

# Operation of the Hamilton City Experimental Ion Exchange Plant

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*Received for publication April 18, 1968*

## Introduction

The ion exchange process was used by the American Beet Sugar Industry to deionize process juices and liquors from 1941 to 1949. A total of six ion exchange plants were described by Dickenson (4)<sup>2</sup>. All of these plants terminated operations, after several years, due to adverse operating economics. Various reasons were given for the plant failures. Chief among these were: short resin life, high regenerant costs, uneconomical white sugar to molasses price ratios, inadequate equipment design and general lack of basic knowledge concerning ion exchange.

In the past fifteen years the state of the art has improved significantly. Progress has been made in decreasing ion exchange resin sensitivity to oxidation (1, 6), and irreversible organic fouling (2). Physical and chemical stability of the more expensive weak anion exchangers has been enhanced by the development of the epoxy-amine and macroporous polystyrene resin matrix. Improvements in ion exchange basic hardware and design concept has also been noteworthy. Fixed bed systems (10) and continuous contactors (3) have been modified in design for greater efficiency. The beet sugar technologist has contributed pertinent basic knowledge concerning non-sugar constituents in ion exchange influent and effluent streams (8).

On the basis of this general improvement in the state of the art, coupled with increased practical knowledge of ion exchange operations (9), the Holly Sugar Corporation installed a commercial size fixed bed ion exchange system at their Hamilton City, California plant. This plant went on stream during the spring campaign of 1967. Plant operation during the spring and fall campaigns of 1967 proceeded with only minor operating problems.

## Plant Description

The ion exchange plant is fed with a 30 brix 80 purity mixture of No. 2 green and thin juice. Plant capacity is approximately 20 to 24 tons of non-sugars eliminated per day. Under normal circumstances 50% to 60% of the non-sugars entering

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<sup>2</sup> Numbers in parentheses refer to literature cited.

the sugar end are eliminated. White sugar production increases as a direct function of non-sugar elimination, while amount of molasses produced is reduced accordingly.

The plant consists of three cation columns operating in service, regeneration and stand-by sequence. Cation regeneration is two stage in execution. The initial cation stripping is accomplished by counterflow of anion ammonium salt waste regenerant solution from the storage tank through the cation resin bed. After counterflow rinsing of the ammonia stripping from the cation, once-used acid, followed by 10% fresh sulfuric acid and rinsing, completes the cation resin regeneration.

The three anion cells operate independently of the cation cells in a merry-go-round manner. At all times two anion cells are in service, one in a primary position, and the other in a secondary position, with respect to juice flow. When the effluent pH of the anion cell in the secondary position indicates leakage, (pH 7.0), this partially exhausted cell is rotated to the primary position where total utilization of anion resin hydroxyl sites takes place. The secondary position is occupied by a freshly regenerated anion cell. The exhausted primary cell is sweetened off in preparation for regeneration with 6% aqua ammonia. Periodically the regular anion ammonia regeneration is combined with a 5% salt strip to eliminate residual color-body build-up within the anion resin matrix.

Effluent juice from the secondary anion is diverted automatically by a set point transmitter to either the sweet water or treated juice hold tanks. Figure 1 indicates the general plant flow. The juice from the sweet water and treated juice tanks is pumped through plate-type heat exchangers counterflow to hot incoming juice. Low brix sweet water is diverted to the evaporator supply tank, while higher brix treated juice is pumped to the high raw melter. By using counterflow heat exchangers, the hot incoming juice is cooled before refrigeration, and the cold juice returning to the factory is heated.

Decationized water for anion rinse and regeneration requirements is supplied from two small automatic cation columns. Decationized water supply from the large cation service cells is optional. Decationized water, 23% aqua ammonia, and 66 Be° sulfuric acid are supplied from near-by storage tanks. A water cooled anhydrous ammonia converter supplies the system with necessary 23% aqua ammonia for weak anion resin regeneration. The 23% aqua ammonia is diluted to 6% aqua ammonia just prior to weak anion regeneration. System material balance is accomplished by temperature-corrected volume meters paired with continuous samplers on all sugar bearing lines into and out of the system.

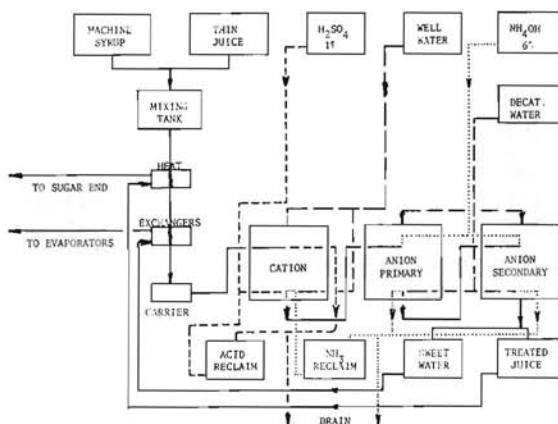


Figure 1.—Hamilton City experimental ion exchange plant.

All valve sequence operations and regenerations are automatic. Any operating sequence may be changed by a simple timer adjustment at the central control panel. In case of automation failure, column valve sequences may be operated from small individual switch panels located in front of each cell.

### Operating Results

The following results were obtained from the Hamilton City ion exchange system under steady state operating conditions. Ion exchange feed varied from 30.0 to 29.5 brix while feed purity varied from 79.2 to 81.6 apparent purity. With reference to operation economics, the four most important operating results and the variables affecting these results will be examined in this paper:

1. Sugar losses.
  - a. Physical
  - b. Chemical (inversion)
2. Dilution.
  - a. Sweet on
  - b. Sweet off
3. Cation regenerant utilization.
4. Non-sugar elimination.

Referring to Figure 2, cation sweet off percent sugar ineffluent vs bed volumes of cation effluent, it is evident that the idealized plug displacement of sugar liquor is not in effect. McGarvey (7), in an excellent discussion, outlines the variables which affect the displacement dilution of sugar liquors. In practice, the cut-off point in sweet off and sweet on must be determined in each factory by comparing the value of sugar recovered with the cost of additional water evaporated.

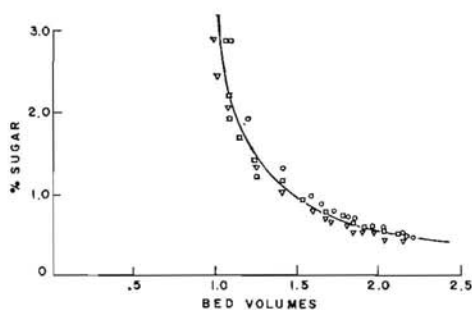


Figure 2.—Typical sweeten-off elution curve, % sugar in ion exchange column effluent vs. bed volumes of sweet-off water.

Figure 3 indicates a typical cation sweet on percent sugar in effluent vs effluent bed volumes. In sweet on, idealized plug displacement of column water is approached. Apparently, diffusion (5) of sucrose into the resin proceeds at a more rapid rate than the reverse situation in sweet off. The pronounced tailing effect in sweet off (Figure 2) is primarily due to slow diffusion of sucrose from the inner labyrinth-like channels of the resin bead. Measurements indicate that 90% of the total ion exchange dilution load is due to cation and anion sweeten off, while cation and anion sweeten on contribute only 10%.

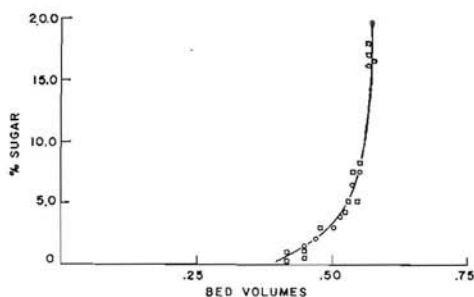


Figure 3.—Typical sweeten-on elution curve, % sugar in ion exchange column effluent vs. bed volumes on feed juice.

Figure 4 shows the graphic relationship between percent sugar in column effluent at cut-off point, and percent dilution calculated as tons-water in ion exchange system effluent divided by the tons of water in the ion exchange influent multiplied by 100. These two variables are related to percent total treated physical sugar loss. Both cation and anion sweet on and sweet off cycles are included in this graphic relationship. The graph, as such, gives the entire dilution, physical loss and effluent percent sugar cut-off relationship for the ion exchange system. As

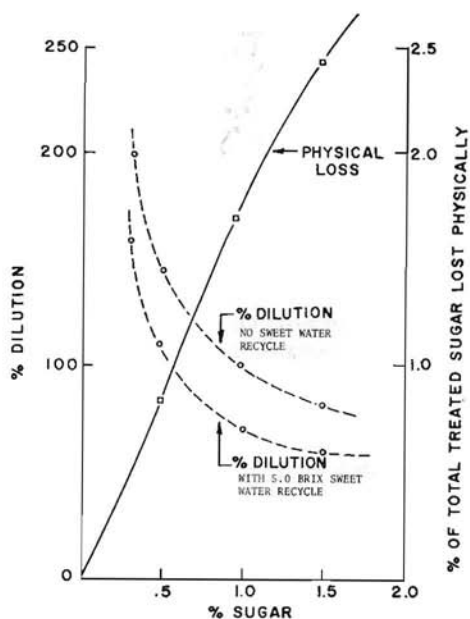


Figure 4.—Graphic relationship between the dependent variables of % dilution, % sugar in column effluent and % of total treated sugar lost physically.

an example, assume anion and cation cut-off points at 1.0% sugar in effluent; then a perpendicular line drawn from 1.0% sugar on the abscissa intersects the physical loss curve and the dilution curves at ordinates of 1.8% loss and, 100% and 65%, dilution respectively. The lower dilution curve assumes the judicious use of 5.0 brix sweet water recycle during the initial part of the cation and anion sweeten off cycle. The 5.0 brix sweet water is reclaimed from the latter part of the sweeten off cycle. In this way, total dilution may be substantially reduced.

Figure 5 portrays the relationship temperature and remaining cation capacity have upon total inversion loss in the cation exchanger (7, 8, 11). Contact time during these runs was 13.5 minutes as calculated from cation resin void volume. The magnitude of heterogeneous catalysis is indicated by assuming that homogeneous catalysis is a fixed value represented by the flat part of the curve at 3.5 bed volumes. At this point column effluent pH of 1.3 has not varied significantly from the first bed volume effluent pH. However, total volume of regenerated cation resin has decreased from 80% to 20%. It is apparent that the decrease in invert formation is by and large due to the decrease in heterogeneous catalysis.

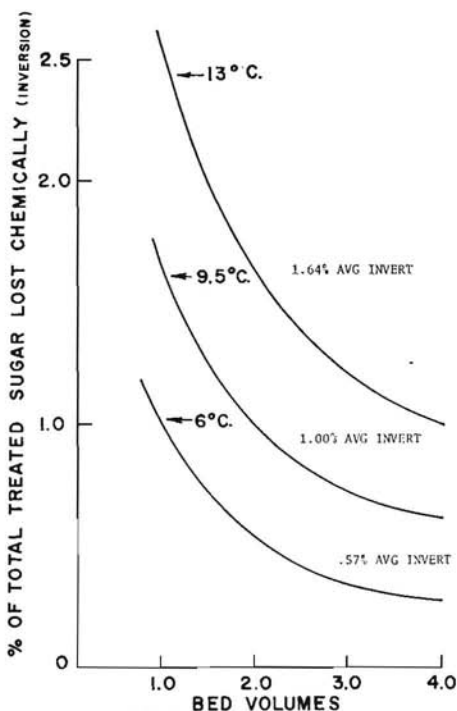


Figure 5.—Percent of total sugar lost through chemical inversion in cation columns vs. bed volumes of cation treated juice effluent.

Figure 6 compares cation column regeneration level (7), expressed as pounds 66 Be° sulfuric acid per cubic foot plus once used acid from the succeeding regeneration, with non-sugar loading to cation conductivity breakthrough capacity and actual cation plus anion total non-sugar elimination. The difference between the two curves (B) indicates the relationship between cation non-sugar capacity to conductivity break and total ion exchange system non-sugar elimination. In this case, 70% of the cation non-sugar breakthrough capacity was eliminated by the total ion exchange system. Intersection of the capacity curves at point (A) with the regeneration level ordinate indicates the minimum amount of total cation regenerated volume at which instantaneous conductivity breakthrough occurs. In this instance the regenerated volume corresponded to a cation resin depth of one foot.

The economic fallacy of total cation resin regeneration is much in evidence in Figure 6. Fresh acid usage far exceeding economical acid utilization would be necessary to obtain total resin regeneration. At the other extreme, lower acid regeneration rates severely limit column capacity as well as equipment

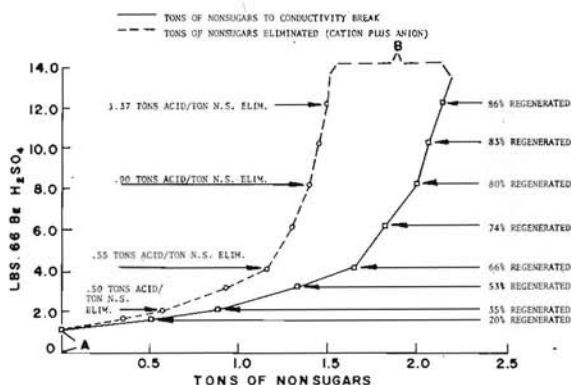


Figure 6.—Graphic relationship between the dependent variables of lbs 66 Be<sup>o</sup> H<sub>2</sub>SO<sub>4</sub>/cu ft cation resin regeneration level and tons of non-sugars eliminated.

investment payout. The best compromise seems to be regeneration operation between 6.0 and 7.0 lbs of fresh 60 Be<sup>o</sup> sulfuric acid per cubic foot plus once used acid. At these points the ratio of fresh acid used to non-sugars eliminated is reasonable.

Weak anion exchangers pose few regeneration problems; 110% of theoretical equivalent requirements are usually adequate for complete regeneration.

Table 1 shows the non-sugar elimination obtained during three normal 8 hour operation periods. Unfortunately, due to the extremely poor quality of the beets processed during the 1967 fall campaign, this data may not be representative of normal northern California operations.

Table 1.—Ion exchange nonsugar elimination.

Run <sup>1</sup>	Influent			Effluent			Percent nonsugars eliminated		
	1	2	3	1	2	3	1	2	3
Purity A.P.	81.6	80.8	81.0	95.1	94.0	93.6	---	---	---
N.S./100 Sugar	22.54	23.76	23.38	5.15	6.38	6.83	77.2	73.2	70.8
Organic (N)/100 sugar	1.27	1.38	1.17	.29	.30	.29	77.2	73.2	75.2
Total amino (N)/100 sugar <sup>2</sup>	.31	.39	.24	.06	.08	.06	80.7	79.5	75.0
Betaine (N)/100 sugar	.36	.36	.39	.15	.16	.14	58.3	55.6	64.1
Unk. organic (N)/100 sugar	.60	.63	.54	.08	.06	.09	86.7	90.5	83.3
Calcium/100 sugar	.08	.08	.09	0	0	0	100.0	100.0	100.0
Sodium/100 sugar	.97	1.00	.89	.05	.04	.03	94.9	96.0	96.6
Potassium/100 sugar	1.49	1.55	1.59	.03	.04	.03	98.0	97.4	98.1
Chloride/100 sugar	1.01	.95	.90	.01	.01	.01	99.0	99.0	99.0
Color index <sup>3</sup>	2523	2420	2348	51	49	64	98	98	97
pH	8.9	8.7	8.8	7.2	7.1	7.3	---	---	---

<sup>1</sup> Eight hour composite.

<sup>2</sup> Includes amino acid(N) plus P.C.A. (N).

<sup>3</sup> Adjusted to ICUMSA tentative method No. 3.

### Summary

Operating results pertaining to dilution, physical and chemical sucrose loss, regenerant utilization, and total non-sugar elimination have been presented. Emphasis was placed on variables affecting the operating results reported.

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