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Sources and Content of Iodine in California Milk and Dairy Products[1]

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ABSTRACT

In recent years, milk and milk products have been implicated as a major contributor to dietary iodine. The possible sources of iodine in milk are supplemental iodine in dairy feeds, iodophor-containing sanitizers used at the dairy farm and/or the processing plant, iodophor-containing teat dips used to control the spread of mastitis among dairy cows, and iodine-containing medications used by veterinarians. A five-year program to determine the California raw milk iodine concentration and identify the sources of adventitious iodine has resulted in the California dairy industry deciding late in 1980 to reduce iodine supplementation of dairy feeds. This resulted in a decrease in milk iodine concentration in samples received in 1981 to 256 ± 234 μg/kg compared to 1980, when the concentration was 474 ± 304 μg/kg. The industry has set up a program to monitor the raw milk iodine concentration at the producer level, thus ensuring that the concentration will continue to decline.

The importance of iodine in human nutrition is well established (*30*). During the last few years concerns have been expressed that the population in the United States might be exposed to more iodine in their diet than is necessary or desirable (*27*), and that a major contributor of dietary iodine is milk and dairy products (*21,27*).

Iodine is a natural constituent of milk. The concentration of iodine in milk has been reported to range from as low as 8 μg/kg to as high as 220 μg/kg (*24*). Values reported before 1970 were generally less than 100 μg/kg, and no higher than 165 μg/kg. The increase in milk iodine concentration since 1970 has been attributed to supplemental iodine in dairy rations (*17*), iodophor-containing sanitizers used on the dairy farm and/or at the creamery (*10,17*) iodophor-containing teat dips used to control the spread of mastitis among dairy cows (*7,12*), and medications containing iodine used by veterinarians in the treatment of various bovine health problems (*7,11*).

The dairy industry in California realized some might suggest that high levels of iodine in milk could be a public health problem, so we set out to determine the iodine concentration of raw milk, market milk and dairy products in California, and if unsatisfactory levels were found, determine the source and necessary corrective steps to reduce the levels. This report covers our findings from the beginning of this program to the present time (1977 to 1981).

MATERIALS AND METHODS

Raw milk samples were collected from dairy farm holding tanks using procedures that have been reported (*4*) to avoid possible contamination of the milk. Samples were stored and shipped at 4°C or less (frozen storage preferred) to our laboratory where the iodide concentration was determined using a selective ion electrode and an Orion 701 Ionalyzer by weighing duplicate 100-g samples into 250-ml beakers. After adjusting the ionic strength by adding 100 mg of $NaNO_3$ or 1 ml of 2 *M* $Ni(NO_3)_2$, the electrode potential was read after the electrodes had been allowed to equilibrate in the stirred sample for 5 min. After the addition of 10 ml of KI solution of known concentration (ca. 10 × the concentration of the sample, as judged from the potential) the new potential was read after another 5-min equilibration period. The difference in potential and the known concentration of the added KI solution were used to determine the concentration of iodine in the unknown by the procedure in the Orion instruction Manual, p. 15 (*26*).

Market milk and dairy product samples (see Table 7) were collected from retail dairy cases and held and shipped at 4°C to our laboratory where their iodine concentration was determined using a procedure based on those of Stabel-Taucher (*28*) and Stolc and Nemeth (*29*).

A trial to determine whether milk samples could be frozen and stored as a method of preservation before iodine determination using the iodide selective ion electrode was performed. Raw milk collected from the University dairy barn holding tank (not more than two milkings old) and pasteurized homogenized whole milk purchased from a local market were each split into 24 separate 250-ml samples. Three samples were analyzed immediately, and the other 21 were frozen and stored at -29°C. Each week three more samples of each milk were analyzed until all the samples had been used.

The effects of pasteurization and homogenization on the selective ion electrode detectable iodide in milk were measured using a pilot plant scale (113 L/h) HTST pasteurizer-homogenizer at 90°C, 281 kg/cm^2, and 10 sec holding time.

The distribution of iodine in cows' milk was determined by separating the cream centrifugally and then separating the casein from the soluble

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TABLE 1. *Comparison of iodine concentrations in raw milk using two methods.*

Sample	Iodine concentration (μg/kg)	
	Direct	Method of addition
1	160	129
2	246	174
3	79	104
4	147	184
5	83	110
6	100	121
7	137	169
8	97	124
9	127	155
10	146	216
11	161	164
12	194	265

proteins centrifugally, using the conditions suggested by Holt et al. (*18*). Iodine in each fraction was estimated using the microchemical procedure used for market milks.

Experiments to determine whether a backflush installation contributed to the raw milk iodine concentration were conducted by having a cooperative dairy farmer milk his herd for 9 d without using the backflush system. Samples of the tank milk were collected on each of these 9 d, and for 7 d after the backflush system was again in use. A second experiment was conducted by collecting foremilk samples from six cows in a herd, then milking them into a bucket using equipment that had been backflushed. The difference in iodine concentration between the bucket- and foremilk samples was presumed to be due to residual iodine in the backflush equipment.

All statistical analyses were performed using computer programs available in the Statistical Package for the Social Sciences (*25*).

RESULTS AND DISCUSSION

Other researchers have determined the iodine concentration in milks using selective ion electrodes and compared the results to those obtained using x-ray fluorescence (*8,9*), neutron activation analysis (*23*) and microchemical procedures (*5,23*). In some reports (*5,8,9,23*), the method of additions approach is advised. In others (*22,31*) a direct measurement approach is advised. Our research (Table 1) demonstrated that with raw milk samples the method of additions is preferable. The data in Table 1 are for 12 raw milk samples that were analyzed using 100 mg of $NaNO_3$ to adjust the ionic strength. The iodine concentration measured directly is different from that measured by the method of known addition. The mean difference, 19.8 μg/kg is 14.2% of the mean concentration determined by direct measurement and the difference varies from 29.3% low to 36.6% high. This is beyond the tolerance of 4% suggested by Orion (*25*), so we, along with others (*8,9,23*), have adopted and recommended that the method of standard addition be used routinely. Our research confirmed that the iodide selective ion electrode responds to solutions of iodide or iodophor added to milk, as has been demonstrated (*8,22,23*).

The data (Table 2) show that no change in iodine concentration occurred during 7 weeks of frozen storage. Analyses of variance indicated that time of storage accounted for only 2.7% of the variation in iodine concentration, and consequently was not statistically significant ($F = 1.67$, $P > .25$). We were pleased to note this since we had found that the commonly used chemical means of preservation (both formaldehyde and potassium dichromate) led to errors in the measurement of milk iodine concentrations with the iodide selective ion electrode. Craven and Griffith (*8*) reported significant ($P<.01$) losses of iodine after 2 weeks frozen storage; we cannot explain the different between these observations, and can only suggest that anyone planning to use this procedure test it first, since the difference might be related to the rate of freezing and/or the temperature of frozen storage.

Using a pilot plant scale HTST pasteurizer-homogenizer, we observed a four-fold increase (from 150 to 600 μg/kg) in the selective ion electrode detectable iodine in raw milk after heating. In (*22*), the holding time was 5 min at 90°C and the increase was from 190 to 480 μg/kg. The observed agreement between electrode and microchemical measurements (*5*) or between electrode and x-ray fluorescence measurements (*8,9*) or between electrode and neutron activation measurements (*23*) indicate that the increase in the apparent iodide concentration was probably not due to the release of organically-bound iodine. Although the literature is unclear on the distribution of iodine in milk, no one has reported finding 75% of the iodine bound organically. An electrode very similar to the iodide ion selective electrode (lacking only the AgI) has been used to measure sulfhydryl and disulfide concentrations (*13,14*). When milk is heated, sulfhydryl groups are

TABLE 2. *Effect of frozen storage on the selective ion electrode measurement of the iodine concentration of raw and pasteurized milk.*

Storage time (wk)	Iodide concentration (μg/kg)			
	Raw		Pasteurized	
	Mean	SD[a]	Mean	SD[a]
0	245.3	6.4	532.0	15.1
1	257.3	8.0	587.7	11.2
2	261.0	5.2	570.0	15.0
3	235.3	1.2	495.0	21.8
4	231.7	6.0	473.3	2.3
5	238.7	5.0	502.0	17.3
6	262.7	13.3	499.3	2.3
7	268.3	3.5	491.0	7.0

[a]Standard Deviation based on analysis of three samples.

TABLE 3. *Iodine concentration of California raw milk by year.*

Year	Iodine concentration (μg/kg)					
	N[a]	Mean	SD[b]	Min[c]	Max[d]	%>500
1977	644	360.5	336.7	58.0	3886.0	21.1
1978	426	387.6	375.8	65.0	2924.0	22.1
1979	96	568.7	320.2	26.0	2104.0	58.3
1980	473	474.0	303.7	49.0	2038.4	38.3
1981	1389	255.9	233.7	21.6	4048.0	9.0

[a]Number of samples analyzed.
[b]Standard deviation.
[c]Minimum concentration found.
[d]Maximum concentration found.

liberated as proteins are denatured (*31*). More particularly, the conditions used in high-temperature-short-time (HTST) pasteurization can lead to denaturation of serum proteins and activation of sulfhydryl groups (*31*). We therefore concluded that the ion selective electrode was not suitable for processed milks or dairy products and decided to use the microchemical procedure (Materials and Methods) for processed dairy foods.

Our data on distribution of iodine in raw milk (55 samples) indicate that about 17% is associated with the cream, 22% is associated with the casein, and the remaining 61% remains in the supernatant liquid formed when the casein is separated from the skim milk using the procedure reported by Holt et al. (*18*).

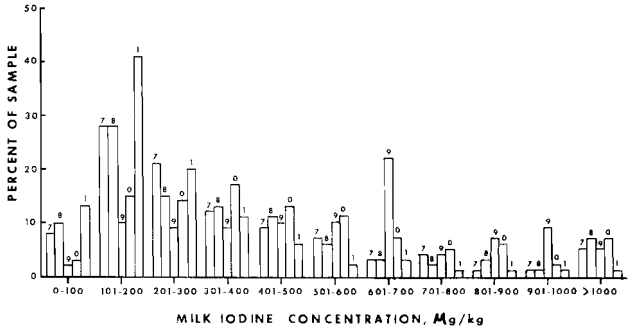

Figure 1. *Changes in the distribution of raw milk iodine concentration in the years 1977 to 1981. The number at the top of each bar indicates the last digit of the year.*

Since 1977 the iodine concentration of more than 3000 raw milk samples collected from dairy farms in California has been measured using selective ion electrode. The data (Table 3) indicate that the mean iodine concentration in the California raw milk samples received for analysis peaked in 1979 and has been declining since then. Also,the proportion of samples having iodine concentrations higher than 500 μg/kg peaked in 1979 and was lower in 1981 than in any previous year of our study. A histogram (Fig. 1) was prepared by partitioning each years' samples according to their iodine concentration. In 1981, over 50% of the samples had concentrations below 200 μg/kg. In every year of the study, there were some herds producing milk with this little iodine. There were no pronounced geographical influences of milk iodine concentrations. Milk from adjoining dairy farms differed by as much as 1000 μg/kg.

Veterinarians at U.C. Davis are working on a program to reduce the spread of bovine mastitis by flushing the milking unit with an iodophor after each cow is milked. This iodophor "backflush system" is designed to control the spread of mastitis among dairy cows in the same herd during milking.

Briefly, this is the principal of operation; after a cow has been milked out, the milking unit is connected to the sanitizer line and flushed with potable water to remove the residual milk. Then iodophor sanitizer (25 ppm active iodine) is flushed through the unit for a time to kill remaining vegetative bacterial cells (about 50 sec). Finally, the unit is flushed with potable water to remove residual iodophor.

We collaborated with veterinarians to determine whether the backflush systems used in California were increasing the iodine concentration of milk.

TABLE 4. *A representative test of a backflush installation.*

Day	Backflush	Tank milk iodine concentration (μg/kg)
1	Off	986
2	"	1004
3	"	988
4	"	868
5	"	874
6	"	712
7	"	802
8	"	881
9	"	1071
10	On	1243
11	"	1392
12	"	1167
13	"	1278
14	"	1061
16	"	1082

The data (Table 4) show representative results. In 9 d with the backflush sytem off, the tank milk iodine concentration averaged 904 ± 104 μg/kg. In 6 d with the backflush system working, the tank milk iodine concentration averaged 1204 ± 126 μg/kg. The mean difference, 300 μg/kg, was very highly significant. To test the effect of corrective measures taken in the operation of the backflush system at another farm, fore- and bucket-milk samples were collected from six cows whose milk production varied from 12.2 to 20.4 kg per milking. The average foremilk iodine concentration (Table 5) was 130 μg/kg; the average bucketmilk iodine concentration was 180 μg/kg. The mean difference, 50 μg/kg, was not statistically signifi-

TABLE 5. *Second test of a representative backflush installation.*

Cow	Milk iodine concentration (μg/kg)		
	Foremilk	Bucket	Bucket-foremilk
1	132	197	65
2	113	116	3
3	140	178	38
4	150	166	16
5	150	157	7
6	93	265	172

cant. We concluded that properly used the backflush system would contribute no more than 50 μg/kg to the milk iodine concentration.

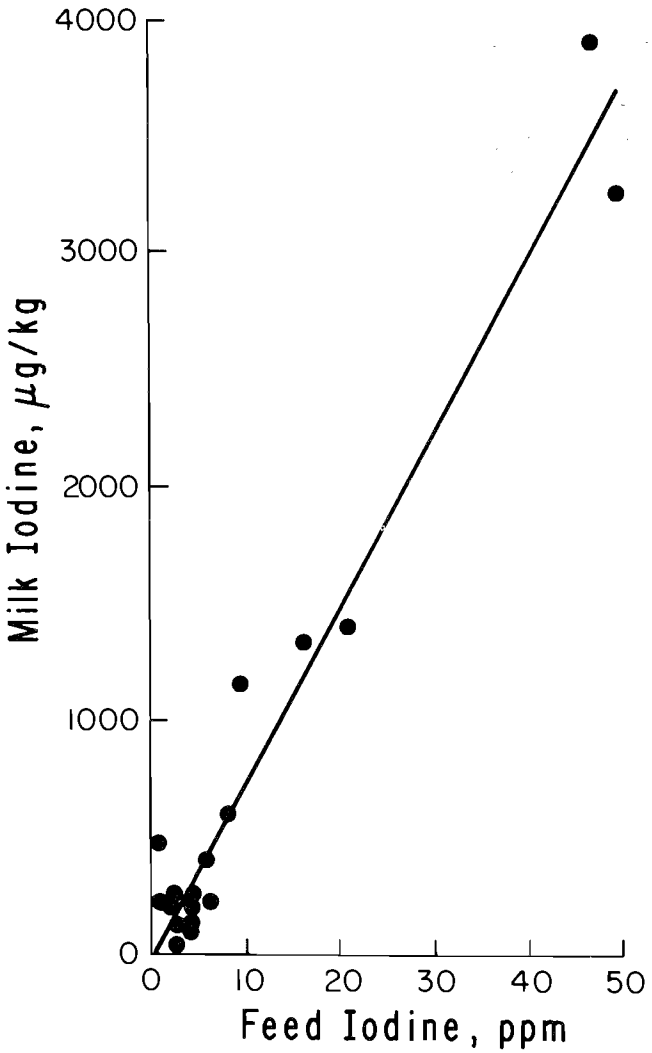

Figure 2. *Correlation between milk iodine concentration and feed iodine concentration in samples taken in 1979 at seventeen California dairy farms.*

To measure other sources of iodine in milk, arrangements were made to collect feed and milk samples from 17 representative dairy farms. Iodine in feed was measured using Hoover's (*19*) iodide selective ion electrode. Iodine in milk was measured using the iodide selective electrode. The results (Fig. 2) confirmed previous reports (*1,3,16*) that milk iodine concentrations are directly correlated to iodine concentrations in cows' feed. In our sample, the correlation coefficient (r) was .89; and the regression equation was:

$$\mu g\ I/kg\ milk = 56\ (ppm\ I\ in\ feed) + 72$$

Iodophor teat dips have been reported to be a source of increased iodine concentrations in milk (*7,12,20*). Our research confirms the report by Cantor and Most (*6*) that when these products are used according to the manufacturer's instructions, the amount of iodine contributed to the total iodine concentration in milk is negligible.

Some question arose concerning the amounts of iodine that might be added to California market milks and dairy products during processing. In 1977 we collected samples from retail dairy cases in local stores and measured the iodine concentrations using the microchemical procedure. Concentrations of iodine in nonfat, lowfat, regular and extra rich milks did not differ significantly. The results (Table 6) of a comparison of four plants over a period of 5 months indicate that the concentration of iodine in market milk was higher in the fall than in the summer and that the plants ranked (from lowest to highest) A, C, B, D. The mean iodine concentration in the 111 samples analyzed was 414 μg/kg, standard deviation 104 μg/kg. The difference between raw (Table 3) and processed (Table 6) milk iodine concentrations was 54 μg/kg; this difference, while large, was not statistically ($P>.05$) significant. We concluded that any iodine added to market milks during processing was negligible, and that the place to reduce the iodine concentration of milk was at the dairy farm.

The iodine concentrations of dairy foods available to the California consumer in 1977 is shown in Table 7. The iodine values for the instant dry milk and powdered infant formula appear high, but the reconstituted products would have iodine concentration similar to the market milks (Table 6) and the raw milk supply for 1977 (Table 3). The mozzarella cheese appears to have a higher iodine concentration than we might expect. However, when we separated casein from skimmilk centrifugally, we found iodine concentrations in the casein as high as 10 mg/kg. The mean value in 55 individual cow samples was 1704 μg/kg, which agrees with the mozzarella cheese values well enough that we feel justified in our conclusion that the iodine concentration in dairy products vailable to the California consumer in 1977 reflected the iodine concentration in the raw milk supply, and was not increased during the processing of the milk.

In 1979, University of California representatives met with dairy industry leaders to communicate the concerns regarding the high iodine concentrations being found in the California raw milk supply. From these meetings some creameries, cooperatives and feed companies undertook an educational program with the dairy farmer. The educational program for the dairy farmer focused on reducing iodine supplementation in feeds and exercising prudent use of iodine-containing materials at the dairy farm. We believe that the observed decrease in iodine from 1979 to 1980 was in part due to this industry's educational effort.

Late in 1980, dairy feed manufacturers, dairy farmers, creamery representatives and University personnel again

TABLE 6. *Iodine concentrations in California market milks from four plants during 1977*[a].

Month	Milk iodine concentration (μg/kg) for plants			
	A	B	C	D
July	270	410	377	402
August	281	418	404	428
September	254	406	380	366
October	462	402	447	472
November	548	371	518	584

[a]Means of nonfat, lowfat, regular, and extra rich.

TABLE 7. *Concentrations of iodine in dairy products available to California consumers in 1977.*[a]

Product	Number of observations	Concentration (μg/kg)			
		Low	High	Mean	Standard deviation
Cottage cheese[a]					
Dry curd	3	312	418	380	59
Low fat	21	248	678	356	120
Regular	21	174	683	325	134
Yogurt[a]	20	212	592	458	131
Instant dry milk	8	375	9920	5569	2803
Powdered infant formula	2	1360	2300	1812	507
Prepared infant formula	3	161	612	380	194
Milk beverages (diet foods)	5	294	1225	713	343
Evaporated milk	9	428	1795	829	344
Sour cream[a]	5	216	478	284	109
Cultured buttermilk[a]	5	278	524	370	77
Cheddar cheese	11	170	810	492	213
Feta cheese	2	46	86	66	28
Mozzarella[a]	5	805	1570	1218	310
Other cheeses	6	104	712	306	225
Processed Cheddar	5	210	1190	571	446
Kefir[a]	3	106	257	158	86
Ice cream[a]	10	177	500	387	105
Ice milk[a]	7	282	564	466	213

[a]Samples that were California-manufactured.

met to determine means to reduce further the iodine in raw milk. It was agreed that iodine would be added to dairy feeds at concentrations to meet the cow's nutritional needs and would not exceed 500 μg/kg of feed except in those instances recognized as exceptional by National Research Council (NRC). We then noted a decrease in raw milk iodine concentrations in 1981 due to this industry effort.

During 1981, monitoring programs were set up by some California creameries to detect increases in iodine in farm tank milk. When increases were noted, creamery and cooperative field representatives would contact the dairy farmer and determine what corrective action could be taken. It is hoped that through the cooperative efforts of dairy farmers, processor representatives and feed company personnel, the iodine concentration in the California raw milk supply will continue to be low.

CONCLUSION

Our objectives were to determine the iodine concentrations in California milk and dairy products, the source of, and practical methods to reduce, the adventitious iodine in these products. During a 5-year period (1977-1981), we succeeded in identifying feed supplements as the major source of adventitious iodine in the California raw milk supply. In late 1980, members of the California dairy industry voluntarily decided to eliminate iodine supplementation of dairy feeds except in those instances that the NRC recognizes as requiring iodine beyond their normal recommendation. The result was that in 1981, the raw milk iodine concentration in California was lower than in any of the previous 4 years. The dairy industry in California has set up the necessary programs to monitor raw milk iodine concentration at the producer level.

We recognize that the California dairy industry is unique and do not suggest that the California solution (elimination of iodine supplementation of dairy feeds) would apply everywhere. Indeed, several workers (*2,3,15*) have reported evidence of iodine deficiencies among dairy cattle, cases in which the California solution would obviously be contraindicated. However, we do feel that the approach taken in California to determine the raw milk iodine concentration and what measures, if any, were required to lower it could be successfully applied anywhere.

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