γ-ray spectrometry results versus sample preparation methods

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Abstract According to recommended conditions two bio-samples, tea leave and flour, are prepared with different methods: grounding into powder and reducing to ash, then they were analyzed by γ ray spectrometry. Remarkable difference was shown between the measured values of tea samples prepared with these different methods. One of the reasons may be that the method of reducing to ash makes some nuclides lost. Compared with the "non-destructive" method of grounding into powder, the method of reducing to ash can be much more sensible to the loss of some nuclides. The probable reasons are discussed for the varied influences of different preparation methods of tea leave and flour samples.

Keywords γ ray spectrometry, Tea, Reducing to ash, Grounding into powder
CLC numbers R144, O652, O657.35

1 Introduction

As is well known, many methods of preparing sample, including ashing, compressing and evaporating etc., are frequently adopted by analysts to improve sensitivity or cut down limitation. No matter which method is adopted, one of the most important premises is that the loss of the matter which is concerned in the measurement ought to be negligible, or else quantitative corrections can be done within error permitted. In the inter-comparison on low level environmental sample with γ ray spectrometry[1], two classical methods of preparing sample were adopted accidentally. However, we obtained two results with a big difference. This makes us pay attention to the the suitability of some classical methods of sample preparing for special kinds of samples and nuclides.

2 Analysis results of tea leave with different methods of preparing sample

As a part of technical exchange between the Laboratory of Industrial Hygiene (LIH) and the Japan Chemical Analysis Center (JCAC), an inter-comparison on radioactivity analysis of environmental samples (tea leave) was done in 1996. The results obtained with different methods is introduced here.

Manuscript received date: 1999-08-16

In order to test the sensitivity of our low background HPGe γ spectrometer with anti-coincidence shield, and to accomplish the inter-comparison on time also, we decided to adopt two classical methods to prepare samples. One method is that the samples are grounded into powder and then dried at about 105°C, which is called as a "quasi-nondestructive technique for preparing samples". The other is that the sample is ashed at 450°C[2~4], which is called as "ashing method for preparing samples".

The tea leave used was bought from a tea shop in Beijing, and it was produced in Fujian province of China. The total weight including stems was 2.5 kg. Both LIH and JCAC obtained a half of the samples, respectively. At LIH, a part of the samples was grounded into powder and dried at about 105°C, and then the powder sample of 157.9 g was filled in a box of ϕ 75×50 to be determined. About 8.6% of the tea leave mass was lost during the preparation.

At the same time, another part of the sample, of which the weight was 782.5 g, was reduced to ash at 450°C[4] and total ash weight of 38.5g was obtained, and then filled into a box of ϕ75×25 to be determined too. Both samples were sealed for more than a month to achieve equilibrium between ^{226}Ra and ^{222}Rn.

All samples were determined with the low background HPGe γ spectrometer with anti-coincidence shield[5]. Each kind of samples was determined 4 or 5 times. Collection time of each spectrum was 85000~86400 s. The spectrum of background was collected in 72 h. The analysis results of the powder are shown in Table 1, and those of the ash are shown in Table 2.

From Ref.[7], the statistical test of coherence of average values between powder and ash was carried out. For example, the final average result of ^{226}Ra in the powder is 2.47±0.46, and that in the ash is 1.53±0.12. Obviously, 2.47-1.53=0.94>0.46+0.12=0.58, then we can draw our conclusion that the averages of them have remarkable difference. Because no radionuclide would be lost in the powder sample and some radionuclides might be lost in the ash sample, we favor the results in Table 1 even more.

Table 1 Analysis results of the powder of dried tea leave

Radio nuclides	Initial average results /Bq·kg^{-1} (1)	Density correction factors[6]	Final average results /Bq·kg^{-1} (1)
^{226}Ra	2.41±0.45	1.027	2.47±0.46
^{137}Cs	0.737±0.089	1.038	0.765±0.092
^{228}Ra	4.34±0.76	1.021	4.43±0.78
^{40}K	595±13	1.011	602±13

(1)Reported are the total errors at the confidence level of 95%

Table 2 *Analysis results of the ash of dried tea leave*

Radio nuclides results /Bq·kg^{-1} [1]	Initial average factors[6]	Density correction /Bq·kg^{-1} [1]	Final average results
^{226}Ra	1.61±0.13	0.9515	1.53±0.12
^{137}Cs	0.562±0.063	0.9851	0.554±0.062
^{228}Ra	3.29±0.32	0.9794	3.23±0.31
^{40}K	485±11	0.9863	478±11

[1]Reported are the total errors at the confidence level of 95%

And we found that different measuring condition, say, geometry, did not influence measured results of the tea leave. The results of the dried tea leave in a polyethylene box of $\phi 75\times 25$ are listed in Table 3. It shows that the results agree with the results by using polyethylene box of $\phi 75\times 50$ very well.

Table 3 Analysis results of the powder of dried tea leave within box* of $\phi 75\times 25$

Radio nuclides	Initial average results /Bq·kg^{-1} [1]	Density correction factors[6]	Final average results /Bq·kg^{-1} [1]
^{226}Ra	1.84±0.76	1.027	1.89±0.78
^{137}Cs	0.689±0.288	1.038	0.715±0.299
^{228}Ra	4.13±1.88	1.021	4.22±1.92
^{40}K	609±51	1.011	615±52

[1]Reported are the total errors at the 95% confidence level

*The sample had not achieved equilibrium between ^{226}Ra and ^{222}Rn

3 Analysis results of flour with different methods of preparing sample

As known from the analysis results of γ spectrometry of tea leave with two different methods of preparing sample, sample prepared with ashing method might lose some matter to a non-negligible degree for tea leave, even if no non-negligible error exists in all other procedures. The fact makes us sprout a thought to detect the suitability of classical methods of sample preparing for some other kinds of samples and nuclides.

A kind of high quality flour, called as SIGNE FUXING, was first used as the sample to be measured. The difference from the measuring of tea leave is that both dried flour and its ash were sealed in sample boxes of the same size $\phi 75\times 50$ in order to avoid more indefinite factors influencing analysis results.

The analysis results of dried flour are listed in Table 4, and those of the ash are listed in Table 5. The data following sign "<" are LLD of the system with this method. Tables 4 and 5 show obviously that their results for ^{40}K are consistent between dried flour and its ash, and the sensitivity of the method of "quasi-nondestructive technique in preparing samples" is comparable to that of "ashing method in preparing samples" in

respect to the detection of ^{40}K. Because no definite data of ^{226}Ra, ^{137}Cs, and ^{228}Ra from the method of "quasi- nondestructive technique in preparing samples" could be obtained, the influences might be brought on with both preparation methods were not decided yet.

Table 4 Analysis results of dried flour

Radio nuclides	Initial average results /Bq·kg^{-1} (1)	Density correction factors[6]	Final average results /Bq·kg^{-1} (1)
^{226}Ra	< 0.72	-	
^{137}Cs	< 0.18	-	-
^{228}Ra	< 1.4	-	-
^{40}K	43.8±3.9	1.0092	44.2±3.9

(1)Reported are the total errors at the confidence level of 95%

Table 5 Analysis results of the ash of dried flour

Radio nuclides	Initial average results /Bq·kg^{-1} (1)	Density correction factors[6]	Final average results /Bq·kg^{-1} (1)
^{226}Ra	0.0575±0.0042	-	
^{137}Cs	0.0150±0.0014	-	-
^{228}Ra	0.0694±0.0080	-	
^{40}K	44.2±1.2	0.9768	43.2±1.2

(1)Reported are the total errors at the confidence level of 95%

4 Discussion and conclusion

If we only concern with ^{40}K in flour, the two preparation methods do not bring on any remarkable difference in the results. It seems that ^{40}K in flour is absorbed only from soil by the plant so that there is not so much volatile matter. This may be a possible reason that leads to the similar results when different methods were used. It seems that tea leave absorbs the radionuclides not only from soil, but also from the atmosphere around leaves. Because there isn't cleaning procedure during the production of tea leave and there is much volatile matter, the remarkably different results come into being. In short, the different chemical states of the nuclides in tea leave are possible reasons that cause notable different results when using the different preparation methods.

According to the principle of "quasi-nondestructive technique for preparing samples", if the sensitivity of a system can be fulfilled, then the results should be fully reliable, just like the detection of flour. Whereas, it is shown that sensitvity of the system has restricted the application range of that method, only in this case, "ashing method in preparing samples" can be a choice.

In our preliminary study of the influences on analysis results using both classical sample preparation methods (for tea leave and flour samples), it is discovered that some

limitations exist in applying the method of reducing to ash on environmental samples. We should be very careful with the specific limits to the samples and nuclides concerned though the ashing method is used by many analysts for its notable high sensitivity. Furthermore, the method had been previously applied to many works as recommended in references, but the limitations of the method had not been pointed out or discovered in those works. For example, radioactivities of samples are the most basic data in estimation of internal or external dose, radiation protection and environment monitoring etc. So it's possible that the ashing method had been applied to the samples and nuclides which are unsuitable, then incorrect results were obtained. When the incorrect results are applied or cited elsewhere, the new non-negligible discrepancy would be spread.

To sum up, in order to gain a clear idea of stuitability of "ashing method for preparing samples", or to obtain corresponding correction factors, it's quite significant to study the influences on analysis results by using different classical sample preparation methods.

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