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Uncertainty Evaluation in Determination of Boron and Lead in Plastic by ICP-AES

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Abstract: The content of toxic trace elements is an important index to measure the quality of plastic products. For a long time, the determination of toxic trace elements is the focus of third party detection and so on. It is very important to evaluate the accuracy of detection. In this paper, a mathematical model of the uncertainty of trace elements based on regression equation, constant volume and weighing weight is established. The results show that the content of Boron and Lead in the plastic samples is (66.3 ± 5.4) mg/kg and (500 ±30) mg/kg, respectively.

1. Introduction

Plastic pollution is one of the environmental issues of common concern around word [1-3]. The United Nations Environment Programme (UNEP) 2018 World Environment Day[4] is the theme "Beat Plastic Pollution", calling for concerted efforts by all countries in the world to combat the problem of plastic pollution. In recent years, the research of plastic testing in China has developed rapidly[5,6]. Some progress has been made in the aspects of the monitoring and analysis methods of plastics, the pollution characteristics and distribution of pollutants in the environment, the environmental process and behavior , the accumulation and the toxic effects of plastics. The content of toxic trace elements is an important index to measure the quality of plastic products. For a long time, the determination of toxic trace elements is the focus of third- party detection institution and so on. It is very important to evaluate the accuracy of detection. In this paper, a mathematical model of the uncertainty[7,8] of trace elements is established by evaluating the uncertainty of the determination results, which provides an effective method for evaluating the content of trace elements in the future.

2. Methods and results:

2.1 Measurement methods

0.2000g plastic sample were used in the digestion tank for three times, and nitric acid was decomposed into a 50ml volumetric flask. The concentration of the element was measured by ICP-OES for three times.

0, 0.10, 0.20, 0.30, 0.40, 0.50 mL standard reserve solution(1000 mg/L) was removed by 1mL pipette respectively,and then transfer the standard solution of different quantity in the 100mL volumetric flask with the scale pipette and dilute it to the scale with water.The intensity of the calibration solution is measured under the same conditions , each solution is measured for three times, and the calibration curve is drawn using the average value.

2.2 Results

Table 1. The content of the element B and Pb

Element	c（mg/L)	ω（mg/kg）
B	0.2644	61.2
B	0.2662	66.7
B	0.2666	71.1
Pb	2.086	535
Pb	1.959	460
Pb	2.040	505

3.Mathematical model of uncertainty

Calculation formula of element content is：

$$\omega = \frac{cV}{m}$$

ω(mg/kg), c(mg/L), V(mL), m：The quality of the sample(g).

3.1 Source of uncertainty

The sources of uncertainty by the mathematical model include：

1.Uncertainty in the concentration of elements in a sample solution:u(c)；

2.Uncertainty in the concentration of elements in a sample solution:u(V)；

3.Uncertainty of the quality of the sample:u(m)；

4.Uncertainty of repeated experiments:u(rep).

4.Evaluation of the relative standard uncertainty components

4.1 Evaluation of relative standard uncertainty of concentration in solution:$u_{rel}(c)$

4.1.1 Relative standard uncertainty of linear fitting of calibration curve:$u_{rel}(c)_1$

From calibration curve $I = a + bc$, we can see:

$$b = \frac{\sum_{i=1}^{n}(c_i - \bar{c})(I_i - \bar{I})}{\sum_{i=1}^{n}(c_i - \bar{c})^2}$$

$$a = \bar{I} - b\bar{c}$$

So, uncertainty of element concentration in solution from Calibration curve linear fitting $u(c)_1$ is

$$u(c)_1 = \frac{s_R}{b}\sqrt{\frac{1}{m} + \frac{1}{n} + \frac{(c_0 - \bar{c})^2}{\sum_{i=1}^{n}(c_i - \bar{c})^2}}$$

s_R means standard deviation of calibrated solution:

$$s_R = \sqrt{\frac{\sum_{i=1}^{n}(I_i - =1c_i + a))^2}{n-2}}(m=9,n=18)$$

$u(c)_1$ of element B and Pb was 0.0008 and 0.0004 mg/L, $u_{rel}(c)_1$was 0.004 and 0.002, respectively.

4.1.2 Uncertainty of standard solution concentration: $u_{rel}(c)_2$

The standard solution concentration was 1000 mg/L，k=2，so the standard uncertainty: $u_{rel}(c)_2$=2 mg/L, relative standard uncertainty: $u_{rel}(c)_2$=0.002.

4.1.3 Uncertainty of removing the volume of standard solution: $u_{rel}(c)_3$

we removed 0,0.10 ,0.20,0.30, 0.40 , 0.50 mL standard reserve solution of element B by using 1mL pipette respectively to build calibration curve , According to GB/T 12805, $u_{rel}(c)_3$ of element B =0.005 . similar to element B, $u_{rel}(c)_3$ of element Pb is 0.005.

4.1.4 Uncertainty of instrument variability

Since the repeatability of the measurement method has been calculated, the variability of the instrument has been included and is no longer calculated.

4.1.5 Relative Standard uncertainty of element concentration in sample solution:

$$u_{rel}(c) = \sqrt{u_{rel}^2(c)_1 + u_{rel}^2(c)_2 + u_{rel}^2(c)_3}$$

So, $u_{rel}(c)$ of element B and Pb is 0.007 and 0.05，respectively.

4.2 Evaluation of relative standard uncertainty of constant volume of sample solution:$u_{rel}(V)$

The test solution was diluted with water in a 50 ml volumetric flask. According to GB/T 12806, tolerance of 50 mL volumetric flask in class A is ±0.5 mL. $u(V)$ = 0.02ml. $u_{rel}(V)$ = 0.0004.
If the sample is repeatedly measured several times, usually several 50ml volumetric flasks are used. It can be considered that the volume error and reading repeatability have been randomized, and the volume uncertainty component can be ignored.

4.3 Evalution of relative standard uncertainty resulted from weighing sample quality by balance:$u_{rel}(m)$

The quality weighed of the samples is 0.2000g. According to the certificate, the maximum allowable difference of the balance is ±0.5mg, weigh twice, once empty, once with plate, and evenly distributed，k=$\sqrt{3}$.

The standard uncertainty of the sample quality weighed : $u(m) = \sqrt{2 \times \left(0.5/\sqrt{3}\right)^2}$ = 0.41mg.
Relative standard uncertainty:$u_{rel}(m)$ = 0.41/200=0.002

4.4 evaluation of relative standard uncertainty introduced by repetitive experiments : $u_{rel}(rep)$

The standard uncertainty introduced by repetitive experiments is

$$u(rep) = s(\omega)/\sqrt{n}$$

$u_{rel}(rep)$ is both 0.03 .

5.Evalution of synthetic standard uncertainty

The components are not related to each other and the combined uncertainty is calculated according to the square root.

$$u_{crel}(\omega) = \sqrt{u_{rel}^2(c) + u_{rel}^2(V) + u_{rel}^2(m) + u_{rel}^2(rep)}$$

$$u_c(\omega) = u_{crel}(\omega) \times \omega$$

$u_{crel}(\omega)$ is 0.04, 0.06, respectively, $u_c(\omega)$ is 2.7, 30mg/L , respectively.

6.Evalution of extended uncertainty

95% confidence interval, including factor K= 2, U= 5.4, 60 mg/L, respectively.

7.Expression of analysis results

The content of element Boron and Lead by ICP-OES is:
B： （66.3±5.4）mg/kg， k=2

Pb：（500±30）mg/kg， k=2

8.Conclustions

In this paper, we established an uncertainty model for content of element B and Pb which can be used for evaluation of plastic pollution.

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