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DETERMINATION OF TIN, CHROMIUM, CADMIUM AND LEAD IN CANNED FRUITS FROM THE CZECH MARKET

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ABSTRACT
The global production of metal cans is more than 300 billion cans. Benefits of metal packaging consist mainly from the great strenght, excellent barrier properties and good thermal conductivity. The main problem of used metal packaging are the corrosion processes. The corrosion of metal container causes dissolution of tin which is used as a protective layer of the steel shell of the can and other metallic elements used in the manufacture of cans. In this work 31 samples of canned fruit was analysed and the concentration of tin, chromium, cadmium and lead was determined in fruit and in syrup using ICP-OES and ICP-MS techniques. The results showed no difference between the concentration of analysed elements in fruit and in syrup. In none of the analyzed samples the permitted maximum concentration of tin 200 mg.kg^{-1} was exceeded. Maximum concentration of tin was measured in canned grepfruit (59.8 ±1.9 mg.kg^{-1}). The age of cans had no significant effect on the concentration of tin in canned fruit. The concentration of tin in fruit packaged in cans with protective layer of lacquer was significantly lower than the concentration of tin in fruit packaged in cans without protective layer of lacquer. Concentration of chromium, cadmium and lead in the analysed samples was very low at the natural levels of occurrence of these metals in fruit and it was impossible to determine unequivocally that the measured concentrations of these metals in canned fruit originate from the corrosion of can. The corrosion of the tinplate was studied using scanning electron microscopy with an energy dispersive spectrometer. By analyzing the SEM pictures and EDS spectra, critical areas of tin plate corrosion were observed. Based on the measured results it can be concluded that the consumption of fresh canned fruit is not a major problem for the inhabitants of the Czech Republic in terms of intake of potentially hazardous metals.

Keywords: corrosion; can; fruit; tin; spectrometry

INTRODUCTION
Metals are one of the most important packaging materials in food industry besides of plastics and paper. Properties for which the metal packaging's are used are mainly their strength, toughness, ductility and impermeability **(Coles and Kirwan, 2011)**. The most important food packaging in the present time is can make from low carbon mild steel sheet. Practical use of cans as packaging is however limited by corrosion processes **(Mannhaim et al., 1983)**. To avoid corrosion the steel sheet is tinned. The tinplate surface consists of a large area of tin, tiny areas of tin-iron alloy and steel. According to the electrochemical laws, in aerated aqueous environment tin is noble to iron and the anodic corrosion of steel results in iron dissolving which may lead to perforation of the can **(Robertson, 2005)**. In hermetically sealed can the food is deaerated and the headspace oxygen is limited. In anoxic conditions and in the present of citric acid, malic acid, tartaric acid, tannins and flavonoids, tin becomes the anode and protects the steel because of anodic dissolution of the tin **(Robertson, 2005; Che et al., 2012)**. However the protective tin coating prevents damage to the packaging it causes dissolution of tin and other metallic elements such as zinc, chromium, lead and cadmium to the inner contents of cans. The increase of these metals content in canned food poses a hazard of a chemical type. From this reason, in the European Union a limit of 200 mg.kg^{-1} of tin in canned food must be followed by food manufacturers (**Council Directive 1881/2006/EC**, **2006)** amended by **Commission regulation 629/2008/EC, 2008**). Other metals having legislative limits in relation to food are lead and cadmium. The maximum allowable concentration of lead in fruits is 0.1 mg.kg^{-1} and 0.2 mg.kg^{-1} in berries. In fruit juices the maximum level of lead is 0.05 mg.kg^{-1}. The maximum allowable concentration of cadmium in fruits and vegetables is 0.05 mg.kg^{-1} (**Council Directive 1881/2006/EC**, **2006** amended by **Commission regulation 629/2008/EC, 2008**). These limits can be also applied on canned fruit. Concentration of chromium in foodstuff is not covered by the legislation. A parametric value of 50 μg.L^{-1} for total chromium in water intended for human consumption is laid down in **Council Directive 98/83/EC**, **1998**.

The aim of this study was to determine the concentration of tin, chromium, cadmium and lead in canned fruits sold in the Czech Republic and to estimate the potential health risk to residents of the Czech Republic associated with canned fruit consumption. Moreover, we tested two hypothesis. The first hypothesis consisted of fact that the the concentration of tin in canned fruit is affected by the use of a lacquer layer and the second hypothesis consisted of fact that the concentration of tin in canned fruit depends on the time after manufacture.

MATERIAL AND METHODOLOGY

Samples were purchased in local stores in the city of Brno in February 2014 (Table 1). Each canned fruit sample was purchased in 2 pieces.

The amount of 5 g of canned fruit or syrup from the canned fruit was transfered into the 50 mL erlenmayer flask and 10 mL of the mixture of nitric and hydrochloric acid (Analytika Praha, Czech republic, Analpure grade) was added. The sample was heated on the heating plate until its complete decomposition and then transfered to 25 mL volumetric flask and filled up to the mark with an ultrapure water. Both solid and liquid part of the sample was analysed separately. Each sample was analyzed 3 times.

Analysis of tin was performed on an ICP-OES (Ultima 2, Horiba Jobin Yvon, France) equipped with Mainhard type nebuliser and cyclonic spray chamber. The gas flow rate (Ar) was set to 13 $L.min^{-1}$ for cool gas, 0.2 $L.min^{-1}$ for auxilary gas and 0.88 $L.min^{-1}$ for nebuliser gas. The radiofrequency power applied to the load coil was 1300 W. The instrument was calibrated using standard addition calibration methods. For the measuring wavelength 189.930 nm was used. The LOD of method used for the analysis was 0.024 $mg.kg^{-1}$ Sn. Extended uncertainty of measurement at a significance level of 95% with the extension coefficient k = 2 was 11%.

Analysis of chromium, cadmium and lead was carried out with a Thermo X-series quadrupole configuration ICP-MS with hexapole collision cell working on He/H mode (Thermo Fisher Scientific, Waltham, Massachusetts, USA). Instrument was equipped with an autosampler and MicroMist concentric nebulizer connected to Scott-type spray chamber. The gas flow rate (Ar) was set to 14 $L.min^{-1}$ for cool gas, 0.7 $L.min^{-1}$ for auxilary gas and 0.9 $L.min^{-1}$ for nebuliser gas. The flow of collision cell gas was 5 $mL.min^{-1}$. The radiofrequency power applied to the load coil was 1300 W. Data were acquired by Plasma lab software (Thermo Fisher Scientific, USA). An internal standards ^{45}Sc and ^{115}In (Analytika Praha, Czech republic) introduced to the plasma by Internal standard kit (Thermo Fisher Scientific, USA) were used for the drift corrections. Before the measurement on ICP-MS the instrument was optimized in order to increase the sensitivity on ^{56}Fe mass while maintaining oxide ratio for CeO/Ce <0.01. Flow of collision gas and collision cell setting was tuned for achieving <500 cps on mass 80. The standards of Fe and Ce were purchased by Analytika Praha. The LODs of the method used for the analysis were 0.002 $mg.kg^{-1}$ (Cr), 0.004 $mg.kg^{-1}$ (Cd) and 0.0003 $mg.kg^{-1}$ (Pb). Extended uncertainty of measurement at a significance level of 95% with the extension coefficient k = 2 was 6% for all elements.

For the surface analysis of tinplate scanning electron microscope (Zeiss EVO LS10, Germany) with an energy dispersive spectrometer (Oxford Instruments XMAX 80mm, United Kingdom) was used.

Accuracy of the analytical methods used for the analysis was verified using recovery test. The liquid part of canned fruit was spiked by metals of interest and analysed. The recovery reached values from 94 to 102%.

All concentrations were expressed as the average of three independent measurements. The concentrations in $mg.kg^{-1}$ of fresh weight were calculated as $c_m = c_s$. V/m, where c_m is the concentration of element of interest in $mg.kg^{-1}$, c_s is the concentration of element of the interest in the analysed solution ($mg.L^{-1}$), V is the volume of analysed solution (L) and m is the weight of the sample used for the analysis (kg). Obtained data were further analyzed with the XLStat (Addinsoft, USA) and Microsoft Excel software. Testing for significance of mean effects and interactions on all variables was calculated using ANOVA analysis of variance. Statistical significance was set at $p = 0.05$.

RESULTS AND DISCUSSION

Concentration of tin in canned fruit

Daily dietary tin intake of an adult is estimated to be about 4 mg. Canned fruits contributed more than 80% of the dietary intake of tin **(EFSA, 2005)**. Tolerated daily dose of tin is not specified, however increased intake of tin in the diet leads to digestive problems, vomiting, headache, fever and other problems **(Blunden and Wallace, 2003)**. The highest concentration of tin was measured in canned grepfruit (59.8 ±1.9 $mg.kg^{-1}$) while the lowest in strawberry compote: 1.10 ±0.12 $mg.kg^{-1}$ (Table 2). Except of one ananas compote sample and mandarine compote sample there was no statistically significant difference between the concentration of tin in solid and liquid part of the canned fruit samples ($F = 0.015 \leq F_c = 4.013$, data not shown) which is in contrast with results published by **Trafandir et al. (2012)**. Results published by **Mino (2006)** are ambiguous as in some cases significant difference between the concencentration of tin in syrup and in fruit was found, on the other side in some cases there was no significant difference. The concentration of tin in fruit packaged in cans with a protective layer of lacquer was statistically significantly different in comparison to the fruit packaged in tinned cans without protective layer of lacquer ($F = 37.696 > F_c = 4.149$). The average tin concentration in canned fruit was 1.91 $mg.kg^{-1}$ for cans with a protective layer of lacquer in contrast to 24.23 $mg.kg^{-1}$ in cans without a protective layer of lacquer. An average age of cans was 1.9 year. The oldest can was 3.1 year old, the latest one 1.0 year old. The age of can had no effect to the measured tin concentration in caned fruits ($p = 0.1590$). In some cans with older date of production lower concentration of tin in compote was found in comparison with cans of earlier date production. The pH value of syrup ranged between 3.15 and 3.98 and had no effect to the measured tin concentration in caned fruits ($p = 0.4509$). In none of the analyzed samples the maximum allowable concentration of tin 200 $mg.kg^{-1}$ was exceeded, however it must be mentioned that after opening the can the atmosphere in the can changes to aerobic from the anaerobic, which results in rapid dissolution of tin from the surface of the can **(Knápek et al., 2009)**. For this reason, it is not convenient to store the open canned fruit in the original package for an extended period of time and, if necessary, transfer it to plastic or glass container.

The data obtained in this study can be directly compared by data published by **Knápek et al. (2009)** who analysed tin in canned food from Czech market by AAS technique. The highest concentration of tin in canned fruit samples was similary as in the present study found by **Knápek et al. (2009)** in grapefruit compote (44.3 – 311 mg.kg^{-1}). **Knápek et al. (2009)** found also excessive amount of tin (in comparison with the permissible maximum limits) in some samples of peach compote (30.5 – 209 mg.kg^{-1}) and pineapple compote (24.1 – 238 mg.kg^{-1}). The concentration of tin in strawberry or raspberry compote found by **Knápek et al. (2009)** was low (<4 – 6 mg.kg^{-1}), similary as in the present study. Other works that deal with the determination of tin in canned fruit state the tin concentration in the range of 41 – 148 mg.kg^{-1} **(Boutakhrit et al., 2011)** or 25 – 199 mg.kg^{-1} **(Roncevic et al., 2012)**.

The inner surface of the cans was analysed by scanning electron microscope with an energy spectrometer. On the picture 1 a surface of one tinplate without protective layer of lacquer is shown. Blackening of the tinplate and pitting corrosion of the tinplate is visible on different areas of the tinplate. The blackening of tinplate is caused by reaction of iron in tinplate and other fruit constituents like sulfur, phosphorous or oxygen and do not lead to the failure of the container. More serious is the problem of pitting corrosion. The pitting corrosion is visible in area II on Figure 1. The measured spectra from the area II consists from large peaks of carbon and oxygen (Figure 2). These peaks indicate that the analysed tinplate could contain some residue of the food in the hole crated by process of pitting corrosion, even after cleaning of the tinplate. The spectra from the area I in the analysed tinplate contains no large peak of carbon and in contrast to the spectra from area II of analysed tinplate it contains higher intensity tin peaks (Figure 3). This testifies to the fact that the protective layer of lacquer in area II is demaged and dissolution of tin occurs here. For a comparison on the Figure 4 the surface of tinpate protected by yelow lacquer is shown. The measured spectra consists only from the peaks of oxygen and carbon and no tin or significant amount of iron is detected (Figure 5) indicating a perfect protective function of yelow lacquer against corrosion.

Concentration of chromium, cadmium and lead in canned food

At human dietary exposure levels chromium absorption is relatively low and depends on its valence state and ligands. Most of the ingested Cr(VI) is considered to be reduced in the stomach to Cr(III), which is poorly bioavailable and presents low ability to enter cells. In contrast to Cr(III), Cr(VI) is able to cross cellular

Table 1 Overview of analyzed samples and some basic parameters.

Sample	Tin plate protection	Manufacture date	Years after manufacture	pH
peach compote	tin side and bottom lacq. lid and seam	2013/01	1.1	3.86
peach compote	yelow lacquer	2012/10	1.3	3.94
peach compote	yelow lacquer	2012/04	1.5	3.83
peach compote	tin side and bottom lacq. lid and seam	2012/07	1.6	3.75
peach compote	tin side and bottom lacq. lid and seam	2012/08	1.6	3.72
peach compote	tin side and bottom lacq. lid and seam	n.a.	***	3.85
apricot compote	tin side and bottom lacq. lid and seam	2011/04	2.7	3.64
apricot compote	lacquered	2012/09	1.3	3.71
apricot compote	lacquered	2011/07	2.6	3.76
strawberry compote	lacquered	2012/03	1.8	3.52
strawberry compote	lacquered	2011/11	2.3	3.64
strawberry compote	lacquered	2012/03	1.9	3.80
strawberry compote	lacquered	2011/01	3.1	3.51
strawberry compote	lacquered	2012/06	1.7	3.68
strawberry compote	lacquered	2012/04	1.8	3.56
pineapples compote	tin side and bottom lacq. lid and seam	2012/09	1.3	3.96
pineapples compote	tin side and bottom lacq. lid and seam	2012/02	2.0	3.98
pineapples compote	tin side and bottom lacq. lid and seam	2012/10	1.3	3.87
pineapples compote	tin side and bottom lacq. lid and seam	n.a.	***	3.81
mandarin compote	tin side and bottom lacq. lid and seam	2011/09	2.4	3.61
mandarin compote	tin side and bottom lacq. lid and seam	2012/05	1.5	3.51
mandarin compote	tin side and bottom lacq. lid and seam	2012/07	1.6	3.93
mandarin compote	tin side and bottom lacq. lid and seam	2012/03	1.9	3.72
grapefruit compote	tin side and bottom lacq. lid and seam	2011/04	2.8	3.26
grapefruit compote	tin side and bottom lacq. lid and seam	2011/01	3.1	3.15
mango compote	lacquered	2011/09	2.4	3.65
mango compote	lacquered	2012/04	1.5	3.59
pear compote	tin side and bottom lacq. lid and seam	2013/02	1.0	3.52
pear compote	tin side and bottom lacq. lid and seam	2012/10	1.3	3.49
blueberry compote	white lacquer	2012/09	1.3	3.21
blackberry compote	white lacquer	2011/09	2.3	3.52

Table 2 Concentration of tin, chromium, cadmium and lead ($mg.kg^{-1}$) in analysed samples (fruit).

Sample	Sn	Cr	Cd	Pb
peach compote	12.49 ±0.11	0.012 ±0.006	0.009 ±0.003	0.0074 ±0.0012
peach compote	5.88 ±0.07	0.034 ±0.007	0.013 ±0.004	0.0041 ±0.0008
peach compote	1.69 ±0.03	0.025 ±0.005	0.008 ±0.003	0.0032 ±0.0005
peach compote	11.89 ±0.06	0.076 ±0.012	0.033 ±0.008	0.0099 ±0.0015
peach compote	19.33 ±0.09	0.085 ±0.015	0.019 ±0.005	0.0082 ±0.0017
peach compote	22.37 ±0.13	0.055 ±0.003	0.017 ±0.004	0.0054 ±0.0013
apricot compote	50.33 ±0.07	0.021 ±0.005	0.012 ±0.003	0.0076 ±0.0008
apricot compote	1.63 ±0.05	0.015 ±0.004	0.015 ±0.004	0.0027 ±0.0007
apricot compote	1.97 ±0.06	0.019 ±0.007	0.009 ±0.003	0.0015 ±0.0005
strawberry compote	1.62 ±0.04	0.013 ±0.004	0.014 ±0.005	0.0009 ±0.0003
strawberry compote	1.28 ±0.06	0.017 ±0.005	0.009 ±0.003	0.0012 ±0.0004
strawberry compote	1.11 ±0.12	0.018 ±0.004	0.012 ±0.005	0.0008 ±0.0003
strawberry compote	1.43 ±0.07	0.021 ±0.005	0.008 ±0.003	0.0011 ±0.0005
strawberry compote	1.19 ±0.05	0.023 ±0.003	0.013 ±0.004	0.0009 ±0.0005
strawberry compote	1.56 ±0.16	0.017 ±0.004	0.007 ±0.003	0.0013 ±0.0004
pineapples compote	21.58 ±0.14	0.072 ±0.012	0.022 ±0.007	0.0032 ±0.0011
pineapples compote	52.3 ±2.3	0.049 ±0.008	0.035 ±0.009	0.0024 ±0.0009
pineapples compote	19.2 ±0.5	0.028 ±0.005	0.014 ±0.004	0.0019 ±0.0008
pineapples compote	24.6 ±0.9	0.035 ±0.007	0.021 ±0.005	0.0027 ±0.0005
mandarin compote	22.3 ±0.5	0.016 ±0.006	0.029 ±0.007	0.0033 ±0.0007
mandarin compote	13.7 ±0.7	0.023 ±0.004	0.017 ±0.006	0.0027 ±0.0006
mandarin compote	16.5 ±0.3	0.018 ±0.005	0.031 ±0.008	0.0019 ±0.0003
mandarin compote	13.66 ±0.12	0.027 ±0.006	0.023 ±0.004	0.0036 ±0.0013
grapefruit compote	15.9 ±0.4	0.032 ±0.007	0.017 ±0.006	0.0028 ±0.0007
grapefruit compote	59.8 ±1.9	0.021 ±0.005	0.015 ±0.005	0.0019 ±0.0004
mango compote	1.83 ±0.06	0.018 ±0.004	0.008 ±0.004	0.0008 ±0.0005
mango compote	1.61 ±0.03	0.015 ±0.005	0.009 ±0.003	0.0011 ±0.0007
pear compote	23.5 ±0.3	0.028 ±0.003	0.013 ±0.005	0.0037 ±0.0009
pear compote	12.6 ±0.05	0.031 ±0.007	0.016 ±0.004	0.0018 ±0.0006
blueberry compote	2.07 ±0.04	0.012 ±0.004	0.007 ±0.003	0.0007 ±0.0003
blackberry compote	1.88 ±0.06	0.015 ±0.003	0.011 ±0.005	0.0009 ±0.0004

membranes. The acute toxicity of chromium(VI) is due to its strong oxidative properties. After it reaches the bloodstream, it damages blood cells by oxidation reactions. Some tinplates used for manufacturing cans may contain the thin chromium oxide film to prevent corrosion of the can. Maximum concentration of chromium in analysed samples was 0.085 ±0.015 $mg.kg^{-1}$. The average concentration was 0.029 $mgkg^{-1}$. The measured data are in accordance with the results published by **Jorhem and Slorlach (1987)** who determined the average concentration of chromium in fruit and vegetables packaged in lacquered welded tinplate cans to be 0.018 $mgkg^{-1}$ and in un-lacquered welded tinplate cans to be 0.091 $mg.kg^{-1}$.

The main toxic effect of cadmium is its toxicity to the kidney, although it has also been associated with lung damage and skeletal changes in occupationally exposed populations. The main source of cadmium in food is atmospheric deposition into the soil and crops or application of municipal sewage sluge to agricultural soil. Cadmium may be also present in the can as the impurity of materials used for making cans. Maximum concentration of cadmium in analysed samples was

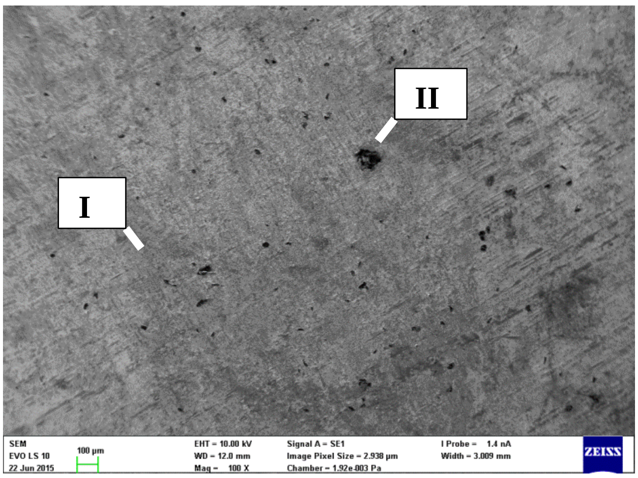

Figure 1 Picture of tinplate without protective lacquere taken by scanning electron microscope. Magnification = 100x.

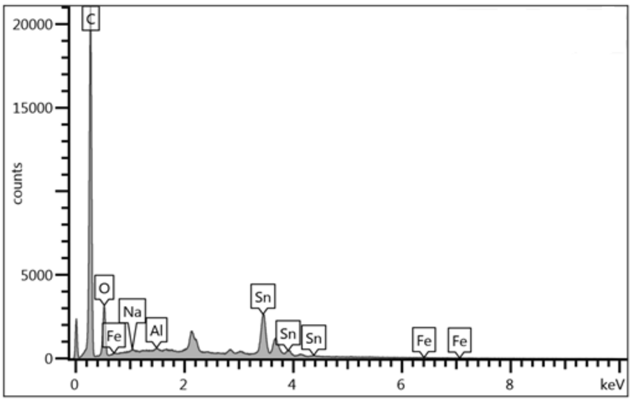

Figure 2 The EDS spectra of area II shown in Figure 1.

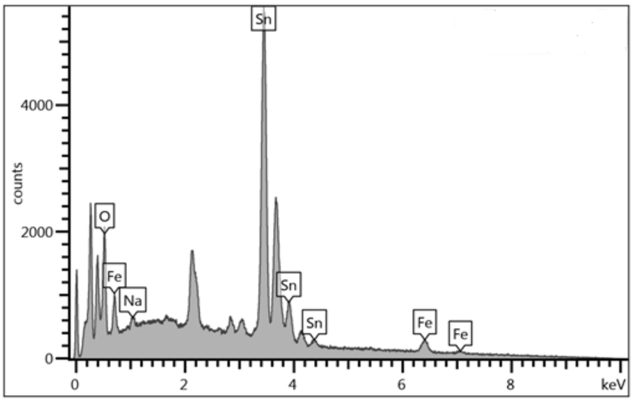

Figure 3 The EDS spectra of area I shown in Figure 1.

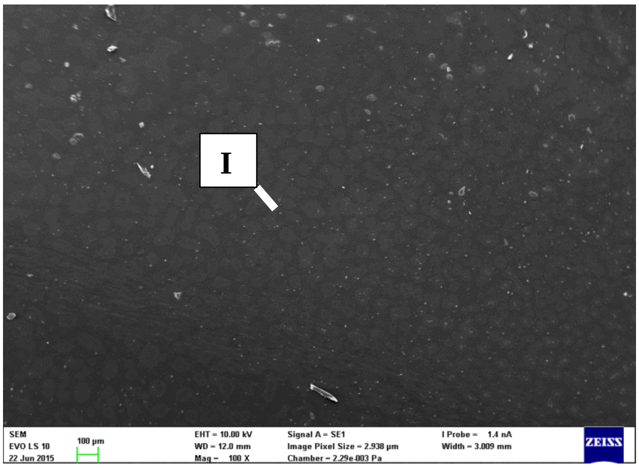

Figure 4 Picture of tinplate protected by yelow lacquere taken by scanning electron microscope. Magnification = 100x.

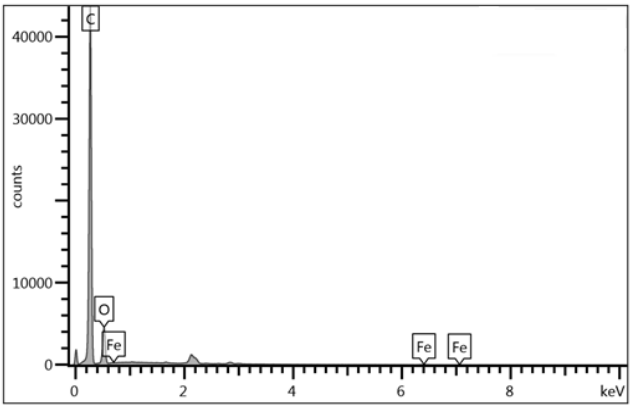

Figure 5 The EDS spectra of area I shown in Figure 4.

0.033 ±0.008 mg.kg^{-1}. The average concentration was 0.017 mg.kg^{-1} which is under the maximum limit of 0.05 mg.kg^{-1} set by EU. **Rafique at al. (2009)** analysed canned strawberry and pineaple from local markets of Pakistan. The concentration of cadmium in canned strawberry was 0.014 mg.kg^{-1} and in canned pineapple 0.017 mg.kg^{-1}. **Jorhem and Slorlach (1987)** studied the concentration of cadmium in fruit and vegetables packaged in welded tinplate cans and they found the mean concentration of cadmium to be 0.004 mg.kg^{-1} for foods in lacquered cans and 0.006 mg.kg^{-1} for foods in un-lacquered cans.

Short-term exposure to high levels of lead can cause brain damage, paralysis, anaemia and gastrointestinal symptoms. Longer-term exposure can cause damage to the kidneys, reproductive and immune systems in addition to effects on the nervous system. The most critical effect of low-level lead exposure is on intellectual development in young children. The main source of lead in food is soil from which the lead may be taken up into plants or lead particles in air which can be deposited on the surface of leaves, stems and fruits. Important source of lead contamination is soldering in the canning process. The average concentration of lead in canned fruits was 0.003 mg.kg^{-1} which is significantly lower concentration in comparison with results published by **Rafique et al. (2009)** or by **Jorhem and Slorlach (1987)** who determined the lead concentration in canned fruit in the range of 0.011 to 0.222 mg.kg^{-1}.

CONCLUSION

In none of the analyzed samples the maximum allowable concentration of tin 200 mg.kg^{-1} was exceeded. The maximum measured concentration of tin was detected in grepfruit sample (59.8 ±1.9 mg.kg^{-1}). Concentration of chromium, cadmium and lead in the analysed samples was very low at the natural levels of occurrence of these metals in fruit and it was impossible to determine unequivocally that the measured concentrations of these metals in canned fruit originate from the corrosion of can. The measured results from ICP-OES and ICP-MS together with an analysis of SEM pictures and EDS spectra proved the perfect protective properties of lacquers used in tinplate cans against corrosion. No significant relationship was found between the age of the can and the tin concentration in the canned fruit (the age of the samples was 1 to 3 years). Based on the measured results it can be concluded that the consumption of canned fruit is not a major problem for the inhabitants of the Czech Republic in terms of intake of potentially hazardous metals.

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