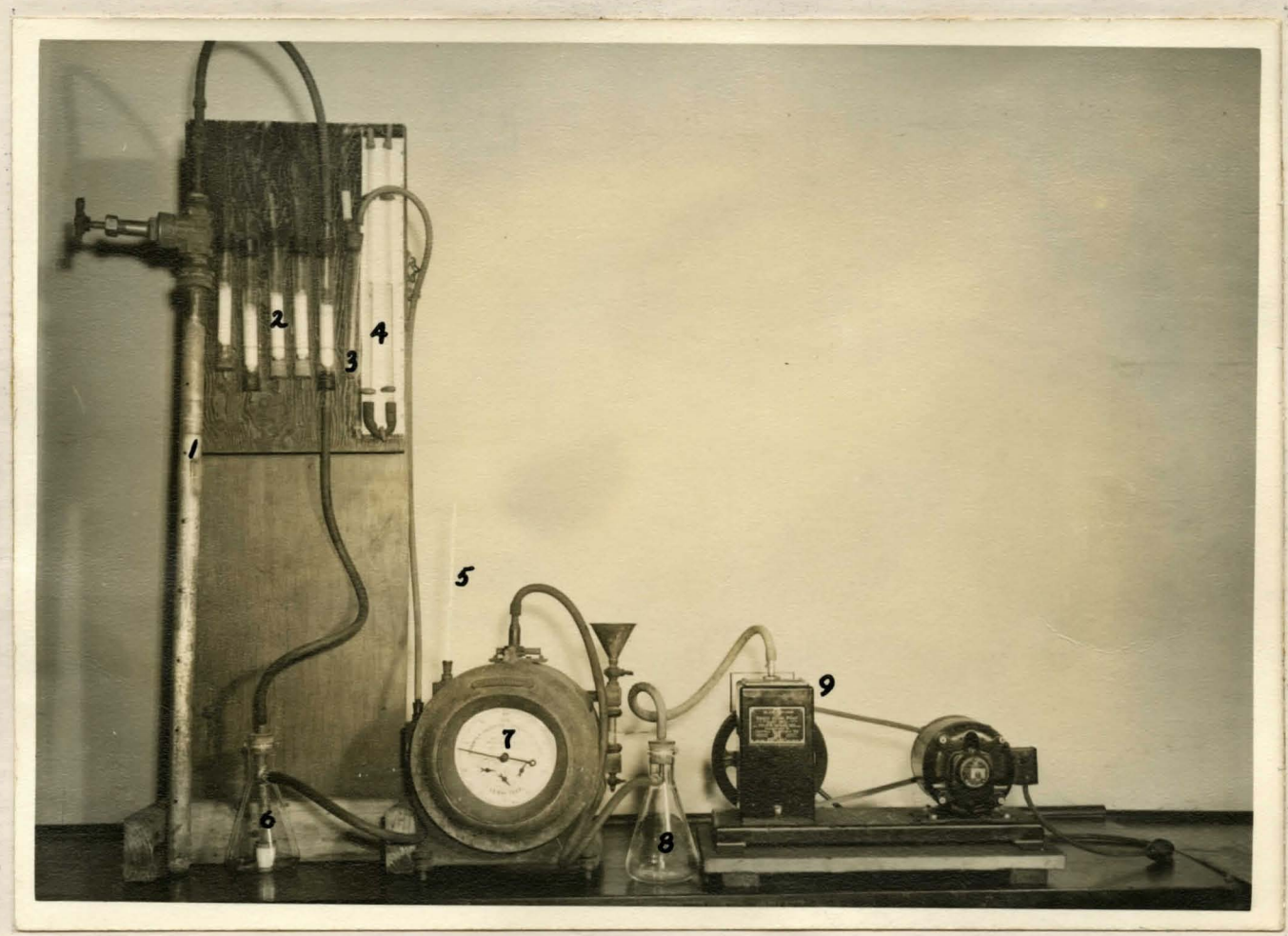

FIGURE 7 DRUM DRYER STACK SAMPLING APPARATUS

(1) PERFORATED SAMPLING TUBE (2) GLASS WOOL FILTERS (3) WATER TRAP TO MANOMETER (4) MANOMETER (5) THERMOMETER (6) ABSORBER (7) WET GAS METER (8) PUMP TRAP (9) VACUUM PUMP

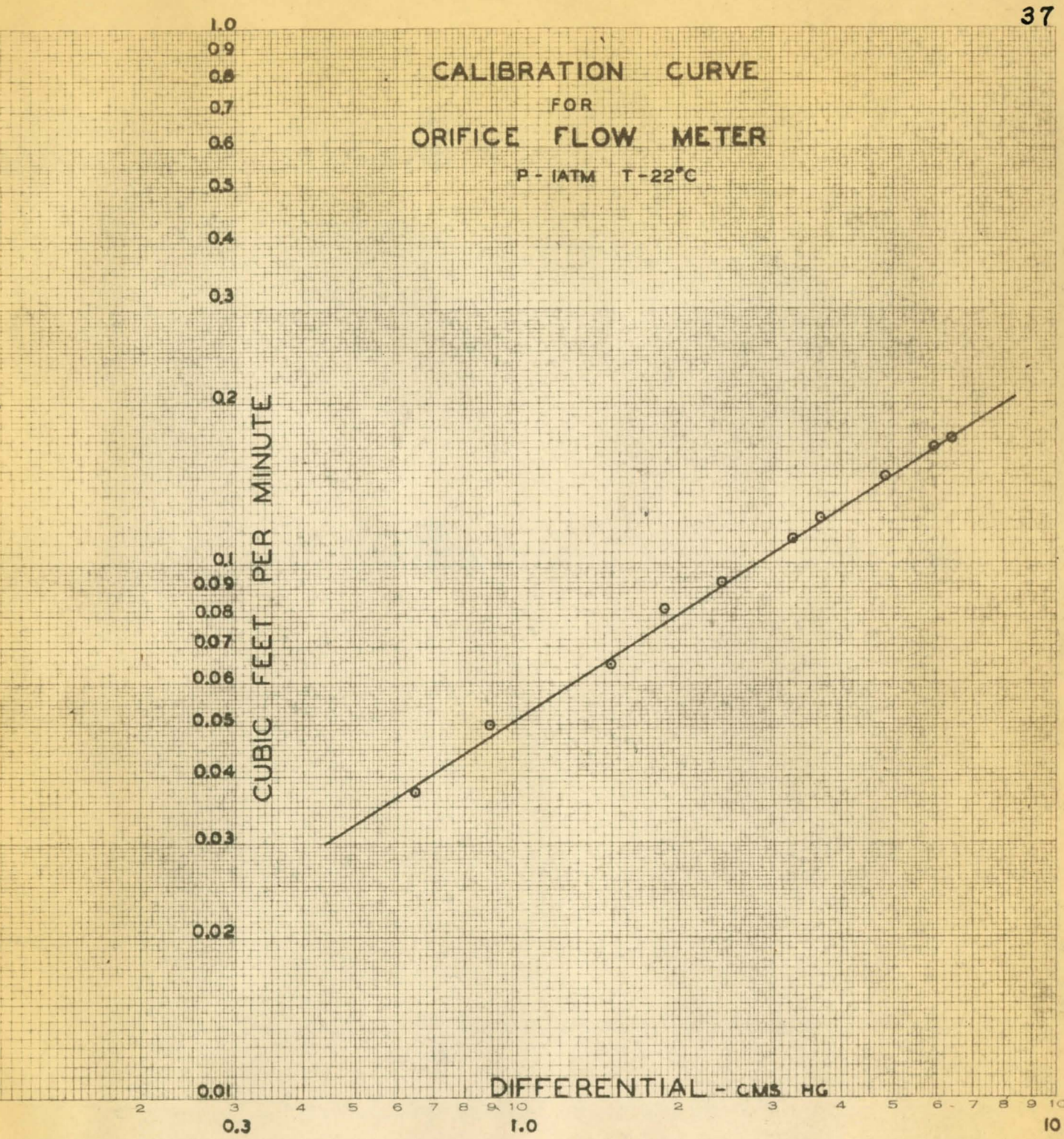

FIGURE 8
CALIBRATION CURVE
FOR
ORIFICE FLOW METER

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in color and the odor of the dust is definitely stronger than the solids from the rotary steam tube dryer. The vapor velocity of the stack gases were 693 feet per minute at an average temperature of 52°C. The volume of stack gases passing through the stack, referred to 1 atm and 20°C. was calculated to be 3390 cubic feet per minute.

The range of values for solids content of the vapor is presented in Table IV.

TABLE IV

DRUM DRYER STACK LOSSES

Run No.	Sampling Rate cu.ft/min	Solids Content gm/cu.ft(20°C 1atm)
1	0.065	0.00391
2	0.065	0.00194
3	0.065	0.00215
4	0.065	0.00315
5	0.075	0.00260
6	0.130	0.00224
8	0.130	0.00410
9	0.200	0.00224
10	0.230	0.00390

Samples from the vapor line carrying flashed cooker steam to the low vacuum spirits beer still were obtained at a point approximately twelve feet beyond flash chamber No.2 (entrainment chamber). Sampling procedures were modified in order to obtain material loss expressed in terms of grams of material per gram of flashed steam. Initial sampling runs were made with a glass wool filter in series with the sampling tube,

water-jacketed condensers, 0.1N NaOH solution absorbent, and vacuum pump. The use of the glass wool filters proved to be inadequate for (a) the distillate contained some entrained solid particles (b) the particles would distribute over the surface of the glass wool and plug up the filter. The sampling procedure finally used consisted of passing the vapors from the sampling tube directly to the condensers and collecting the distillate with the suspended solids. The non-condensibles were bubbled by means of a No.2 Gooch crucible through a known volume of 0.1N NaOH absorbent. The arrangement of the apparatus is shown in Figure 9. Sampling technique for the flashed cooker steam consisted of (a) flushing the sampling tube with water under pressure (b) withdrawing a sample equal to that of a sampling run and discarding the distillate (c) removal of approximately 350 grams of distillate over a period of 30 minutes.

The distillate was analyzed for solids content and pH. The 0.1N NaOH absorbent was back titrated with 0.1N H_2SO_4 to determine the milligram equivalents of volatile acids. During the sampling runs, it was observed that at intermittent intervals slugs of cooked grain were drawn into the condensers. This irregularity of flow was reflected in the results. It was also observed that during each sampling run the original colorless 0.1N NaOH absorbent became discolored to straw yellow. During the titration of the absorbent with 0.1N H_2SO_4, the solution emitted an offensive odor. Further investigation

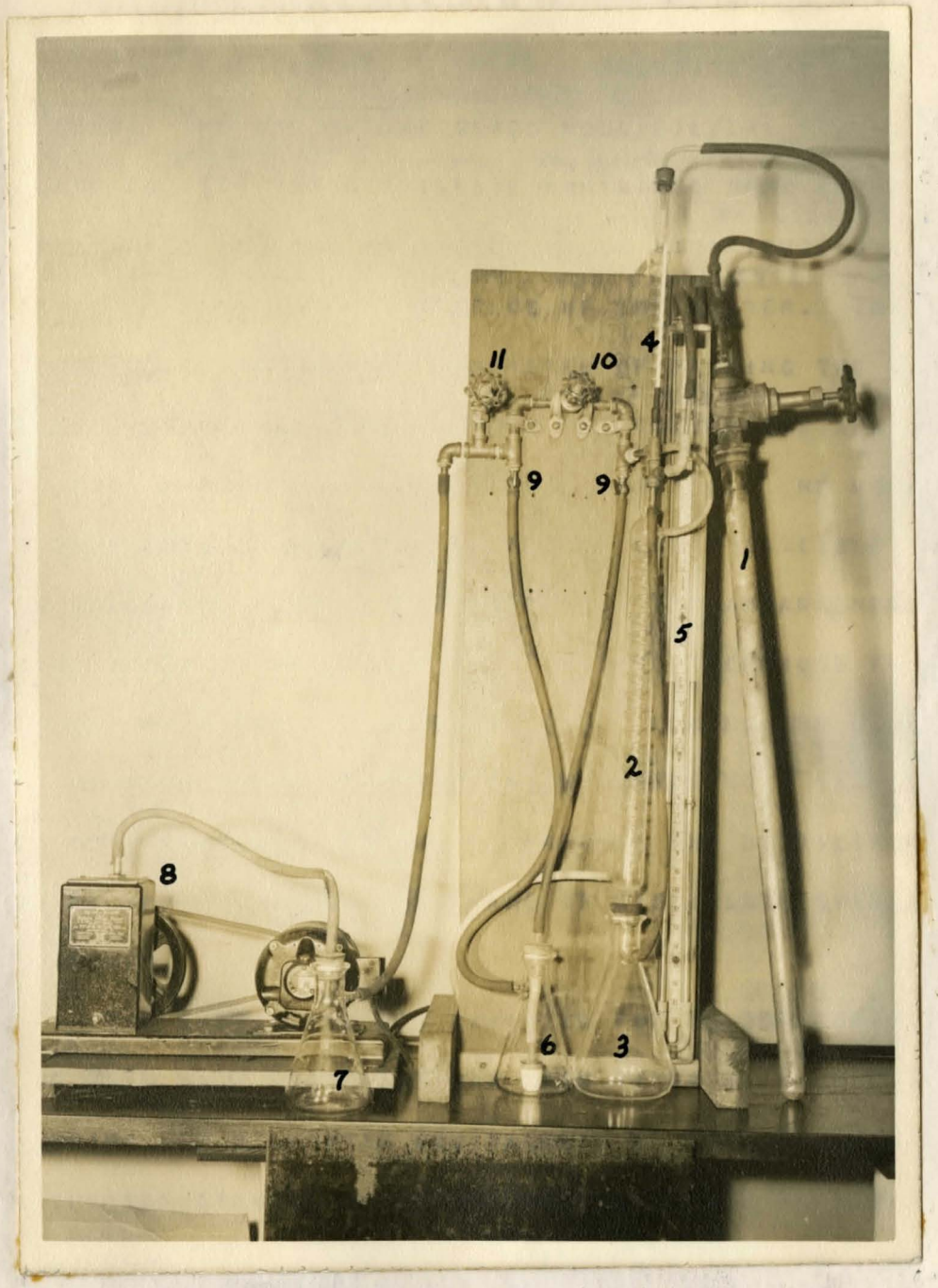

FIGURE 9 FLASHED STEAM AND BEER STILL VAPOR SAMPLING APPARATUS

(1) PERFORATED SAMPLING TUBE (2) WATER-JACKETED CONDENSERS
(3) DISTILLATE RECEIVER (4) THERMOMETER (5) MANOMETER (6) ABSORBER
(7) PUMP TRAP (8) VACUUM PUMP (9) PETCOCK (10) BY-PASS VALVE
(11) CONTROL VALVE

REVEALED THAT UPON HEATING, THE VOLATILE ACID VAPOR WHICH THE ABSORBENT HAD TAKEN UP, COULD BE REMOVED. THE RESULTS OF THE SAMPLES TAKEN FROM THE FLASHED COOKER STEAM LINE ARE PRESENTED IN TABLE V.

TABLE V

FLASHED COOKER STEAM LOSSES

Run No.	Running Rate pounds/min	Solids Content of Distillate gm/gm of Dist.	PH of Dist.	Milligram-equivalent of acid vapor per gm Dist.
5	450	0.000454	5.1	
6	450	0.000522	6.15	0.0935
7	450	0.000371	5.5	
8	471	0.000523	5.35	0.0327
9	471	0.001509	6.05	0.0274
10	471	0.000934	5.1	0.0288
11	486	0.000379	5.33	0.0472
12	541	0.000671	5.55	0.0480

PROTEIN DETERMINATIONS MADE ON AN ALIQUOT PORTION OF THE ABSORBENT, INDICATED NO PRESENCE OF PROTEIN.

VAPOR SAMPLES FROM THE SPIRITS BEER STILL STEAM EJECTOR VENT WERE REMOVED FROM THE FOUR INCH VENT LINE LEADING FROM THE SECOND STAGE SURFACE CONDENSER TO THE ROOF OF THE STILL BUILDING. THE APPARATUS USED WAS SIMILAR TO THAT IN FIGURE 9. IN PLACE OF THE ABSORBER, THE CALIBRATED ORIFICE FLOW METER WAS TIED IN AT POINTS 9-9. A TEST TUBE WAS INSERTED IN THE DISTILLATE RECEIVER TO CATCH THE CONDENSIBLE PORTION OF THE VAPOR. THE SAMPLING RATE WAS APPROXIMATELY 0.4 CUBIC FEET PER

minute. Sampling runs varied from 45 to 120 minutes. Sampling runs No.2 and No.3 were made with the spirits beer still receiving straight power house steam; run No.4 was made with the beer still on flashed cooker steam. Anemometer and temperature readings were taken at the discharge of the vent to the atmosphere. The results are tabulated in Table VI.

TABLE VI

SPIRITS BEER STILL STEAM EJECTOR VENT LOSSES

Run No.	Volume Sampled cubic feet (20°C. 1atm)	Volume of Distillate milliliters	Specific Gravity of Distillate 25°/25°
2	34.6	38	0.9826
3	19.2	28	0.9819
4	18.5	27	0.9756

The distillates obtained from runs No.2 and No.3 were decidedly different from run No.4. The odor from distillates No.2 and No.3 were identical, i.e., a sharp pungent odor bordering that of "heads" or perhaps near fusel oil. Distillate No.4 had the characteristic nauseating malodor first experienced during the neutralization of the 0.1N NaOH absorbent in the flashed cooker steam investigation. Distillates No.2 and No.3 were colorless; No.4 was slightly cloudy. It was also observed that when the vacuum on the distillate receiver was increased, the distillate exhibited the tendency to evaporate at room conditions.

The vapor velocity through the ejector vent was 440 feet a minute at 51°C., with a beer feed to the still of 175 gallons per minute. The volume of vapor reverred to 20°C. and 1 atmosphere, was 34.7 cubic feet per minute.

The vapors from the Badger low vacuum spirits beer still going to the dephlegmator were removed through the perforated sampling tube described in Figure 3. The sampling tube was inserted dianetrically across the three foot diameter collar connecting the eight foot diameter still to the vapor line leading to the dephlegmator. The sampler was located approximately eight inches above the top spray nozzle. The initial sampling runs were made with a glass wool filter trap in series with the sampler, water-jacketed condensers, distillate receiver, absorber, and vacuum pump. This procedure proved unsatisfactory for the entrained solids passed through the filter and were present in the distillate. The system finally used was to pass all the vapors from the sampling tube directly to the condensers. The non-condensibles were bubbled through 0.1N NaOH solution and finally to the vacuum pump. The arrangement of the apparatus was similar to that used in sampling flashed cooker steam as shown in Figure 9. The sampling procedure consisted of (a) opening sampling valve to allow air to be drawn through perforations, (b) flushing sampling tube with water under pressure, (c) withdrawing one liter of distillate over a period of approximately one hour and discarding, (d) removal of the vapor sample during a period of one hour. The solids content of the

distillate was determined by drying known volumes of distillate in weighed evaporating dishes at 105°C. The alcohol content was determined by the A.O.A.C. Method XVI, No.5.(11) The pH of the distillate was also determined. The 0.1N NaOH solution was back-titrated for absorbed acid constituents. Sampling runs were made under varied operating conditions in an attempt to obtain a trend of the entrainment tendencies. The variables observed were (1) feed rate to the still (2) straight power house steam flow rate, and (3) source of steam (flashed cooker steam or straight power house steam). The results obtained in the investigation of the spirits beer still are tabulated in Table VII.

TABLE VII

SPIRITS BEER STILL SOLIDS LOSS

Run No.	Feed Rate G.P.M	Distillate Per cent Alcohol by volume 60/60	Solids Content gms/100ml	Solids Content pounds per proof-gal.	PH	Acid Absorbed
7	170	12.28	0.4096	0.1380		0.0645
8	170	16.60	0.2156	0.0733	4.3	
9	170	10.05	0.2873	0.0966	4.5	
10	170	18.34	0.3816	0.0873	4.5	
11	175	13.45	1.1562	0.415	4.5	
12	175	15.69	1.1142	0.201	4.5	
13	175	17.38	1.1762	0.263	4.5	
14	175	20.10	0.7989	0.167	4.5	
15	175	20.09	0.8121	0.170	4.5	
16*	180	27.84	0.4231	0.0833	4.5	
17	180	29.44	0.3287	0.0647	4.6	0.0214
18	180	33.32	0.3841	0.0681	4.7	0.0368
19	180	33.16	0.4237	0.0731	4.7	0.0380
20*	185	16.44	0.2488	0.0827	4.6	0.0207
21*	185	16.44	0.2337	0.0777	4.3	0.0223
22	185	19.39	0.2747	0.0913	4.5	0.0305
23	185	16.44	0.2995	0.0766	4.4	0.0308
24*	190	13.06	0.2810	0.289	4.4	0.0192
25	190	11.00	0.6066	0.229	4.4	0.0151
26*	190	20.88	0.0756	0.0151	4.3	0.0174
27	190	12.12	0.2766	0.0947	4.2	0.0352
28	190	16.28	0.1479	0.0472	4.3	0.0174
29	190	17.79	0.2086	0.0675	4.4	0.0306
30	190	17.70	0.2522	0.0796	4.4	0.0261

*Runs made with straight power house steam.

Absorbed acid is expressed as NO-equivalents per proof-ml

The runs made with straight power house steam yielded a distillate with an odor characteristic of the normal beer still distillates. During the back-titration of the absorbents no abnormal odor was detected. However, those runs made with the spirits beer still on flashed cooker steam, yielded distillates bearing the offensive odor encountered in the flashed cooker

steam investigation. The absorbents used in these runs yielded the same offensive odor during neutralization as did those of the flashed cooker steam investigation.

Table VIII presents results obtained from additional spirits beer still runs with the still on controlled metered flow of straight power house steam.

TABLE VIII

SPIRITS BEER STILL SOLIDS LOSS

Run No.	Feed Rate G.P.M	Feed % Alcohol by Volume 60/60°F	Steam Flow Pounds per Hour	Distillate % Alcohol by Volume 60/60	Distillate Solids Content Grs per 100 ml	Distillate Solids Content Pounds per Proof-Gal
31	110	5.89	20,000	11.18	0.5906	0.2210
32	110	5.89	20,000	13.38	0.5000	0.1586
33	110	5.89	20,000	15.69	0.5066	0.1570
34	110	5.89	20,000	11.68	1.6693	0.3819
35	110	5.89	20,000	15.82	0.5298	0.1398
36	130	5.89	20,400	14.12	1.1076	0.3261
37	130	5.89	20,400	17.46	0.5476	0.1311
38	130	5.89	20,400	14.62	0.5825	0.1345
39	145	5.89	19,800	14.76	0.4924	0.1304
40	130	6.01	19,800	22.04	0.4414	0.0838
41	130	6.01	19,800	11.00	0.5460	0.2079
42	130	6.01	19,800	17.69	0.5456	0.1296
43	150	6.01	19,700	19.58	0.4482	0.0977
44	150	6.01	19,500	18.58	0.4978	0.1116
45	150	6.01	19,500	19.00	0.4348	0.0975
46	150	6.01	19,500	16.42	0.4904	0.1247
47	160	6.01	20,500	15.68	0.3199	0.0872
48	160	6.01	20,700	15.28	0.3297	0.0901
49	160	6.01	21,300	16.78	0.2591	0.0762
50	160	6.01	21,000	15.18	0.2436	0.0670
51	170	6.01	22,200	13.62	0.5540	0.1694
52	170	6.01	22,200	14.42	0.5672	0.1730
53	170	6.01	22,200	14.08	0.4800	0.1450
54	180	5.66	22,200	14.56	0.5429	0.1562
55	180	5.66	22,200	15.38	0.4705	0.1280
56	180	5.66	22,200	16.06	0.4137	0.1154
57	180	5.66	22,200	16.12	0.3576	0.0927

THE EXPERIMENTAL PROCEDURE FOLLOWED IN THE DETERMINATION OF A PRACTICAL "THEORETICAL" DRIED GRAIN YIELD WAS RESOLVED INTO (1) AN INVESTIGATION OF THE SOLIDS AND STARCH CONTENT OF THE GRAINS IN THE PRECOOKER, (2) SOLIDS AND STARCH CONTENT OF THE GRAIN SLURRY AFTER HIGH PRESSURE AND HIGH TEMPERATURE COOKING WITH IMMEDIATE FLASH COOLING, (3) SOLIDS AND STARCH CONTENT OF THE CONVERTED MASH ENTERING THE FERMENTER, AND (4) DETERMINATION OF ALCOHOL AND RESIDUAL SOLIDS YIELDS.

THE EXPERIMENTAL PROCEDURE FOR PART ONE INVOLVED A SOLIDS MATERIAL BALANCE BETWEEN THE WEIGHED GRAIN AND THE METERED WATER AND BACKSET ENTERING THE PRECOOKER TO THE ACTUAL SOLIDS CONTENT OF THE PRECOOKER. THE STARCH CONTENT OF THE WHOLE PRECOOKER SLURRY WAS MADE. THE MILLED GRAIN SAMPLE WAS REMOVED FROM THE CHUTE BETWEEN THE MILL AND THE VANE FEEDER TO THE PRECOOKER. THE SAMPLES FROM THE PRECOOKER WERE REMOVED FROM THE 1½ INCH DRAIN VALVE LOCATED ON THE LEG FROM THE PRECOOKER TO THE MIXING PUMP.

THE SAMPLES FROM FLASH CHAMBER No.1 WERE REMOVED FROM THE CONE OF THE FLASH CHAMBER. THE SAMPLING POINT WAS LOCATED 29 INCHES ABOVE THE MALT INFUSION INLET. THE SAMPLES WERE REMOVED THROUGH A 1/4 INCH DIAMETER PIPE INTO A ONE LITER SUCTION FLASK BY MEANS OF A VACUUM PUMP. THE SAMPLING PROCEDURE CONSISTED OF (A) OPENING VALVE TO SAMPLING TUBE AND ALLOWING AIR TO BE DRAWN THROUGH (B) COLLECTING ONE LITER OF SLURRY AND DISCARDING (C) REMOVAL OF SLURRY SAMPLE TO TOTAL TWO LITERS.

The solids content of the grain was determined by the A.O.A.C. Method XXVII, No.7 (12). The starch content of the slurry was determined by the A.O.A.C. Method XXVII, No.31 (13) using the diastase-acid hydrolysis procedure. Dextrose determinations were made using the A.O.A.C. XXXIV Munson-Walker method with subsequent thiosulphate titration.(14) Solids determination of the slurry was made by adding a known weight of slurry to a weighed evaporating dish and drying at 105°C.

The results obtained for parts one and two are tabulated in Table IX.

TABLE IX

PRECOOKER AND FLASH CHAMBER ANALYSIS

Mashing Data To Precooker (Exclude Malt)	% Solids Incoming	% Solids Calculated	% Solids Dried Slurry In Precooker	% Starch Bone-Dry In Precooker	% Solids Dried Slurry In Flash Chamber	% Starch Bone-Dry In Flash Chamber
Corn- 410 lbs/min Malt- 34 lbs/min Water- 70 g.p.m. Backset- 22 g.p.m.	85.2 3.6	30.2	27.1	61.0	24.7	72.5
Corn- 380 lbs/min Malt- 24 lbs/min Water- 75 g.p.m. Backset- 0	86.6	32.7	31.1	56.8	25.6	58.8
Corn- 160 lbs/min Rye- 107 lbs/min Malt- 12 lbs/min Water- 74 g.p.m. Backset- 0	87.9 88.8	26.5	24.4	49.7	20.9	54.7

The experimental procedures for parts three and four necessitated that the converted mash samples and plant yeast samples be obtained under aseptic conditions. Sterile containers were used to secure plant converted mash and plant yeast. The samples of plant converted mash were obtained from the filling spout leading into the fermenter. The yeast samples were removed from the yeast tub containing finished yeast scheduled to be dropped to the filling fermenters. Initially, only two liter samples of converted mash were removed. The final technique was to remove four liters of plant converted mash and one liter of plant yeast.

The converted mash was weighed and added to a sterile four-liter glass container provided with an agitator. To the mash was added the plant yeast equal to approximately three per cent of the weight of converted mash. The mixture was kept in constant agitation before and during the inoculation of the laboratory fermenters. Sterile laboratory fermenters were prepared as described by Stark, Adams, Scalf, and Kolachov(15). In place of the 500 ml centrifuge bottles, 500 ml erlenmeyer flasks were used. The technique used to inoculate the fermenters was (1) provide a flexible connection consisting of 1/4 inch diameter glass tubing and rubber tubing between the four-liter mixing container and fermenter, (2) apply sufficient suction to fermenter to draw over mixed mash, (3) inoculate fermenter with approximately 300 ml of mash, (4) withdraw suction and connection to fer-

menter, and (5) seal innoculated fermenter with alcohol and carbon dioxide trap. After a number of fermenters were innoculated, samples of the mixed mash and yeast were removed for solids and starch determinations. After sampling for solids and starch determinations, additional fermenters were innoculated. The fermenters were placed in a constant temperature water bath for incubation. The fermenters were incubated at 86°F for the first twenty hours and at 90°F for the remaining period of incubation. These conditions were established to duplicate plant operating conditions. At the end of the incubation period, the liquid was distilled from the original fermenter flask. A sample of 100 ml of distillate was collected in a 100 ml volumetric flask. The stillage remaining in the fermenter flask was quantitatively transferred by repeated washings to weighed evaporating dishes. The stillage was dried at 110°C.

The starch content of the mash mixture was determined by the A.O.A.C. Method XXVII, No.31 together with the A.O.A.C. Method XXXIV. The alcohol content of the distillate was determined with a pycnometer. The alcohol content of the original yeast sample was also determined in order to calculate the actual alcohol production. The results obtained from the laboratory fermentations of plant converted mash with plant yeast are tabulated in Table X.

TABLE X

SUMMARY OF LABORATORY FERMENTATIONS

	Fermenter No.	1	2	4	5
A.	Weight of mash to fermenter - grams	292.21	292.30	180.35	157.80
B.	Solids content of mash - per cent	19.2	18.4	19.71	19.71
C.	Solids in fermenter (A x B)	56.1	53.8	35.60	31.10
D.	Starch in mash - %	75.9	64.6	74.0	74.0
E.	Starch in fermenter (C x D) grams	42.59	34.80	26.30	23.10
F.	Theoretical alcohol yield (0.568 C) gms	24.2	19.75	14.95	13.10
G.	Alcohol produced-gms	18.18	18.20	8.94	8.20
H.	Alcohol introduced in yeast mash-grams	0.54	0.71	0.41	0.36
I.	Actual alcohol produced (G-H) grams	17.64	17.49	8.53	7.84
J.	Fermentation efficiency (I/F) x 100 per cent	73	88.5	57.0	59.8
K.	Solids recovered-gms	25.50	24.30	14.70	14.6
L.	Per cent solids recovered (K/C)x100	45.4	45.3	41.3	47.0
M.	Fermentation time-hrs.	48	48	48	48
N.	Mash bill				
	Rye - pounds per min.	160	160		
	Wheat - pounds per min	-	-	170	170
	Malt - pounds per min	44	44	35	35
	Corn - pounds per min	260	260	190	190
	Milo - pounds per min	-	-	-	-
	Backset - gals per min	18	18	0	0

TABLE X - (CONTINUED)

SUMMARY OF LABORATORY FERMENTATIONS

	Fermenter No.	6	7	8	9
A.	Weight of mash to fermenter - grams	141.80	369.90	291.90	241.60
B.	Solids content of mash - per cent	19.71	16.9	16.9	16.9
C.	Solids in fermenter (A x B) grams	27.95	62.5	49.4	40.7
D.	Starch in mash - %	74.0	73.5	73.5	73.5
E.	Starch in fermenter (C x D) grams	20.65	45.94	36.3	30.0
F.	Theoretical alcohol yield (0.568 C) gms	11.75	26.1	20.6	17.04
G.	Alcohol produced-gms	7.46	16.98	14.73	13.18
H.	Alcohol introduced in yeast mash-grams	0.32	1.16	0.91	0.75
I.	Actual alcohol produced (G-H) grams	7.14	15.82	13.82	12.43
J.	Fermentation Efficiency (I/F) x 100 per cent	60.6	60.7	67.3	73.0
K.	Solids recovered- gms	13.20	35.30	25.70	20.50
L.	Per cent solids recovered (K/C)x100	47.3	56.5	52.2	50.5
M.	Fermentation time-hrs	48	20	24	44
N.	Mash bill				
	Rye - pounds per min	-	240	240	240
	Wheat-pounds per min	170	160	160	160
	Malt -pounds per min	35	38	38	38
	Corn -pounds per min	190	-	-	-
	Backset -gals per min	0	13	13	13

TABLE X - (CONTINUED)

SUMMARY OF LABORATORY FERMENTATIONS

	Fermenter No.	10	11	12	13
A.	Weight of mash to fermenter - grams	259.50	268.20	277.30	333.30
B.	Solids content of mash - per cent	16.9	16.9	16.9	16.39
C.	Solids in fermenter (A x B) grams	43.7	45.4	46.8	54.50
D.	Starch in mash - %	73.5	73.5	73.5	71.3
E.	Starch in fermenter (C x D) grams	32.2	33.3	34.4	38.85
F.	Theoretical alcohol yield (0.568 C) gms	18.3	18.95	19.6	22.14
G.	Alcohol produced-gms	14.36	14.29	17.35	12.86
H.	Alcohol introduced in yeast mash-grams	0.81	0.84	0.87	0.41
I.	Actual alcohol produced (G-H) grams	13.55	13.45	16.48	12.45
J.	Fermentation Efficiency (I/F) x 100 per cent	74.0	71.1	84.0	56.4
K.	Solids recovered - gms	21.90	22.70	22.60	29.10
L.	Per cent solids recovered (K/C)x100	50.0	50.0	48.4	53.5
M.	Fermentation time-hrs	45	68	72	20
N.	Mash Bill				
	Rye - pounds per min	240	240	240	-
	Wheat-pounds per min	160	160	160	110
	Malt -pounds per min	38	38	38	63
	Milo -pounds per min	-	-	-	120
	Backset-gals per min	13	13	13	19

TABLE X - (CONTINUED)

SUMMARY OF LABORATORY FERMENTATIONS

	Fermenter No.	14	15	16	17
A.	Weight of mash to fermenter - grams	360.50	379.40	280.95	299.75
B.	Solids content of mash - per cent	16.39	16.39	16.39	16.39
C.	Solids in fermenter (A x B) grams	59.00	62.18	46.05	49.13
D.	Starch in mash - %	71.30	71.30	71.30	71.30
E.	Starch in fermenter (C x D) grams	42.07	44.33	32.83	35.03
F.	Theoretical alcohol yield (0.568C) gms	23.89	25.18	18.65	19.89
G.	Alcohol produced-gms	15.08	19.41	14.75	15.87
H.	Alcohol introduced in yeast mash-grams	0.45	0.47	0.35	0.37
I.	Actual alcohol produced (G-H) grams	14.63	18.94	14.40	15.50
J.	Fermentation Efficiency (I/F) x 100 per cent	61.4	75.4	77.3	78.0
K.	Solids recovered-gms	29.25	24.20	18.10	19.10
L.	Per cent solids recovered (K/C)x100	49.6	38.95	39.3	38.9
M.	Fermentation time-hrs	22	43	44	45
N.	Mash bill				
	Wheat-pounds per min	110	110	110	110
	Malt -pounds per min	63	63	63	63
	Milo -pounds per min	120	120	120	120
	Backset-gals per min	19	19	19	19

TABLE X - (CONTINUED)

SUMMARY OF LABORATORY FERMENTATIONS

	Fermenter No.	18	19	20	21
A.	Weight of mash to fermenter - grams	246.15	297.55	258.70	356.40
B.	Solids content of mash - per cent	16.39	16.39	16.39	20.85
C.	Solids in fermenter (A x B) grams	40.34	48.77	42.40	74.31
D.	Starch in mash - %	71.30	71.30	71.30	74.50
E.	Starch in fermenter (C x D) grams	28.76	34.77	30.23	55.36
F.	Theoretical alcohol yield (0.568C) gms	16.34	19.75	17.17	31.45
G.	Alcohol produced-gms	13.11	16.07	13.64	14.95
H.	Alcohol introduced in yeast mash - grams	0.31	0.37	0.32	1.06
I.	Actual alcohol produced (G-H) grams	12.80	15.70	13.32	13.89
J.	Fermentation Efficiency (I/F) x 100 per cent	78.4	79.5	77.5	44.1
K.	Solids recovered - gms	15.10	18.40	14.70	45.90
L.	Per cent solids recovered (K/C)x100	37.5	37.75	34.7	61.8
M.	Fermentation time-hrs	68	69	72	20
N.	Mash bill				
	Wheat-pounds per min	110	110	110	480
	Malt -pounds per min	63	63	63	51
	Milo -pounds per min	120	120	120	-
	Backset-gals per min	19	19	19	0

TABLE X - (CONTINUED)

SUMMARY OF LABORATORY FERMENTATIONS

	Fermenter No.	22	23	24	25
A.	Weight of mash to fermenter - grams	300.60	365.00	299.50	287.90
B.	Solids content of mash - per cent	20.85	20.85	20.85	20.85
C.	Solids in fermenter (A x B) grams	62.68	76.10	62.45	60.03
D.	Starch in mash - %	74.5	74.5	74.5	74.5
E.	Starch in fermenter (C x D) grams	46.49	56.70	46.52	44.72
F.	Theoretical alcohol yield (0.568C) gms	26.52	32.20	26.43	25.40
G.	Alcohol produced-gms	14.36	18.86	20.76	19.72
H.	Alcohol introduced in yeast mash - grams	0.90	1.10	0.90	0.86
I.	Actual alcohol produced (G-H) grams	13.46	17.76	19.86	18.86
J.	Fermentation efficiency (I/F) x 100 per cent	50.8	55.2	75.2	74.3
K.	Solids recovered-gms	35.70	41.70	22.90	22.15
L.	Per cent solids recovered (K/C)x100	56.9	54.8	36.7	36.8
M.	Fermentation time-hrs	23	24	44	44
N.	Mash bill				
	Wheat-pounds per min	480	480	480	480
	Malt -pounds per min	51	51	51	51
	Backset-gals per min	0	0	0	0

TABLE X - (CONTINUED)

SUMMARY OF LABORATORY FERMENTATIONS

	Fermenter No.	26	27	28	29
A.	Weight of mash to fermenter - grams	344.00	317.85	296.60	297.85
B.	Solids content of mash - per cent	20.85	20.85	20.85	20.85
C.	Solids in fermenter (A x B) grams	71.72	66.27	61.84	62.10
D.	Starch in mash - %	74.5	74.5	74.5	74.5
E.	Starch in fermenter (C x D) grams	53.44	49.37	46.07	46.27
F.	Theoretical alcohol yield (0.568 C) gms	30.35	28.04	26.17	26.28
G.	Alcohol produced-gms	23.28	21.94	20.52	20.87
H.	Alcohol introduced in yeast mash - gms	1.03	0.95	0.89	0.89
I.	Actual alcohol produced (G-H) gms	22.25	20.99	19.63	19.98
J.	Fermentation efficiency (I/F) x 100 %	73.5	74.9	75.2	76.0
K.	Solids recovered-gms	26.60	23.70	23.00	23.40
L.	Per cent solids recovered (K/C)x100	37.1	35.7	37.2	37.6
M.	Fermentation time-hrs	45	68	68	69
N.	Mash bill				
	Wheat-pounds per min	480	480	480	480
	Malt -pounds per min	51	51	51	51
	Backset-gals per min	0	0	0	0

DISCUSSION

The results obtained from the investigation of solids losses from the evaporator, as tabulated in Table I, have been divided into those losses from the condensate pump and those losses from the condensed vapors emmitted from No. 4 effect.

In the feed range of 8000 to 8300 gallons per hour, the solids loss through the condensate pump was constant at the value of 0.0073 gm per 50 ml of condensate. This solids loss is attributed solely to entrainment. The method of feed has no effect upon the magnitude of the solids loss. This situation may be attributed to the operating conditions. When the evaporator is operated with backward feed (4-3-2-1B-1A-1AA), the thin liquor enters effect No.4, is concentrated from approximately 3% to 4% solids, and is then pumped to effect No.3. With the evaporator operating with mixed feed, (2-3-4-1B-1A-1AA), the thin feed enters effect No.2 at the same 3% solids and is concentrated to approximately 4% solids, which is then fed to effect No.3 under the same conditions of solids concentration as the feed from effect No.4 to effect No.3. Although the temperature of the liquor in effect No.2 is higher than that in effect No.3 and may cause partial flashing as it enters the region of lower pressure in effect No.3, the quantity of liquor flashing is small and would not add materially to the entrainment loss from effect No.3.

THE ENTRAINMENT LOSSES FROM EFFECTS 1B-1A-1AA ARE ASSUMED TO BE SUBSTANTIALLY CONSTANT, SINCE THE SOLIDS CONCENTRATIONS TO EACH EFFECT WHETHER FED FROM EFFECT No.2 OR No.4 WILL BE SUBSTANTIALLY CONSTANT. WHEN OPERATED WITH MIXED FEED, THE TEMPERATURE OF THE LIQUOR ENTERING EFFECT No.1B WILL BE LOWER THAN WHEN OPERATED WITH BACKWARD FEED, BUT THIS SHOULD HAVE NO EFFECT ON THE SOLIDS LOSS FROM EFFECT 1B FOR THE MAJOR PORTION, IF NOT ALL, OF THE ENTRAINED SOLIDS IS DERIVED FROM THE FORCED CIRCULATION OF THE LIQUOR THROUGH THE TUBES. THE IMPACT OF THE CONTINUOUS HIGH VELOCITY STREAM OF APPROXIMATELY 10 TO 15% SOLIDS CONCENTRATION AGAINST THE DEFLECTOR PLATE IS SUFFICIENT TO PRODUCE A FINE MIST OF ENTRAINED LIQUOR WHICH IS CARRIED OUT IN THE VAPOR TO THE STEAM CHEST OF EFFECT No. 2 AND ULTIMATELY TO THE CONDENSATE PUMP. THE SAME REASONING HOLDS FOR EFFECTS 1A AND 1AA, ALTHOUGH THE SOLIDS LOSS SHOULD BE LOWER THAN THAT EXPECTED FROM 1B BECAUSE OF THE HIGHER VISCOSITY AT THE GREATER SOLIDS CONCENTRATION.

IN THE FEED REGION OF 9,000 TO 10,600 GALLONS PER HOUR, THE SOLIDS CONTENT OF THE CONDENSATE PUMP DISCHARGE INCREASED TO AN AVERAGE VALUE OF 0.0137 GMS PER 50 ML OF CONDENSATE. THIS INCREASE MAY BE ATTRIBUTED TO THE HIGHER FEED RATES AND MORE VIGOROUS BOILING AS A RESULT OF CLEANER HEATING SURFACES, BETTER OPERATING CONDITIONS OR A COMBINATION OF ALL THREE FACTORS.

THE SOLIDS LOSS OBTAINED FROM THE VAPORS OF THE FOURTH EFFECT INDICATE, WITH THE EXCEPTION OF RUN No.1, A

TREND TOWARDS A CONSTANT REGION OF 0.0329 TO 0.0381 GMS PER 50 ML OF CONDENSED VAPOR. THIS SEEMS TO BE CONSTANT REGARDLESS OF THE METHOD OF FEED, FEED RATE, AND SMALL CHANGES IN VACUUM IN THE FOURTH EFFECT.

THE MAGNITUDE OF THE COMBINED LOSSES FROM THE CONDENSATE PUMP AND FROM THE SURFACE CONDENSER MAY BE ESTIMATED FROM THE OPERATING CONDITIONS OF RUN No.3 (TABLE I).

FEED --- 10,600 GALLONS PER HOUR AT 3% SOLIDS
= 10,600 x 8.33 x 0.03 = 2,650 POUNDS OF SOLIDS PER HOUR

WATER RATE TO SURFACE CONDENSER ------ 1,780 GALS/MIN

TEMPERATURE OF WATER IN ---------- 78°F.

TEMPERATURE OF WATER OUT ---------- 103°F.

B.T.U. INCREASE OF THE SURFACE CONDENSER IN CONDENSING VAPOR FROM EFFECT No.4 AT 110°F.,

= 1780 x 8.33 x 60 x (103-78) = 22,200,000 B.T.U./HR

POUNDS PER HOUR OF CONDENSATE FROM SURFACE CONDENSER,

$$= \frac{22,200,000 \text{ BTU/HR}}{1051.6 \text{ BTU/LB}} = 21,500 \text{ POUNDS PER HOUR}$$

OR $\frac{21,500 \text{ LBS/HR}}{8.33 \text{ LBS/GAL}}$ = 2,670 GALLONS PER HOUR

FEED CONCENTRATED FROM 3% TO 25% SOLIDS, OR

POUNDS OF 25% SYRUP PRODUCED PER HOUR

$$= \frac{2,650 \text{ LBS/HR}}{0.25} = 10,600 \text{ LBS PER HOUR}$$

POUNDS OF WATER EVAPORATED PER HOUR,

= (88,300 LBS FEED/HR - 10,600 LBS SYRUP/HR)

= 77,700 LBS/HR

Pounds of condensate delivered by condensate pump from effects No. 2, No.3 and No.4,

$$= (77{,}700 - 21{,}500) = 56{,}200 \text{ pounds per hour}$$

Loss of solids from condensate pump,

$$= \frac{0.0137 \text{ gms}}{50 \text{ ml}} \times \frac{1 \text{ lb}}{454 \text{ gms}} \times \frac{1000 \text{ ml}}{0.264 \text{ gal}} \times \frac{1 \text{ gal}}{8.33 \text{ lbs}}$$

$$= 0.000276 \text{ pound solids per pound of condensate}$$

Loss per hour from condensate pump,

$$= 56{,}200 \text{ lbs/hr } (0.000276 \text{ lbs/lb}) = \frac{15.5 \text{ lbs solids}}{\text{hour}}$$

Loss from surface condenser,

$$= 0.0355 \text{ gms/50 ml} = \frac{0.000714 \text{ pounds solids}}{\text{pound of condensate}}$$

Loss per hour from surface condenser,

$$= 0.000714 \times 21{,}500 \text{ pounds per hour} = \frac{15.3 \text{ lbs solids}}{\text{hour}}$$

The calculated magnitude of solids lost from the condensate pump and surface condenser, using average values of 0.0137 gm/50ml and 0.0355 gm/50ml respectively, indicates some definite relationship between pounds of condensate from condensate pump and surface condenser to their respective solids losses. Within a region of relatively close approximation the relationship may be expressed by

$$A/B = a/A$$

where A is the pounds of condensate from condensate pump
B is the pounds of condensate from surface condenser
a is the pounds of solids loss per pound of condensate from the surface condenser
A is the pounds of solids loss per pound of condensate from the condensate pump

The relatively narrow range of pH values for both condensate pump and surface condenser condensates may be attributed to the buffer action of the dissolved salts present in the entrained liquor droplets.

The rotary steam tube dryer stack losses tabulated in Table II show little consistency. The sampling runs made with the steam jet open to the stack to provide an induced draft through the dryer with the resultant decrease in solids loss indicates the presence of other variables which have greater influence upon the entrainment of the dust through the dryer than the opening or closing of the steam jet. The most apparent uncontrollable variable is the wind across the roof of the Foods and Feeds building. The mean value for the solids loss through the dryer stack may be taken as 0.0024 gms per cubic foot for the determination of the solids lost up the dryer stack per hour. With the steam jet closed, the stack gas velocity was 2595 cu.ft per minute (20°C., 1 atm). The loss per hour is then

$$= \frac{2595 \text{ cu.ft}}{\text{min}} \times 60 \times \frac{0.0024 \text{ gm}}{\text{cu.ft.}} \times \frac{1 \text{ lb}}{454 \text{ gms}}$$

$$= 0.825 \text{ pounds per hour}$$

The pH of 4.5 to 5.9 with no detectable titratable acids in the case of the water absorbents for the rotary dryer stack gases is readily understood from the definition of pH. Since the $(H^+) = 10^{-pH}$, the concentration of the H^+ ion with a pH of 5 would have to be 0.00001 mol per liter.

The drum dryer stack losses tabulated in Table IV indicate a narrow range of fluctuation in the solids content of the stack vapors. An overall range would be between 0.002 to 0.004 gm solids per cu.ft. This narrow range of solids losses may be attributed to the constant stream of air which is blown over the dried sheet and to the dust particles which are generated as the dried sheet is broken up in the conveyor beside the drums.

With a stack gas velocity of 3390 cu.ft. per min., the drum dryer stack losses may be determined.

$$\text{Loss per hour} = \frac{0.0029 \text{ gms}}{\text{cu.ft.}} \times \frac{3390 \text{ cu.ft.}}{\text{min}} \times \frac{1 \text{ lb}}{454 \text{ gms}}$$

$$= 1.305 \text{ pounds solids loss per hour}$$

The solids from the flashed cooker steam line, as tabulated in Table V, indicates a range of values from 0.000371 to 0.000523 gm of solids per gram of distillate. Within this narrow range a mean value of 0.00045 gm per gm of distillate may be taken. The loss of solids from the entrainment chamber is relatively small, particularly so when consideration is taken of the great pressure and temperature differences between the cooker U-tube and flash chamber No.1, the high velocity of the cooked mash from the discharge of the U-tube into the flash chamber No.1, and the excessive use of steam during the cooking process. A typical set of cooker operating data will aid the discussion at this point.

Backset rate to precooker ---------- 25 gal per min

Weight of grain to precooker ---------- 450 lbs per min

Water rate to precooker ---------- 75 gal per min

Temperature in precooker ---------- 130°F.

Temperature in U-tube ---------- 360°F.

Pressure of steam to jet heater ---------- 185 lbs/sq.in.

Metered steam flow rate to jet heater ------- 24,000 lbs/hr

Temperature in flash chamber No.1 ---------- 150°F.

Material and heat balance:

Specific heat of precooked slurry,

Weight of grain x 0.35 = 450 x 0.35 = 157.5 BTU/min/°F.
Weight of water and backset x 1.00 = 833.0 BTU/min/°F.
990.5

Specific heat of precooked slurry = $\frac{990.5}{1283}$ = 0.771 BTU/lb/°F.

Latent heat of 185 lbs/sq.in. gage steam ------ 843 BTU/lb

Temperature of 185 lbs/sq.in. gage steam ------ 381.8°F.

Pounds per hour of steam needed to raise slurry to 360°F.

$$= \frac{12{,}831 \text{ lbs/min} \times 0.771 \times (360-130) \times 60}{(843 \nearrow 21.8)}$$

= 15,800 pounds per hour

Assuming the steam flow meter to be accurate to 1%, the excess steam used is (23,760 - 15,800) = 7,960 lbs/hr.

The flow of excess steam to the cooker jet heater may be explained by the following conditions:

(1) The steam flow is automatically controlled by the temperature existing at the discharge end of the cooker U-tube.

(2) At the discharge end of the U-tube is a manually controlled

COCK WHICH IS USED TO CONTROL THE FLOW FROM THE U-TUBE TO THE FLASH CHAMBER.

WHEN THE MANUALLY CONTROLLED COCK IS OPENED TO A POINT GREATER THAN THAT NECESSARY TO ATTAIN COOKING TEMPERATURE OF 360°F. FOR A PERIOD OF ONE MINUTE COOKING INTERVAL THE TEMPERATURE OF THE DISCHARGE END OF THE U-TUBE WILL DROP AWAY FROM THE CONTROL POINT SET ON THE TEMPERATURE RECORDER OF THE AUTOMATIC CONTROLLER INSTRUMENT. THIS DROP IN TEMPERATURE FROM THE CONTROL POINT CAUSES, BY MEANS OF THE CONTROLLER MECHANISM, TO OPEN THE AUTOMATIC STEAM VALVE TO PERMIT ADDITIONAL STEAM TO FLOW INTO THE JET HEATER IN ORDER TO BRING THE TEMPERATURE AT THE DISCHARGE END OF THE U-TUBE UP TO 360°F. SINCE THE SETTING OF THIS MANUALLY CONTROLLED COCK VARIES CONSIDERABLY, AN EXCESS OF STEAM IS PRESENT.

HOWEVER, ASSUMING THAT 24,000 LBS/HR OF STEAM IS BEING USED, THE QUANTITY OF FLASHED STEAM GENERATED IN THE FLASH CHAMBER No.1 MAY BE CALCULATED.

LET M = WEIGHT OF COOKED MASH ENTERING FLASH CHAMBER No.1

= (76,980 LBS/HR + 24,000 LBS/HR) = 100,980 LBS/HR

LET F = POUNDS OF FLASHED VAPOR GENERATED IN FLASH CHAMBER AT TEMPERATURE OF 150°F.

LATENT HEAT OF VAPORIZATION AT 105°F = 1008.2 BTU/LB

THEN, (M - F) = WEIGHT OF MASH LEAVING THE FLASH CHAMBER

THE SPECIFIC HEAT OF THE MASH ENTERING THE FLASH CHAMBER:

WEIGHT OF GRAIN ---------- 27,000 LBS
WEIGHT OF WATER ---------- 73,980 LBS

BTU GAINED BY GRAIN -------- 27,000 x 0.35 = 9,450
BTU GAINED BY WATER -------- 73,980 x 1.00 = 73,980
83,430

SPECIFIC HEAT OF MASH = 83,430/100,980 = 0.829

HEAT BALANCE REFERRED TO A DATUM PLANE OF 150°F.:

$0.829 \times M(360 - 150) = F(1008.2) + (M - F)(150 - 150)c_{M-F}$

$0.829 \times 100{,}980 \times 210 = 1008.2F$

$F = 17{,}500$ LBS PER HOUR

THE SOLIDS LOST IN THE FLASHED COOKER STEAM LINE IS THEREFORE,

$\frac{0.00045 \text{ GM SOLID}}{\text{GM DISTILLATE}} \times \frac{17{,}500 \text{ LBS}}{\text{HOUR}} = 7.88$ POUNDS PER HOUR

THE MALODOROUS VAPOR PRODUCED IN THE COOKER FLASHED STEAM IS A PRODUCT RESULTING FROM THE HIGH TEMPERATURE COOKING AND SUBSEQUENT IMMEDIATE FLASH WASHING PROCESS. IT MAY BE THE RESULT OF EITHER OR A COMBINATION OF THE TWO SEPARATE CONDITIONS. IT WAS BELIEVED THAT THE MALODOR MAY BE A DECOMPOSITION PRODUCT OF A NITROGENOUS NATURE. THIS WAS DISPROVED BY THE NEGATIVE PROTEIN DETERMINATIONS MADE ON THE STRAW-COLORED 0.1 N NaOH ABSORBENT. ALTHOUGH NO INVESTIGATION HAS BEEN MADE TO DETERMINE THE EXACT NATURE OF THE MALODOR GENERATED, IT IS NOW BELIEVED THAT THE OFFENSIVE ODOR MIGHT BE A SULFUR COMPOUND.

THE VAPORS ESCAPING FROM THE SPIRITS BEER STILL STEAM EJECTOR VENT CONTAINED NO SOLIDS. THIS RESULT WAS EXPECTED IN VIEW OF THE TORTUOUS PATH THE "NON-CONDENSIBLES" MUST FOLLOW THROUGH THE DEPHLEGMATOR, BAROMETRIC CONDENSER,

and two-stage steam ejectors and intercondensers. However, as presented in Table VI, all "non-condensibles" are not non-condensible, that is, there is a portion of the vapors escaping up the vent which can be condensed at room conditions. The distillate has a high vapor pressure for a decrease in pressure on the distillate receiver accelerated evaporation of the distillate at room temperatures.

The offensive odor first experienced in the flashed cooker steam was present in the distillate from the ejector vent when the spirits beer still was receiving flashed cooker steam. Further investigations on the spirits beer still proved that the malodorous vapor present in the vent is only a portion of that entering the beer still, the remaining portion being present in the alcohol distillate in the dephlegmator.

The preliminary sampling runs, No.7 through No.15, of the spirits beer still vapors indicated the need for a concentrated investigation and if possible, an indicated trend of solids loss with such variables as feed rate, steam flow rate and general operating conditions of the beer still. Sampling runs No. 16 through No.30 were made under the conditions listed in Table VII, in an attempt to obtain sufficient data for correlation of results. An auxiliary table of results derived from Table VII, is presented in Table XI. For each feed rate, the vapor velocity was determined from the mean value of the per cent alcohol in the vapor, temperature at top of the still, and vacuum existing at the top of the still.

THE MEAN VALUES OF THE SOLIDS CONTENT OF THE CONDENSED VAPOR WAS ALSO DETERMINED FOR EACH FEED RATE AND EXPRESSED AS GRAMS PER 100 ML OF DISTILLATE AND POUNDS PER PROOF-GALLON. A RATIO OF SOLIDS CONTENT EXPRESSED AS GRAMS PER 100 ML TO VAPOR VELOCITY EXPRESSED AS CUBIC FEET PER SECOND WAS ALSO FORMED.

TABLE XI

SPIRITS BEER STILL SOLIDS LOSS (RUNS 7 - 30)

Feed Rate Gals/Min	Vapor Velocity Cubic Feet/Sec (A)	Solids Loss gms/100ml (B)	Solids Loss Lbs/P-Gal	Ratio B/A x 10^{-4}
100	1040	0.2186	0.0507	2.10
145	1615	0.2636	0.0644	1.63
150	2180	0.7539	0.2590	3.46
170	1780	0.3231	0.0694	1.82
175	1605	1.0511	0.2253	6.55
180	930	0.3899	0.0528	4.19

THE RESULTS PRESENTED IN TABLE XII WERE DERIVED FROM TABLE VIII AND PRESENTED IN THE SAME MANNER AS TABLE XI.

TABLE XII

SPIRITS BEER STILL SOLIDS LOSS (RUNS 31 - 57)

Feed Rate Gals/Min	Vapor Velocity Cubic Feet/Sec (A)	Solids Loss gms/100ml (B)	Solids Loss lbs/p-gal	Ratio B/A x 10^{-4}
110	1175	0.5601	0.1789	4.77
130	1105	0.5267	0.1351	4.77
138	1410	0.5110	0.1404	3.62
153	1405	0.4673	0.1069	3.33
160	1530	0.2871	0.0796	1.88
170	1775	0.5437	0.1618	3.06
180	1890	0.4537	0.1208	2.40

From a study of the individual sampling runs in Table VII and VIII, it is readily observed that within the same feed rate, with the beer feed composition relatively constant from one fermenter to another, the alcohol concentration of the vapor differs from one run to another. The beer feed recorder charts and the steam flow recorder charts indicated constant values of feed rate and steam flow at the respective control points. The runs made with the beer still using flashed cooker steam, Table VII, not asterisked, the steam flow rate could not be measured or controlled. Any change in cooker operation of reasonable magnitude could be expected to reflect in the operation of the beer still and possibly account for fluctuations in the alcohol concentration

OF THE DISTILLATES TABULATED IN TABLE VIII. THE ONLY PLAUSIBLE CONCLUSION THAT MAY BE DRAWN FROM THESE OBSERVATIONS IS THAT STEADY FLOW CONDITIONS DO NOT EXIST IN THE SPIRITS BEER STILL. IT IS ALSO RECOGNIZED THAT ALTHOUGH THE FEED RATE TO THE STILL, STEAM FLOW RATE, AND VACUUM AT THE TOP OF THE STILL ARE CONTROLLED WITHIN THE ACCURACY OF COMMERCIAL CONTROLLERS, NOTHING IS KNOWN OF THE EFFECTIVENESS OF THE INDIVIDUAL SPRAYS, THE PATTERN OF DISTRIBUTION OF A PARTIALLY PLUGGED SPRAY, THE CONDITION OF THE WOOD GRIDS, THE EFFECTIVE DISTRIBUTION OF THE BEER OVER AND THROUGH THE WOOD GRIDS, WHAT HOLD-UP, IF ANY, EXISTS, AND OTHER FACTORS WHICH HAVE BEEN OVER-LOOKED BUT WHICH MAY BEAR EQUAL WEIGHT TO OR GREATER WEIGHT THAN THOSE INVESTIGATED.

IT IS BECAUSE OF THIS UNSTEADY FLOW STATE WHICH DEFINITELY EXISTS WITHIN THE STILL THAT LITTLE CORRELATION OF THE DATA IS POSSIBLE. THE RESULTS OBTAINED FROM SAMPLING RUNS No.31 THROUGH No.57 PRESENTED IN TABLE VIII TEND TO CONCENTRATE WITHIN A NARROWER REGION THAN DO RUNS No.7 THROUGH 30. WITH THE EXCEPTION OF RUNS 47 THROUGH 50 MADE WITH A FEED RATE OF 160 GALS/MIN, THE SOLIDS LOSS PER 100 ML OF DISTILLATE RANGES ABOUT THE MEAN VALUE OF 0.5104 GRAM, AND 0.1406 POUND PER PROOF-GALLON OF DISTILLATE. ALSO, FROM TABLE XII, EXCLUDING RUNS MADE AT A FEED RATE OF 160 GALS/MIN, THE RATIO OF SOLIDS LOSS PER 100 ML OF DISTILLATE TO THE VAPOR VELOCITY DECREASES WITH INCREASE OF FEED RATE AND WITHIN THE FEED RATE REGION OF 110 TO 180 GALS PER MINUTE THIS RATIO IS WITHIN THE

interval of 2.40 to 4.77 x 10^{-4}.

The solids present in the spirits beer still vapors consist of fine particles from undistilled beer. The particles are so fine that only a portion of these solids may be removed from suspension by centrifuging at 1750 R.P.M. It is believed that the only effective method of removing the entrained solids from the vapor is to install in place of the sixteen-inch deep packing at the top of the column, Figure 2, two or possibly three conventional tunnel-cap or bubble cap plates to wash the solids from the rising vapors. The installation of three liquid sealed plates would, at the most, increase the differential across the column six to eight inches of water or about eleven millimeters of mercury. The effectiveness of the existing four layers of grids with the top water spray is practically negligible. A close inspection of the top grid section revealed that a major portion of the grid area was partially plugged with some sections completely plugged with solids.

It is also believed that the beer feed inlet is located too close to the top of the still body. Reference to Figure 2, reveals that the spraying of the beer into a fine stream together with reasonably high vapor velocity of the rising vapors produces the inherent solids entrainment loss. The lowering of the beer feed spray nozzles with a deeper section of packing consisting of two sections with an intermediate free space is an alternate method to reduce entrainment. The wash plate method is preferred to the water-spray-grid method

IN THAT THE RISING VAPORS WITH THE ENTRAINED SOLIDS MUST COME IN CONTACT WITH THE LIQUID AND THUS BE WASHED. THE SPRAY-GRID METHOD HAS THE DISADVANTAGE OF INCOMPLETE WASHING OF ALL THE VAPORS AT ALL TIMES. WHATEVER METHOD IS USED, THE GRAIN PARTICLES MUST BE REMOVED FROM THE VAPORS BEFORE CONDENSATION AND INTRODUCTION TO THE PURIFYING COLUMN AND RECTIFYING COLUMN. SINCE THE SPIRITS BEER STILL IS OPERATED UNDER LOW VACUUM TO RETARD THE DECOMPOSITION OF THE STILLAGE WHICH TAKES PLACE AT TEMPERATURES EXPERIENCED IN AN ATMOSPHERICALLY OPERATED BEER STILL, THE PRESENCE OF GRAIN PARTICLES IN THE PURIFYING AND RECTIFYING COLUMNS, WHICH ARE OPERATED AT ATMOSPHERIC CONDITIONS, TENDS TO NULLIFY THE ADVANTAGES DERIVED FROM THE LOW TEMPERATURE DISTILLATION ACCOMPLISHED IN THE SPIRITS BEER STILL.

THE MAGNITUDE OF THE SOLIDS LOSS PER HOUR FROM THE SPIRITS BEER STILL MAY BE ESTIMATED FROM A DAY'S TYPICAL MASHING RATE. THE SAME MASHING RATE USED IN THE FLASHED COOKER STEAM CALCULATIONS WILL SERVE ADEQUATELY AS THE BASIS FOR THE BEER STILL. IT IS TO BE NOTED THAT THE TOTAL BEER FEED IS DIVIDED BETWEEN THE SPIRITS BEER STILL AND THE WHISKEY BEER STILL.

Grain to precooker	450 lbs per min
Weight of malt for conversion (10% of grain)	45 lbs per min
Total grain to system	495 lbs per min

Taking as a basis one bushel of grain at 12% moisture to weigh 56 pounds, the total mashing rate over a 20 hour mashing

DAY = 495 LBS/MIN x 1 BU./56LBS x 1200 MIN/20 HR DAY

= 10,620 BUSHELS

ON THE BASIS OF A BEER FEED CONCENTRATION OF 35 GALLON BEER PER BUSHEL, THEN OVER A PERIOD OF 24 HOURS OF CONTINUOUS DISTILLATION, THE BEER FEED RATE IS,

$$= \frac{10{,}620 \text{ BUSHELS}}{24 \times 60} \times \frac{35 \text{ GALS}}{\text{BUSHEL}} = 258 \text{ GALS/MIN}$$

IF THIS BEER FEED IS DIVIDED BETWEEN THE SPIRITS BEER STILL AND THE WHISKEY BEER STILL IN A TYPICAL PLANT OPERATING RATIO SUCH THAT THE BEER FEED TO THE SPIRITS BEER STILL IS 180 GAL/MIN AND THE FEED TO THE WHISKEY BEER STILL IS 78 GAL/MIN, THE NUMBER OF BUSHELS DISTILLED IN THE SPIRITS BEER STILL PER HOUR

$$= \frac{180 \text{ GAL/MIN} \times 60}{35 \text{ GAL/BUSHEL}} = 309$$

ASSUMING AN AVERAGE ALCOHOL YIELD OF 5.0 PROOF-GALLONS PER BUSHEL MASHED, THE SOLIDS LOSS FROM THE SPIRITS BEER STILL MAY BE DETERMINED USING THE MEAN VALUE OF 0.1406 LBS SOLIDS PER PROOF-GALLON,

$$= \frac{309 \text{ BUSHELS}}{\text{HOUR}} \times \frac{5.0 \text{ PROOF-GAL}}{\text{BUSHEL}} \times \frac{0.1406 \text{ LBS}}{\text{PROOF-GAL}}$$

= 217 POUNDS OF SOLIDS PER HOUR

The samples of precooked slurry removed from the precooker and the samples of pressure-cooked and flashed mash removed from the flash chamber No.1 were obtained primarily to investigate a suitable basis upon which to develop the practical "theoretical" dried grain yield. The results of the investigation of the solids in the precooker and flash chamber presented in Table IX, revealed that the automatic scale readings of the weighed grain to the precooker were not accurate enough for the dried grain determination. This lack of accuracy was attributed to the manner in which the grain was weighed. The whole grain was fed into a counter-balanced hopper of known weight and when full, was dumped to Merchen continuous feeders which fed the grain to the mills. Each dump of grain was recorded and the number of dumps over a period of time multiplied by the weight of each dump divided by the period of time was the mashing rate. The accuracy of this method was none too good, for a dump might have been recorded before or after an assigned interval of time.

The decrease in the per cent solids between the precooked slurry and the flashed-cooked mash is attributed to the poor conditions of vacuum in the flash chamber. This poor vacuum level resulted in the decrease of flashed cooker steam from the cooked mash.

The investigation further revealed that there is an increase in starch content of the mash after flash pressure cooking. This result may indicate that (a) the A.O.A.C.

Diastase-Acid Hydrolysis method is incomplete, (b) the instantaneous flash after pressure cooking is instrumental in the liberation of latent starch granules, or (c) the flash pressure cooking process decomposes the grain during cooking to produce some aldehydes or other materials capable of reducing Fehling's solutions.

It was decided that the most accurate and reliable basis for the dried grain yield determination was the starch content of the converted mash going into the fermenter.

The results obtained from the series of laboratory fermentations upon plant processed mash which had been inoculated with plant yeast indicates that at the end of a forty-four hour fermentation period, no change was observed in the alcohol produced in the fermenter and no change in the dried grain residue in the fermenter. This is derived from the constant values of fermentation efficiency and per cent dried grain recovered.

The values of per cent starch in the converted mash to the fermenter, fermentation efficiency, and per cent dried grain recovered were plotted as shown in Figure 10. The method of plotting used was (a) draw a guide line connecting the per cent starch to fermentation efficiency, (b) draw a vertical up to this guide line from the value of per cent dried grain recovered (c) at the intersection of the two lines mark a point. From this curve it is now possible to obtain the practical "theoretical" dried grain yield knowing

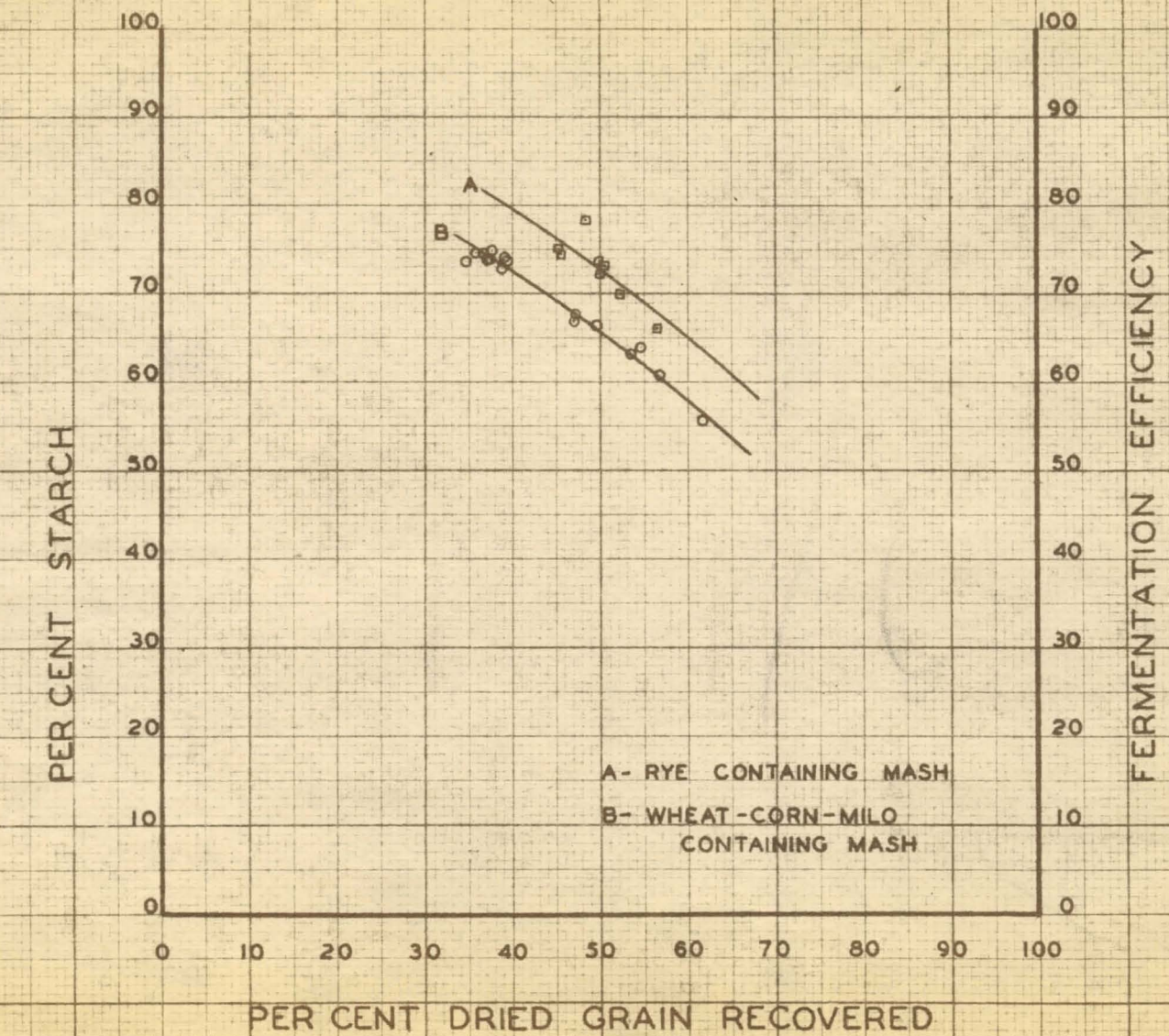

FIGURE 10 - GRAIN RECOVERY CORRELATION

THE PER CENT STARCH ON A BONE DRY BASIS IN THE MASH TO THE FERMENTER, THE SOLIDS CONTENT OF THE ENTERING GRAIN, THE FERMENTATION EFFICIENCY, AND THE SOLIDS CONTENT OF THE RECOVERED DRIED GRAIN. AT THE END OF FORTY-FOUR HOURS OF FERMENTATION, THE MEAN VALUE OF THE PER CENT DRIED GRAIN RECOVERED FOR WHEAT-CORN-MILO CONTAINING MASHES WAS 37.4% AT 73% STARCH AND 76% FERMENTATION EFFICIENCY.

TO CONVERT THIS RESULT TO A POUNDS PER BUSHEL BASIS, ASSUME ONE BUSHEL OF GRAIN AT 12% MOISTURE CONTENT WEIGHS 56 POUNDS AND THE MOISTURE CONTENT OF THE DRIED GRAIN IS 9%. UNDER THE CONDITIONS OBTAINED IN THE DETERMINATIONS OF 73% STARCH TO FERMENTER WITH A FERMENTATION EFFICIENCY OF 76% BASED ON THE PER CENT STARCH TO THE FERMENTER, THE DRIED GRAIN YIELD SHOULD BE, NEGLECTING ALL LOSSES INVESTIGATED ABOUT THE DISTILLERY,

$$= \frac{56 \text{ LBS}}{\text{BUSHEL}} \times 0.88 \times 0.374 = 18.45 \text{ LBS ON A BONE DRY BASIS}$$

OR, $\frac{18.45}{0.91} = 20.3 \frac{\text{POUNDS OF DRIED GRAIN AT 9\% MOISTURE}}{\text{BUSHEL OF GRAIN AT 12\% MOISTURE}}$

SUMMARY AND CONCLUSIONS

From the overall investigation of the Joseph E. Seagram & Sons, Inc. Louisville Distillery, the following average solids losses were found with a mashing rate of 530 bushels per hour:

Evaporator surface condenser	15.3 lbs/hr
Evaporator condensate pump	15.5 lbs/hr
Rotary steam tube dryer stack	0.83 lbs/hr
Drum dryer stack	1.31 lbs/hr
Flashed cooker steam	7.88 lbs/hr
Spirits beer still -- (309 bushels/hr)	217.00 lbs/h
Total	257.82 lbs/hr

or $\frac{257.82 \text{ lbs solids per hour}}{309 \text{ bushels per hour}}$ = 0.835 pounds per bushel

The spirits beer still is the greatest contributor to solids loss and the loss is great not only in dried grain but greater still in the deleterious effect the presence of grain in the atmospherically operated stills must have upon the quality of the spirits.

The practical "theoretical" dried grain yield was determined to be 20.3 pounds of dried grain at 9% moisture from a bushel of grain weighing 56 pounds at 12% moisture.

The Foods and Feeds Department can be expected to recover (20.3 - 0.84/0.91) or 19.4 pounds of dried grain per bushel under the present operating conditions as compared with the usual recovery of 17 to 18 pounds.

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APPENDIX

SAMPLE CALCULATIONS

ROTARY STEAM TUBE DRYER - RUN NO.3

VOLUME COLLECTED - 8 LITERS AT 5.6 INCHES H_2O VACUUM, 21°C.
8 LITERS AT 5.6 INCHES H_2O VACUUM, 21°C.
8 LITERS AT 5.6 INCHES H_2O VACUUM, 21°C.
24 LITERS

VOLUME COLLECTED, CORRECTED TO 760 MM Hg AND 20°C.

$$24 \times \frac{293}{294} \times \frac{749.6}{760} = 23.6 \text{ LITERS}$$

WEIGHT OF GLASS WOOL FILTER NO.3 PLUS RESIDUE -- 23.1310 GMS
WEIGHT OF GLASS WOOL FILTER NO.3 EMPTY ---------- 23.1291 GMS
WEIGHT OF SOLIDS RESIDUE ---------- 0.0019 GMS

$$\text{LOSS} = \frac{0.0019 \text{ GM}}{23.6 \text{ LITERS}} \times \frac{28.32 \text{ LITERS}}{\text{CUBIC FOOT}} = 0.00228 \text{ GM/CU.FT}$$

VOLUME OF VAPOR ESCAPING FROM THE ROTARY STEAM TUBE DRYER STACK

DIAMETER OF STACK ---------- 30 INCHES
AREA ---------- 4.89 SQ.FT.

MEAN VAPOR VELOCITY IN STACK ----- 636 FEET PER MIN AT 81°C.

CORRECTED VOLUME PASSING UP STACK TO 760 MM Hg AND 20°C.

$$(636 \times 4.89) \times \frac{293}{354} = 2595 \text{ CU.FT/MIN}$$

DRUM DRYER RUN NO. 4

FLOW METER DIFFERENTIAL ---------- 4.2 CMS Hg
EQUIVALENT VAPOR RATE ---------- 0.130 CU.FT/MIN, 760MM, 22°C
VACUUM ---------- 12.3 INCHES WATER
TEMPERATURE OF VAPOR ---------- 33°C.
DURATION OF RUN ---------- 30 MINUTES

WEIGHT OF GLASS WOOL FILTER NO.4 PLUS RESIDUE --- 22.2709 GMS
WEIGHT OF GLASS WOOL FILTER NO.4 EMPTY ---------- 22.2560 GMS
WEIGHT OF SOLIDS RESIDUE ---------- 0.0149 GM

VOLUME OF VAPOR CORRECTED TO 760 MM Hg AND 20°C.

$$3.90 \text{ CU.FT} \times \frac{293}{306} \times \frac{32.9 \text{ FT OF WATER}}{33.9 \text{ FT OF WATER}} = 3.69 \text{ CU.FT}$$

$$\text{LOSS OF SOLIDS} = \frac{0.0149 \text{ GM}}{3.69 \text{ CU.FT}} = 0.0041 \text{ GM/CU.FT}$$

VOLUME OF VAPOR ESCAPING UP THE DRUM DRYER STACK,

DIMENSIONS OF STACK ---------- 32 INCHES BY 32 INCHES
CROSS-SECTIONAL AREA ---------- 5.43 SQ.FT

MEAN VELOCITY OF VAPOR UP STACK -- 693 FT/MIN AT 52°C.

VOLUME CORRECTED TO 760 MM Hg AND 20°C.

$$(5.43 \times 693) \times \frac{293}{325} = 3390 \text{ CU.FT/MIN}$$

SPIRITS BEER STILL RUN NO. 17

BEER FEED ---------- 180 GAL/MIN
STEAM SOURCE --------- FLASHED COOKER STEAM
WEIGHT OF DISTILLATE COLLECTED --- 739.2 GMS
SPECIFIC GRAVITY OF DISTILLATE --- 0.975 AT 60°F.
VOLUME OF ABSORBENT ---------- 200 ML OF 0.1017 N NAOH
SOLIDS CONTENT OF 100ML OF DISTILLATE --- 0.3287 GRAM

ALCOHOL DETERMINATION OF DISTILLATE:

USED 50 ML OF ORIGINAL SAMPLE PLUS 100 ML OF WATER.
COLLECTED 100 ML OF DISTILLATE

WEIGHT OF SPECIFIC GRAVITY BOTTLE WITH WATER AT 25°C -	82.7850
WEIGHT OF SPECIFIC GRAVITY BOTTLE EMPTY ----------	32.8938
WEIGHT OF VOLUME OF WATER ----------	49.8912
WEIGHT OF SPECIFIC GRAVITY BOTTLE WITH DISTILLATE, 25°-	81.8708
WEIGHT OF SPECIFIC GRAVITY BOTTLE EMPTY ----------	32.8938
WEIGHT OF VOLUME OF DISTILLATE AT 25°C. ----------	48.9770

SPECIFIC GRAVITY OF DISTILLATE AT 25°C. = $\frac{48.9770}{49.8912}$

= 0.9817

EQUIVALENT TO 14.72 % ALCOHOL BY VOLUME AT 60°F.

ACTUAL PER CENT ALCOHOL BY VOLUME OF ORIGINAL SAMPLE = 29.44

PROOF OF VAPOR ORIGINALLY CONDENSED IN SAMPLE = 58.88 AT 60°F

SOLIDS CONTENT OF DISTILLATE PER PROOF-GALLON OF DISTILLATE,

$$\frac{0.3287 \text{ GM}}{100 \text{ ML}} \times \frac{1 \text{ LB}}{454 \text{ GMS}} \times \frac{1 \text{ GAL}}{0.5888 \text{ P-GAL}} \times \frac{3785 \text{ ML}}{\text{GAL}}$$

= 0.0467 POUNDS PER PROOF-GALLON

ACID CONTENT OF NON-CONDENSIBLE VAPORS:

TITRATION OF 25 ML OF ABSORBENT REQUIRED 13.4 ML OF 0.1009 N H_2SO_4. OR 1.1905 MILLIGRAM-EQUIVALENTS.

MILLIGRAM-EQUIVALENTS OF ACID VAPOR PER PROOF-MILLILITER OF DISTILLATE = $\frac{1.1905 \times 8}{(739.2/0.975) \times 0.5888}$ = 0.0214

VAPOR VELOCITIES THROUGH THE SPIRITS BEER STILL

RUNS NO. 24 THROUGH NO.26

BEER FEED ---------- 180 GAL/MIN

ALCOHOL IN BEER ---------- 5.66% BY VOLUME --- 4.53% BY WT.

ALCOHOL CONCENTRATION IN VAPOR -- 15.54% BY VOL.-12.58% BY WT.

ASSUME NEGLIGIBLE LOSSES, ALL ALCOHOL LEAVES AT THE TOP OF THE STILL.

FEED INTO STILL = 180 GAL/MIN x 60 x 0.99 x 8.34

= 89,000 LBS/HR

ALCOHOL IN WITH FEED = 89,000 x 0.0453 = 4,030 LBS/HR

VAPOR LEAVING AT TOP = $\frac{4,030 \text{ LBS ALCOHOL/HR}}{0.1258 \text{ LB ALCOHOL/LB VAPOR}}$

= 32,100 LBS/HR

POUND MOLS OF ALCOHOL LEAVING = 4030/46 = 87.6 LB-MOL/HR

POUND MOLS OF WATER LEAVING = (32,100-4030)/18 = 1560

TOTAL NUMBER OF POUND MOLS LEAVING AS VAPOR = 1647.6

VACUUM AT TOP OF STILL ---------- 80 MM Hg

TEMPERATURE AT TOP OF STILL ----- 58°C.

VAPOR VELOCITY =

$$\frac{359 \text{ CU.FT}}{\text{LB-MOL}} \times \frac{(460+136.4)}{492} \times \frac{760 \text{ MM}}{80 \text{ MM}} \times \frac{1647.6 \text{ LB-MOL/HR}}{3600 \text{ SECS/HR}}$$

= 1890 CUBIC FEET/SEC

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VITA

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He attended Brooklyn public schools, Seth Low Junior High School and finished his secondary education in the New Utrecht High School in Brooklyn, New York. He received his Bachelor of Chemical Engineering degree from the College of the City of New York in September, 1938.

Mr. Brenbilder was employed in the U.S. Naval Inspection Service as a Junior Inspector of Naval Engineering Materials. In December, 1941, he resigned to accept employment in the Production Department of the Calvert Distilling Co., Relay, Maryland. In March, 1943, he was transferred to the Central Engineering Department of the Joseph E. Seagram & Sons, Inc., in the capacity of Supervisory Engineer. In November, 1944, he was awarded the Joseph E. Seagram & Sons, Inc. Fellowship at the University of Louisville Institute of Industrial Research.

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