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Direct-Acid-Set Cottage Cheese Whey as a Base for a Shelf-Stable Athletic-Type Drink[1]

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ABSTRACT

A shelf-stable athletic-type beverage was developed from direct-acid-set cottage cheese whey. First, the pH of the whey was adjusted to 5.2 with a saturated potassium hydroxide solution. The whey was heated with stirring to 90°C and held for 10 min to coagulate the protein, which then was removed by filtering or centrifuging. Calcium hydroxide was added to increase the pH to 5.6, and then potassium hydroxide was added to bring the pH up further to 6.5. The whey was filtered or centrifuged again to remove the cloudiness caused by addition of calcium hydroxide and additional protein precipitation. Beta-galactosidase was added and whey was held at 5°C for 18 h to hydrolyze the lactose. Then, one part water was mixed with two parts whey before saturated citric acid was added to make an acceptable orange-flavored beverage. The beverage then was heated to 88°C and stored in 8-fl. oz. bottles capped with Teflon-lined closures. The levels of electrolytes, such as sodium and potassium, in this product were similar to those in commercially available athletic-type drinks. In two separate trials, involving 28 persons each, the whey-based drink, when compared with a commercial product, was preferred 64% and 46% of the time, respectively. During storage some of the added sucrose was hydrolyzed into glucose and fructose; however, a taste panel did not detect a change in sweetness in the stored products. The heat process used (88°C for 5 min) appeared to be adequate for commercial sterility. The stability of the product during storage was good and estimated to be longer than 6 months. Ingredient cost of the whey-based athletic-type drink was $0.14 per 32 fl. oz.

Approximately 3.9 billion lb of acid whey were produced in the United States in 1981 as a by-product of cottage cheese manufacture (*4*). However, most of this acid whey was not further-processed. About 85% of unprocessed whey came from small cheese plants with daily production of 19,800 lb of whey or less (*14*). Milk processing plants that produce cottage cheese are normally among these small whey producers. It may not be economically feasible for these plants to purchase equipment to further process the whey into dried or condensed products, so it most often is disposed of through municipal sewage systems. Disposing of whey in this manner is costly in the form of surcharges, as well as being a waste of the nutrients (*14*).

As an alternative to disposal of whey, researchers have suggested that it could be used as a base for manufacture of a variety of beverages. Holsinger et al. (*12*) reviewed extensively the uses of cultured cheese whey in beverages. Since then, several researchers have reported the use in beverages of cottage cheese whey resulting from the widely used direct-acid-set (DAS) method of making cottage cheese (*7,9*). Demott (*11*) formulated an orange-flavored and lemon-lime-flavored drink from DAS whey by adding appropriate amounts of orange or lemon-lime concentrate, sugar, and saccharin. Chen et al. (*9*) developed an imitation milk that contained neutralized DAS whey, whole milk, and non-fat dry milk. Blackburn and Bassette (*7*) prepared a chocolate-flavored dairy drink. It contained neutralized DAS whey, whole milk, sugar, chocolate, and non-fat dry milk.

In this research, a non-refrigerated, shelf-stable product was developed using DAS cottage cheese whey as a base. Methods are described for preparing the whey base, formulation of the drink and heat-processing for shelf stability. The product is similar in composition to electrolyte-carbohydrate containing athletic-type drinks currently on the market.

MATERIALS AND METHODS

Preparation of beverage base from whey

Fresh DAS whey was obtained from two local plants. The DAS whey was warmed to 25°C, and saturated potassium hydroxide solution was added to obtain a pH of 5.2. With continuous stirring on a hot-plate, the whey was heated and held at 90°C for 10 min to coagulate protein. In the KSU milk plant, a steam-jacketed vat with an agitator was used for heating. The coagulated protein was removed by filtration or centrifugation with a cream separator. Filtered whey was allowed to cool to room temperature. Calcium hydroxide was added at 0.10 to 0.15 g/100 ml of filtered whey, and it was stirred for 15 min. This resulted in a pH of 5.6 to 5.7. The pH was further raised to 6.5 with saturated potassium hydroxide solution. The liquid was filtered or separated again to remove the cloudiness caused by calcium hydroxide and some protein precipitation at that pH. Maxilact L 2000 beta-galactosidase (GB Fermentation Industries, Inc., Charlotte, NC) was added at a level of 0.085%, v/v. The hydrolysis was done at 5°C for 18 h.

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Formulation and processing of whey-based beverage

The finished product was formulated by adding one part of water to two parts of deproteinated, lactose-hydrolyzed whey; adjusting the pH to 3.7 with saturated citric acid; adding 1% (w/v) glucose and 4% (w/v) sucrose; and adding sufficient orange flavoring (0.2%, v/v, Norda Natural and Artificial Orange Emulsion #EP-10,806, East Hanover, NJ; and 0.3%, v/v, Blanke Baer Orange Extract, Fenton, MO) to make an acceptable orange-flavored athletic-type drink. The beverage was heated to 88°C in a beaker on a hot plate or in a steam-jacketed kettle in the plant, and poured into a 8-fl. oz. bottles. The bottles were capped with Teflon-lined metal closures, inverted and held for 5 min, and then turned upright and cooled to room temperature. The overall flow diagram of this whey-based beverage process is shown in Fig. 1.

Product sterility tests

To test product sterility, 7 bottles chosen randomly from the 7 cases of the product processed in the plant were incubated at 37°C for 15 d, and checked for visual changes after 7 and 15 d. On day 15, each was sampled aseptically and 1 ml plated in each of four different media: Standard Plate Count agar for aerobic viable cell counts (*18*); orange serum agar for acid tolerant bacterial counts (*16*); anaerobic agar in an anaerobic jar for anaerobic bacterial counts (*17*); and potato dextrose agar (pH adjusted to 3.5 with 10% tartaric acid, 0.1% tetracycline and 0.1% chlorohexanimide added to prevent bacterial growth) for yeast and mold counts (*16*). Duplicates were made of each analysis.

Product acceptability tests

The paired preference test (*15*) was used to determine if the whey-based drink was comparable to a leading commercial athletic-type drink. Two different sets of 28 panelists compared the two drinks. The two tests were done 6 months apart using two different batches of whey beverage and two different code numbers of commercial product. Each panelist was given two coded samples in 2-fl. oz. cups, and asked to indicate which one he/she preferred.

A consumer acceptability test was conducted by scoring the whey-based beverage on a 7-point hedonic scale. The beverage was distributed in 8-fl. oz. bottles to the consumer along with an evaluation form, instructions, and a self-addressed stamped envelope for returning the evaluation form. An average value was determined from total responses as well as percentages of responses for each category.

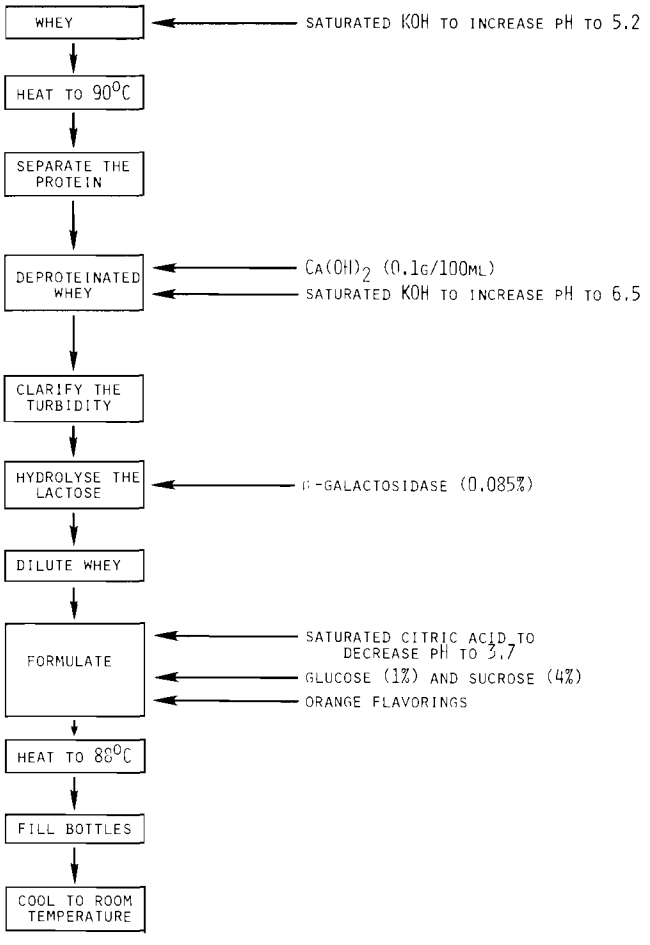

Figure 1. *Flow diagram of the whey-based beverage process.*

Analysis of product composition

The whey-based athletic drink was analyzed for sodium, potassium, chloride, and protein. Sodium and potassium were quantitated with a Jarrell-Ash Atomic Absorption Instrument according to A.O.A.C. procedures (*2*). Chloride was determined by a titrimetric kit from Sigma Chemical Company, St. Louis, MO. Titration was done with a standardized mercuric nitrate solution using diphenylcarbazone to indicate the endpoint visually (*5*). Protein was analyzed by the Kjeldahl procedure (*2*).

Organoleptic storage studies

A multiple comparison test (*15*) using 8 panel members trained in food product evaluation was conducted to evaluate stored samples of the beverage. The reference sample was stored at 5°C for the duration of the test. Test samples were stored at 21°C as well as 32 and 37°C to accelerate changes in shelf life. Panelists tasted the reference and 3 samples every 2 weeks during a 10-week period to determine changes in overall flavor and sweetness. Reference scores were predetermined by having the panelists evaluate the 0-day samples for these characteristics. An average of the scores was calculated and used as the reference for the duration of the experiment.

Chromatographic analysis of sugars during storage

Stability of sugars during storage was analyzed by high performance liquid chromatography (HPLC). Samples of the beverage stored at 5, 21 and 37°C for 4 and 8 weeks were diluted, one part beverage to four parts water. The diluted samples were passed through Sep-Pak C_{18} cartridges (Waters Associates, Inc., Milford, MA), as described by the manufacturer (*3*), to remove lipids, colored materials, and residual proteins.

Standard carbohydrate solutions were prepared from analytical-grade reagents. The sugars involved were dissolved in water (w/v) at a concentration near that estimated in the diluted samples. The standard solution contained 0.05% lactose, 0.34% galactose, 0.55% glucose, 0.15% fructose, and 0.54% sucrose. Before injection, the standard was eluted through a Sep-Pak as done for samples.

The HPLC system consisted of a Beckman Model 100A solvent delivery system, An Altex Scientific 210 septumless injector with a 20-μl loop, an Altex Scientific refractive index detector Model 156, and a Fisher Recordall Series 5000 chart recorder. A LiChrosorb Anion Exchange AX-GU cartridge (Brownlee Labs, Inc., Santa Clara, CA) was used as a guard column to remove organic acids and filter out harmful particles. Carbohydrates were separated on an Altex Scientific μ Spherogel Carbohydrate 7.5% column (7.5 mm i.d. × 30 cm) which was held at 80°C by a 30-cm Altex Scientific water jacket on the column and a Fisher Model 80 circulation water bath. The mobile phase was deionized, boiled, and degassed water that was held at 62°C in the solvent reservoir. The flow rate was 0.6 ml/min, and chart speed was 0.5 cm/min. A Hamilton 25-μl syringe was used to inject a sample into a 20-μl sample loop. Retention time was used to identify sugars in the sample.

RESULTS AND DISCUSSION

It was necessary to remove proteins from whey for this beverage to prevent them from precipitating during heat processing and storage, and also to eliminate their buffering capacity so that the appropriate sourness could be achieved when adjusting the pH to 3.7 with citric acid. Calcium hydroxide was used in conjunction with potassium hydroxide as neutralizing agents to help reduce salti-

ness in the finished product. According to our preliminary study, less citric acid was needed for lowering the pH when calcium hydroxide was used along with potassium hydroxide. Lactose in whey was hydrolyzed to eliminate lactose-intolerance problems that might be associated with drinking the beverage and to increase sweetness, since both glucose and galactose are sweeter than lactose.

The heat processing method used appeared to preserve the beverage in 8-fl. oz. bottles. There was no microbiological growth detected from the beverage samples stored for 15 d at 37°C. All four microbiological tests conducted (viable cell count, acid tolerant bacterial count, anaerobic bacterial count, and yeast and mold counts) showed no growth of microorganisms in seven replicates. There were no visible changes in the color or appearance. These results indicated that the heat treatment employed was adequate to maintain "commercial sterility" (*20*).

The finished product is orange in color, and translucent. It has an orange flavor; it is sweet and slightly sour, and is similar to a leading commercial product. Most of the sugars present are in the form of glucose and sucrose. A slight amount of lactose remains in the final formulation, but not enough to cause problems for lactose-intolerant persons. Results of sensory analysis indicated that the whey-based beverage is comparable in flavor to a leading commercial product. Of the 28 panelists who participated in the first preference test, 64% preferred the whey-based athletic-type drink while 36% preferred the commercial athletic drink. The difference was not statistically significant. In the second test 46% preferred the whey-based athletic-type drink and 54% preferred the commercial drink. Again, the difference was not statistically significant. The results are summarized in Table 1. Results of the consumer acceptance test showed that of the 69 panelists who responded, 47.8% liked the whey-based drink to some degree, 14.5% neither liked nor disliked it, and 37.5% disliked it to some degree. The average response on the 7-point hedonic scale was 4.26, which was between "like slightly" and "neither like nor dislike". However, in the consumer test it was not determined if the panelists used or even like athletic-type drinks. Some of the consumers may have rated it low because of the bland flavor of athletic drinks in general, especially if they were unfamiliar with that flavor.

The results of the chemical analysis indicated that the levels of electrolytes, such as sodium and potassium, in the whey-based beverage were similar to those in a commercially available drink (Table 2). The potassium level of this beverage (2.5 meq/L) is no different from that of the commercial athletic-type drink. The sodium and chloride contents (14.5 meq/L and 2.3 meq/L, respectively) are slightly lower than those of the commercial drink. The sodium level that the American Dietetic Association (ADA) recommends for athletic-type drinks is 10 meq/L (*1*). The whey-based drink is slightly more than the level recom-

TABLE 1. *Comparison of whey-based athletic-type drink to a leading commercial athletic-type drink by preference test*[a].

Product	Number of panelists		
	1st Trial	2nd Trial	Total of both trials
Whey-based beverage	18 (64%)	13 (46%)	31 (55%)
Commercial beverage	10 (36%)	15 (54%)	25 (45%)
Total	28 (100%)	28 (100%)	56 (100%)

[a]Note: The results are not statistically significant at $P<0.05$.

TABLE 2. *Comparison of minerals, protein, and caloric content of whey-based athletic-type drink and a leading commercial drink.*

Component	meq/L	
	Whey-based	Commercial[a]
Calcium	2.7	–[b]
Phosphorus	21.7	–[b]
Sodium	14.5	22.7
Potassium	2.5	2.5
Chloride	2.3	13.5
Magnesium	2.2	–[b]
Calories	25 cal/100 ml	21 cal/100 ml
Protein	0.17%	–[b]

[a]From Coyle et al. (*10*).
[b]Not availble.

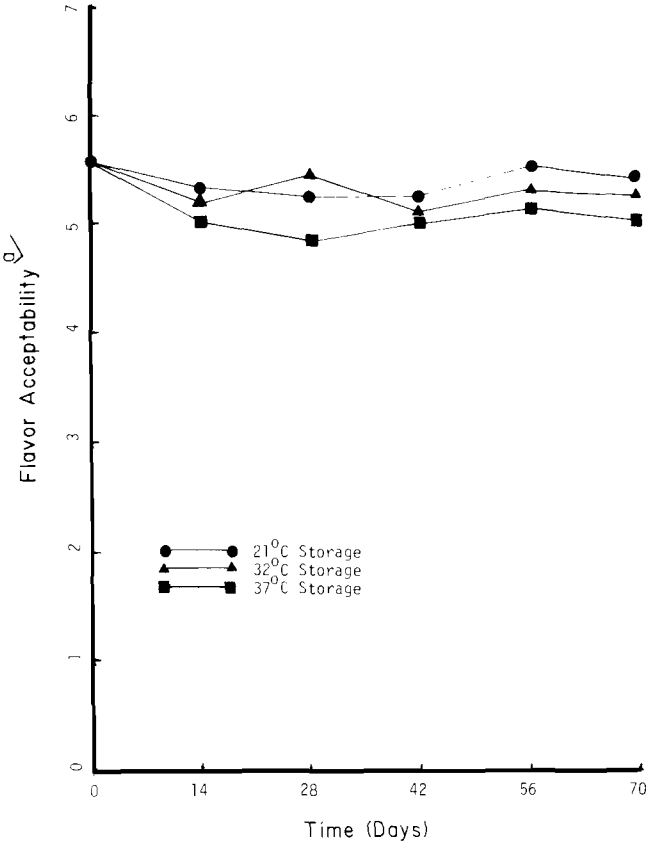

Figure 2. *Stability of overall flavor during storage at various temperatures. Each data point indicates the average of the scores of eight panelists.* [a]*Relative to the reference sample (5°C storage): 1 = definitely not acceptable; 7 = extremely acceptable.*

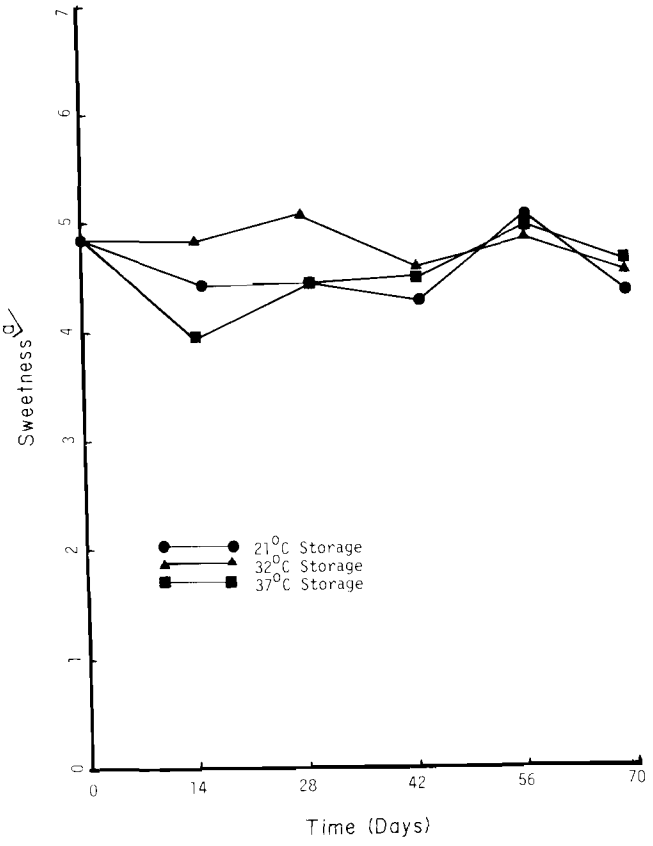

Figure 3. *Stability of sweetness during storage at various temperatures. Each data point indicates the average of the scores of eight panelists.* [a]*Relative to the reference samples (5*°C storage): 1 = *definitely not sweet; 7 = extremely sweet.*

mended (14.5 meq/L) but closer to the ADA recommendations. The potassium level recommended by the ADA is 5 meq/L. The whey-based drink had a level slightly less than that recommended at 2.5 meq/L (*1*). There was 0.17% protein in the whey-based drink, which added to the mouth feel without the addition of gums.

Organoleptic studies showed that no change occurred in the overall flavor and sweetness during 10 weeks of storage at any of the storage temperatures used. As depicted in Fig. 2 and 3, panelists detected little difference among the beverages stored for 10 weeks at 21, 32, and 37°C. If we assume the rate of quality deterioration in this beverage follows the Q_{10} theory [(rate of reaction doubles for each 10°C rise in temperature (*8*)], it appears to have a shelf life of more than 25 weeks at 21°C (10 weeks times 2.5). Because of the time limit on this study, testing of the theory was restricted. However, according to the results obtained thus far, the product appears to be stable at room temperature for at least 6 months.

Figure 4 shows the HPLC separation of sugars in the whey-based beverage stored for 8 weeks. Beverages stored at different temperatures for 8 weeks have different concentrations of the five sugars present. According to our preliminary studies, there was no interference on the peaks coming from the whey base used. The first eluting peak on the chromatograms (unnumbered peak) was always present in the whey-based beverage samples (Fig. 4B, 4C, and 4D). It may be an anion peak coming from salts in the whey base (*19*). The sucrose peak (peak no. 1) is from sucrose that was added for sweetness. The lactose shoulder (peak no. 2) represents the residual lactose remaining after hydrolysis by the beta-galactosidase. The glucose peak (peak no. 3) is from glucose added for sweetness, plus glucose that resulted from enzymatic hydrolysis of lactose, and from acid hydrolysis of the sucrose. Galactose (peak no. 4) is present as a product of the lactose hydrolysis. The amount of sucrose in the beverage decreased as the storage temperature increased. Accordingly, the amount of fruc-

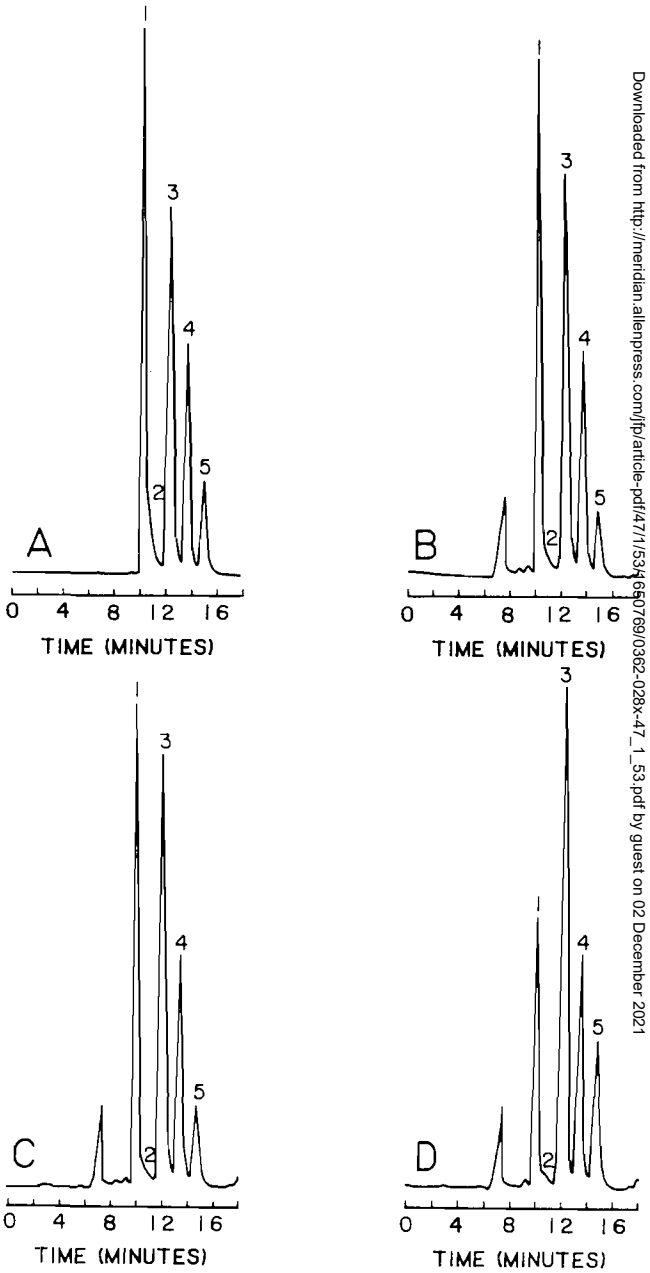

Figure 4. *High performance liquid chromatograms of standard carbohydrate solution and product stored for eight weeks: 1) sucrose, 2) lactose, 3) glucose, 4) galactose, and 5) fructose; A - standard solution, B - product stored at 5°C, C = product stored at 21°C, and D - product stored at 37°C.* μ*-Spherogel Carbohydrate column (80°C); solvent,* H_2O*; flow rate, 0.6 ml/min; injection volume, 20* μ*l; RI detector, 16* ×.

tose (peak no. 5) and glucose increased. This is due to acid hydrolysis of the sucrose by the acidic conditions present in the beverage (*13*). Since fructose is a sweeter sugar than sucrose, and glucose is much less sweet then sucrose (*6*), the changes in concentration of the three sugars during storage should result in an overall decrease in sweetness. However, the shift in sweetness of the sample stored at 37°C was not detected by the panel. This indicates that the difference in sweetness caused by acid hydrolysis of sucrose was minimal.

The whey-based athletic-type drink ingredient cost was $0.14 per 32 fl. oz. This does not include the savings from sewage plant surcharges for whey disposal. A cost breakdown is shown in Table 3. It is difficult to make a direct comparison between the ingredient costs of this whey-based drink and commercial products on the market. However, at the present time the retail price of a leading commercial athletic drink is considerably higher than the ingredient cost of this whey-based athletic-type drink.

TABLE 3. *Estimated ingredient cost for producing a 32 fl. oz. quantity of the whey-based athletic-type drink.*

Ingredient	Unit cost[a]	Cost
Whey	_[d]	_[d]
Water	_[e]	_[e]
Potassium hydroxide	$ 1.69/lb	$0.009
Calcium hydroxide	3.47/lb	0.003
Maxilact L2000	14.52/lb	0.017
Sucrose	0.33/lb	0.028
Glucose	0.24/lb	0.005
Citric acid	5.73/lb	0.055
Orange flavor & color[b]	12.50/gal	0.006
Orange extract[c]	19.20/gal	0.014
Total cost		$0.14

[a]Based on wholesale prices as of March 1, 1983.
[b]Norda, East Hanover, NJ.
[c]Blanke Baer, Fenton, MO.
[d]Savings on surcharge by sewage plant not included.
[e]Negligible.

REFERENCES

1. Anonymous. 1980. Nutrition and physical fitness--A statement by the American Dietetic Association. J. Am. Dietet. Assoc. 75:437-443.
2. Anonymous. 1980. Official methods of analysis of the Association of Official Analytical Chemists, 13th ed. Association of Official Analytical Chemists, Washington, DC.
3. Anonymous. 1981. SEP-PAK C_{18} Cartridges--A new dimension in sample cleanup. Waters Associates, Inc., Milford, MA.
4. Anonymous. 1982. Estimated U.S. fluid whey and whey solids production and resulting quantity of whey solids further processed. Bulletin No. 0882. Whey Products Institute, Chicago, IL.
5. Anonymous. 1982. Chlorides. Sigma Technical Bulletin No. 830-T. Sigma Chemical Co., St. Louis, MO.
6. Aurand, L. W., and A. E. Woods. 1973. Food chemistry. AVI Publishing Co., Inc. Westport, CT. p. 101.
7. Blackburn, L. C., and R. Bassette. 1982. Neutralized direct-acid-set whey as an extender for a chocolate-flavored dairy drink. J. Food Prot. 45:12-13.
8. Brady, I. E., and G. E. Humiston. 1975. General chemistry: Principles and structures. John Wiley and Sons, Inc., New York, NY. p. 307.
9. Chen, F. H., R. Bassette, and J. T. Marshall. 1979. A milk-like beverage from neutralized direct-acid-set cottage cheese whey. J. Food Prot. 42:299-301.
10. Coyle, E. F., D. L. Costill, W. J. Fink, and D. G. Hoopes. 1978. Gastric emptying rates for selected athletic drinks. Res. Quart. 49(2):119-124.
11. Demott, B. J. 1975. Acceptability of flavored drinks made with cottage cheese whey produced by the direct acidification process. J. Milk Food Technol. 38:691-692.
12. Holsinger, V. H., L. P. Posate, and E. D. DeVelbiss. 1974. Whey beverages: A review. J. Dairy Sci. 57:849-859.
13. Hodge, J. E., and E. M. Osman. 1976. Carbohydrates. *In* O. Fennema (ed.) Principles of food science, Part I, Food chemistry. Marcel Dekker, Inc., New York, NY. p. 72.
14. Jelen, P., and M. LeMaguer. 1976. Feasibility evaluation of cheese whey processing in small plants. J. Dairy Sci. 59:1347-1352.
15. Larmond, E. 1977. Laboratory methods for sensory evaluation of foods. Publication No. 1637. Research Branch--Canada Department of Agriculture, Ottawa, Ont., Canada.
16. Leininger, H. V. 1976. Equipment, media, reagents, routine tests and stains. *In* M. L. Speck (ed.) Compendium of methods for the microbiological examination of foods. American Public Health Association, Washington, DC.
17. Lennette, E. H., E. H. Spaulding, and J. P. Truant (ed.). 1974. Manual of clinical microbiology, 2nd ed. American Society for Microbiology, Washington, DC. p. 369.
18. Marth, E. H. (ed.) 1978. Standard methods for the examination of dairy products, 14th ed. American Public Health Association, Washington, DC.
19. Richmond, M. L., D. L. Barfuss, B. R. Harte, J. I. Gray, and C. M. Stine. 1982. Separation of carbohydrates in dairy products by high performance liquid chromatography. J. Dairy Sci. 65:1394-1400.
20. Tanner, F. W. 1944. The microbiology of foods, 2nd ed. Garrard Press, Champaign, IL.