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Project Summary

Testing and Evaluation of an Alcohol Production Facility Utilizing Potatoes as a Feedstock

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This study presents the sampling and analysis results for the characterization of the liquid effluents and solid residuals from a process in which culled potatoes are used as a feedstock for the production of ethanol fuel. Gaseous emissions were not studied. The facility, located in eastern Idaho, produces approximately 1 million gallons of ethanol per year. The effluents were sampled in December 1981.

Liquid and solid samples were taken from sluice/flume water, chopper product, makeup water, cooker product, fermenter product, beer tank, stillage, interim and final product, washwater, fusel oil, acid bath and Sparkle* bath. The effluents from the plant were analyzed for ethanol and sugar content, conventional parameters, metals, cyanide, phenols, nutrients, oil and grease, priority pollutant organics and selected pesticides. The effluents from this plant in general showed the following significant characteristics: oxygen demand (TOC, COD, BOD), solids (TSS, TS, specific conductance), nutrients (nitrogen and phosphorus) and metals (Al, Cd, Ca, Cr, Fe, Mg, Mn, Hg, Ti, and Zn).

This Project Summary was developed by EPA's Industrial Environmental Research Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

*Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Introduction

The U.S. Environmental Protection Agency (EPA) conducted a study of the fuel alcohol industry to determine the environmental impact of alcohol production from grain and waste products. As part of this study, the Industrial Environmental Research Laboratory in Cincinnati, Ohio (IERL-Ci) and the Effluent Guidelines Division (EGD) conducted sampling and analyses to characterize the air, water, and solid waste streams from alcohol facilities and to obtain treatability information.

Since many of the newer facilities incorporate feedstocks such as sugar beets, culled potatoes and fruit, and sweet sorghum, rather than the grain and waste products feedstocks analyzed in the past, EPA has begun to evaluate the environmental impacts of these nongrain feedstock facilities. This report describes the results of sampling and analyses of the liquid effluents and solid residuals from an ethanol facility utilizing one of these feedstocks - potatoes. The full report includes a description of the process at the potato facility and the environmental impact associated with the effluents, presents the techniques used to acquire the samples for analysis, outlines the analytical techniques employed, and presents the results. An appendix contains the quality assurance and quality control (QA/QC) results.

Process Description

The alcohol productions facility converts potatoes and grains into ethanol. During the sampling period of December 2, 3,

and 4, 1981, potatoes were used as the feedstock. Figures 1 and 2 are flow diagrams of the process.

Sampling and Analyses

The purpose of the sampling was to characterize solid and liquid discharges and potential discharge streams throughout the process. Table 1 indicates the analytical sample matrix and the following paragraphs discuss the sampling points and techniques used during the December 1981 sampling period.

Sluice/Flume Water Sample

The flume water was sampled at sampling point 15 as potatoes were transferred to the chopper tank. Four 1-liter grab samples were taken and composited. The water was sampled twice as it came off the conveyer belt and twice from the agitated catch basin below the conveyor. The flume water, which is reused through three batches, was to be discharged after the next batch.

Chopper Product

The chopped potatoes are transferred as a batch to a cooker tank. The valve located at the bottom of the batch tank, sampling point 1, was used for sampling.

Makeup Water

The site used an onsite water well. The tap located closest to the well pump (near the batch tank) was sampled after allowing the water to run for 10 minutes. This is sampling point 2.

Cooker Product

Four 1-liter samples of the cooker product were taken at sampling point 3, one at the beginning, two in the middle, and one near the end of the cycle.

Fermenter Product

The fermenter product was sampled at the sampling port on the side of the fermenter tank at sampling point 4. The sample was composited as the batch was being pumped to the beer tank. Two composite fermenter product samples were taken simultaneously. The first set (4C) was maintained on ice during and after sampling. The second set (4W) was maintained at ambient temperatures for 36 hours in an attempt to approximate the beer tank effluent. The samples are referred as "cold" and "warm," respectively, in this report. This sampling protocol was necessary due to the inaccessibility of the actual beer tank effluent (beer still feed) stream. Chemical analysis in the laboratory revealed few differences between the "cold" and "warm" fermenter product samples.

Beer Tank

The beer tank effluent sampling was minimal due to difficulties in reaching the effluent. Two 500-ml grab samples were taken 16 hours apart at sampling point 5.

Stillage

The three stillage (beer still bottoms) streams were sampled at sampling points 6 and 7. The excess stillage, or stillage overflow, (material which the solid-liquid separator [SOMAT] could not process) was sampled by compositing hourly for 10 hours. The solid material or thick stillage stream was sampled hourly for 10 hours at the SOMAT outlet. Also, one grab sample of the thin stillage stream was taken. This was sampled at the pipe where the SOMAT discharged the liquid to the main waste stream.

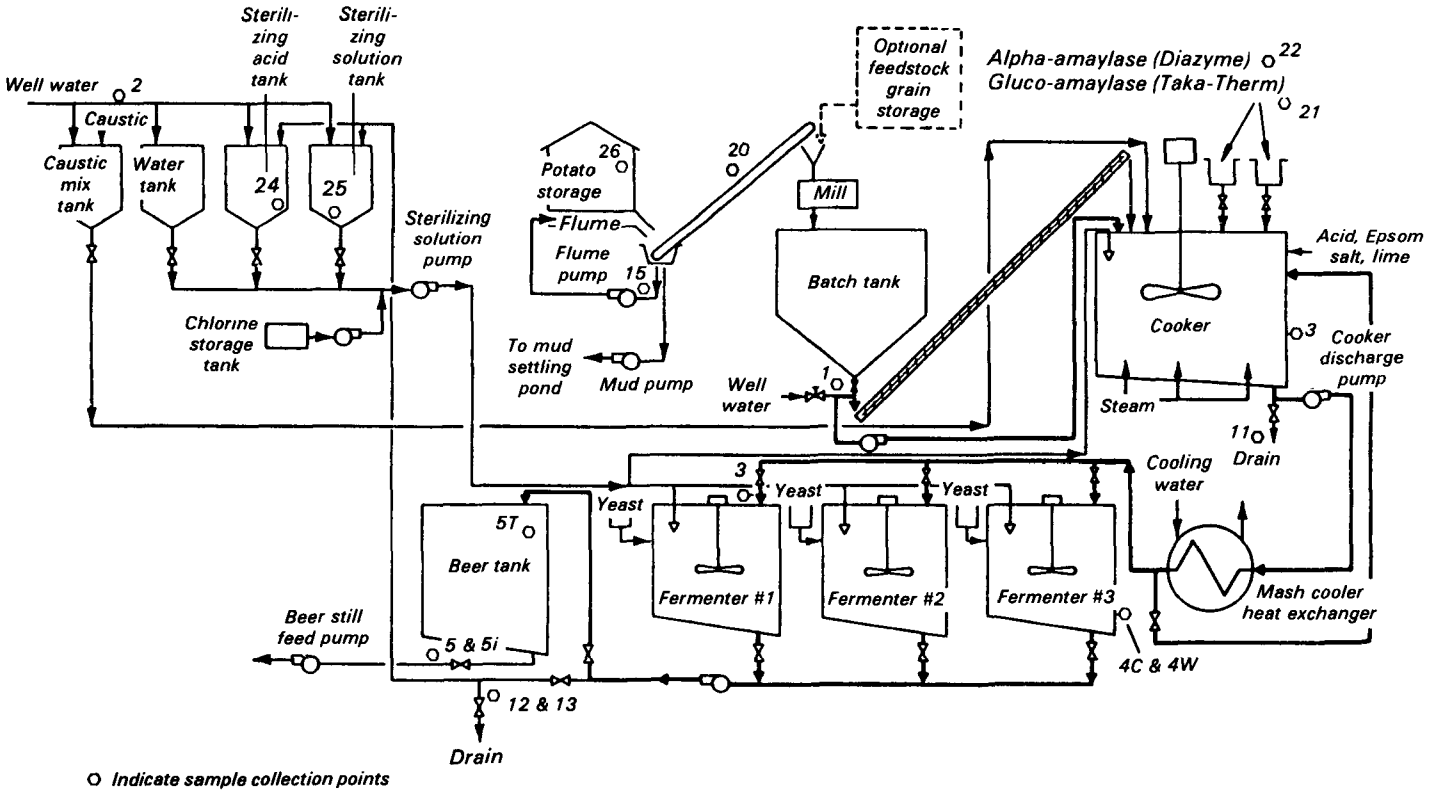

Figure 1. Feedstock processing schematic.

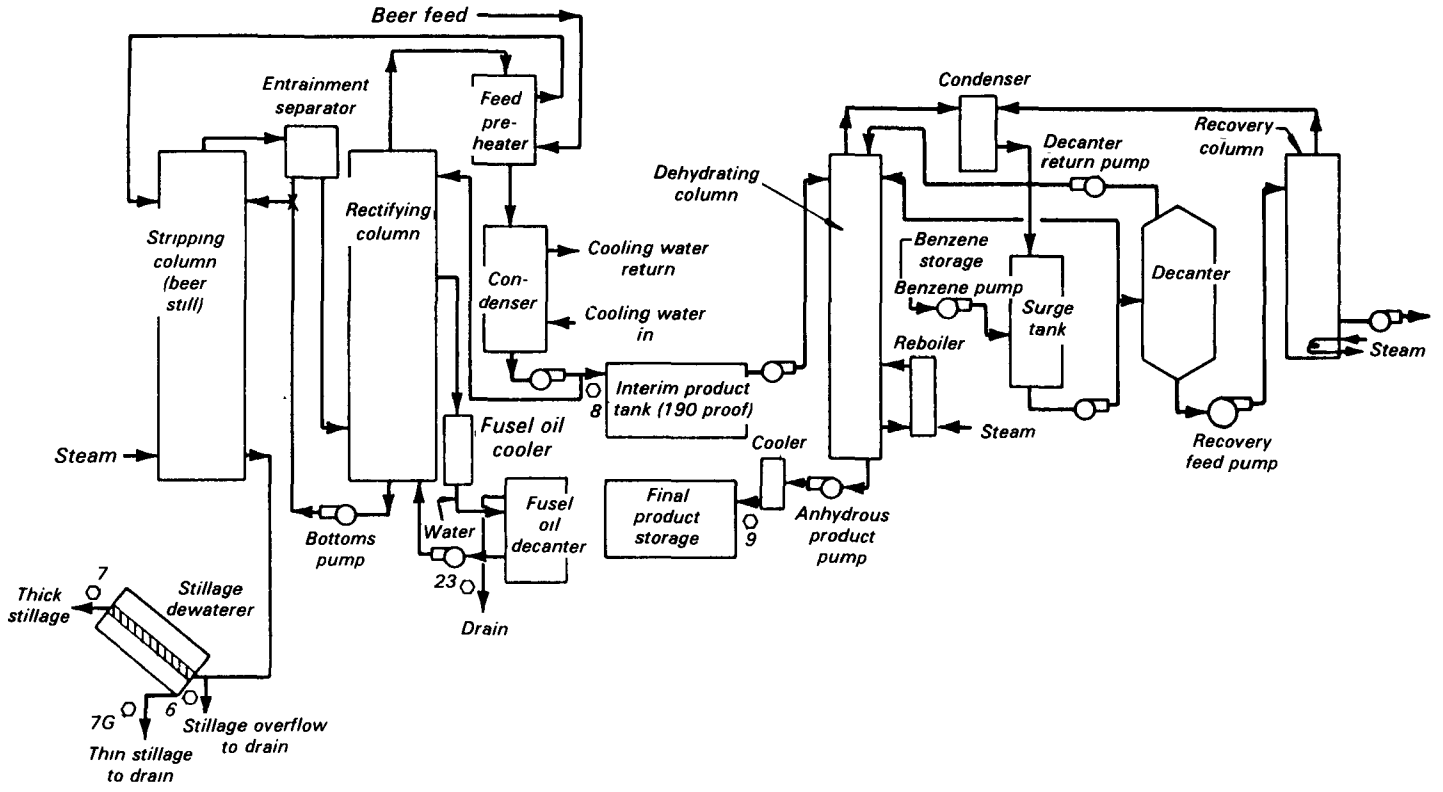

Figure 2. Distillation and dehydration process flow diagram.

Interim Product

The interim product (rectifier overhead) sample was composited hourly for 10 hours by taking 50-ml grab samples at a test valve, sampling point 8, located on the outside control panel. The composited sample was sealed and shipped to the laboratory by land carrier.

Final Product

During the sampling period, the dehydration system malfunctioned and no composite sample could be taken. The final product was, however, sampled from a product storage tank at sampling point 9. A 500-ml grab sample was placed in an amber glass bottle and shipped by land carrier to the laboratory.

Cooker Washout

The washout cycle for the cooker tank consists of three rinse-and-dump cycles; before, between, and after the two sterilizing bath cycles. The dump water for the three cycles was composited during one batch cleaning by taking two 400-ml grab samples three times during each dump. The sample was taken at the drain pipe sampling point 11. The three sets were composited, sealed, and placed on ice.

Fermenter Washout

The fermenter washout cycle for the fermenter tank is identical to the cooker washout cycle. The sampling and compositing methods were the same as those for the cooker washout sample. Samples were taken at point 12.

Combined Washout

After delivery to the laboratory, the two washout cycles were combined. Initially performed analyses were repeated on the combined sample with the exception of the coliform analysis.

Fusel Oil

A grab sample of the accumulated fusel oil was taken at sampling point 23 after the material was mixed for approximately 8 hours. This sample was broken in transit and was replaced with a sample from a later process run; results, therefore, are only generic rather than test specific.

Acid Bath

A 500-ml grab sample of the agitated acid bath was taken at sampling point 24 sterilization cycle. The sample batch was to be dumped the following day.

Sparkle Bath

A 500-ml grab sample of the agitated Sparkle bath was taken at sampling point 25 during the sterilization cycle. The sampled batch was dumped after a few days.

Combined Bath

After delivery to the laboratory, the two bath samples were combined. Subsequently, all initial analytical tests performed on the bath solutions were repeated on the combination sample.

Other Samples

Other samples were taken at various points for possible analysis should the mainstream analyses have shown poten-

Table 1. Analytical Sample Maxtrix

ID No.	Stream	Parameter																					
		BOD_5	COD	TOC	TSS	TS	pH	Conductivity	21 metals[a]	P.P. organics[b]	Oil and grease	Coliform	Phenolics	Cyanide	SO_4^{-2}	NO_3^{-1}	P, total	NH_3, TON[c], TKN	EtOH, R-H's	Sugar	Four pesticides	Density[d]
1	Chopper product	x	x	x		x	x														x	x
2	Makeup water	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x				
3	Cooker product	x	x	x	x	x	x														x	x
4C	Fermenter product (cold)	x	x	x	x	x	x													x	x	x
4W	Fermenter product (warm)	x	x	x	x	x	x													x	x	x
5i	1st beer tank effluent	x				x	x													x[e]		x
5	2nd beer tank effluent	x			x	x	x													x[e]		x
5T	Beer tank tops					x	x															
6	Excess stillage	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x
7	Thick stillage	x	x	x		x	x															
7G	Thin stillage	x	x	x	x	x	x	x														x
8	Distillation product																			x		
9	200-proof dehydration product																			x		
11	Washout cooker	x				x	x					x										x
12	Washout fermenter	x				x	x					x										x
13	Washout combination	x	x	x	x	x	x	x														x
15	Sluice water	x	x		x	x	x	x				x									x	
23	Fusel oil																			x		
24	Acid bath	x				x	x															
25	Sparkle bath	x				x	x															
27	Bath combination	x	x	x	x	x	x	x														
30	Field/lab blank	x	x	x	x	x		x	x	x	x	x	x	x	x	x	x	x	x	x	x	x

[a]*21 metals are for "total" analyses and are: Al, Sb, As, Ba, Be, Bi, Cd, Ca, Cr, Cu, Fe, Pb, Mg, Mn, Hg, Ni, Se, Ag, Tl, Ti, and Zn.*
[b]*Acid extractables, base/neutral extractables, and VOA — by fused silica capillary column, Pesticides (organochlorine) — GC/EC.*
[c]*TON, Total organic nitrogen derived from TKN less NH_3.*
[d]*Densities have been determined but are not reported. They are used for conversions from weight/weight units to weight/volume units (i e. mg/kg to mg/L).*
[e]*Ethanol only.*
x = Being measured.

tial problems. No unexpected results were noted, and additional analyses were not necessary.

Analytical Techniques

The analytical techniques employed were standard methodologies or optimized methods chosen from available alternatives. Table 2 presents the technique chosen for each parameter to be studied.

Approximately 40 percent of the analytical effort was involved with quality assurance/quality control (QA/QC) activities. Some features of the QA/QC procedures are summarized in the following paragraphs.

Upon completion of the sampling trip, the project chemist reviewed the analytical requirements with the sample custodian during the logging-in process. The chemists and analysts discussed with the laboratory supervisors alternative procedures before the analyses commenced. Thus, the samples requiring analysis and procedures to be followed were clearly specified at the outset of the analytical program.

The laboratory analysis portion of the QA/QC program included glassware preparation to reporting the results utilizing formalized procedures. Any alteration of procedures was reviewed with the project chemist before testing began.

The analytical QA/QC procedures entailed blank, duplicate, spike, method standard, and reference sample analyses. Calibration standards were of primary standard grade, NBS traceable, or certified purity.

To ensure the integrity of the reported results, the project chemist reviewed data with the analysts and discussed the results. Reported values were transcribed directly from the laboratory book to the handwritten laboratory draft. The typed draft for review was then checked against the original laboratory draft.

By using these specific procedures and several other integral parts of the laboratory QA/QC program, the quality of the analytical work was documented and assured.

Discussion of Analytical Results

Plant waste streams showed characteristics that could adversely impact the water quality of the receiving stream (Table 3). The most significant was oxygen demand (for all process and waste streams). This is apparent from the BOD, COD, and TOC results. Direct discharge of these streams could severely affect waters by depleting available oxygen.

The discharge of nutrients (e.g., free and bound nitrogen and phosphorus) can have significant adverse effects on the environment. The excess stillage stream, as would be expected, exhibited high nutrient content.

The pH throughout the system (liquid and solid stream) was in the range of 4.4 to 6.0 to facilitate the action of enzymes and yeast in the fermentation process. The effects of these wastes would depend

Table 2. *General Chemical Analysis*

Parameter	Method
BOD_5	5-day incubation, sample analyzed for oxygen depletion
COD	Acid dichromate reflux, back titrate with ferrous ammonium sulfate
TOC	Conversion to CO_2, infrared quantitation
Total suspended solids	Gravimetric, 105°C, weigh residue on filters
Total solids	Gravimetric, 105°C, weight of residue
Phenols (total)	Distill, aminoantipyrine color, $CHCl_3$ extraction
Cyanides (total)	Distill, barbituric acid colorimetry
Ammonia	Distill, followed by nesslerization
Nitrate	Brucine colorimetric
Sulfate	Turbidimetric
Phosphorus (total)	Nitric/sulfuric acid digest, ascorbic acid colorimetry
Specific conductance	Wheatstone bridge conductivity
Metals[a]	Atomic adsorption spectrophotometry following acid digestion. Analysis by cold-vapor flameless AA (Hg), flame and graphite furnace analyses as appropriate for others
pH	Electrometric
Total Kjeldahl nitrogen	Sulfuric acid mercuric oxide digestion, distillation, nesslerization
Oil and grease	Partition/gravimetric, freon extraction
Total and fecal coliform	Multiple tube fermentation (gas producers)
Sugars, reducing	Condense with orthotoluidine in acetic acid followed by spectrophotometry on liquid portion of samples

[a]*Metals: Al, Sb, As, Ba, Be, Bi, Ca, Cd, Cr, Cu, Fe, Pb, Mg, Mn, Hg, Ni, Se, Ag, Tl, Ti, and Zn.*

on the relation of their acidity to the buffering capacity of the receiving environment. A pH drop of any magnitude could result in aquatic population shifts and destruction, corrosion of metallic systems, and agricultural crop damage.

The results of the metals analyses indicate that the wastes have significant levels of several metals including aluminum, cadmium, calcium, chromium, copper, iron, magnesium, manganese, mercury, titanium and zinc. Without further testing the source of metals cannot be definitively isolated; however, most can be attributed to the materials added (e.g., Epsom salt) and to leaching from the process equipment (primarily stainless steel and aluminum). The levels of these metals ranged from 180 to 67,000 μg/liter in the makeup water and 7 to 176,000 μg/liter in the excess stillage.

No priority pollutant organics were detected in the samples. One unknown sulfur compound was detected at 12 μg/L

Table 3. *Values for Conventional Parameters (mg/L, Unless Noted Otherwise)*

		Parameter								
ID No.	Stream	BOD_5[a]	COD	TOC	TSS (103° to 105°C)	TS[b] (103° to 105°C)	pH[c] electrometric @ 25°C	Specific conductivity μmhos/cm @ 25°C	Total Coliform[f]	Fecal Coliform[f]
1	Chopper product	47,000	160,000	33,000	—	16% wt	5.50	—	—	—
2	Makeup water	<2	<5	3.3	<5	308	7.39	442	8.1	<2
3	Cooker product	107,000	209,000	57,500	18,000	18% wt	4.97	—	—	—
4C	Fermenter product (cold)	102,000	216,000	36,000	35,000	6.3% wt	4.88	—	—	—
4W	Fermenter product (warm)	102,000	201,000	34,000	32,000	6.1% wt	4.88	—	—	—
5i	1st beer tank effluent	59,000	—	—	—	5.8% wt	4.58	—	—	—
5	2nd beer tank effluent	54,000	—	—	24,800	6.2% wt	4.78	—	—	—
5T	Beer tank tops	—	—	—	—	10.7% wt	5.16	—	—	—
6	Excess stillage	20,000	59,800	22,000	22,000	5.5% wt	4.74	13,600	<20	<20
7	Thick stillage	25,000[g]	74,700[g]	29,000[g]	—	8.1% wt	4.65	—	—	—
7G	Thin stillage	18,000	54,800	21,000	24,700	54,000	4.67	14,900	—	—
11	Washout cooker	17,100	—	—	—	29,100	5 51	—	≥24,000	9,200
12	Washout fermenter	4,200	—	—	—	4,700	2.76	—	460	20
13	Washout combination	9,900	22,000	7,700	3,080	10,100	4.45[d]	1,950	—	—
15	Sluice water	1,980	7,500	—	11,200	12,200	6.90	1,130	≥24,000	270
24	Acid bath	780	—	—	—	1,750	6.64 4.99[e]	—	—	—
25	Sparkle bath	2,700	—	—	—	5,600	6.14 4.46[e]	—	—	—
27	Bath combination	2,200	4,600	1,600	880	3,100	5.43[d] 4.63[e]	1,020	—	—
30	Field/lab blank	<2	<5	1.9	NA	<10	—	<1	<2	<2

[a]*Most of the BOD determinations were performed a second time to bring results in a qualifiable range.*
[b]*Total solids method was 24 hr at 103°-105°C.*
[c]*First pH determinations were performed on 12/5/81.*
[d]*Combinations were measured on 12/9/81.*
[f]*Rechecks were measured on 2/22/81.*
Units are MPN/100ml (most probable no. per 100ml).
[g]*mg/kg.*

in the flume water. With the exception of alcohols, no compounds of significance were detected in the samples, including in the fusel oil.

Bacteriological quality is of concern from the flume water and washout waters. Coliform analyses are performed as indicated tests for possible health-related organisms such as fecal streptococcus. More testing would be required to preclude bacterial sources other than the feedstock itself.

Conclusions and Recommendations

The preliminary results of this sampling and analysis are consistent with those of other fuel alcohol plants using a variety of other feedstocks. Thus, the problems to be addressed and methods of addressing them may be similar.

The effluents from this plant contain the following water quality variables and pollutants that could degrade receiving waters:

- Oxygen demand (TOC, COD, BOD)
- Solids (TSS, TS, specific conductance)
- Nutrients (nitrogen and phosphorus)
- Metals (Al, Cd, Ca, Cr, Cu, Fe, Mg, Mn, Hg, Ti and Zn)
- Bacteria (total and fecal coliform)
- Corrosivity, low pH

Although primary concern for environmental impacts should be with those normal discharges from the process, one must additionally be concerned with the "dumping" of poor batches and the disposal of the solid materials other than by byproducts use (i.e., as a feedstock).

The low pH of the liquid stream potentially has two effects:

- adverse effect on crops if used in land farming
- corrosion of process equipment

It would appear that these streams should be neutralized to a pH of 6 to 8 as early as possible. Since the acidic level is required for the fermentation process, a realistic point for neutralization would be downstream of the fermentation tank in the beer tank. The chemical (a buffer salt) used for neutralization at this point must be benign with respect to the byproduct use.

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***Mary Ann Curran** is the EPA Project Officer (see below).*
The complete report, entitled "Testing and Evaluation of an Alcohol Production Facility Utilizing Potatoes as a Feedstock," (Order No. PB 84-187 962; Cost: $10.00, subject to change) will be available only from:
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