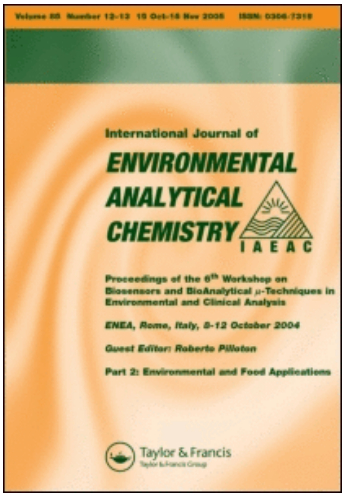


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# The Use of Ion-Exchange Filters for the Determination of Iodide in Milk by X-ray Fluorescence Spectrometry

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An X-ray fluorescence spectrometric method has been developed for the quantitative determination of iodide in milk which makes use of anion-exchange filters for the isolation and concentration of iodide from the liquid samples. The milk is deproteinized with trichloroacetic acid, filtered then passed through an anion-exchange paper disc to remove the iodide. The disc is removed, dried then analysed by X-ray fluorescence spectrometry. Six samples, consisting of homogenized, 2% fat and skim milk were analysed and found to contain between 0.37–0.67 ppm iodide. Detection limits in milk were estimated to be 0.05 ppm.



## INTRODUCTION

It has been reported that the iodine content of the diet appears to be increasing and is attributed to the use of food additives[1,2] and, in milk and other derived products, to the use of iodine-containing sanitizers in the dairy industry.[3–5] High levels of iodine in the diet have been linked to thyrotoxicosis.[4,6] Because of this, it is important that suitable methodology for monitoring of the diet be available in order that health assessments may be made.

X-ray fluorescence spectrometry has been employed in our laboratory for the routine analysis of a number of elements in foods

and the technique is particularly attractive in terms of speed and the low degree of sample preparation required. The approach has been applied to total iodine in milk samples[7,8] where the freeze-dried milk is compressed into pellets for X-ray measurement. We have found that the sample preparation was somewhat time consuming since difficulty in preparation of the pellets was encountered because of fat present in the freeze-dried residue. Since it has been reported that 97% of the iodine in natural bovine milk is secreted as iodide,[9] we decided to investigate methodology for iodide determinations in milk by removing the anion from milk samples by means of filtration with anion-exchange resin impregnated filter paper[10] which may then be washed and directly analysed by X-ray fluorescence. Results of this work are reported herein.

## EXPERIMENTAL

### Reagents and materials

Trichloroacetic acid solution (5%) was prepared by dissolving 5 g of reagent grade trichloroacetic acid (TCA) in water and diluting to 100 ml in a volumetric flask. Potassium iodide (KI) stock solution (1 mg I/ml) was prepared by dissolving 262 mg analytical grade KI in water and diluting to 200 ml in a volumetric flask. Standard working solutions were prepared by appropriate dilutions of the stock. The milk samples consisted of homogenized, 2% and skim milk and were purchased from local supermarkets. Spiked milk samples were prepared at 1 mg I/ml and 0.5 mg I/ml).

### Apparatus

A Phillips X-ray spectrometer PW 1410 was operated under the following conditions. Chromium target tube: 50 kV and 40 mA. LiF 200 analysing crystal. Flow counter, 1620 V,dc (10% methane, argon gas). Vacuum and spinner on. Iodide peak intensity measured at 93.73° for 1 minute. Background was determined as the average of counts at 92° and 95°.

Ion-exchange paper discs (5.0 cm dia.) were cut out from SB-2 ion-exchange resin loaded paper (Reeve Angel Products). A Millipore filter assembly (Millipore Corporation) was fitted to a 500 ml filter

flask and a regulating valve (Swagelok) was inserted in the pressure tubing between the filter flask and the aspirator.

### Sample analysis

Duplicates (20 ml) of the milk sample and 20 ml each of two spiked milk samples were pipetted separately into individual 150 ml Erlenmeyer Flasks. Then, 25 ml distilled water was added to each flask and left to stand for 5 minutes in a constant temperature water bath at 60°C. A 10 ml volume of TCA solution was added to each flask, which was swirled and left for another 10 minutes. The flasks were removed, cooled and the solutions filtered through No. 1 Whatman filter paper and the clear filtrate retained.

One SB-2 disc was placed with the smooth side up on the base of the Millipore filter assembly and the funnel connected. The disc was conditioned as follows. Three ml of 5*N*-NaOH were placed on the disc and after one minute the vacuum was turned on using the regulating valve. The disc was washed 3 times with 5 ml distilled water, then the vacuum turned off. For sample analysis, the clear milk filtrate was poured into the funnel and the valve opened to allow filtration at a rate of 2 drops per second until all of the liquid had passed through the filter disc. The disc was removed and dried in a fumehood. The remaining samples were treated in the same way. For the blank, 20 ml distilled water was used. The X-ray counts were obtained on all discs and the net counts determined by subtracting the background counts from the counts at 93.73°. A standard addition curve was prepared for quantitation as shown in Figure 1.

## RESULTS AND DISCUSSION

Our initial attempt to isolate iodide ion from milk was simply to pass the liquid samples through the ion-exchange filter. However, the filter paper quickly became clogged, preventing the filtration from proceeding. Precipitation of the milk fat and protein with trichloroacetic acid beforehand however, resulted in a clear filtrate which readily passed through the ion-exchange filter. Trichloroacetic acid proved to be superior to 5*N*-$H_2SO_4$ which was initially used. The latter treatment occasionally resulted in cloudy filtrates and in most cases when neutralization with 5*N*-NaOH was done, a further

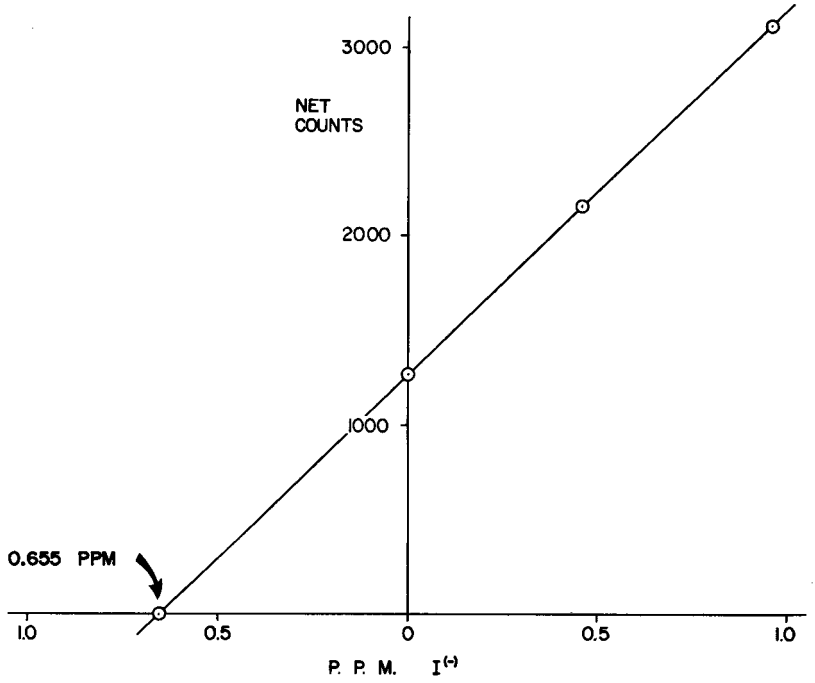


FIGURE 1 Typical standard addition curve for iodide in a homogenized milk sample. Concentration of iodide found was 0.655 ppm I.



precipitation occurred, requiring another filtration before the solution was passed through the ion-exchange paper. No neutralization of the filtrate was done using the trichloroacetic acid precipitation, thus saving significant time during the analysis. However, under the strongly acid conditions the retention of the iodide was reduced by about 25% (the optimum pH for iodide removal from solution with the SB-2 ion-exchange paper employed herein being slightly less than pH 7).[10] Nevertheless, standard solutions of KI gave linear responses in the concentration range of 0.25–1.5 ppm I which was the range of interest. The detection limit of 0.05 ppm (3 × std. dev. of background counts) was more than adequate for the sample analyses.

The iodide content of four homogenized (different dairies), one 2%, and one skim milk sample is shown in Table I. The average results

TABLE I
Iodide content of milk samples by X-ray fluorescence spectrometry

| SAMPLE | IODIDE, ppm I[a] | TOTAL IODINE ppm I[b] |
|---|---|---|
| Whole Homogenized | 1. 0.435, 0.458 | 0.509 ± 0.005 |
| | 2. 0.564, 0.515 | |
| | 3. 0.510, 0.470 | |
| | 4. 0.655, 0.672 | |
| 2% Homogenized | 0.400, 0.374 | 0.384 ± 0.002 |
| Homogenized Skim Milk | 0.479, 0.493 | 0.448 ± 0.004 |

[a]Duplicate values obtained by this method.
[b]Total iodine results (± std. dev.) from reference 11; obtained on similar but different samples.



ranged from 0.37–0.67 ppm I. These results compare well to the total iodine results found in similar milk samples using an acid digestion-colorimetric procedure[11] and illustrate that the iodine content of milk is indeed essentially iodide as reported earlier.[9] This also indicates that for milk samples acid digestion or ashing techniques are not required.[9]

Although the detection limits are similar for both the method described here and the pellet technique, we have found the former to be preferred in terms of ease of sample preparation and speed of analysis. This method should be applicable to other dairy products such as infant formula, ice cream, yogurt and cheese. With selective optimization, the approach used in this work may be used for the isolation of a number of other anions for quantitation by X-ray fluorescence spectrometry.

## References

1. J. A. Pittman, G. E. Dailey, and R. J. Beschi, *New Eng. J. Med.* **280,** 1431 (1969).
2. R. L. Vought, F. A. Brown, and J. Wolff, *J. Clin. Endocrin. Metabol.* **34,** 747 (1972).
3. D. G. Dunsmore and S. M. Wheeler, *Aust. J. Dairy Technol.* **32,** 166 (1977).
4. R. J. Connolly, *Med. J. Aust., March* 633 (1971).
5. M. M. Joerin and A. F. Bowering, *N.Z. J. Dairy Sci. Technol.* **7,** 118 (1972).
6. J. Wolff, *Am. J. Med.* **47,** 101 (1969).

7. E. A. Crecelius, *Anal. Chem.* **47,** 2034 (1975).
8. G. S. Craven and M. C. Griffith, *Aust. J. Dairy Technol.* **32,** 75 (1977).
9. E. W. Bretthaver, A. L. Mullen, and A. A. Maghissi, *Health Phys.* **22,** 257 (1972).
10. P. L. Warren, *Proc. Analyt Div. Chem. Soc. Jan.* 13 (1977).
11. P. W. F. Fischer and M. R. L'Abbé, *J. Assoc. Offic. Anal. Chem.* **64,** 71 (1981).