SHORT COMMUNICATION

Determination of Seventeen Elements in Edible Oils and Margarine by Instrumental Neutron Activation Analysis

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Instrumental neutron activation analysis was used to determine the concentration of As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hg, K, Na, Rb, Sb, Sc, Se, Sr and Zn in almond, sunflower, peanut, sesame, linseed, soy, corn and olive oils, as well as in three margarine brands. The concentration of As, Ba, Ce, Cs, Eu, Hg, Rb, Se and Sr were below the system detection limit under the experiment conditions. Chromium was detected only in one of the margarine samples (171 μg/g); Sb only in corn oil (18 ng/g) and Sc only in linseed oil (19 ng/g). Cobalt, Fe, K, Na and Zn were detected in all oil and margarine samples investigated. The concentration ranges for Co, Fe, K, Na and Zn in oils were: 0.016–0.053; 4.45–19.1; 5.93–47.2; 2.44–12.9 and 0.48–1.54 μg/g, respectively. For margarine, the concentration ranges for Co, Fe, K, Na and Zn were 0.09–0.012; 4.53–10.6; 58.3–1140; 13.2–9870 and 0.38–0.47 μg/g, respectively. The elemental contents of the analyzed samples are within the ranges reported in the literature for edible oils and fats.

KEY WORDS: Edible oils and fats, instrumental neutron activation analysis, toxic elements, trace elements.

In the last decade or so, there has been an increasing interest in identifying and measuring various trace elements in foodstuffs due to their effects on the enzymatic and other human biosystems. For edible oils and fats, trace elements may also affect the flavor stability during storage (1). Recently, the concentrations of some toxic and other elements during the refining process (2) and in commercially available products were reported (3–10). The various elements detected in edible oils and fats may originate from the oil itself (*i.e.*, the uptake of these elements from soil, fertilizers or water used for irrigation), from metal pressing and processing equipment or from processing additives and catalysts used to modify the degree of hydrogenation.

Literature data on the concentration of various elements in edible oils and fats are scant, and the elements reported are mostly limited to those measured by atomic absorption (AA) spectroscopy (2–4,7–9). Instrumental neutron activation analysis (INAA) is a powerful analytical method to measure several elements at trace concentration level with the advantages of no added reagents, low detection limits and minimal sample handling and processing. Thus, applying INAA to identify and measure trace elements in edible oils provides data on the concentration (or the upper concentration limit) for elements not reported previously by AA.

In this study, INAA was employed to measure seventeen elements in eight different commercially available oils and three margarines. The elements measured were As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hg, K, Na, Rb, Sb, Sc, Se, Sr and Zn. The analyzed oils were almond, sunflower, peanut, sesame, linseed, soy, corn and olive oils.

EXPERIMENTAL PROCEDURES

Eight different edible oils and three margarine samples were purchased from local supermarkets and used for this study. Approximately 300–500 mg oil or margarine was accurately weighed into a 2/5-dram high-purity polyethylene vial (Olympic Plastics, Los Angeles, CA) and thermally sealed. Each vial was inserted into a 2-dram vial and thermally sealed. An array of certified standard solutions was used. In addition, several multi-elements Standard Reference Materials (SRMs) from the National Institute of Standards and Technology (Gaithersburg, MD) were used. The SRMs were bovine liver (SRM 1577a), citrus leaves (SRM 1572a), oyster tissue (SRM 1566a), rice flour (SRM 1568) and wheat flour (SRM 1567). Good agreement was observed between the certified and the determined values for the elements measured. Blank experiments were carried out to check the purity of the polyethylene container and to make a blank correction if warranted.

Samples, standards and blank vials were irradiated for eight hours in the TRIGA Mark I nuclear reactor on the campus of the University of Texas (Austin, TX). The neutron flux was approximately 2×10^{12} n cm^{-2} s^{-1}. Neutron bombardment of the stable isotopes in the samples results in neutron capture and the production of radioactive isotopes. Such isotopes decay with the emission of gamma radiation. For example, iron in the sample contains ^{58}Fe, which captures a neutron to produce the radioactive nuclide ^{59}Fe. This indicator radionuclide decays by beta emission and releases gamma radiation with characteristic energy of 1099 keV:

$$^{58}Fe + n \xrightarrow{\text{(neutron capture)}} {}^{59}Fe \xrightarrow{\text{(beta decay)}} {}^{59}Co + \gamma \quad [1]$$

stable isotope — neutron — indicator radionuclide — gamma-rays

The indicator nuclides used for measuring the seventeen elements in this study and their nuclear characteristics are given in Table 1.

To minimize the effect of spectral interference and to maximize selectivity, the counting scheme allowed for a decay period of 24–36 h to elapse before the first count was taken for 1 h. Data collected from this counting period were processed to measure As, K and Na. After a second decay period of 4–6 wk, all samples, standards and blanks were counted again for 8 h. Data collected were processed to measure Ba, Ce, Co, Cr, Cs, Eu, Fe, Hg, Rb, Sb, Sc, Se, Sr and Zn.

Emitted gamma rays were counted with a high-purity germanium detector. The detector possessed a relative efficiency of 30%, with full width at half maximum of 1.9 at 1.33 MeV for the ^{60}Co line, and the peak-to-Compton ratio was 54:1. The detector was connected to a 4096 pulse-height multichannel analyzer and set to monitor

TABLE 1

Nuclear Reaction and Experimental Parameters for the Determination of Seventeen Elements in Edible Oils and Margarines

Element	Nuclear reaction and indicator nuclide	Half-life	γ-Ray-monitored (keV)	Irradiation time	Decay time	Counting time
As	^{75}As (n,γ) ^{76}As	26.3 h	559.1	8 h	24-36 h	1 h
Ba	^{130}Ba (n,γ) ^{131}Ba	11.7 d	496.3	8 h	28-35 d	8 h
Ce	^{140}Ce (n,γ) ^{141}Ce	32.50 d	145.4	8 h	28-35 d	8 h
Co	^{59}Co (n,γ) ^{60}Co	5.271 y	1332.5	8 h	28-35 d	8 h
Cr	^{50}Cr (n,γ) ^{51}Cr	27.70 d	320.1	8 h	28-35 d	8 h
Cs	^{133}Cs (n,γ) ^{134}Cs	2.065 y	795.9	8 h	28-35 d	8 h
Eu	^{151}Eu (n,γ) ^{152}Eu	13.48 y	1408.0	8 h	28-35 d	8 h
Fe	^{58}Fe (n,γ) ^{59}Fe	44.516 d	1099.2	8 h	28-35 d	8 h
Hg	^{202}Hg (n,γ) ^{203}Hg	46.61 d	279.2	8 h	24-36 h	8 h
K	^{41}K (n,γ) ^{42}K	12.36 h	1524.6	8 h	28-35 d	1 h
Na	^{23}Na (n,γ) ^{24}Na	14.96 h	1368.8	8 h	24-36 h	1 h
Rb	^{85}Rb (n,γ) ^{86}Rb	18.65 d	1076.7	8 h	28-35 d	8 h
Sb	^{123}Sb (n,γ) ^{124}Sb	60.20 d	1691.0	8 h	28-35 d	8 h
Sc	^{45}Sc (n,γ) ^{46}Sc	83.81 d	889.3	8 h	28-35 d	8 h
Se	^{74}Se (n,γ) ^{75}Se	120.0 d	264.7	8 h	28-35 d	8 h
Sr	^{84}Sr (n,γ) ^{85}Sr	64.84 d	514.0	8 h	28-35 d	8 h
Zn	^{64}Zn (n,γ) ^{65}Zn	243.8 d	1115.5	8 h	28-35 d	8 h

gamma ray energies up to 2048 keV. Data collection and data reduction was performed with a computer-controlled EG&G ORTEC 7010 system (Oak Ridge, TN).

RESULTS AND DISCUSSION

The concentrations of the seventeen elements measured in the eight edible oils investigated are listed in Table 2. The table also lists the concentration of these elements in three different margarine samples. It appears that none of the samples examined contains As, Ba, Ce, Cs, Eu, Hg, Rb, Se or Sr.

The concentration of Co in the samples examined ranges from 9 to 53 ng/g, with linseed oil showing the highest Co concentration. These values are within the range of 0-0.94 μg/g reported for oils and fats (5). No Cr was detected in any of the oil samples examined. Only one margarine brand showed 171 ng Cr/g. This concentration is much less than the 4.9-151 μg/g range reported for oils and fats (5), but higher than the <0.1 μg/g reported for crude oil (2). Iron was detected in all oil and fat samples examined, with concentration ranging from 4.45-19.1 μg/g. This is higher than the 0.61-0.83 μg/g (9), 3.7 μ/g (7) and <3.96 μg/g (6) values reported in Japanese oils and the 1.4 μg/g reported for Egyptian oils (4). However, the measured Cr concentration is much less than that of 49-151 μg/g as reported by Cunningham and Stroube (5).

Sodium was detected in all oil and margarine samples investigated, with concentration ranging from 2.44-12.9 μg/g for oils and 13.2-9870 μg/g for margarine. The

TABLE 2

Concentrations of Seventeen Elements in Eight Edible Oils and Three Margarines

Element	Almond oil	Sunflower oil	Peanut oil	Sesame oil	Linseed oil	Soy oil	Corn oil	Olive oil	Margarine A	Margarine B	Margarine C
As μg/g	<0.10	<0.11	<0.095	<0.087	<0.11	<0.13	<0.10	<0.093	<1	<1	<1
Ba μg/g	<2.6	<1.9	<1.7	<2.3	<2.1	<1.7	<1.4	<1.8	<1.5	<3.7	<3.9
Ce ng/g	<62	<49	<49	<68	<210	<69	<44	<32	<56	<56	<56
Co ng/g	34	41	21	19	53	32	20	16	12	9	9
Cr ng/g	<79	<72	<93	<62	<81	<71	<60	<75	<90	<73	171
Cs ng/g	<10	<8	<6	<12	<9	<7	<6	<6	<5	<6	<5
Eu ng/g	<5	<4	<3	<4	<5	<3	<4	<5	<3	<2	<2
Fe μg/g	9.11	7.23	5.56	5.53	19.1	6.76	6.98	4.45	6.22	4.53	10.6
Hg ng/g	<8	<8	<7	<10	<7	<9	<7	<5	<6	<7	<7
K μg/g	12.6	5.93	8.22	29.6	23.9	16.8	10.8	47.2	58.2	788	1140
Na μg/g	3.22	4.66	2.44	3.08	10.6	5.17	3.07	12.9	13.2	8930	9870
Rb μg/g	<0.31	<0.25	<0.34	<0.22	<0.23	<0.44	<0.22	<0.20	<0.31	<0.57	<0.54
Sb ng/g	<17	<15	26	<17	<19	<12	18	<14	15	<9	<10
Sc ng/g	<0.5	<0.5	<0.4	<0.6	19	<9.5	<0.4	<0.5	<0.5	<0.4	<0.5
Se ng/g	<68	<66	<71	<96	<76	<54	<61	<57	<55	<53	<41
Sr μg/g	<5.4	<1.8	<5.1	<4.9	<4.4	<5.6	<5.0	<4.1	<4.2	<4.0	<3.9
Zn μg/g	1.08	0.79	0.93	1.11	1.54	0.69	0.48	0.61	0.47	0.39	0.38

exceptonally high concentration for margarine brands B and C (8930 and 9870 μg/g, respectively), as compared to brand A (13.2 μg/g), may be attributed to the addition of table salt. Other studies have reported the concentration of sodium in oils as 3.15–4.90 μg/g (9) and in oils and fats as 4090–8220 μg/g (5). The concentration of potassium in vegetable oil was reported to range from 1.00 to 1.80 μg/g (9), and from 84–3220 μg/g for oils and fats (5). The measured concentration range of potassium ranged from 5.93 to 1140 μg/g, with brands B and C margarine showing exceptionally high concentrations of K.

Antimony was detected in peanut and corn oils (26 and 18 ng/g, respectively) and in brand A margarine (15 ng/g). These values are within the limit of <200 ng/g given in another study (5). The same discussion can be applied to Sc, which was detected only in linseed oil at a concentration of 19 ng/g as compared to <400 ng/g reported previously (5). Zinc was present in all oil and margarine samples investigated, with a concentration range from 0.38 to 1.54 μg/g. Crude vegetable oil was reported to contain 0.4 μg/g (2). Other studies reported edible oils to contain 0.11–0.18 μg Zn/g (9), and oils and fats to contain 2.62–4.54 μg/g (5).

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