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DETERMINATION OF IODINE IN MILK PRODUCTS AND BIOLOGICAL STANDARD REFERENCE MATERIALS BY EPITHERMAL NEUTRON ACTIVATION ANALYSIS

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The biologically essential trace element, iodine, has been determined in various milk products by epithermal neutron activation analysis /ENAA/ after sealing in quartz and irradiating under cadmium cover. The method was extended to several IAEA and NBS biological reference materials.

INTRODUCTION

Iodine is regarded as an essential trace element although its role is not fully understood[1]. However, iodine concentrations are usually so low /<5 μg g^{-1}/ that recorded values are sparse or of doubtful validity. That this applies particularly to milk products was illustrated in a recent summary of results for the reference material, IAEA milk powder A11, for which values for the iodine content were estimated to be from 0.05 to 2.6 μg g^{-1} depending on the analytical technique used[2]. An earlier measurement using radiochemical neutron activation

analysis[3] gave a value of 0.082±0.003 μg g^{-1} for this reference material.

We have experienced similar problems in the determination of iodine in commercially available milk powders. The results of a recent round-robin between local laboratories, summarised in Table 1, show the analytical techniques used and the variation produced.

Iodine has been determined non-destructively in many biological materials by activation analysis with resonance neutrons[4-8], epithermal neutron activation analysis /ENAA/. This technique relies on a value of the resonance integral for iodine which is much larger than those for the major interfering elements, Na, Cl, Mn and Br. We have extended this method to milk products, ensuring that no loss of iodine occurred during the activation process by sealing the sample in quartz tubes. A series of standard reference materials was also examined.

EXPERIMENTAL

All liquid milk samples were lyophilised before irradiation. Milk products and a series of IAEA and NBS standard reference materials /IAEA SRMs: Oyster tissue MA-M-1, Fish homogenate MA-A-2, Blood, Milk powder A11; NBS SRMs: 1566 Oyster tissue, 1571 Orchard leaves, 1572 Citrus leaves, 1577 Bovine liver /were weighed /300-400 mg/ into prewashed /nitric acid, distilled water, AR ethanol/, high purity, quartz vials and heat sealed. An iodine standard solution was prepared by dissolving high purity KI in distilled water. An aliquot containing 10 μg of iodine was pipetted into a quartz vial before heat sealing.

All samples were inserted individually into a cadmium box with a minimum wall thickness of 0.7 mm and placed

TABLE 1

Concentration of iodine in milk powders /μg g^{-1}/

Sample	Colorimetric analysis				Distilln	XRF
	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6
A	0.80	1.02	1.16	0.93	2.20	1.34
B	0.67	0.74	0.88	1.25	2.10	1.66
C	0.89	1.08	1.28	1.50	4.20	2.40
D	0.78	0.92	1.09	1.20	1.70	2.10
E	0.39	0.41	0.48	0.33	1.80	1.09

inside an aluminium rabbit for transport into the pneumatic transfer facility of Moata, a 100 kW Universities Training Reactor /Argonaut/ located at the Lucas Heights Research Laboratories. The samples were irradiated at a thermal flux of 6×10^{11} n $cm^{-2}s^{-1}$ and an epithermal flux of 2×10^{10} n $cm^{-2}s^{-1}$. Optimum irradiation, cooling and counting times were calculated using the Advance Prediction Computer Program /APCP/ of Guinn et al.[9]. After experimental testing, the following standard protocol was adopted. Samples were irradiated for 15 min and, after a decay period of 20 min, each quartz-encapsulated sample was counted for 1000 s on a 20% Ge/Li/ detector, 1.9 keV resolution for the 1.332 keV photon peak of ^{60}Co, coupled to a 4 K multichannel analyser. The full-energy peak area under the 443 keV peak for ^{128}I from samples and standard was evaluated and the total iodine concentration measured.

RESULTS AND DISCUSSION

Results obtained for a range of biological reference materials are summarised in Table 2. The figures agree well with literature values. Unfortunately, the levels of iodine in the IAEA milk powder A11 were lower than the detection limits of 0.1 μg g^{-1} obtained for ENAA conditions and the composition of this material. However, the result casts doubt on the validity of the values of 0.7 and 2.6 μg g^{-1} reported in the literature[2] for IAEA A11.

Table 3 summarises the results of measurements obtained for a selection of milk products. Samples of the milk powders used in the earlier round-robin exercise were reexamined by ENAA. Our results supported those laboratories that obtained the lower levels of iodine.

TABLE 2

Concentration of iodine in a range of biological reference materials /μg g^{-1}/

Sample	This work	Literature values
NBS SRMs :		
1566 Oyster tissue	2.5 ± 0.2	2.34[3]
1571 Orchard leaves	0.19 ± 0.07	0.17[3], 0.17[10]
1572 Citrus leaves	1.29 ± 0.05	
1577 Bovine liver	0.21 ± 0.06	0.22[3], 0.18[10]
IAEA SRMs :		
Oyster tissue MA-M-1	7.7 ± 0.4	7.5[3]
Fish homogenate MA-A-2	0.62 ± 0.1	
Blood	23.5 ± 0.7	
Milk powder All	<0.10	0.082[3], 0.05[2], 0.7[2], 2.6[2]

Four samples of market cow's milk were selected randomly for testing and were lyophilised within 24 h of collection. Values for the iodine levels in these samples ranged from 0.058 to 0.12 μg g^{-1}. These compared favourably with the range of iodine concentrations reported for normal cow's milk[11] /0.014-0.270 μg g^{-1}/.

Earlier investigations in this laboratory had involved the measurement of biologically essential trace elements in mother's milk[12]. The current method for iodine determination was used on three of the samples collected for the earlier study. The concentration of iodine for these samples fell within the literature range of 0.04-0.08 μg g^{-1} recorded for human milk[11].

TABLE 3

Concentration of iodine in a range of milk products by epithermal neutron activation analysis

Sample		Iodine, $\mu g\ g^{-1}$
Milk powder	A	0.91 ± 0.05
	B	0.82 ± 0.04
	C	1.11 ± 0.04
	D	1.02 ± 0.05
	E	0.42 ± 0.01
Cow's milk	A	0.057± 0.006
	B	0.12 ± 0.01
	C	0.086± 0.007
	D	0.12 ± 0.01
Human's milk	A	0.075± 0.007
	B	0.080± 0.007
	C	0.038± 0.003

CONCLUSION

The use of ENAA for measuring the concentration of iodine in a range of milk powders, cow's milk and human milk and other biological material has been studied. To ensure no loss of iodine during the activation process, all samples were sealed and irradiated in high purity quartz and subsequently analysed by high resolution γ-spectrometry while encapsulated. The technique was shown to be accurate, precise and rapid for these types of samples.

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