Uncertainty Evaluation in Determination of Chromium by Inductively Coupled Plasma Mass Spectrometry(ICP-MS)

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Abstract: The content of Chromium in plastic products is (276±6) mg/kg, which is determinated by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) using Microwave digestion. A mathematical model of the uncertainty containing variables such as regression equation, constant volume , weighing weight and so on is established.

1. Introduction

Plastic products have deeply affected our lives. Although"the plastic limit order" (which has been implemented for more than a decade) has greatly reduced the white pollution[1-3], it is inevitable that a large number of plastic products still exist and cannot be eliminated. Poor-quality plastic products contain a large amount of heavy metal ions, which greatly affect people's health. They also pollute the environment after they are discarded. Therefore, the detection of heavy metals in such samples has always been the focus of the inspection industry. The evaluation of heavy metal content, which is an important indicator of the manufacturer's product quality. In this paper, a mathematical model of the uncertainty of Chromium is established[4].

2. Methods and Results

2.1 Measurement methods

0.2g sample was treated by nitric acid with microwave digestion for three times, and decomposed into a 50mL volumetric flask.

0, 1, 2, 3, 4, 5 mL standard reserve solution(1 mg/L) , which is prepared from 1000 ppm solution, was removed by 1mL pipette, respectively, and then set the volume to 100 mL.The intensity of the calibration solution is measured under the same conditions by ICP-MS for three times.

2.2 Results

The content of the element Chromium is 278.626, 276.685, 273.684 mg/kg, respectively.

3. Mathematical model of uncertainty(Identification and Evaluation of Uncertainty Components)

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:

Uncertainty in the concentration of elements in a sample solution based on standard curve line :u(c);

Uncertainty in the concentration of elements in a sample solution based on the sample

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volume:u(V);

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

Uncertainty of repeated experiments:u(rep).

4. Evaluation of 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:

$$b = \frac{\sum_{i=1}^{n}(c_i-\bar{c})(I_i-\bar{I})}{\sum_{i=1}^{n}(c_i-\bar{c})^2} = 8621.43$$

$$a = \bar{I} - b\bar{c} = 3161.366$$

the calibration equation I= 8621.43c+3161.366

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:

$$s_R = \sqrt{\frac{\sum_{i=1}^{n}(I_i-=1c_i+a))^2}{n-2}} = 3042.577(m=9,n=18)$$

$u(c)_1$ of element Cr was 0.145441 ug/L, $u_{rel}(c)_1$was 0.007.

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

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

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

According to GB/T 12805[5], The volume errors are 0, ±0.008, ±0.008, ±0.008, ±0.0080, ±0.050ml , respectively, the corresponding standard uncertainty is 0, 0.0033, 0.0033, 0.0033, 0.0033, 0.020ml based on the distribution of triangles, respectively:

$$u_{rel}(c)_3 = \sqrt{(0+0.033^2+0.0033^2+0.0033^2+0.0033^2+0.002^2)\ /6} = 0.0027$$

4.1.4 Relative standard uncertainty of element concentration in sample solution:

$$u_{rel}(c) = \sqrt{u_{rel}^2(c)_1 + u_{rel}^2(c)_2 + u_{rel}^2(c)_3} = 0.008$$

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

According to GB/T 12806 and the distribution of triangles, The volume errors of 50 mL volumetric flask in class A is ±0.5 mL. u(V) = 0.02ml:

$$u_{rel}(V) = 0.020/50 = 0.0004$$

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

We weigh the sample with a scale with a volume of 0.1 mg. The allowable difference is ±0.1 mg, According to the certificate, the standard uncertainty is $(\frac{0.1}{\sqrt{3}})$ mg, so

$$u(m) = \sqrt{(\frac{0.1}{\sqrt{3}})^2 \times 2} = 0.082(mg)$$

and the relative standard uncertainty:

$$u_{rel}(m) = 0.082 / 500 = 0.00016$$

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

Weigh 3 samples, determined by the determination method, the results are shown in Table 1:
According to table 1, the standard uncertainty introduced by repetitive experiments is

$$u(rep) = s(\omega)/\sqrt{n}=1437.6$$

$$u_{rel}(rep) =0.006.$$

Table1 Repeated results of the sample for three times

average value	Standard deviation of single measurement	The standard deviation of the average of the measured values
276332	2490	1437.6

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.

$$u_{rel}(w) = \sqrt{u_{rel}^2(c) + u_{rel}^2(V) + u_{rel}^2(m) + u_{rel}^2(rep)} =0.01$$

$$u(w) = 2763 ug / kg$$

6. Evalution of extended uncertainty

95% confidence interval, including factor K= 2, U = 2763*2=5527 ug / kg

7. Expression of analysis results

The content of Chromium by ICP-MS is:

$$w = (276 \pm 6) mg / kg, k = 2$$

8. Conclusions

The content of element Chromium is 276±6 mg/kg, k=2, and we successfully established a uncertainty model for uncertainty evaluation based on regression equation, constant volume, weighing weight.

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