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DETERMINATION OF MELAMINE RESIDUE IN WATER SAMPLES BY UPLC/MS/MS

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The melamine contamination of milk that occurred in 2008 was undoubtedly a serious food safety situation in China as safe drinking water is an important part of food safety. Since both the drinking water and the food chain may cause a direct or indirect impact on human health, it is urgent to carry out the study on the analysis method of melamine residue in water samples.

EXPERIMENTAL

Equipment and Reagents

An Ultra Performance Liquid Chromatography/tandem mass spectrometry (ACQUITY®/Quattro Premier™); MassLynx™ 4.1 workstation; solid-phase extraction (Zymark); Waters Oasis® MCX SPE column (200 mg, 6 mL); pressure blowing concentrator (Organomation).

Methanol and acetonitrile are both in liquid chromatography pure (Fisher); water is obtained from a Millipore water purification system; ammonium acetate is in guaranteed grade (Merck), hydrochloric acid and ammonia are in analytical pure (purchased from East China Pharmaceutical Company); and the standard reference is purchased from J&K.

Sample of the Pretreatment Methods

Solid-phase extraction: Add 5 mL of methanol and 5 mL water, at the speed of 5 mL/min to the column to make it active. Acidification of water sample: Add 0.25 ml of concentrated hydrochloric acid to 500 ml of water. Add the water sample with the speed of 4 mL/min and the volume of 500 ml. Rinse the sample with 5 mL of water and methanol respectively. Elute with 5 mL of methanol (containing 5% of ammonia) twice and concentrate the eluent with a pressure blowing concentrator until it is almost dried up. Dilute to 1 mL with mobile phase and filter it. Inject.

CHROMATOGRAPHY CONDITIONS

UPLC®/MS method: column: UPLC column (ACQUITY UPLC® BEH HILIC, 1.7 µm, 2.1 50 mm); column temperature: 40 °C; mobile phase: a mixture of acetonitrile and water (containing 10 mM of ammonium acetate) (90:10); flow speed: 0.4 ml/min.

The target contaminator is quantitatively analyzed with the MRM mode. Detect the target with ESI^+. Capillary voltage: 3.0 KV, source temperature: 120 °C, the temperature of desolvent gas: 400 °C, flow speed: 800 L/h, cone gas flow: 50 L/h; argon flow speed: 0.38 mL/min at the MRM mode; cone current: 40 V, CID: 17; characteristic ions pair: 127>85, as in the following Figure 1.

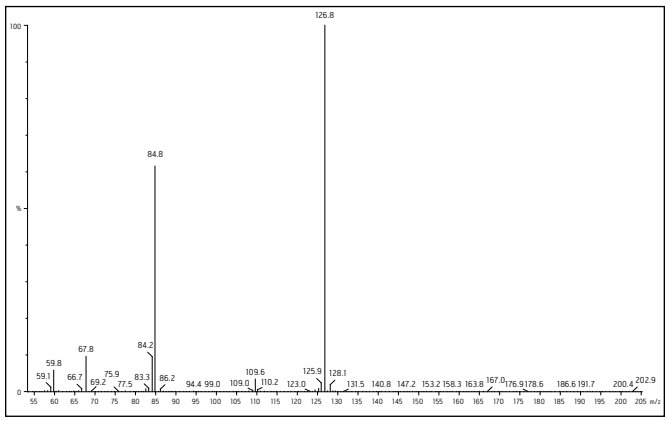


Figure 1. Mass spectra of melamine.

PERFORMANCE

Accuracy

Add 6.0 µg of target substance to 500 ml of testing water, and the recovery of a series of pre-sampling is 101.8±10.7% (n=6), as showed in Table 1.

Precision

Inject 1.0 µg/ml of standard solution for 6 times repeatedly and the RSD of peak area is 3.2%.

Detective limit

The signal/noise ratio is 15 for 1.0 ng/mL of standard solution and 3 for 0.2 ng/mL of standard solution, as shown in Figure 2. Taking the pre-sampling procedure into consideration, the quantitative detective limit of this method is 0.4 ng/L.

Linearity

Prepare 4 standard solutions with different concentrations ranging from 1.2 ng/mL to 2400 ng/mL, the equation for the standard curve is y=1350.3x+161822,R=0.9925.

Blank

The blank is lower than the detective limit.

Determination of sample

The water sample is obtained from a reservoir in Zhejiang province with a concentration of melamine of <0.4 ng/L. Add some standard solution to this water sample and analyze the content of melamine. The concentration of melamine is 0.03 μg/L and the total ion flow is showed in Figure 3.

Analytes	Precision %(n=6)	Recover% (n=6)	Detective limit (ng/L)	Linearity
Melamine	3.2	101.8±10.7	0.4	y = 1350.3x + 161822, r=0.9925

Figure 1. Performance of this method.

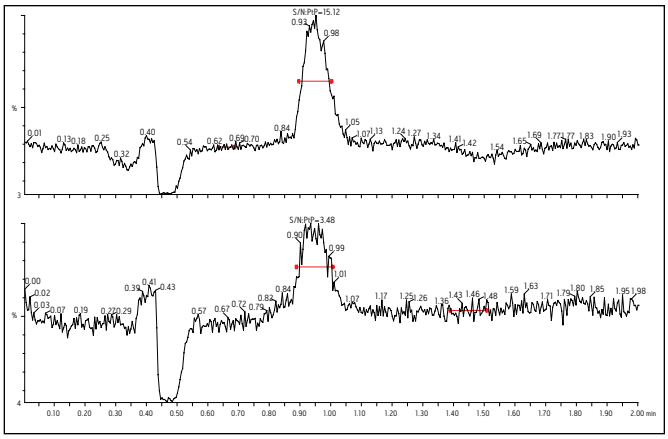


Figure 2. The S/N of melamine in different concentration.

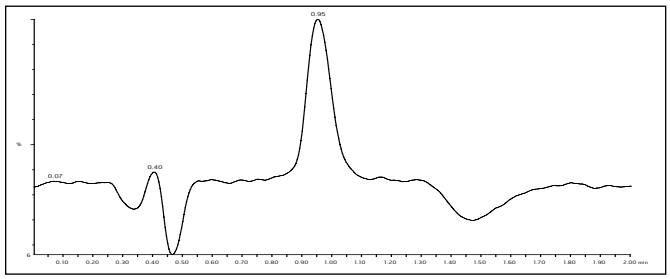
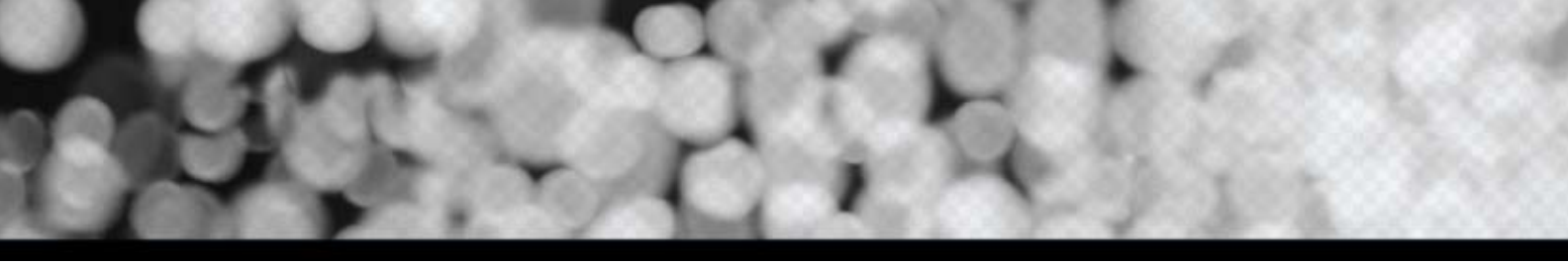


Figure 3. TIC of testing sample.

CONCLUSIONS

The UPLC/MS/MS method used for the determination of melamine residue in water sample has been proved to be practicable. The analyses can be fulfilled in 1.0 min with a recovery of 101.8±10.7% and a precision of 3.2%. With the range covering three magnitudes, the linear coefficient is greater than 0.99 and the detective limit is up to 0.4 ng/L. All of these indicated that this method meets the requirement of practical analyses.



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