

## Radioactive cesium concentrations in purchased tea leaves and the other plant materials for brewing tea

購入した茶葉とその他飲茶用植物材料における放射性セシウム濃度

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(received on 1, September,2016) / (accepted on 10, January,2017)

### Summary

Frequently drinking tea is an ordinary custom for a large number of Japanese people. Since the radioactive cesium (Cs) in tealeaves may harm human health, the concentration should be examined as precisely as possible. In order to determine  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations, a method to isolate Cs from soil, dead leaves and leaf mold was applied for Cs isolations from green tea leaves and the other kinds of plant materials for brewing tea. Sample was carbonized in a covered stainless steel beaker (300 cm<sup>3</sup>) heated for 3 hours in an electric furnace at 450 °C, and the Cs atoms extracted by nitric acid were collected into 1 g of ammonium phosphomolybdate trihydrate. Gamma rays from Cs sample were measured by a pure Ge detector with volume of 160 cm<sup>3</sup> shielded by lead blocks. Results in a supplemental experiment suggested that mass reduction rate by carbonization scarcely depended on the amount of dried sample in the beaker. The Japanese products used in this study were manufactured by companies located in or western from Ibaraki prefecture. The products were obtained on or after 4 May 2013. Among 21 tea leaves products obtained for this preliminary study, the highest concentration of  $^{137}\text{Cs}$  was  $17.8 \pm 0.8$  Bq/kg in a product purchased in Okayama Prefecture on 28 September 2013. (The concentrations and activity ratios shown in this summary are those decay-corrected to the end of March 2011.) In this product, the  $^{134}\text{Cs}/^{137}\text{Cs}$  activity ratio was 1 within uncertainty. The  $^{137}\text{Cs}$  concentration was as low as 0.1-0.2 Bq/kg in two Sen-tea leaves products. One was manufactured in Hyogo Prefecture and the other was manufactured in Yamaguchi Prefecture. The  $^{137}\text{Cs}$  concentrations in the other tea leaves products were higher than or equal to 0.3 Bq/kg. In four tea leaves products of which raw material tea leaves were produced in Shizuoka, Yamaguchi and Kagoshima Prefectures, and in two oolong tea products, the  $^{137}\text{Cs}$  concentrations were higher than 0.5 Bq/kg and the differences from each  $^{134}\text{Cs}$  concentration were 0.5 – 1.3 Bq/kg. The differences were probably caused by the  $^{137}\text{Cs}$  atoms emitted by the Chernobyl disaster or atmospheric nuclear weapon tests. Except tealeaves, four plant materials for brewing tea were examined in this study. The products of dried mugwort leaves for brewing tea manufactured in Oita prefecture was obtained on 26 August 2013. Both  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations in the mugwort leaves were approximately 0.4 Bq/kg. In the other three plant materials for brewing tea, both  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations were less than 0.3 Bq/kg.

Keywords: Fukushima nuclear disaster/ radioactive cesium/ tealeaves product/ chemical isolation

## 1. Introduction

Shobara City in the Bihoku area of the Chugoku region in western Japan is approximately 770 km distant from the Fukushima Daiichi Nuclear Power Plant (FDNPP). Kato and Ishihara (2012)<sup>1)</sup> observed the radioactive cesium (Cs) from the FDNPP in both of the rainwater and air dusts samples at a location in Shobara City in April of 2011. In San Francisco Bay that is nearly 7700 km distant from the FDNPP, the radioactive Cs concentration in rain reached a maximum level on 24 March 2011<sup>2)</sup>. The concentration was nearly twice the maximum level found in the rain of Shobara City on 19 April 2011<sup>1, 2)</sup>. This suggested that the Pacific Ocean was contaminated with vast amounts of radioactive Cs from the FDNPP. However, the radioactive Cs concentration in the Pacific Ocean will not increase because of the effects of currents of seawater<sup>3)</sup>. On the other hand, the pollutions in foods and beverages on the land of Japan have been influenced by various factors, such as weather effects and human activities. Therefore, precise and continuous measurements of radioactive Cs concentrations are necessary in order to estimate how foods and beverages have been contaminated with radioactive Cs. Tea is one of the favorable beverages in Japan, and so the radioactive Cs concentrations in tea leaves products should be determined as precisely as possible. Chemical isolation of Cs from the tea leaves may allow us to reduce the uncertainty of measured concentration. A method to isolate Cs ions was previously applied for determining the radioactive Cs concentrations in soils, leaf molds and dead leaves<sup>4, 5)</sup>. In this preliminary study, measurements of radioactive Cs concentrations in tea leaves products were performed using the Cs isolation method. The measurements were also performed for the Cs samples isolated from the other plant materials for brewing tea.

## 2. Materials and Methods

Figure 1 shows the prefectures where the companies supplying the tea leaves products used in this study are located. Some tea leaves products were labelled to show the tea leaf gardens for the raw tea leaves included in the products. Some of the other products were made from the raw tea leaves from some gardens inside a prefecture. The labels on the other tea leaf products show that the raw tea leaves were grown somewhere in Japan. There are numerous tea leaves production areas in Japan. The Shizuoka Prefecture is famous for producing Japanese green tea. One of the samples was made by a company in Ibaraki prefecture using the raw materials grown in Shizuoka Prefecture. More measurements were tried for two oolong tea products of which raw tealeaves were grown in Fujian province of the People's Republic of China. Fujian province lies far from and to the west of the FDNPP. The radioactive Cs concentrations were also determined in four dried plant products for making tea. Three were manufactured in Oita Prefecture and one was manufactured in Hyogo Prefecture.

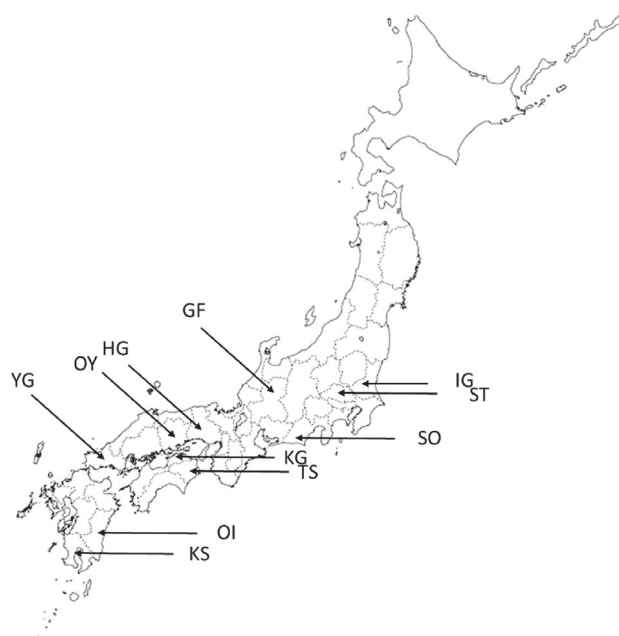


Fig.1 Locations of companies that manufactured tea leaves products used in this study. The arrows indicate the prefectures where the companies are located. Names of prefectures are abbreviated by two characters, such as IG for Ibaraki.

Every sample was heated in a covered stainless steel beaker using a 450 °C electric furnace for 3 hours. After turning off the switch of the furnace, the beaker was left to stand overnight in the electric furnace for cooling the carbonized sample. The Cs atoms were extracted by nitric acid, and were collected into 1 g of ammonium phosphomolybdate trihydrate (APM), (Nacalai Tesque, Extra Pure Reagent), as with the study by A. Tsumura, et al <sup>6)</sup>. The Cs isolation method from the carbonized sample was almost same as that in the previous study<sup>5)</sup>. The gamma rays from the samples were measured by a pure coaxial-type Ge detector (Oxford, CPVDS 30-0190, 160 cm<sup>3</sup>), which was shielded by lead blocks of 15 cm in thickness.

The stainless steel beaker containing samples was heated in an electric oven. If air outside of the beaker can freely enter into the inside, a considerable amount of leaves can be incinerated. In that case, the amount of incinerated leaves may have related to the volume of free space in the beaker. Since the beaker was covered, tea leaves in the beaker were scarcely incinerated. In order to examine how the mass of sample in the beaker relates to the mass reduction rate, that is, the mass ratio of carbonized tealeaves to dried tea leaves before carbonization, an supplemental experiment (1<sup>st</sup> supplemental experiment) was performed using two stainless beakers and homogenized dried green tea leaves in products manufactured by a company in Ibaraki Prefecture. The raw material tealeaves for the products were produced in Shizuoka Prefecture. The 400g of manufactured tealeaves purchased on 2 March 2015 were homogenized, and 123.1g and 61.6g of the dried tealeaves (IG11 and IG12) were put into covered stainless steel beaker, respectively. There was few free space in the 300 mL (313 mL of practical inner volume) stainless steel

beaker containing 123.1g of tealeaves. The beakers were heated at 450 °C for 3 hours and were kept in the electric oven over night.

Another supplemental experiment (2<sup>nd</sup> supplemental experiment) was performed in order to examine the extraction rate of  $^{137}\text{Cs}$  ions from the solution by using 1 g of APM. In the first place, the Cs ions in the sample solution were extracted with 1 g of APM, and the Cs sample with APM was prepared by a filtration. Again one gram of APM was added to the filtrate, and the remaining Cs ions were collected. The gamma-ray measurements were performed for these two Cs samples. The  $^{137}\text{Cs}$  661.7 keV gamma-ray full energy peaks in the two spectra were analyzed, and the count rates were compared with each other.

### 3. Results and Discussion

Figure 2 shows the gamma-ray spectrum for Cs sample isolated from a tea leaves (Green tea) manufactured at a location in Saitama Prefecture obtained on 16 August 2014. As shown in Fig. 2, the tealeaves included not only  $^{137}\text{Cs}$  but also  $^{134}\text{Cs}$ . The sample was named ST1(Green,UNK). In this study, the sample name includes two characters indicating the prefecture where the company manufacturing

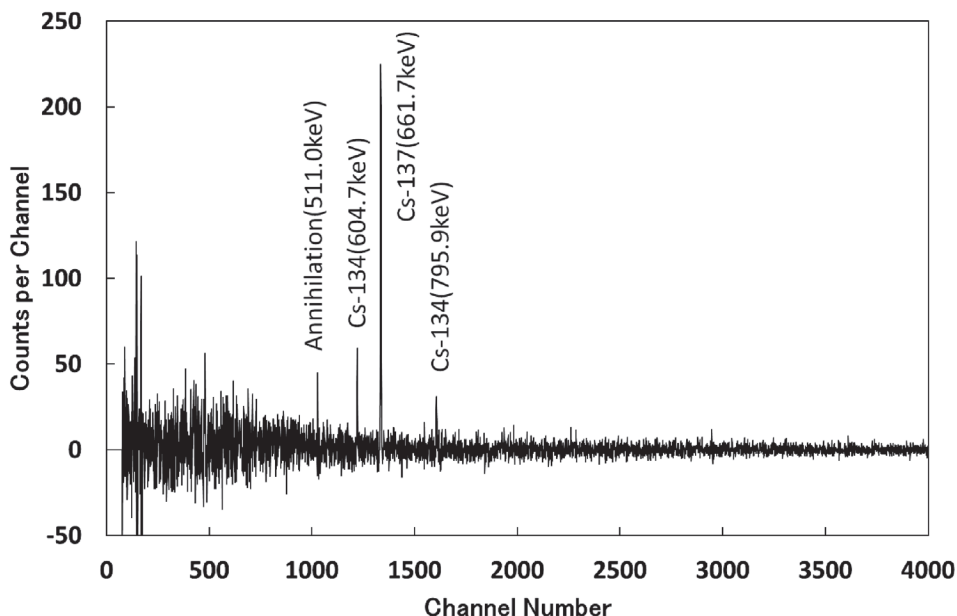


Fig.2 Gamma-ray spectrum for Cs sample made from tealeaves product.

The sample name was ST1(Green, UNK), and the measured  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations were shown in Table 1. The method for naming samples is explained in the footnote of Table 1 and in the text. The tea leaves product produced in Saitama Prefecture was obtained on 7 August 2015, and the gamma rays from the Cs sample were measured for 20.5 hours on 6 to 7 January 2016. The background gamma rays were also measured, and were subtracted after adjustment of difference in measurement time.

the sample is located. In parenthesis of the name, the first word indicates the kind of tealeaves shown in the label on the envelope of product, and a set of two characters indicates the prefecture where the raw material tea leaves were produced. It should be noted that the production places were estimated based on label information, except those for ST11(Green, UNK) and HG11(Hoji, HG). The labels on the products of ST11(Green, UNK) and HG11(Hoji, HG) did not show the places where the raw material tea leaves were produced. The information on the raw material tea leaves for HG11(Hoji,HG) was obtained by an interview with the saleswomen at the direct sales store in front of the factory. “UNK” in ST11(Green, UNK) means that the production place for the raw material tea leaves was unknown. “Green tea” is a general term of unfermented tealeaves including “Sen tea” and “Hoji tea”.

In the 1<sup>st</sup> supplemental experiment for green tealeaves product manufactured by a company in Ibaraki, the masses of the carbonized samples in filled and half-filled beakers decreased to be 36.5% and 40.7% of each original mass before carbonization, respectively. Those mass reduction rates were similar to those in the dead leaves of which radioactive Cs concentrations were determined in the previous study<sup>5)</sup>. This result suggested that the amount of tea leaves in a stainless steel beaker scarcely influenced the mass reduction rate. The mean mass reduction rate in all samples examined in this study was 0.38 (SD: 0.03). The samples IG11 and IG12 were taken out from 400g of tea leaves purchased on 2 March 2015. The other tea leaves

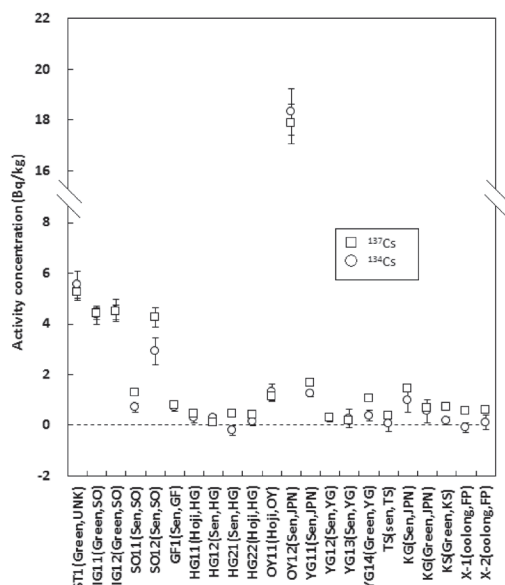


Fig.3 Radioactive cesium concentrations in tea leaves products.

The marks indicate the concentrations decay-corrected to the end of March 2011. The <sup>137</sup>Cs and <sup>134</sup>Cs concentrations were shown by open squares and by open circles, respectively. The naming method for the products was explained in the footnote of Table 1 and in the text. Note that the vertical axis has a break.

products in Fig. 3 and Table 1 described below were different from each other concerning brand name or date purchased.

The 2nd supplemental experiment was performed using the sample of IG12(Green, SO). The results showed that the count rate for the Cs sample from the filtrate was  $0.8 \pm 2.3$  % of that for the first Cs sample. In this study, this percentage and the uncertainty were used for compensating the data of  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations for Cs ions missed during the Cs extraction with 1 g of APM. As known well, the APM is useful for collecting Cs ions in solution <sup>6)</sup>. The result of the 2<sup>nd</sup> supplemental experiment suggested that almost all Cs ions could be extracted with 1 g of APM in the case of the present study.

Table 1 shows the  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations in the dried tealeaves on the date when the product was obtained. Table 1 and Fig. 3 show the  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations, decay-corrected to the end of March 2011. As shown in Table 1 and Fig. 3, several Japanese tea leaves products clearly included the  $^{134}\text{Cs}$  atoms from the FDNPP. The locations of tea leaves plantations in Shizuoka Prefecture are close to or in Shizuoka City where the monthly fallouts in March and April 2011 brought a large amount of radioactive Cs atoms ( $550 \text{ MBq/km}^2$  of  $^{134}\text{Cs}$  and  $540 \text{ MBq/km}^2$  of  $^{137}\text{Cs}$  in March, and  $87 \text{ MBq/km}^2$  of  $^{134}\text{Cs}$  and  $86 \text{ MBq/km}^2$  of  $^{137}\text{Cs}$  in April) <sup>7)</sup>. In March of 2011, radioactive Cs was not detected in monthly fallout in Kakamigahara City of Gifu Prefecture. In April 2011, the amount of radioactive Cs in monthly fallout on Kakamigahara City was only 11 % of that in Shizuoka City <sup>8)</sup>. The samples of SO11(Sen, SO) and GF1(Sen, GF) were purchased in 2013. The  $^{137}\text{Cs}$  concentrations in these samples were similar to each other, as shown in Table 1. However, the  $^{137}\text{Cs}$  concentration in SO12(Sen, SO) purchased in 2014 was clearly higher than those in GF1(Sen, GF) and SO12(Sen, SO) in 2013.

As shown in Table 1, the activity ratios of  $^{134}\text{Cs}$  to  $^{137}\text{Cs}$  in the samples SO11(Sen, SO) and SO12(Sen, SO) from Shizuoka Prefecture seemed to have been less than one, decay-corrected to the end of March, 2011. The differences ( $0.6 \text{ Bq/kg}$  and  $1.3 \text{ Bq/kg}$ ) were probably due to the Chernobyl disaster or the atmospheric nuclear weapon tests. In a Sen tea product manufactured in Hyogo Prefecture (HG12(Sen, HG)), the  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations were as low as  $0.09 \pm 0.04$  and  $0.10 \pm 0.04 \text{ Bq/kg}$ , respectively. This low uncertainties were due to the long gamma-ray measurement (81.6 hours). The samples of oolong tealeaves used in this study were made of raw materials grown in Fujian Province of the People's Republic of China. The  $^{134}\text{Cs}$  concentrations in the oolong tea leaves were zero within the uncertainties in October of 2013. This suggests that almost all of the  $^{137}\text{Cs}$  atoms in the oolong tea leaves ( $0.5 \text{ Bq/kg}$ ) were released either from the Chernobyl disaster on 26 April 1986 or from the atmospheric nuclear weapon tests on or before 16 October 1980. The tea trees for tealeaves are usually younger than 30 years old <sup>9)</sup>. This suggests that the  $^{137}\text{Cs}$  atoms in the raw tealeaves for these oolong tea leaves products may have come from the soils in and around the tea plantations.

Table 1 Radioactive Cs concentrations in tea leaves products.

No.	Sample name*	Sampling date	$^{137}\text{Cs}$ (Bq/kg) <sup>§</sup>	$^{134}\text{Cs}$ (Bq/kg) <sup>§</sup>	$^{137}\text{Cs}$ (Bq/kg) <sup>&amp;</sup>	$^{134}\text{Cs}$ (Bq/kg) <sup>&amp;</sup>
	Tea leaves					
1	ST1(Green,UNK)	2015/8/7	4.77 ± 0.31	1.30 ± 0.13	5.24 ± 0.32	5.55 ± 0.53
2A	IG11(Green,SO)	2015/3/2	4.02 ± 0.21	1.17 ± 0.10	4.40 ± 0.23	4.35 ± 0.36
2B	IG12(Green,SO)	2015/3/2	4.07 ± 0.25	1.21 ± 0.12	4.46 ± 0.27	4.53 ± 0.43
3	SO11(Sen,SO)	2013/12/16	1.11 ± 0.10	0.22 ± 0.06	1.28 ± 0.12	0.68 ± 0.18
4	SO12(Sen,SO)	2014/8/16	3.99 ± 0.35	1.17 ± 0.22	4.25 ± 0.38	2.91 ± 0.55
5	GF1(Sen,GF)	2013/7/22	0.75 ± 0.09	0.32 ± 0.07	0.79 ± 0.10	0.69 ± 0.15
6	HG11(Hoji,HG)	2014/4/26	0.42 ± 0.09	0.11 ± 0.07	0.45 ± 0.10	0.30 ± 0.19
7	HG12(Sen,HG) <sup>‡</sup>	2014/4/26	0.09 ± 0.04	0.10 ± 0.04	0.10 ± 0.05	0.27 ± 0.11
8	HG21(Sen,HG)	2014/3/1	0.41 ± 0.08	-0.08 ± 0.07	0.44 ± 0.09	-0.21 ± 0.18
9	HG22(Hoji,HG)	2014/3/1	0.36 ± 0.09	0.05 ± 0.06	0.38 ± 0.09	0.14 ± 0.17
10	OY11(Hoji,OY)	2013/9/28	1.04 ± 0.17	0.56 ± 0.13	1.10 ± 0.18	1.29 ± 0.31
11	OY12(Sen,JPN)	2013/9/28	16.84 ± 0.73	7.92 ± 0.40	17.84 ± 0.78	18.32 ± 0.92
12	YG11(Sen,JPN)	2013/5/4	1.59 ± 0.13	0.61 ± 0.10	1.67 ± 0.14	1.22 ± 0.10
13	YG12(Sen,YG)	2013/5/4	0.29 ± 0.07	0.13 ± 0.07	0.30 ± 0.08	0.27 ± 0.13
14	YG13(Sen,YG)	2013/12/7	0.14 ± 0.09	0.07 ± 0.11	0.16 ± 0.10	0.26 ± 0.37
15	YG14(Green,YG)	2015/4/25	0.96 ± 0.10	0.09 ± 0.05	1.05 ± 0.11	0.36 ± 0.21
16	TS(sen,TS)	2014/12/4	0.34 ± 0.11	0.02 ± 0.09	0.37 ± 0.13	0.07 ± 0.30
17	KG(Sen,JPN)	2016/8/24	1.27 ± 0.12	0.16 ± 0.07	1.42 ± 0.13	0.95 ± 0.44
18	KG(Green,JPN)	2016/9/13	0.60 ± 0.10	0.09 ± 0.07	0.68 ± 0.12	0.54 ± 0.45
19	KS(Green,KS)	2015/5/23	0.63 ± 0.08	0.04 ± 0.04	0.68 ± 0.08	0.17 ± 0.14
20	X1(oolong,FP)	2013/10/19	0.52 ± 0.10	-0.05 ± 0.08	0.55 ± 0.11	-0.11 ± 0.19
21	X2(oolong,FP)	2014/4/11	0.54 ± 0.10	0.04 ± 0.11	0.58 ± 0.11	0.10 ± 0.29
	Other tea					
T1	HG23(BSB,HG)	2014/3/1	0.12 ± 0.08	0.01 ± 0.06	0.13 ± 0.08	0.02 ± 0.17
T2	OI(HC,OI)	2013/8/26	0.12 ± 0.12	-0.30 ± 0.10	0.13 ± 0.13	-0.68 ± 0.22
T3	OI(Mugwort,UNK)	2013/8/26	0.34 ± 0.09	0.20 ± 0.09	0.36 ± 0.09	0.44 ± 0.19
T4	OI(Kaki,OI)	2013/8/26	0.06 ± 0.07	0.06 ± 0.08	0.07 ± 0.07	0.13 ± 0.18

\* Sample name is composed of abbreviation of prefecture where the company supplying the product exists. Two oolong tea products used in this study were named X1(oolong,FP) and X2(oolong,FP), respectively.

The descriptions of content and prefecture where raw tealeaves were produced are shown in parentheses. "JPN" and "FP" are abbreviations for Japan and for Fujian Province of the People's Republic of China, respectively. "UNK" is abbreviation for "unknown".

Abbreviations used for expressing description of samples are as follows:

"Sen" and "Hoji" mean Sen tea and Hoji tea, respectively.

"Kaki" : Dried leaves of Japanese persimmon (kaki) tree for brewing tea.

"BSB" : Black soybean for brewing tea.

"HC" : Dried *houttuynia cordata* for brewing tea.

"Mugwort" : Dried mugwort (*artemisia princeps*) for brewing tea.

§ Concentration in dried sample on the sampling date.

& Concentration in dried sample decay corrected to 31 March 2011.

‡ Measurement lasted for 81.6 hours.

The present Cs isolation method was also applied for measuring the radioactive Cs concentrations in four kinds of products for brewing tea except tealeaves. The  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  activities in a purchased dried mugwort leaves were approximately 0.4 Bq/kg decay corrected to the end of March in 2011. Although the product was manufactured in Oita Prefecture, the production place for the raw mugwort leaves was unknown. The  $^{137}\text{Cs}$  and  $^{134}\text{Cs}$  concentrations in the other three samples in western Japan were lower than 0.3 Bq/kg, as shown in Table 1.

Relatively large amounts of nitric acid, sodium hydroxide and sodium carbonate are required for Cs isolation from 25 g of carbonized plant sample. Procedure to prepare each Cs sample requires long time. These facts have suppressed the number of samples of which radioactive Cs concentrations could be determined. A more conventional method to isolate Cs atoms with less reagents will be developed in near future.

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## 要 旨

日本には頻繁にお茶を飲む人が多い。お茶の葉に含まれる健康を害する可能性がある放射性セシウム(Cs)の濃度は可能な限り正確に測る必要がある。茶葉とその他の飲茶用植物の製品を入手し<sup>137</sup>Csと<sup>134</sup>Csの濃度を測定するために、土壌、枯葉そして腐葉土からCsを抽出する方法を適用してCsを抽出した。乾燥試料は蓋をした300 cm<sup>3</sup>ステンレススチールビーカー内に入れて電気炉で3時間450℃に加熱して炭化し、硝酸で抽出したCsは1gのリンモリブデン酸アンモニウム3水和物で回収した。回収したCs試料からのγ線とバックグラウンドγ線は鉛遮蔽した160 cm<sup>3</sup>高純度Ge半導体検出器で測定した。補足的な実験を行い、炭化物の収率(炭化後と炭化前の質量比)はビーカー中の乾燥試料の量にほとんど依存しないことが分かった。日本茶製品は茨城県以西の会社で製造され2013年5月4日以降に購入されたものを用いた。本予備的研究で調べた21の茶葉製品の中で最も高かった<sup>137</sup>Cs濃度は2013年9月28日に岡山県内で購入した茶葉製品中の17.8 ± 0.8 Bq/kgであった。(本要旨中に示す放射能濃度と<sup>134</sup>Cs/<sup>137</sup>Cs放射能比は2011年3月末まで崩壊補正した値である。)この茶葉製品中の<sup>134</sup>Cs/<sup>137</sup>Cs放射能比は誤差の範囲で1であった。兵庫県と山口県で生産された2つの煎茶中の<sup>137</sup>Cs濃度は0.1-0.2 Bq/kgであった。これ以外の茶葉製品中の<sup>137</sup>Cs濃度は0.3 Bq/kg以上であった。茶葉の原産地が静岡県、山口県そして鹿児島県であった4試料と2つのウーロン茶で<sup>137</sup>Cs濃度は0.5Bq/kg以上であり<sup>134</sup>Csとの濃度差が0.5 ~ 1.3 Bq/kgであった。<sup>134</sup>Csとの差はチェルノブイリ原発事故または大気圏核実験で放出された<sup>137</sup>Csによって生じたと考えられる。本研究では茶葉以外の4つの飲茶用植物製品についても調べた。2013年8月26日に購入した大分県で製造された飲茶用蓬生葉では<sup>137</sup>Csと<sup>134</sup>Csの濃度がいずれも約0.4 Bq/kgであった。それ以外の3試料では、<sup>137</sup>Csと<sup>134</sup>Csの濃度がいずれも0.3 Bq/kg未満であった。

