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Preparation of bovine liver candidate reference material*

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Abstract—The bovine liver candidate reference material specially for micro-analytical techniques was prepared. The preparation process including material collection, dried, pulverize, sieve, homogenization and preliminary test was described in detail. The more effective grinding methods were established to achieve the median particle size of 22μm.
Keywords: standard reference material; bovine liver; quality assurance.

1 Introduction

Standard reference materials (SRM) are an indispensable element for quality assurance. They play a key role in demonstration of accuracy of analytical work. Up to now, the reference materials are mostly satisfactory for various analytical techniques, but there is an increasing demand for micro quantitative information of nuclear analysis techniques. Most RMs are certified for minimum sample sizes larger than 100 mg in their certificates by producers. A minimum sample size, which is compatible with the respective analytical technique, however, is one of the most important requirements (Watjen, 1990; 1993) for a suitable RM. Therefore RMs with such large sample size are useless for methods such as XRF, NAA, PIXE and other accelerator-based methods, which commonly use and analyse samples in the mg mass range or even smaller samples. So much work still remains to be done to improve measurements capability for the determination of trace components present at the mg/kg to μg/kg levels in environmental and biological samples. Some specific natural matrix reference materials containing very low levels of trace elements and having high degree of homogeneity are developed for many micro-analytical procedures (Valkovic, 1992; Zeisler, 1995).

The Co-ordinated Research Programme (CRP) organized by IAEA specifically addresses the question of quality control materials for micro-analytical nuclear techniques. As a task we make efforts to prepare a new candidate reference material-bovine liver to meet this purpose.

2 Process of preparation

2.1 Material collection

Fresh bovine livers were collected from normal male calves (just born in a week) in Second Animal Farm, Shanghai suburb, in 1996. The average wet weight of each fresh bovine liver was about 800 g. Approximately 83 kg of sample including in total 104 bovine liver individuals was kept in cleaning polythene bags and stored in low temperature refrigerator (bellow -40℃) until preparation.

To keep mineral contamination and the loss of mineral elements from the samples to a

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minimum, instruments used for the collection of specimens and their handing were acid cleaned and washed with high purity water. All operations were performed in a special cleaning-room to avoid any contamination of the material with metals.

2.2 Cleaning and dried

First we took out the sample from refrigerator to thaw them at normal temperature (10—20℃) for 24 hours. After removal of storage bags and visible contaminants, all liver samples were rinsed by high purity water to remove blood or surface fluid drainage to obtain the wet weight. Then we cut the liver tissues into small pieces with titanium knife and removed the blood tube at the same time. Later we beat these liver tissues using food pulverizer with titanium spinning knife. About 63 kg homogeneous mixture of liver tissue was obtained and kept in plastic drum.

The sample dry was performed by freeze-drying machine in Shanghai Biological Production Institute. After being unfreezed, the liver mixture was even placed on the some aluminum trays, which were put into the machine and dried at −40℃ for 35 hours. Finally the liver mixture reached constant weight. The ratio R = 0.25 of dry to wet weight was determined, and about 16 kg of dried liver was obtained.

2.3 Pulverize and sieve

The grinding of the dried liver tissue into powder was accomplished by using a agate ball mill pulverizer (QM-1SP. produced by Instrument Factory of Nanjing University, China). This mill machine is designed by planet principle as Fig. 1. Forty agate balls of 20 mm and 10 mm diameters were put inside of each agate pot. Four agate pots in all were mounted on the machine plate and revolved round the main axis on their own axis in the opposite direction. The rotational speed is adjustable from 50—300 r/min.

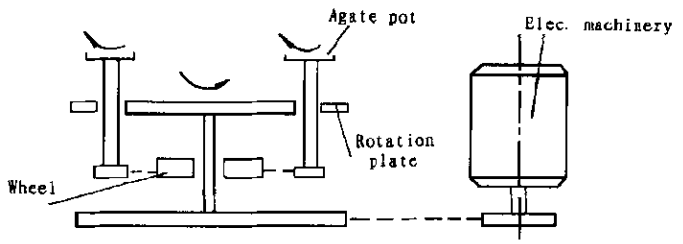

Fig. 1 A working principle of the ball mill pulverizer

The bovine liver powder was sieved through 80 μm plastic nylon sieve. The fraction retained on the sieve was returned to agate pots for further grinding again. The ground and sieved material about 12 kg was collected in a plastic drum for further treatment.

2.4 Homogeneity and particle size determination

The homogeneity which is one of the basic requirements for a candidate reference material can be achieved by a thorough mixing of the powdered material. The bovine liver powder was blended in two ways to achieve the fine powdered material. First, the fractions were put into a polyethylene rotation drum which was placed in a specially constructed homogenizer. It is able to rotate in two directions thus assuring good mixing of the material. Then the mixing material was transferred to a *Y* type homogenizer with teflon lined (produced by Japan), and blended automatically for more than 3 hours to achieve a high degree of homogeneity.

Mastersizer *X* (Malvern Instruments Ltd.) laser light scattering instrument in connection

with a dry powder sampling unit was used as the determination of particle size distribution in powder liver (Fajgelj, 1994). The mass of the sample analyzed was approximately 40 mg in each case. Four samples in all were performed to determine particle size distribution, and average of the median particle's diameter for four samples is about 22μm and the size corresponding to the largest peak in the distribution is about 35μm.

3 Conclusion

Bovine liver powder has been prepared as a part of CRP reference materials for microanalytical nuclear techniques. The results show that its particle size distribution was improved significantly than these of other RMs. For reasons of quality control and better assessment of resulting data, its certification campaigns on world-wide will be organized by IAEA.

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