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## Determination of acrylonitrile in solid waste by automatic headspace gas chromatography

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### Abstract

This study established a method to determine the acrylonitrile in solid waste by automatic headspace gas chromatography. This method adopted the automatic headspace sampling, separated by DM - FFAP chromatographic column, detected by FID detector. The conditions of headspace balance time and equilibrium temperature were optimized. The linear range, detection limit, recovery, sensitivity and stability were tested. Under the conditions of 80 °C and balance time of 30 min, the method detection limit of acrylonitrile was 1.66 µg /kg. The relative standard deviation was under 5%. The standard addition recovery was 92.7% ~ 101.5%. The results showed that the before processing of this method is simple, rapid, no consumption of organic solvents, accurate and applicable. The method can meet the requirements of Identification standards for hazardous wastes-Identification for toxic substance content.

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*Keywords:* automatic headspace sampling; gas chromatography; acrylonitrile; solid waste

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### 1. Introduction

Acrylonitrile is a public basic organic chemical raw materials, and belongs to the basic and important raw materials of three synthetic materials-synthetic fiber, synthetic rubber and plastics<sup>1</sup>. It is widely used in organic synthesis industry and people's economic life. The acrylonitrile is a kind of colorless volatile liquid<sup>2</sup>, and its steam

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has acute toxicity<sup>3</sup>. It is also one of the specified test items in the Identification standards for hazardous wastes – Identification for extraction toxicity (GB5085.3-2007)<sup>4</sup> because acrylonitrile is abundant in the solid waste and it is one of the difficult part to deal with.

Recently, most of acrylonitrile is determined by method of Gas Chromatography(GC-MS)<sup>5-6</sup>, Headspace Gas Chromatography(GC-MS)<sup>7-8</sup>, Purge and Trap Gas Chromatography(GC-MS)<sup>9-10</sup> at home and abroad. It is suggested that it should use static headspace, purge and trap technique when analyzing the fluid samples and solid samples of acrylonitrile in the Identification standards for hazardous wastes-Identification for extraction toxicity. Headspace Gas Chromatography is one of the most widely used and the rapid development separation and analysis methods, and it is also the main method to determining the acrylonitrile. There are more introduction in the determination of fluid samples, but for the determination of solid waste is rarely reported<sup>11</sup>.

In this study, the acrylonitrile in solid waste is determined by Automatic Headspace Gas Chromatographic DM-FFAP chromatographic column and FID detector, and the method are studied systematically. The results showed that this method eliminated the tedious pretreatment process, greatly reduced the sample period, no consumption of organic solvents, accurate and applicable, compared with the method of Purge and Trap Gas Chromatography. The method also can meet the requirements of Identification standards for hazardous wastes-Identification for toxic substance content.

## 2. Experimental

### 2.1. Instruments and reagents

Gas Chromatograph: PerkinElmer 600, with FID detector. Automatic headspace sampler: PE Turbo Matrix HS40. Chromatographic column: DM-FFAP (50m×0.32mm×0.5 μ m). Acrylonitrile standard solution: 1020mg/L, BW3436. The matrix modifier: adding phosphoric acid to 500ml in deionized water until  $\text{PH} \leq 2$ , adding 180g of sodium chloride, dissolving and mixing.

### 2.2. Chromatographic condition

Chromatographic column: DM-FFAP capillary column 50m×0.32mm×0.5μm, column temperature: at 40°C for 2min, with 10°C/min to 100°C for 2min. Sample inlet: at 200°C, carrier gas(high pure nitrogen), constant current, the flow rate was 4.5mL/min, split injection, the split ratio 5:1. Detector: at 250°C, the flow rate of hydrogen was 40mL/min, the flow rate of air was 400mL/min, the flow rate of make-up gas(high pure nitrogen) was 30mL/min.

### 2.3. Automatic headspace condition

Temperature: the temperature of sampling probe was 120°C, the temperature of transmission line was 150°C, furnace temperature was 80°C. Timing: pressuring for 2min, sampling 0.1min, pulling the probe for 0.2min, thermal insulation for 30min, the cycle of GC for 10min. Options: operator schema: constant, The sampling schema: time(control). Gas circuit: the pressure of sampling was 14.0psi, the pressure of chromatographic column was 15.0psi.

### 2.4. Determination method

#### 2.4.1. Preparation of standard solution

Using the concentration of 1020mg/L standard solution of acrylonitrile diluted into 10.2mg/L standard solution, for the preparation of standard curve.

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