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RECOVERY OF FREE AMINO COMPOUNDS FROM POTATO
STARCH PROCESSING WATER BY USE OF ION EXCHANGE.
II. LARGE-SCALE LABORATORY EXPERIMENTATION.¹

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The authors have previously reported an ion-exchange method for concentrating the free amino compounds in dilute potato extract simulating starch factory processing water (3). In this method, the solubles were extracted from ground, sulfited potatoes by centrifuging and washing the centrifuge cake with water equal to one-third the weight of potatoes ground. The protein was removed by heat coagulation to prevent clogging during subsequent passage of the liquor through the ion-exchange column. The amino compounds were concentrated and separated from most of the other potato solubles by absorption on a 300 ml column of Dowex 50³ resin (H⁺ form) followed by elution with two normal NH₄OH. In a typical experiment, 28% of the total solids and 75% of the amino compounds originally present in six liters of the dilute liquor were recoverable in 350 ml of eluate combined from the more concentrated middle fractions. The most concentrated fraction contained 8.3% solids and the combined middle fractions contained 4.7% solids.

This note presents results obtained with a column of 7½ cm inner diameter containing 3 liters of wet resin (20-50 mesh). There were three changes in the technique as outlined in our previous paper. First, for convenience in handling, the potato juice was diluted to 3.0-3.5% in the extraction instead of 1% as before. Second, 100 ml fractions of eluate were collected instead of 50 ml. Third, all eluate fractions containing less than 2% solids were discarded, as compared with a 1% solids limit in the earlier work.

Demonstration that the 3% level of liquor solids could be advantageously used to charge the column is of considerable importance. In the new potato starch factories, operators are seeking to alter conventional practices so that most of the soluble constituents will be discharged at about 3% solids instead of the usual 1% solids of starch processing water. Interest in the recovery of the solubles from starch manufacture is now at an all-time high. With it becoming necessary to stop the discharge of these substances into streams, starch factory operators are studying all possible means of converting the waste into a saleable by-product.

Table 1 presents data for a typical run in which amino compounds were recovered by ion exchange.

Figs. 1 and 2 should also be considered along with the examination of Table 1.

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³Mention of this trade name does not imply a recommendation or endorsement by the U. S. Department of Agriculture over others not mentioned.

TABLE 1.—Typical amino compounds recovery run using ion-exchange resin process.

A. Charging the column	
Volume of resin column, liters	3.
Weight of potatoes used, lbs.	70.
Effluent flow rate, liters per hour	4.5
Potato liquor	3.05
Solids content, per cent	26.
Volume put through column, liters	800.
Solids introduced to column, g.	406.
Solids retained by resin, g.	50.8
As per cent of wt. introduced	
B. Eluting the column	
Eluting agent	2N NH ₄ OH
Elution flow rate, liters per hour	2.5
Potato solids eluted	284.
Wt., g.	35.5
Per cent, based on total amount introduced	70.
Per cent, based on amount retained during charging	
Eluate containing 2% or more solids	23 to 42
Fractions, number	2.
Total volume, liters	
Eluate solids content, per cent	14.2
Combined fractions 23 to 42	26.8
Most concentrated fraction (number 38)	
Eluate pH (fractions 23 to 42)	3.1 to 10.2
Range	7.5
Most concentrated fraction	

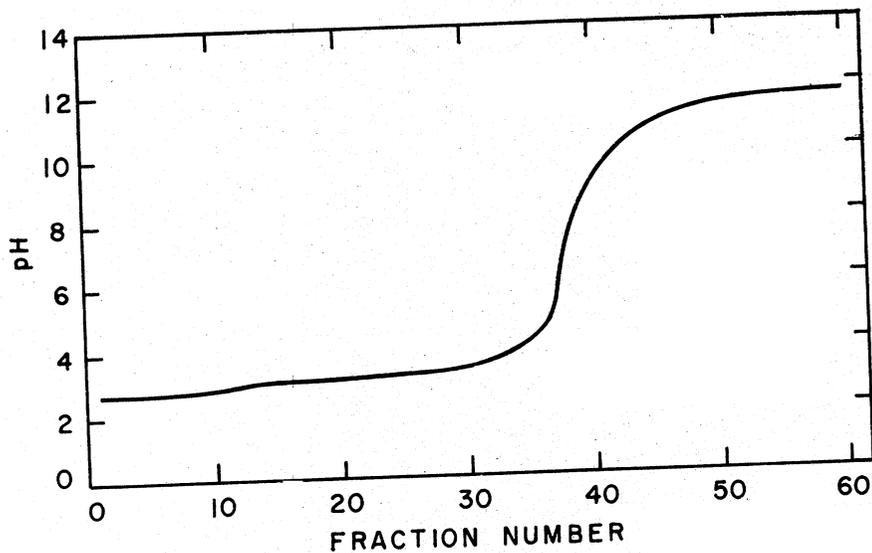


FIG. 1.—Variation of pH during elution of potato amino compounds from Dowex 50 resin with 2N NH₄OH.

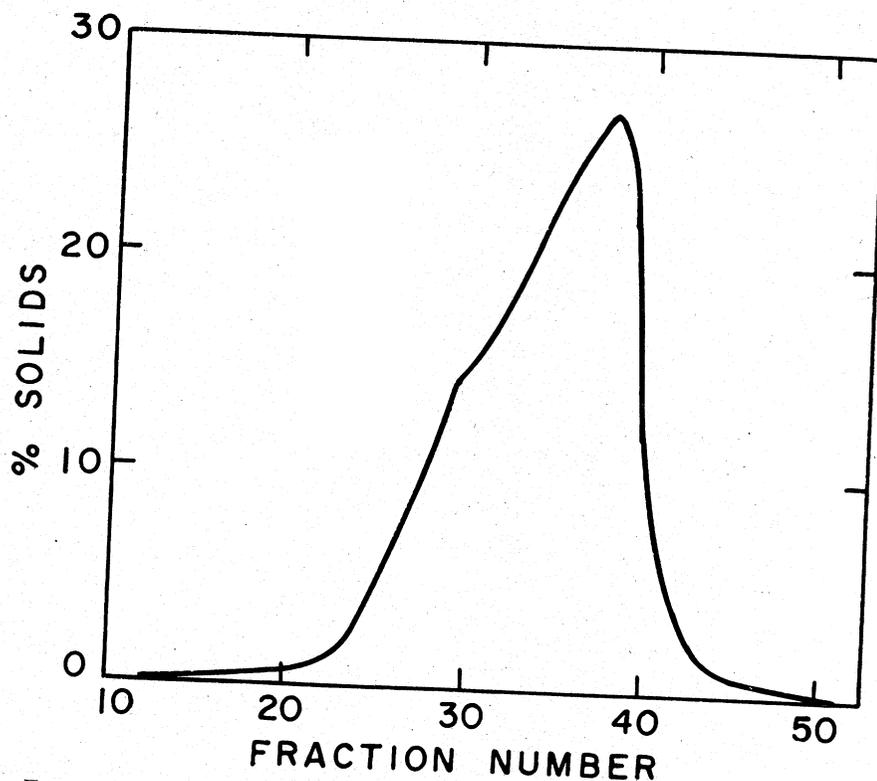


FIG. 2.—Variation of solids contents of fractions obtained during elution of potato amino compounds from Dowex 50 resin with 2N NH_4OH .

Fig. 1 shows the variation of pH with increasing fraction number of the eluate. A sharp change in pH was noted from about fraction 35 to 45, with an initial plateau at around pH 3 and a final plateau around 11.5. Fig. 2 presents a plot of percentage solids in the eluate as a function of the fraction number. The concentration of the eluate began to rise sharply beyond fraction 20, reached a maximum at fraction 38, and then fell sharply.

Crystallization of amino compounds out of fractions 33 to 39 started almost immediately after these fractions left the column. After filtering these combined fractions, 8.2 g of white crystals were obtained. Upon standing at room temperature, fractions 30 to 37 yielded an additional 24.2 g of crystals. Thus, a total of 32.4 g of amino compounds was obtained from the eluate, directly, without cooling or concentrating.

Fractions 23 to 37 were then combined to form a mixture which was acidic; fractions 38 to 42 were combined to form a basic portion. On cooling the acidic portion to 5 C., 20.5 g of white crystals were obtained. The basic portion, however, did not yield crystals on cooling. By concentrating to approximately one-half the original volume, the acidic portion yielded 23.6 g of white amorphous material and the basic portion 5.8 g

of similar material. The 82.3 g of solids thus far obtained contained approximately 60% asparagine, the remainder being mostly glutamine, aspartic acid, and glutamic acid.

The two portions of eluate were now nearly black. They were combined and decolorized with 25 g of activated carbon to yield a light green solution. An additional 48.2 g of white crystals was obtained on concentrating to one-half volume and cooling. The mother liquor was taken to dryness under mild conditions to yield 54 g of a light brown hygroscopic powder. This powder was relatively rich in lysine, histidine, gamma-amino-butyric acid and valine. In addition, it contained alanine, threonine, leucine, isoleucine, tyrosine, serine, methionine, glycine, proline, phenylalanine, and arginine.

It will be noted that a total of 185 g of amino compounds was recovered from the eluate while 284 g were available, as determined by the solids contents of the individual fractions. This loss is probably mostly mechanical, occurring during filtering, transferring, and decolorizing.

A partial analysis of the amino compounds mixture recovered from potato liquor was given in the first paper on this subject. A more complete analysis of this mixture as obtained in the large-scale laboratory process, is presented in Table 2.

TABLE 2.—*Analysis of amino acid mixture recovered from potato liquor by large-scale laboratory process.*

Constituent	Per cent, M.F.B.
Total nitrogen	15.8
Total amino compounds content (as asparagine)	105.8
Individual amino compounds	
Asparagine	28.8
Glutamine	12.8
Aspartic acid	6.6
γ -aminobutyric acid plus valine	6.5
Glutamic acid	3.1
Total polyphenols	6.8
o-dihydric polyphenols	0
Total sugars	2.2
Ash	0.4
Potassium	0

Potassium was determined by the method of Bultasová and Kono-pasek (2). Ortho-dihydric phenols were determined by the Arnow method (1). Total amino compounds content (as asparagine) was estimated by application of Moore and Stein's finding that color development of amino compounds with ninhydrin is about the same (on an equal molar basis) for nearly all amino compounds (4). Methods used in determining other constituents listed in Table 2 were the same as referred to in the previous paper (3).

The recovery of free amino compounds from potato liquor simulating starch processing water was carried out in the laboratory on a much larger scale than previously reported. The solids content of the liquor charged to the column was about 3% compared with only 1% before; this, together with a larger size column meant much faster charging of the column with amino compounds. The combined middle fractions of the eluate from the column contained 14% solids and the most concentrated single fraction 27% solids. Demonstration of the possibility of obtaining most of the amino compounds content in fractions of such high concentrations seems to form a basis for pilot plant studies looking forward to recovery of these compounds from potato starch processing water.

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