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Present status and future trends in biological and environmental reference materials in China

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Summary. The present status of biological and environmental reference materials (BERMs) is summarized, including the institutions involved in the preparation, certification and approval of BERMs, types, analysis, homogeneity, stability and role in analytical practice. The future perspectives on the BERMs in China are also briefly discussed.

The history of the BERMs in China is relatively short in comparison with the developed countries, but the development of preparation, certification and use of the BERMs has been extremely rapid, since the first introductory paper on reference materials was published in 1979 [1]. Up to now, 36 of the first class and 21 of the second class of BERMs have been produced by a number of Chinese institutions and approved by the State Bureau of Technical Supervision (SBTS) [2], which is responsible for organization, supervision and appraisal of the reference materials.

Institutions

Over 100 institutions throughout China are involved in preparation, analysis and certification of reference materials. Table 1 lists the main ones and their activities. China has set up a unified collaboration network, but no institute specifically dedicated to the preparation and certification of the BERMs for trace element analysis, like NIST in the USA.

Table 1. Main institutions for preparation, certification and appraisal of BERMs in China

Name	Main activities
State Bureau of Technical Supervision (SBTS)	Organization, supervision and appraisal of reference materials
National Research Center for Certified Reference Materials (NRCCRM)	Preparation, certification and distribution of RMs, especially gas and water RMs
Chinese Academy of Sciences (CAS) and its subordinate institutes	Preparation, analysis and certification of various types of RMs, e.g. human hair, Tibet soil, river sediment, coal ash, peach leaves, tea, mussel, *Codonopsis pilosuna*
State Bureau of Environmental Protection (SBEP) and its subordinate institutes	Environmental RMs, e.g. polluted farmland soil
Ministry of Hygiene (MOH) and its subordinate institutes	Biological and clinical RMs, e.g. maize, freeze-dried urine, bovine serum, whole blood
Ministry of Geology Minerals (MGM) and its subordinate institutes	Geological RMs and others, e.g. river sediment, soil, shrub leaves, poplar leaves, tea
Ministry of Commerce (MOC) and its subordinate institutes	Food RMs, e.g. cabbage, wheat flour, pork liver

Types of biological and environmental reference materials in China

Tables 2 and 3 list the 19 main environmental and biological reference materials available in China up to 1991, respectively, with the certified multielement contents or reference values in them. In addition, there are many BERMs with only one or a few certified elements, e.g. F with 114 ± 14 μg/g in coal ash (GBW08402) and 1.91 ± 0.18 and 33.7 ± 2.2 μg/g in corn RMs (GBW08506 and GBW08507), Hg with 0.036 ± 0.003 μg/g in rice (GBW08508), Pb with 112 ± 15 μg/L and Cd with 1.05 ± 0.17 μg/L in whole blood (GBW09132), etc.

Preparation and analysis of BERMs

The preparation of BERMs is a tedious and time-consuming task. As an example, the preparation process of the *Codonopsis pilosuna* is briefly introduced below. It is a Chinese herbal medicine with good curative effect, which may be related to the contents of trace elements and its growing environment. After collection, the herb sample was dried in the sun without washing; the fibrous roots were removed.

The dust on its surface was removed with a clean brush. The sample was placed on strictly cleaned steamer trays in an aluminium boiler with deionized water. The water was evaporated by heating to 50–60 °C. Once every 15 min, bidist. water was sprayed onto the herb. Afterwards, the expanded herb sample was brushed and washed again twice in deionized water.

Table 2. The main environmental reference materials in China (up to 1991) (unit: μg/g)

Element	River sediment GBW08301	Tibet soil GBW08302	Polluted soil GBW08303	Coal ash GBW08401
Al	--	71100 ± 600	68600 ± 1700	--
As	56 ± 5	3.8 ± 0.4	10.6 ± 0.6	11.4 ± 0.3
Ba	375 ± 11	(509)	(724)	(1450)
Be	(3.5)	2.96 ± 0.04	(2.5)	10.7 ± 0.5
Br	--	(1.3)	--	--
Ca	--	25900 ± 200	47900 ± 1700	--
Cd	2.45 ± 0.15	0.081 ± 0.007	1.20 ± 0.07	0.16 ± 0.02
Ce	--	83.6 ± 1.7	--	--
Co	16.5 ± 0.7	13.1 ± 0.6	13.0 ± 0.6	33.2 ± 1.4
Cr	90 ± 4	60.8 ± 1.8	112 ± 6	60 ± 4
Cs	--	(7.3)	--	--
Cu	53 ± 3	24.6 ± 1.4	120 ± 6	53 ± 2
Dy	--	(5)	--	--
Eu	--	1.4 ± 0.1	--	--
Fe	39400 ± 600	33400 ± 500	29700 ± 1000	76500 ± 700
Hf	--	(7.3)	--	--
Hg	0.22 ± 0.02	(0.018)	2.15 ± 0.06	(0.039)
K	--	21200 ± 900	15700 ± 800	--
La	--	41.9 ± 2.0	(40)	--
Lu	--	(0.48)	--	--
Mg	--	15300 ± 200	13000 ± 800	--
Mn	975 ± 17	677 ± 12	519 ± 18	1178 ± 20
Mo	--	--	(3.3)	--
N	--	1280 ± 30	--	--
Na	--	15200 ± 500	11000 ± 600	--
Nd	--	42.3 ± 2.4	--	--
Ni	(32)	31.1 ± 0.8	40 ± 2	--
P	--	8600 ± 400	1600 ± 80	--
Pb	79 ± 6	14.2 ± 0.3	73 ± 2	33.8 ± 2.2
Rb	--	135 ± 7	(68)	--
Sb	--	(0.4)	--	--
Sc	--	10.8 ± 0.8	(10)	--
Se	0.39 ± 0.05	0.16 ± 0.02	(1.0)	1.13 ± 0.08
Si	--	305700 ± 500	(259000)	--
Sm	--	7.1 ± 0.3	--	--
Sr	--	163 ± 15	405 ± 16	--
Ta	--	(1.1)	--	--
Tb	--	(0.9)	--	--
Th	--	17.6 ± 0.4	11.6 ± 0.5	--
Ti	--	4000 ± 200	3600 ± 100	--
U	--	3.84 ± 0.20	(3.2)	--
V	(96)	77.5 ± 4.0	--	95 ± 5
Yb	--	3.1 ± 0.3	--	--
Zn	(251)	58.0 ± 3.3	260 ± 11	61 ± 4

Data in parentheses are the reference values; the uncertainties are ± 2 standard deviation; no data exist for elements not mentioned

Table 3. The main biological reference materials in China (up to 1991) (unit : μg/g)

Element	Human hair GBW07601	Human hair GBW09101	Shrub leaves GBW07602	Poplar leaves GBW07604	Tea GBW07605	Peach leaves GBW08501	Rice flour GBW08502
Ag	0.029 ± 0.008	(0.35)	0.027 ± 0.006	(0.013)	(0.018)	--	--
Al	--	13.3 ± 2.3	2140 ± 220	1040 ± 60	(3000)	--	--
As	0.28 ± 0.05	0.59 ± 0.07	0.95 ± 0.12	0.37 ± 0.09	0.28 ± 0.04	0.34 ± 0.03	0.051 ± 0.003
Au	(0.0025)	--	--	--	--	--	--
B	(1.3)	--	34 ± 7	53 ± 5	15 ± 4	(45.8)	--
Ba	17 ± 2	(5.41)	19 ± 3	26 ± 4	58 ± 6	18.4 ± 0.9	--
Be	0.063 ± 0.020	--	0.056 ± 0.014	0.021 ± 0.005	0.034 ± 0.006	--	--
Bi	0.34 ± 0.02	--	(0.022)	0.027 ± 0.002	0.063 ± 0.008	--	--
Br	(0.36)	(0.602)	2.4 ± 0.4	7.2 ± 1.4	3.4 ± 0.5	--	--
Ca	2900 ± 300	1090 ± 72	22200 ± 1300	18100 ± 1300	4300 ± 400	--	55 ± 2
Cd	0.11 ± 0.03	0.095 ± 0.012	0.14 ± 0.06	0.32 ± 0.07	0.057 ± 0.010	0.018 ± 0.004	0.020 ± 0.002
Ce	0.12 ± 0.03	--	2.4 ± 0.3	0.49 ± 0.07	1.0 ± 0.2	--	--
Cl	--	(152)	(11300)	(2300)	--	--	--
Co	0.071 ± 0.012	0.135 ± 0.008	0.39 ± 0.05	0.42 ± 0.03	0.18 ± 0.02	(0.25)	--
Cr	0.37 ± 0.06	4.77 ± 0.38	2.3 ± 0.3	0.55 ± 0.07	0.80 ± 0.03	0.94 ± 0.07	--
Cs	--	--	0.27 ± 0.03	0.053 ± 0.003	0.29 ± 0.02	--	--
Cu	10.6 ± 1.2	23.0 ± 1.4	5.2 ± 0.5	9.3 ± 1.0	17.3 ± 0.8	10.4 ± 0.8	2.6 ± 0.2
Dy	(0.017)	--	--	(0.036)	(0.074)	--	--
Eu	(0.006)	--	0.037 ± 0.002	0.009 ± 0.003	0.018 ± 0.002	--	--
F	--	--	24 ± 3	22 ± 4	320 ± 31	--	--
Fe	54 ± 10	71.2 ± 6.6	1020 ± 67	274 ± 17	264 ± 15	431 ± 15	5.1 ± 0.2
Gd	--	--	--	(0.043)	(0.093)	--	--
Hf	--	--	0.14 ± 0.02	(0.026)	(0.033)	--	--
Hg	0.36 ± 0.08	2.16 ± 0.21	--	0.026 ± 0.003	(0.013)	0.046 ± 0.006	--
Ho	--	--	--	--	(0.019)	--	--
I	--	(0.875)	--	--	--	--	--
K	(20)	(11.8)	8500 ± 500	13800 ± 700	16600 ± 1200	21700 ± 800	656 ± 15
La	0.049 ± 0.011	(0.014)	1.23 ± 0.10	0.26 ± 0.02	0.60 ± 0.04	--	--
Li	2.0 ± 0.1	--	2.4 ± 0.4	0.84 ± 0.15	(0.36)	--	--
Lu	--	--	--	--	(0.007)	--	--
Mg	360 ± 40	105 ± 6	2870 ± 180	6500 ± 500	1700 ± 200	4700 ± 200	120 ± 5
Mn	6.3 ± 0.8	2.94 ± 0.20	58 ± 6	45 ± 4	1240 ± 70	75.4 ± 2.7	9.8 ± 0.2
Mo	0.073 ± 0.014	(0.58)	0.26 ± 0.04	0.18 ± 0.01	0.038 ± 0.007	--	--
N	149000 ± 1000	--	12000 ± 200	25600 ± 600	33200 ± 900	--	--
Na	152 ± 17	266 ± 12	11000 ± 1000	200 ± 13	44 ± 6	--	8.4 ± 0.6
Nd	--	--	(1.1)	(0.22)	(0.44)	--	--
Ni	0.83 ± 0.19	3.47 ± 0.40	1.7 ± 0.4	1.9 ± 0.3	4.6 ± 0.5	--	--
P	170 ± 10	(184)	830 ± 40	1680 ± 60	2840 ± 90	--	--
Pb	8.8 ± 1.1	7.2 ± 0.7	7.1 ± 1.1	1.5 ± 0.3	4.4 ± 0.3	0.99 ± 0.04	0.75 ± 0.05
Pr	--	--	--	--	(0.12)	--	--
Rb	--	--	4.2 ± 0.2	7.6 ± 0.8	74 ± 5	--	--
S	43000 ± 3000	(46900)	3200 ± 300	3500 ± 400	2450 ± 220	--	--
Sb	0.095 ± 0.016	(0.21)	0.078 ± 0.020	0.045 ± 0.006	0.056 ± 0.006	--	--
Sc	0.008 ± 0.001	(0.00287)	0.31 ± 0.03	0.069 ± 0.007	0.085 ± 0.013	--	--
Se	0.60 ± 0.04	0.58 ± 0.05	0.184 ± 0.013	0.14 ± 0.02	(0.072)	(0.04)	0.045 ± 0.008
Si	870 ± 80	--	5800 ± 400	7100 ± 800	(2100)	--	--
Sm	(0.012)	--	0.19 ± 0.01	0.038 ± 0.006	0.085 ± 0.023	--	--
Sn	--	--	--	--	--	--	--
Sr	24 ± 1	4.19 ± 0.14	345 ± 11	154 ± 9	15.2 ± 0.7	61.6 ± 3.9	--
Tb	--	--	(0.026)	--	(0.011)	--	--
Th	--	--	0.37 ± 0.02	0.07 ± 0.01	0.061 ± 0.009	--	--
Ti	2.7 ± 0.6	--	95 ± 18	20.4 ± 2.2	24 ± 4	--	--
U	--	--	(0.11)	(0.028)	--	--	--
V	--	(0.069)	2.4 ± 0.3	(0.64)	(0.86)	--	--
W	--	--	(0.06)	--	--	--	--
Y	0.084 ± 0.016	--	(0.63)	0.145 ± 0.015	0.36 ± 0.04	--	--
Yb	--	--	0.063 ± 0.014	0.018 ± 0.004	0.044 ± 0.005	--	--
Zn	190 ± 9	189 ± 8	20.6 ± 2.2	37 ± 3	26.3 ± 2.0	22.8 ± 1.3	14.1 ± 0.5

Notes: Data in parentheses are the reference values; the uncertainties are ± standard deviation.

Table 3 (cont.)

Wheat flour GBW08503	Cabbage GBW08504	Tea GBW08505	Pork liver GBW08551	Mussel GBW08571	Freeze-dried urine GBW09103	Bovine serum GBW09131	*Codonopsis pilosuna* GBW09501
--	--	--	--	--	--	--	--
--	--	--	--	(231)	--	--	--
0.22 ± 0.02	0.056 ± 0.006	0.191 ± 0.027	0.044 ± 0.004	6.1 ± 0.6	0.36 ± 0.04	--	--
--	--	--	--	(0.1)	--	--	--
--	--	--	--	--	--	--	--
--	--	15.7 ± 1.9	--	--	--	--	109 ± 13
--	--	--	--	--	0.031 ± 0.002	--	--
--	--	--	--	--	--	--	--
--	--	(2)	--	--	--	--	--
441 ± 22	7920 ± 180	2840 ± 210	197 ± 7	1110 ± 20	--	94 ± 16	1780 ± 90
0.031 ± 0.002	0.029 ± 0.003	0.032 ± 0.005	0.067 ± 0.002	4.5 ± 0.3	0.053 ± 0.003	--	--
--	--	0.686 ± 0.092	--	--	--	--	--
--	--	--	--	--	--	--	--
--	--	(0.2)	(0.100)	0.94 ± 0.03	--	--	(0.14)
--	--	(0.8)	(0.20)	0.57 ± 0.04	0.091 ± 0.006	--	--
--	--	(0.13)	--	--	--	--	--
4.40 ± 0.31	3.00 ± 0.10	16.2 ± 1.9	17.2 ± 0.5	7.7 ± 0.5	0.45 ± 0.04	0.66 ± 0.08	6.80 ± 0.62
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
39.8 ± 2.6	52.0 ± 1.6	373 ± 23	1050 ± 40	221 ± 7	--	1.57 ± 0.22	69.5 ± 8.6
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
--	--	(0.004)	--	0.067 ± 0.004	--	--	--
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
1980 ± 140	14500 ± 400	19700 ± 1300	11500 ± 200	4240 ± 100	--	219 ± 25	--
--	--	0.458 ± 0.020	--	--	--	--	--
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
551 ± 21	1840 ± 20	2240 ± 190	747 ± 26	1970 ± 100	--	20.6 ± 1.2	--
19.6 ± 1.0	22.0 ± 0.5	766 ± 28	8.32 ± 0.19	10.2 ± 0.9	0.29 ± 0.03	--	18.8 ± 0.7
--	--	--	3.8 ± 0.4	(0.6)	--	--	--
(23900)	28000 ± 1000	48800 ± 1400	108600 ± 500	5820 ± 70	--	--	--
(10.0)	7570 ± 80	142 ± 13	2330 ± 40	--	--	3260 ± 130	--
--	--	--	--	--	--	--	--
--	--	--	7.61 ± 0.48	--	0.31 ± 0.03	--	--
(1500)	3400 ± 100	4260 ± 230	(13000)	(13500)	--	--	--
0.35 ± 0.08	0.28 ± 0.04	1.06 ± 0.10	0.54 ± 0.02	1.96 ± 0.05	0.112 ± 0.009	--	(0.50)
--	--	--	--	--	--	--	--
--	(31.7)	36.9 ± 1.3	--	--	--	--	--
--	--	3150 ± 80	--	--	--	--	--
--	--	0.037 ± 0.003	--	--	--	--	--
--	--	(0.1)	--	--	--	--	--
(0.10)	0.083 ± 0.004	0.041 ± 0.010	0.94 ± 0.03	3.65 ± 0.09	0.44 ± 0.06	0.0389 ± 0.0023	(0.37)
--	--	--	--	--	--	--	--
--	--	(0.06)	--	--	--	--	--
--	--	--	--	--	--	--	--
--	45.2 ± 1.3	10.8 ± 1.8	--	12.8 ± 0.6	--	--	18.0 ± 0.8
--	--	--	--	--	--	--	--
--	--	0.105 ± 0.012	--	--	--	--	--
--	--	(36)	--	--	--	--	--
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	--
22.7 ± 2.0	26.7 ± 0.8	38.7 ± 3.9	172 ± 4	138 ± 5	2.22 ± 0.10	0.71 ± 0.10	15.2 ± 1.0

The cleaned sample was put on an ultraclean working desk and blown dry for about 1 h. The herb sample was quickly frozen for 2 h in a refrigerator, then dried at 50–60 °C in an infrared oven, and finally in a vacuum desiccator. The dried *Codonopsis pilosuna* samples were ground, sieved with a Nylon sieve (40 mesh), mixed, bottled, irradiated with ^{60}Co at 10^6 rads for disinfecting, tested for uniformity and preserved dry at low temperatures.

The major analytical techniques used in determination of trace elements in BERMs in China are as follows: NAA instrumental and radiochemical, PIXE, AAS, ICP, IDMS (isotopic dilution mass spectroscopy), XRF, AFS, POL (polarography), and COL (colorimetry).

Due to its unique features and popularity in China, NAA, instrumental and radiochemical, has played a significant role as well in examination of the homogeneity as in the analysis of the chemical composition of Chinese BERMs [3–5]. Most elements shown in Tables 2 and 3 (up to 45 or more) were determined by NAA with good accuracy. For example, 42 elements were reported in an IAEA intercomparison study on Lake Sediment SL-3 by a Chinese laboratory with no outlier for the 26 IAEA recommended elements [6].

As a result, NAA has served as a reference method in interlaboratory comparisons in China and provided 30 to 50% data of all certified elements in Chinese BERMs.

BERMs homogeneity and stability

Homogeneity is one of the most important properties of reference materials. Ingamells and Switzer [7] proposed a sampling constant K_s for a well-mixed material, defined as the minimum subsample amount needed to hold the relative sampling uncertainty to 1% at the 68% confidence level in a single determination. It is, however, only valid for a well-mixed sample. Thus, Visman [8] developed a general theory of sampling that takes into account the effects of inhomogeneity to suit both well-mixed and segregated materials. According to this theory, the sampling variance S_s^2 could be related to the individual increment size W and the number of increments n by the following equation

$$S_s^2\ (\%) = \frac{a}{nW} + \frac{b}{n}$$

where a and b are the homogeneity constant and the segregation constant, respectively. Using the above model, we studied the homogeneity of trace elements in some environmental reference materials, e.g. river sediment, soil, etc. [9]. From our results we draw the following conclusions:

1. The homogeneity of the elemental distribution in a well-mixed reference material relates to its own chemical behaviour (species). For example, Hf, Cr, Th and REE often exhibit large variance, while Sc has only small variance. This is due to the lack of an independent mineral of Sc. On the contrary, Hf, Cr, Th and REE exist in zircon, chromite, monazite and other accessory minerals.

2. The relative sampling variances of almost all elements increase with the decrease of sampling amount, which confirms the prediction of Ingamells and Switzer [7].

3. The sampling constants (a and b, or K_s) are meaningful only when the sample variance is discernible in the overall variance.

Like the homogeneity, the long-term stability of trace element contents is another important parameter of BERMs. Thus, the examination of the change of the contents of trace elements during storage constitutes a necessary step in study and use of the BERMs in China. For example, the results given by NAA and AAS indicate that the contents of the certified elements (Ba, Ca, Co, Cu, Fe, Mn, Pb, Se, Sr and Zn) in *Codonopsis pilosuna* (GBW09501) fell into the allowable error range for 20 months, showing the reference material is stable within a specified period [10]. Other Chinese BERMs in powder form exhibit no change of the trace element composition for a long time, either.

Water samples are often considered to be unstable during long time storage, due to the likely absorption of trace elements on the container wall or the desorption from the wall. We studied the concentration variation of trace elements in fresh and sea water samples kept at acidic conditions (pH = 1) and low temperature and found no detectable change in the concentration of Ag, Au, Ba, Cd, Cu, Cs, Cr, Eu, Fe, Hf, La, Lu, Mo, Ni, Rb, Sb, Sc, Sm, Ta, Th, U, Yb, Zn and Zr during a period of over 300 day storage in polyethelene containers [11].

BERMs role in environmental and biological research

BERMs have been and are being used extensively in surveying endemic diseases and monitoring their correlation to the environment. China is a large country with varying landforms, soils and biological communities giving rise to a great range of ecological environments. Generally speaking, man should be in harmony with the environment he inhabits. Human health, however, is sometimes affected due to the abnormality of some elements essential to life, e.g. Se.

A well-known fact is that a number of endemic diseases are caused by, or can be related to environmental chemical factors. These include Keshan Disease, Kaschin Beck Disease, endemic goitre, cretinism and fluorosis.

BERMs are essential in a large-scale national environmental monitoring. Otherwise the data from hundreds of analytical laboratories are not comparable without the analytical quality control.

Taking Se as an example BERMs were used in mapping the distribution of selenium contents of feedstuff and forages in China. A total of 11,473 representative samples, including maize, barley, sorghum, millet, potatoes, oilseed meals, orchard grass, fescue, wild rye, brome grass, straws, haulms, etc., were analyzed for their Se contents from 1782 sampling sites in 1094 counties of 26 provinces and three cities (not including Taiwan) in China. The results indicate that 29% of the sampled counties are severely Se-deficient with less than 0.02 μg/g Se content, 43% are Se deficient with 0.03–0.05 μg/g, 19% are moderate Se-containing with 0.06–0.09 μg/g, and 9% are adequate Se-containing with above 0.10 μg/g [12].

Other examples for the use of BERMs in China are given in [13–15].

Future trends for BERMs in China

1. Preparation of more BERMs meeting the increasing requirements of trace element analysis in biological and environmental research;
2. Preparation of new varieties of BERMs with the certified properties, e.g. chemical species, organic compounds;
3. Further study of homogeneity of BERMs in terms of sampling constants and segregation degrees.

4. Development of some new type of BERMs for microbeam analysis of trace elements with scanning proton microprobe, synchronous radiation induced X-ray fluorescence analysis.

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