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Short communication

# Centrifugal concentrator for the substitution of nitrogen blow-down micro-concentration in dioxin/polychlorinated biphenyl sample preparation

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## Abstract

A centrifugal concentrator was applied for the substitution of nitrogen blow-down micro-concentration in dioxin/PCB sample preparation. Automation was achieved through this apparatus and resulted in significant improvements in sample preparation productivity, along with excellent reproducibility and recovery rate of dioxin/PCB. Mean recoveries of dioxin congeners were 90–106%, respectively. © 2002 Elsevier Science B.V. All rights reserved.

*Keywords:* Centrifugal concentration; Sample preparation; Dioxin; Polychlorinated biphenyls; Nitrogen

## 1. Introduction

The monitoring of substances of environmental concern such as dioxin/polychlorinated biphenyl (PCB) is costly and time consuming. Hence, each stage of sample preparation procedures needs to be improved. There are many publications for the improvement of the extraction, clean-up and/or determination procedures. However, few of them describe the modification of concentration stage. The micro-Snyder column of the K-D apparatus is widely used for micro-concentration [1–3]. The major draw-

back of the use of K-D apparatus is low productivity. An alternative to the micro-Snyder column is a closed nitrogen blow-down device such as Zymark Turbo Vap™. But Suzuki pointed out recently possible dioxin sample loss along with the nitrogen blow-down [4].

The centrifugal concentrator can be substituted for the micro-Snyder column or nitrogen blow-down device. Automation is achieved through the concentrator and consequently laboratory productivity improves significantly. In this work, recovery rates and reproducibility of the centrifugal concentrator were studied. First of all, a centrifugal concentrator was reconstructed in order to install a K-D concentration tube onto its angle-rotor. Then, a concentration test was performed to assess the recovery rates and reproducibility.

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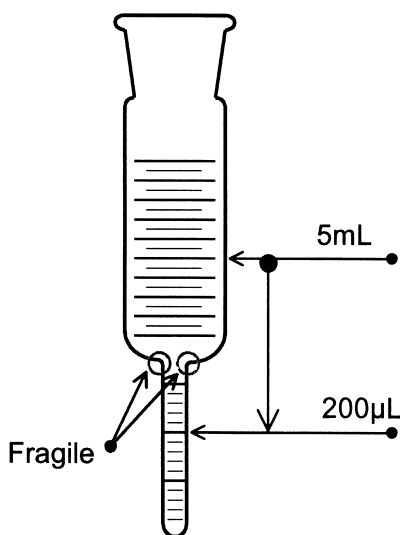


Fig. 1. K-D concentration tube.

## 2. Experimental

### 2.1. Standards

NK-LCS-AD dioxin standard mixture (Wellington Laboratories, Ont., Canada) containing  $^{13}\text{C}$ -labelled 2378-T4CDD/F, 12378-P5CDD/F, 23478-P5CDF, 123478-H6CDD/F, 123678-H6CDD/F, 123789-H6CDD/F, 234678-H6CDF, 1234678-H7CDD/F, 1234789-H7CDF (40 pg/ $\mu\text{L}$ , respectively) and O8CDD/F (80 pg/ $\mu\text{L}$ , respectively) was used as a

recovery standard; 25  $\mu\text{L}$  of the standard was added to a 10-ml K-D concentrator tube (Fig. 1). Then the standard was diluted with toluene.

### 2.2. Apparatus

To protect the long-nosed bottom of the K-D concentration tube, the space between the angle rotor and the isothermal chamber needs to be securely maintained (Fig. 2). So, a commercial centrifugal concentrator (Savant, Holbrook, NY) was reconstructed as follows. A large isothermal chamber (AES2010) was combined with a small angle rotor (AES1010) and the rotor shaft was raised in order to make an additional space between the rotor and the chamber.

### 2.3. Concentration

Dioxin standard mixture was diluted to 200  $\mu\text{L}$  or 5 ml. The 200- $\mu\text{L}$  sample was analyzed immediately after the dilution with GCQ plus ion trap mass spectrometer (ThermoQuest, Austin, TX); 5-ml samples were concentrated to 200  $\mu\text{L}$  prior to GC-MS analysis. The recovery rate was calculated based on the results of the 200- $\mu\text{L}$  sample. Rotational frequency was 3000  $\text{min}^{-1}$ . Pressure/temperature of the isothermal chamber was set at 1333 Pa/43°C. The dilution/analysis was repeated nine times to assess the reproducibility of the centrifugal concentration.

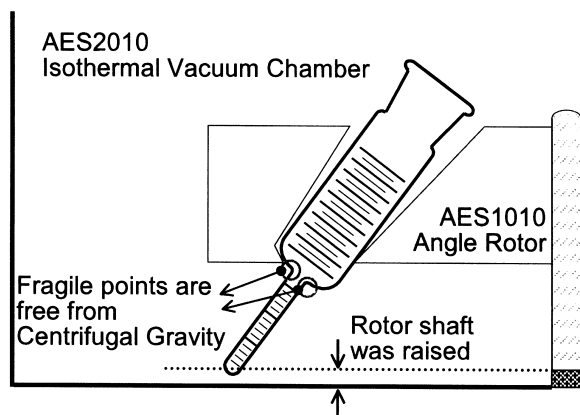


Fig. 2. Centrifugal concentrator.

## 3. Results and discussion

### 3.1. Recovery rate

Mean recoveries of dioxin congeners were 90–106%. As shown in Fig. 3, the recoveries of higher chlorinated congeners were slightly less than lower chlorinated congeners. This was an interesting phenomenon. As far as vapor pressure was concerned, higher chlorinated congeners were hardly dispersed into the air compared with the lower congeners. So, the lost portion of the higher chlorinated congeners was expected to remain in the K-D concentrator tube. It seemed that as the surface of the sample went down, a very small portion of the higher

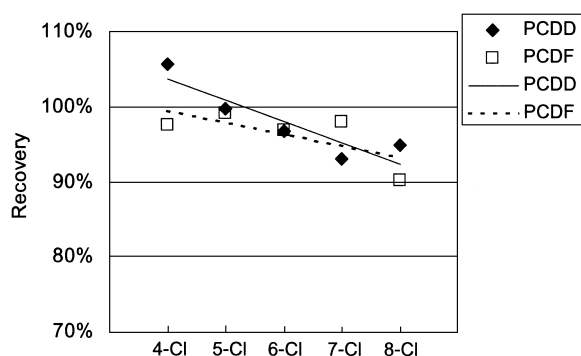


Fig. 3. Recovery rate of PCDD/PCDF.

chlorinated congeners was partially dried out on the wall of the tube and did not go down to the bottom along with the solvent.

### 3.2. Reproducibility

The coefficient of variance of the centrifugal concentration is shown in Fig. 4. These variances were the sum of those of pipetting, concentration and GC–MS determination. The variances of higher chlorinated congeners were considerably higher than lower chlorinated congeners. Higher chlorinated congeners were easy to dry-out compared to the lower chlorinated congeners and were likely to remain on the wall of the concentration tube if the evaporation speed was too fast, though this was an irregular behavior. As a result, the coefficient of variance of higher chlorinated congeners turned out to be higher than that of lower chlorinated congeners. In this study, the evaporation speed was

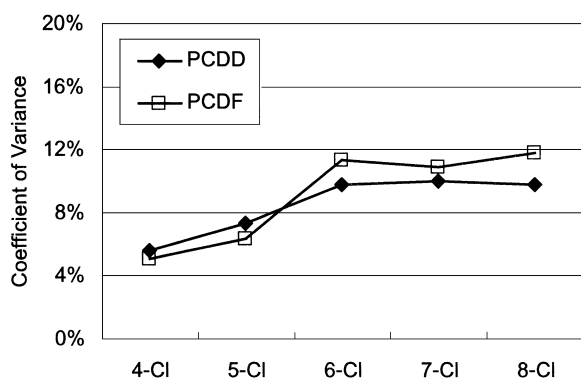


Fig. 4. Reproducibility.

approximately 0.2–0.3 ml/min and both recovery and variance were acceptable. However, if the evaporation was boosted by installing an additional heat source such as an infrared lamp, both the recovery and the variance of higher chlorinated congeners are expected to be worse than those of this study; 0.2–0.3 ml/min seems to be the most appropriate considering the concentration time, the recovery rate and the coefficient of variance.

### 3.3. Productivity

The evaporation rate of toluene was 5–10 times faster than that of the micro-Snyder column. In addition, sample throughput was improved because the concentration step could be automated by the centrifugal concentrator. The evaporation process was quite stable compared to that by the nitrogen blow-down device. As a consequence, the centrifugal concentrator significantly improved laboratory productivity.

## 4. Conclusion

A centrifugal concentrator can be substituted for a micro-Snyder column or nitrogen blow-down device. The recovery rate of lower chlorinated PCDD/PCDF was as good as 100% and the coefficient of variance was less than 5%. As chlorination number increased, recovery rate decreased. In contrast, coefficient of variance increased along with the chlorination number. Laboratory productivity could be improved significantly using the centrifugal concentrator.

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