

## Project Summary

# Analysis of Nitrite in NO<sub>2</sub> Diffusion Tubes Using Ion Chromatography

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A method was developed for the analysis of the NO<sub>2</sub> collected by a passive diffusion controlled atmospheric sampling device. The method measured the total amount of nitrite ion using ion chromatography. The precision obtained under field conditions, defined as twice the average deviation of the blanks, was 30 ppb hr. The overall accuracy of the method in exposure chamber tests was 20 percent.

The Palmes Tube design was altered by surrounding the triethanolamine coated screen with a Teflon liner. The nitrite was transferred from the diffusion tube sampler to a concentrator column by flushing the screen with deionized water in a device which minimized contamination due to handling. The total amount of nitrite was measured using standard ion chromatographic techniques.

*This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).*

### Introduction

A passive diffusion controlled personal sampler for nitrogen dioxide, known as the Palmes Tube, is widely used because it is small and inexpensive, and therefore easily deployed in large numbers. The traditional method for analysis reacts the collected nitrite with

reagents to form a colored solution, which is then measured quantitatively with a spectrophotometer. A number of different laboratories which use the Palmes Tube have reported the lower limit of detection is around 300 ppb hr and the accuracy is around 20 percent for an exposure time of one week. A method of analysis which is more sensitive without sacrificing accuracy would allow shorter exposure times, and would give significantly more information about ambient concentrations of nitrogen dioxide.

The variance of the field blanks determines the sensitivity of the method. A major contribution to the blank levels is exposure of the screens during preparation and storage of the samplers. A limitation of the sensitivity of the spectrophotometric method is the inability to concentrate the entire sample within the light path of the spectrophotometer. Ion chromatography is an alternative method of analysis which may be adapted to overcome the limitations of sample concentration.

### Procedures

The Palmes Tube design was altered by placing the triethanolamine screen within a Teflon liner, which then fit into the closed end of the diffusion tube. The acrylic diffusion tube was replaced by a stainless steel tube. A transfer apparatus was constructed which accommodated the sampler. Deionized water was used to rinse the collected nitrite from the screen. The entire nitrite sample on the screen was transferred and trapped on

a concentrator column in an ion chromatograph. The nitrite was then resolved chromatographically and quantified using a conductivity detector.

To reduce contamination to an acceptable level, all sampler parts were soaked in sodium hydroxide solution prior to preparation of the sampler. The freshly coated screens were dried in a stream of purified nitrogen gas. No convenient method was found which allowed exposed tubes to be stored for a reasonable length of time before analysis.

### Results and Discussion

The limit of detection was 6 ppb hr, which was at the noise level of the instrument. The achievable accuracy under field conditions which included shipping the samplers before and after exposure for a total of four days between preparation and analysis, was 20 percent at the 70 ppb hr exposure level. Field blanks and field blank deviations increased with the time between preparation and analysis. One day lapse time gave an average blank deviation of 6 ppb hr, but at four days, the average blank deviation increased to 30 ppb hr. The overall precision and accuracy of the method was limited by the perishable nature of the sampling device. In order to obtain precision which approaches the sensitivity of the method, ways to store the samplers both before and after exposure will be required.

Interferences collected by the sampler under ambient sampling conditions, both carried as a personnel monitor and at stationary locations, were small and did not affect the precision. Contamination, usually caused by poor handling, was easily recognized by an unusually large chloride peak. The throughput using the ion chromatographic method of analysis was roughly half the throughput using the traditional spectrophotometric method.

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*The complete report, entitled "Analysis of Nitrite in NO<sub>2</sub> Diffusion Tubes Using Ion Chromatography," (Order No. PB 87-188 777/AS; Cost: \$11.95) will be available only from:*

*National Technical Information Service  
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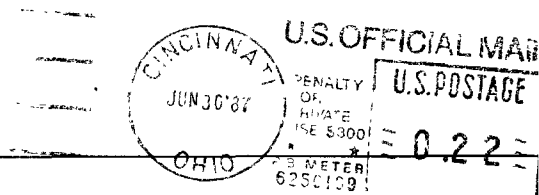
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