Notice of Retraction

After careful and considered review of the content of this paper by a duly constituted expert committee, this paper has been found to be in violation of IEEE's Publication Principles.

We hereby retract the content of this paper. Reasonable effort should be made to remove all past references to this paper.

The presenting author of this paper has the option to appeal this decision by contacting TPII@ieee.org.
Determination of Permethrin Residues in Vegetables by Photochemical Fluorescence Spectrometry

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Abstract : Permethrin is a new kind of pesticide with fluorescence. Its fluorescence intensity can be enhanced in the presence of β-cyclodextrin. Based on this , a spectrfluorimetry method with high sensitivity and selectivity was developed for the determination of permethrin. Under the optimum condition, the permethrin has maximum excitation and emission wavelengths at 281 nm and 330 nm respectively. The linear dynamic range is from 0.004μg/ml to 0.036μg/ml with the correlation coefficient of 0.9993, and the RSD is 1.0 %. The method is obviously feasible in the determination of permethrin residues in vegetables with recoveries for the spiked sample between 98.0% and 101.0 %.

Keywords : Permethrin ; photochemistry; fluorescence spectrometry; β-cyclodextrin

Ⅰ INTRODUCTION
Permethrin is effective, broad-spectrum, low-residue pesticide with a non-cyano pyrethroid structure. It has been playing the role of contact poison and stomach toxicity, no smoking inside the conduction and fumigation, a clearly broad spectrum insecticide. It can be used to prevent and cure a variety of pests on the cotton, vegetables, tea, fruit trees. However, having applied the insecticide, fruits and vegetables still need to be cautioned, especially being purchased during the forbidden period. At present, the determination of permethrin are biosensor method [1], HPLC [2, 3], gas chromatography [4, 5], spectrophotometry [6, 7] and so on. These methods have low detection limits and high accuracy, but with applied limitation due to complicated preparations. Fluorescence analysis has the advantages of high sensitivity and selectivity, the less sample use, fast and easy operation, etc., particularly in the detection of trace material. In this paper, the fluorescence properties of permethrin [8] are studied by mean of photochemical fluorescence analysis, photons as derivative reagent, without the impurities and dilute solutions, which eliminates a number of negative factors adding the chemical reagents determining. The method used to determine permethrin residues in vegetables, with simple operation, high accuracy, precision and less interference, fully meets the national standard requirements of the maximum allowance of the permethrin residue.

Ⅱ EXPERIMENT
2.1 Instruments and reagents
F-4500 fluorescence spectrophotometer (Hitachii, Japan), pH5-3B Precision Digital pH meter (0.01) (Shanghai), ZF-20D black-box-type ultraviolet analyzer (Shanghai).

Permethrin (Developed by the Research and Monitor Institute of environmental protection, Ministry of Agriculture, standards), β-cyclodextrin (Beijing Aobo Star biotechnology Ltd.) 0.01mol / L. Other reagents are analytical grade, and double distilled water is used in experiments.

2.2 Preparation of the solution
1. Permethrin standard solution:
Take 1ml the standard methanol solution with concentration 100μg/ml to a 100ml volumetric flask as the preparation of 1μg/ml stock solution, diluted with distilled water to 0.01μg/ml standard solution when using.
2. Preparation of Britton-Robinson buffer solution:
The mixture of the boric acid, 85% phosphoric acid, 36% acetic acid (concentration of them is 0.04mol / L) and 0.2mol / L NaOH solution are combined at different mixing ratios to form different pH buffer solutions. Calibrate the pH value by pH measurements.

2.3 Experimental Methods
Pipette 1ml permethrin standard solution into 25ml colorimetric tube, then add in sequence 6ml 0.01mol / L β-CD solution, 1ml Britton-Robinson buffer solution (pH = 9.29), 2ml 5% of the formaldehyde solution, diluted with distilled water to the mark, shake it well, at room temperature with a 1cm quartz cuvette at the 10nm pass band of excitation and emission spectra the fluorescence intensity is measured.

III RESULTS AND DISCUSSION
3.1 Excitation and emission spectra
Permethrin in the excitation wavelength 281nm, emission wavelength 330nm can emit fluorescence, the intensity of which would increase significantly in the presence of β-cyclodextrin and exposure of UV light, the peak position not changed. Spectral pass band for the 10nm, excitation and emission spectra shown in Figure 1.

3.2 The effect of β-cyclodextrin
Experiments show that β-cyclodextrin can increase the sensitivity of the fluorescence intensity of permethrin obviously. In the system, with the increasing amount of 0.01mol/L β- cyclodextrin solution , its fluorescence intensity waxes, but the peak position does not change. Figure 2 shows: when the addition of the 0.01mol / L β-CD is 6-7ml, the fluorescence intensity is moderate, so in this experiment, the addition of β-cyclodextrin is 6.0ml.

3.3 The effect of formaldehyde
Experiments show that 5% formaldehyde also has a position effect on the fluorescence properties of permethrin and the peak position does not move as well. In this experiment, the amount of formaldehyde is 2.0ml.

3.4 pH on the fluorescence intensity
Buffer solution with different pH conditions are tested, and the results are shown in Figure 4. In the range of pH from 8.65 to10.14, fluorescence intensity would be at its maximum with the most stability, and then decrease. Therefore this study selects the pH = 9.29 buffer solution to control acidity.
3.5 The influence of the time of UV light exposure

According to experimental results, when irradiated by UV light, the fluorescence intensity would rise markedly, with its highest point after 10min irradiation.

3.6 Interference Experiment

When permethrin is at 0.004 $\mu$g/ml, the relative error $\leq \pm 5\%$, the following coexisting substances do not interfere: 1.0 $\times 10^{-3}$mol / L lactose and 1.0 $\times 10^{-4}$mol / L of K, Ca, Ba, Al, Fe, starch, glucose, while ascorbic acid has a weak interference.

3.7 Method of characteristics

Take 1ml permethrin standard solution with different concentrations into 25ml colorimetric tube, then add 6ml $\beta$-cyclodextrin solution, 2ml 5% formaldehyde solution, 1ml Britton-Robinson buffer solution (pH = 9.29) in turn, dilute to the mark with distilled water and shake it well. At room temperature the intensity would be measured, according to the experimental methods 1.3. The results show that permethrin, its concentration within 0.004$\mu$g/ml to 0.036$\mu$g/ml, together with fluorescence intensity shows a good linear relationship. Linear equation is $F = 20.97X + 298.42$, $R = 0.9993$, of which the detection limit ($S / N = 3$) is 0.14ng / mL. After the parallel determinations of the 11 standard solution at 0.01$\mu$g/ml, a relatively standard deviation (RSD) is 1.0%.

3.8 Sample Analysis

3.8.1 Preparation of samples

Take 2. 6773 g celery and 2. 6706 g lettuce, which are commercially available, torn to pieces, soaked with 50 mL pure methanol, and washed surface of vegetables by a dropper, moved to 50 mL volumetric flask after filtration. After constant volume with methanol, the sample solution are generated.

3.8.2 Determination of sample

In accordance with the experimental methods, add 1ml celery and lettuce sample solutions into two 25ml colorimetric tubes, and then take another two 25ml cuvettes with 1ml celery, 1ml lettuce sample solutions, and 1 ml permethrin standard solution. Finally conduct parallel determination five times. Results are as shown in Table 1.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Found($\mu$g/ml)</th>
<th>Added($\mu$g/ml)</th>
<th>Total($\mu$g/ml)</th>
<th>Recovery(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lettuce</td>
<td>0.00524</td>
<td>0.001</td>
<td>0.00625</td>
<td>101%</td>
</tr>
<tr>
<td>Celery</td>
<td>0.00466</td>
<td>0.001</td>
<td>0.00564</td>
<td>98%</td>
</tr>
</tbody>
</table>

REFERENCES


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