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ION EXCHANGE PROCESSES FOR REMOVING RADIOACTIVE CONTAMINATION FROM MILK [1]

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SUMMARY

These studies on removing radioactive contamination from milk were carried on for the purpose of developing a feasible standby process for use in dairy plants in the event that radioactive fallout should reach hazardous levels. Current levels of all the radioactive contaminants are far below what is considered hazardous levels; Iodine-131 is essentially undetectable in all milk supplies. However, the remote possibility of an unforeseen nuclear reactor accident, or resumption of nuclear testing by any nation, would result in significant increases in fallout.

A fixed-bed column process for removing radiostrontium has received the most attention; commercial feasibility is more nearly established for its use than for other procedures, or for removal of other radionuclides.

The testing of nuclear weapons has yielded atmospheric fallout containing several radioactive substances which constitute potential health hazards. This problem has received serious attention by the Department of Agriculture, the U. S. Public Health Service, the Atomic Energy Commission, other government agencies, and the general public.

The U. S. Public Health Service has established a national pasteurized milk network from which composited samples of milk are provided each week from stations in every state and possession for analyses for radioactive contaminants. The radionuclides reported in significant amounts in milk are Strontium-90, Iodine-131, Strontium-89, Cesium-137, and Barium-140. All of these nuclides are of public health significance when ingested in large quantities; however, Strontium-89, Strontium-90, and Iodine-131 have been given primary attention from the point of view of investigations directed toward minimizing their concentrations in dairy products. The radiation characteristics and a statement concerning the biological significance of each is shown in the following table:

	Half-life	Disposition
Iodine-131	8 Days	Thyroid gland
Strontium-90	28 Years	Bone
Strontium-89	50.5 Days	Bone
Cesium-137	30 Years	Whole body
Barium 140	12.8 Days	Bone

Methods of minimizing the levels of fallout substances in the food chain depend largely on the radioactive, chemical, and biological properties of the element. The use of dry feeds and the storage of milk, or milk products, are effective means of minimizing the levels of Iodine-131 in milk, but have little value for solving the Strontium-90 problem. Therefore, a process for removing Strontium-90 from milk seems highly desirable as a standby countermeasure. Under some circumstances, removal of Iodine-131 from milk may also be the most effective means of controlling the levels of this isotope. Such processes have been the objective of a research program supported by the Atomic Energy Commission, the Public Health Service of the U. S. Department of HEW, and the Agricultural Research Service of the U. S. Department of Agriculture, for the past four years. The work has been carried out in the Beltsville laboratory and pilot plant of the Agricultural Research Service and at the Public Health Service, Robert A. Taft Sanitary Engineering Center in Cincinnati, Ohio.

This presentation reviews the research and development program, including factors affecting the removal of the radionuclides, means of controlling the composition and quality of milk, and the steps in carrying out the removal processes. Commercially available ion exchange resins have been used in the development work for removing both cationic and anionic (Iodine-131) radionuclides.

FIXED-BED CATION REMOVAL PROCESS

The pilot plant developments during the first few months of the program at Beltsville for removing Strontium-90 have been previously reported (3). Some modifications in the procedure and improvements in equipment have

[1] Invitational paper presented at the 59th Annual Meeting of the American Dairy Science Association, University of Arizona, Tucson, June, 1964.

been made since that time. The process consists of passing milk through columns containing a strong acid ion exchange resin. About 95% of the radioactive strontium can be removed with this process while maintaining the milk's chemical composition near normal, and inducing only small changes in the physical stability and flavor of the resin-treated milk. Flavor scores, as judged by a panel of ten judges, have usually averaged about 1.0 point below the controls on the A.D.S.A. score-card.

The nutritional quality of milk processed for removal of Strontium-90 by the fixed-bed method was evaluated by determining the growth rates of baby pigs and rats and by chemical analyses. Results showed no significant differences in the body weight gains between the animals fed on ion exchange processed milk and those fed normal milk, and no marked changes in the vitamin contents of the ion exchange processed milks. More detailed information on the nutritional aspects of this process are being collected and evaluated.

The present pilot plant equipment includes two stainless steel columns, 11.5 in. in diameter by 80 in. long which contain the resin. This will accomodate about 27 gal. of settled resin bed, thus providing for much lower milk flow rates per unit volume of resin than the glass columns previously described (3). Sufficient accessory equipment, including automatically controlled valves, are arranged so that the milk, rinse water, detergent solution, and mixed salt regenerating solution can be passed through the resin bed according to a predetermined time schedule. Four regenerant tanks are provided so that as much as three-fourths of the salt solution can be reused. These pilot plant facilities were designed for a capacity of 100 gal. per hour.

The fixed-bed process for removal of radiostrontium from milk includes the following operations:

1. *Resin type—initial preparation of resin bed.* The resin should be of the nuclear sulfonic acid type, such as Amberlite IR-120 [2] or Dowex 50W.[2] The particle size should be in the 20-50 mesh range, and be of the intermediate porosity type. Commercial grades are normally received in the sodium form. Prior to use for removing Strontium-90, the resin bed is charged with a mixed solution of calcium, magnesium, potassium, and sodium chlorides. The quantities of each are selected so that the resin and milk will be in equilibrium with respect to these major cations. This assures a minimum of change in the cation composition. The ratio of these ions in the regenerant will be different from that on the resin which, in turn, will differ from the ratio in milk, because the affinities of the cations for the resin differ, and because their activities in milk and solutions differ. The optimum ratio to be placed on the resin also varies with the pH of the milk, primarily because the activity of calcium changes greatly with changes in pH (4). The relative proportion (mole ratio) of calcium desired on the resin increases with decreasing pH of the milk to be treated. The total composition of the solution may be varied, but for economy in space utilization, a concentrated solution is recommended. The composition of this solution used during most of the development work at Beltsville was (in grams per liter): $CaCl_2 \cdot 2\ H_2O$, 53.5; KCl, 23.1; NaCl, 8.5, and $MgCl_2 \cdot 6\ H_2O$, 15.1.

After charging the resin column with the salt solution, it is thoroughly rinsed with water.

2. *Acidification of milk.* At the normal pH of milk strontium is combined to a great extent (80%) with the caseinate complex (2). Most of it is, therefore, difficult to remove by ion exchange. Passing 30 resin bed volumes of normal milk through a resin column removes an average of less than 50% of the radiostrontium. By acidifying to a pH of 5.3 with citric acid, about 95% can be readily removed (2, 5). Only about 15% of strontium is bound at this pH.

The amount of strontium removed is significantly affected by the type of acid used. In laboratory experiments, acidifying to pH 5.3 with citric, hydrochloric, and phosphoric acids resulted in average removals of 97.5, 88.4, and 83.1%, respectively, for 25 bed volumes of milk passed through resin columns. Although citric acid is the most expensive, these results recommend its use rather than the other two.

The present pilot plant is equipped for continuous in-line acidification, using a variable speed metering pump for the purpose. At a pH of 5.3 milk casein is sensitive to heat; thus, it is necessary to maintain a low temperature while milk is in the acid condition. Milk temperatures from 40 to 80 F have been found to have no effect on the amount of strontium removed.

3. *Flow of milk through the column.* The acidified, cold (50 F), raw whole milk is pumped through a filter, then downflow through a column of the resin. Either of two flow rates are suggested for practical use: one-eighth and one-sixteenth resin bed volumes (rbv) per min-

[2] Reference to certain products or companies does not imply an endorsement by the Department over others not mentioned.

ute. For a constant total capacity of 100 gal. per hour, these rates require back-washed and settled resin volumes in the column of 13.35 and 26.7 gal., respectively. The slower flow rate, one-sixteenth rbv per min, results in the removal of about 1.0% more strontium than one-eighth rbv per min; however, it has been found that at either rate an average of about 95% removal can be maintained for a total throughput of 30 rbv. Faster flow rates result in significant reductions in the amount of strontium removed (1).

At one-sixteenth rbv per min a total volume of 30 rbv of milk requires 8 hr of continuous flow time without interruption; at one-eighth rbv per min, milk flow would be directed to a second column after 4 hr. The resin inventory per 8 hr of processing time will be the same for either flow rate. The regenerant (salt solution) requirements per unit volume of milk processed will also be the same.

4. *Neutralization and processing of the treated milk.* The effluent milk from the ion exchange column is neutralized by continuous in-line addition of a solution of potassium hydroxide, a mixed solution of sodium and potassium hydroxide, or a combination of sodium and calcium hydroxides. Calcium hydroxide is insoluble and must be added as a powder, or as a slurry.

After neutralization, the milk is pasteurized and homogenized. A vacuum treatment may be employed to remove the water added in the acidification and neutralization steps. However, the dilution resulting from these additions (using 0.75 M citric acid and 1.5 N KOH) amounts to less than 4%.

5. *Cleaning and sanitizing the resin.* Immediately after the milk cycle, the resin is rinsed with warm water, followed by an upflow wash with a nonionic detergent solution at about 160 F. Higher temperatures may be used for controlling bacterial growth since this resin (nuclear sulfonic acid type) will withstand temperatures up to the boiling point of water.

The problems involved in ion exchange resin sanitation are being more thoroughly investigated by the U. S. Public Health Service at the Taft Engineering Center, Cincinnati, Ohio.

6. *Regeneration of the resin.* The resin is regenerated with a salt solution of the same composition as that used for the initial preparation of the resin (Step 1). The chief purpose of this cycle is to remove the radiostrontium from the resin; it also maintains the equilibrium with respect to the minerals in milk. With a fixed-bed procedure, it is necessary from an economic standpoint to reuse as much of the regenerant as possible. The pilot plant at Beltsville provides for reusing three-fourths of the salt solution. The first fraction (one-fourth of the total) through the resin column is discarded. Each of the three remaining fractions is pumped into separate tanks. These are then used for subsequent regeneration in the order in which they are recovered, followed by a tank of fresh solution equal in volume to the amount discarded.

The amount of regenerant required for each cycle is proportional to the amount of resin in the column, and also depends on the amount of strontium to be stripped from the resin. To maintain a high level of removal, it is necessary to strip about 97% of the radiostrontium from the resin. For the conditions described above about 16 rbv of regenerant are required, 4 rbv of which are fresh salt solution. Thus, one volume of salt solution is used for each 7.5 vol of milk.

The first 4 rbv fraction through the column removes about two-thirds of the strontium from the resin, and after a steady state condition has been attained the radio-strontium to be disposed of is concentrated by a factor of 7.5 relative to the concentration in the original milk supply. The health hazard involved in contaminating the waste water from a dairy plant should not be a big problem if attention is paid to dilution of the discarded regenerant with water during the hours of operation.

The following table outlines the steps and suggested time schedules for an 8-hr milk cycle through one column without interruption.

	Quantity *(rbv)*	Time *(min)*	Temp *(F)*
Rinse water	6	24	50
Milk	30	480	50
Rinse water	6	24	100
Detergent	8	32	150
Rinse	6	24	160
Regenerant No. 1	4	40	70
Regenerant No. 2	4	40	70
Regenerant No. 3	4	40	70
Fresh regenerant	4	40	70
Rinse water	6	24	160

Removal of Barium-140 and Cesium-137. The potential health hazard associated with Barium-140 is much less than with Strontium-90 because its yield from nuclear weapons testing is less and its half life—12.8 days—is much shorter. Being chemically similar to Strontium, however, it is effectively removed by the same ion exchange process as is used for Strontium (5).

Cesium-137 is chemically related to potassium, and metabolically distributes itself throughout the body, which is in contrast to the bone-seeking nature of strontium. However, a standby process for removing it from milk would be desirable, since it has been detected in significant amounts in milk and its half-life is about equal to that of Strontium-90—30 years. The ion exchange process described above can be carried out for the removal of cesium. However, above 90% removal can be maintained for only 10 to 12 rbv, after which a rapid decrease occurs. Its affinity for the resin is less than that of strontium, and is less in acidified milk than in normal milk. Consequently, the procedure for optimum removal of radiostrontium is inefficient for removing Cs-137. Further discussion of the effect of acidification and a potential method for maintaining a high level of removal is given in a later section.

Test runs for removing Strontium-90 from milk containing only environmental levels. Practically all of the research carried out during the development of the fixed-bed cation process was done by using Strontium-85, because the method for its assay is simple and rapid compared to that for Strontium-90. Strontium-85 was fed to a Holstein cow either orally or intravenously (in vivo-labeled), or added directly to milk in the laboratory (in vitro-labeled). A comparison of the removals obtained by the two methods of labeling milk is reported in another paper (2). Levels of about 1 μc per quart of milk were normally used for the removal studies. This compares with about 25 $\mu\mu c$ of Strontium-90 in current milk sources; i.e., the levels of Strontium-85 were about 40,000 times the environmental levels of Strontium-90. The percentage removal of Strontium-85 from milk labeled in vivo and of Strontium-90 should be the same. This was confirmed by determining the percentage of Strontium-90 removed from milk obtained from a local dairy. The average removal from three separate runs of 30 rbv each, processed in the pilot plant, was 95%. This is in good agreement with data obtained in the laboratory with the high levels of Strontium-85.

Cost of the process. The cost of operating the fixed-bed process as described above is estimated at 1.0¢ per quart of milk when USP grades of citric acid, potassium hydroxide, and chloride salts of Ca, K, Na, and Mg are used. If technical grades of the chloride salts are used for regeneration the cost is estimated at 0.5¢ per quart. These costs are based on pilot plant experience and include estimates for investment, investment burden, labor, and normal operating costs.

Monitoring for removal of radiostrontium. The analysis for the Strontium-90 content in milk requires about 3 wk. A rapid procedure would be highly desirable so that the efficiency of the removal process could be checked soon after processing. The percentage of stable strontium and of radiostrontium removed from milk will be the same; consequently, analyses for stable strontium were considered a possible means of monitoring for removal. This, conceivably, could be done in about three days. However, the $CaCl_2$ used as a regenerant contains enough stable strontium as an impurity to render this test of no value. Although a rapid procedure for testing the efficiency of removal is not available, a processor can be assured of removing over 90% of radiostrontium by maintaining proper control of the operating conditions. These include, particularly, pH of the milk, flow rate through the column, and adequate regeneration of the resin after each milk cycle.

Commercial scale evaluation. Primary emphasis on the project at Beltsville has been directed toward developing the fixed-bed ion exchange process for removing Strontium-90 from milk. The pilot plant procedure described above is now being evaluated on a scale of 12,500 lb per hour (100,000 lb per day) by a commercial dairy plant. This evaluation is being carried out under terms of a contract supported by the U. S. Departments of Agriculture and Health, Education and Welfare, so that a determination of its feasibility in an average dairy plant can be made.

FIXED-BED IODINE-131 REMOVAL PROCESS

A strong base anion exchange resin may be used to remove Iodine-131 under similar conditions to those used for the removal of cationic radionuclides. For use in fixed-bed columns the resin is regenerated with a mixed solution of the sodium salts of the major milk anions—NaCl, Na_2HPO_4, and sodium citrate. It appears from laboratory studies that approximately 90% removal can be maintained for a quantity exceeding well over 150 rbv (6). It is not necessary to acidify milk for this procedure. The strong base resins are characterized by amine, or fishy odors, particularly in the hydroxyl form. It is advisable to pretreat the resin before use with either a salt solution (NaCl), or alternate acid and base washes.

Pilot plant runs were made for flavor evaluations by passing milk through an anion resin (Dowex 2-×8) in a 6-in. glass column. The resin was regenerated with acid and base rinses.

Satisfactory flavor scores (above 35 on the A.D.S.A. score-card) were usually obtained from these runs. Few criticisms suggesting a fishy flavor were indicated by a ten-member judging panel.

FIXED-BED PROCESSES FOR REMOVING BOTH I-131 AND RADIOSTRONTIUM

An integrated pilot plant process has been arranged for removing both I-131 and radiostrontium by passing milk first through the anion resin column, then acidifying to pH 5.3 and passing it through the cationic resin. This process is currently being evaluated for removal of Iodine-131 and for flavor in the pilot plant.

Since iodine has such a great affinity for the resin, regeneration is more difficult than for the cation resin. It can be stripped from the resin with 2 N HCl, although the corrosive nature of this acid is objectionable. This step also requires complete regeneration with the mixed sodium salt solution before another milk cycle can be made. Further work in this area may be necessary before a completely satisfactory process is worked out.

LABORATORY INVESTIGATIONS WITH CONTINUOUS ION EXCHANGE PROCESSES

Another phase of the isotope removal investigations included the use of a continuous ion exchange contactor. It is a laboratory model having a capacity of about 12 gal. per hour. A schematic drawing of this unit is shown in Figure 1. The contactor loop consists of glass pipe of 1 in. in diameter except for the feed (milk) section, which is 2 in. in diameter. To begin an operation, the loop is filled completely with resin except for a part of the section between Valves 1 and 5. Water is admitted along with the resin so that air is excluded from the system. The loop contains a series of sections, each separated by valves so that all of the steps in the resin cycle—milk treatment, rinsing, detergent wash, and regeneration—are carried out within the unit. The solution pumps are automatically timed to operate for preset periods up to a few minutes, after which they are automatically cut off. During the short interruption—3 to 5 sec—the valves (No. 1, 2, 3, and 4 in Figure 1) separating the various sections are opened. Valve 5 is closed and sufficient water pressure applied as indicated in the diagram, to force the resin to move in a clockwise direction around the loop. The amount of movement depends on the hydraulic pulse time. A pulse time of 1.5 sec normally displaces the resin about 2.4 cu. in. The pulses

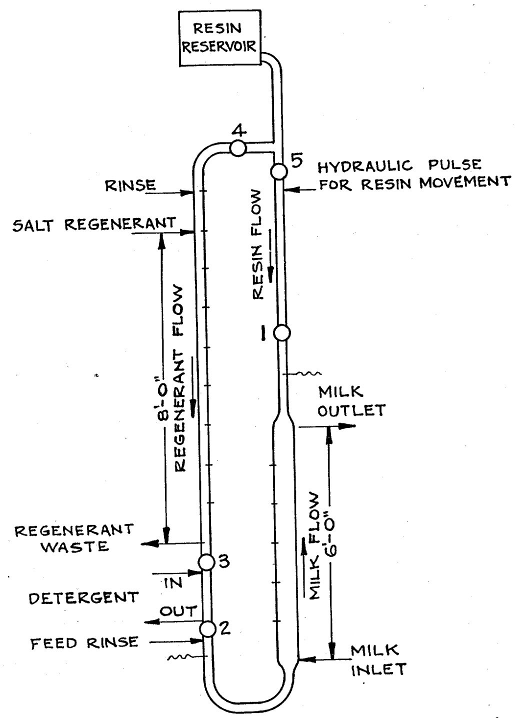

FIG. 1. Schematic drawing of continuous ion exchange contactor.

are usually spaced about 1.5 min apart. Thus, fresh regenerated resin is intermittently added to the milk section. After the interruption, the valves automatically close, except No. 5, which opens, and the solution pumps start again. A countercurrent flow of solutions with respect to resin is thus carried out in mechanical stages. The product flow, although not truly continuous because of the frequent interruptions necessary to accomplish the resin pulses, approaches continuity, since the resin pulse time is small compared to the solution flow time. A continuous countercurrent system has inherent advantages with respect to efficiency and product uniformity.

The continuous system was used to study (1) the removal of cationic radionuclides (Sr-85 and Cs-134) from milk using a strong acid resin in the mixed form, (2) the removal of Strontium-85 with the resin in a sodium form, and (3) the removal of Iodine-131 and simultaneous neutralization with a strong base resin in the hydroxyl cycle. The latter procedure was studied with the idea of using it as a second stage in a cation-anion removal process. Each of these uses is discussed below.

1. *Removal of cationic radionuclides (Sr-85 and Cs-134) by continuous ion exchange.* For removing radiostrontium, several runs were

made using the same resin and regenerant solution as described above for the fixed-bed cation removal process. Also, it was found necessary to acidify the milk in the same manner as is used for the fixed-bed process. Both citric and hydrochloric acids were used and, as discussed above, slightly more strontium was removed by using citric acid than by using HCl. Preliminary trials were made to establish operating conditions with respect to the milk and regenerant flow rates, and the resin pulse rate. Suitable rates for these materials were as follows (approximately):

Milk —750 ml per min
Regenerant— 85 ml per min
Resin — 2.4 cu. in. at 1.5-min intervals

Ninety per cent removal could be maintained when operating under these conditions at a milk pH of 5.3.

The amount of radiocesium removed under the above conditions during the first 3 hr ranged from 97 to 99%, but decreased to levels below 90% after that time. A run made under the same conditions without acidifying milk resulted in the removal of 99% of cesium for a period of 7 hr. Laboratory trials with batch studies confirmed these results, showing that the affinity of cesium for the resin is greater in normal milk than in milk acidified to a pH of 5.3.

2. *Removal of radiostrontium with the continuous contactor using a sodium-form resin.* Laboratory studies with batch systems have shown good removals of radiostrontium from milk with resin in the sodium or potassium forms. Such systems, however, are inherently inefficient, since large resin to milk ratios would be needed. Treatment of milk with such single ion charged resins in a fixed column is also not feasible, since the effluent milk changes continuously in mineral composition and the amount of strontium removed decreases rapidly. A continuous countercurrent system has advantages in both respects. However, any system using a sodium form resin results in a great increase in the sodium content of milk and decreases in the other cations. Thus, subsequent readjustments in mineral composition would need to be made.

Tests for the removal of Strontium-85 with a sodium-form resin were made by passing milk (in vivo-labeled with the isotope) through the continuous contactor containing Amberlite IR-120. The regenerant used was 10% NaCl. An average of over 98% removal was obtained from a 7-hr run. As expected, the sodium concentration in the milk increased greatly, with marked decreases in the other cations. Potassium was reduced to near zero. It should be possible to restore the cation balance by passing the milk from the sodium cycle through a second contactor containing resin regenerated with an appropriate mixed salt solution. Since this was not available, restoration of the cation balance to near normal was accomplished by passing the milk through a fixed-bed resin in the mixed ion form (Ca-K-Na-Mg). The appearance of the pasteurized, homogenized product was normal. The flavor of the milk was considered acceptable.

For a feasible process this procedure will require cycling the milk through two continuous contactors in series. However, it has the advantage that a high level of removal can be obtained without the need for adding acid or neutralizers for pH adjustments. The explanation for this is no doubt the result of the dispersing effect of sodium ion on the caseinate micelle.

3. *Removal of I-131 and simultaneous neutralization of milk with a strong base resin using a continuous contactor.* Early in these investigations some of the acidified milks processed through laboratory fixed-bed columns were neutralized by adding batchwise, and with thorough stirring, small quantities of a strong base (Dowex 2-×8) resin in the hydroxyl form. The resin was then separated by filtering. This type of resin is noted for having an amine or fishy odor. However, milk with an acceptable flavor was usually obtained if the resin was thoroughly rinsed with water before use (5, 7). A batch procedure is not suitable for continuous processing. Also, there would be problems in separating, cleaning, and regenerating the resin.

In principle, a continuous procedure of neutralizing milk with an anion resin appears feasible. An anion resin is also effective for removing I-131 from milk. Investigations of a cation-anion process in series were made by passing acidified milk first through a cation resin and then through an anion resin in the continuous contactor. For most of these studies, the cation process consisted of a fixed-bed column described above, since only one continuous unit was available.

The initial charge of anion resin (Dowex 2-×8-Cl) was pretreated by alternate acid-base cycles and then regenerated with a mixed citrate-phosphate-chloride solution (6). Partial regeneration to the hydroxyl form during the milk run was accomplished with 5% NaOH. Conditions for operating the anion cycle—milk, resin, and regenerane flow rates—were established so that the most effective neutralization was obtained. The frequent interruptions in the cycle necessary for the periodic resin movement cause cyclic variations in the pH of the effluent milk; the pH of the pooled milk, of course, will have an average value. Irregular fluctuations in the resin pulse rate, because of packing, or of spaces in the loop which are void of resin, result in undesirable and uncontrollable changes. Restoration of the pH to near normal, however, was generally successful in these runs.

Flavor evaluations of samples from initial runs were generally good, but some unacceptable scores were obtained after the resin had been in the loop for about 1 wk. The criticisms were identified by some panel members as the characteristic amine, or fishy, flavor. Preliminary work indicated this problem could be minimized by mixing a small amount of a carboxylic acid resin with the anion resin.

Removal of Iodine-131 from milk (in vivo-labeled) by the continuous process described above was quite effective, varying from 91 to 86% during a 10-hr run. The amount of milk passed through during this time was equivalent to about 90 rbv, based on all of the resin in the loop. The regenerant (5% NaOH) removed very little of the iodine from the resin. Althoughly a high level of removal was obtained with ineffective regeneration, for practical processing some means of stripping the iodine from the resin would need to be provided. This conceivably could be done with an HCl cycle preceding the NaOH cycle, or by removing the resin periodically from the contactor for regeneration in columns.

OTHER PROCESSES FOR REMOVING RADIOSTRONTIUM

Several exchangers with special functional groups besides the strong acid resin were also investigated for removing radiostrontium. These included carboxylic, phosphorous, and phosphoric acid resins, zeolite inorganic exchangers, a chelating resin, and a zwitterion. The most promising of these was an intermediate-strength carboxylic acid resin. In excess of 90% strontium removal was obtained for about 18 rbv of milk. This compares with 30 to 35 rbv for the strong acid resins. This type of resin is easily regenerated with an acid solution, which may be an advantage if the Strontium-90 contaminated regenerant should become a waste disposal problem. However, a fixed-bed process using this resin would require an extra regenerating cycle before the resin would be ready for reuse.

A method of removing radiostrontium from milk by precipitating it with an excess of calcium phosphate was also investigated (8). It was found that by adding 80 g per liter of tricalcium phosphate and heating to pasteurization temperature (145 F) for 30 min, about 85% of radiostrontium could be removed by centrifugal separation of the precipitate. However, it was also found that this method results in the precipitation of about one-third of the milk nitrogen (protein). From a practical viewpoint the necessity for adding 80 g per liter (almost 1 g per gram of milk snf) for effective removal appears excessive.

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