

## Nitrogen speciation by microstill flow injection analysis

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Received 22 July 2008; accepted revised 19 November 2008

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### ABSTRACT

A sequential determination of ammonia, nitrite, and nitrate has been carried out using a flow injection manifold incorporating a Microstill. The nitrogen speciation method has been designed using inorganic acid and base reagents, thus avoiding colorimetric reactions whose associated reagents and products are environmentally unacceptable. The robust manifold requires just three valves to switch measurement conditions for each species. UV irradiation was selected for nitrate reduction and gave acceptable resolution of nitrate and nitrite ions. In solutions containing comparable concentrations of both species, reduction of nitrate by UV irradiation gives a systematic error for nitrate due to nitrite interference at the 95% confidence level, nitrite being partially oxidised to nitrate during sample processing. At 98% confidence the error was not significant. A glass micro-electrode provided potentiometric detection in an ammonium chloride post-still collector stream. Ammonium chloride provided adequate baseline recovery when switching from acidic to basic distillation conditions, although resulting in an increase in detection limits over those found for a single analyte by the Microstill technique. The final speciation procedure gave working ranges of 0.03–10 mg/L for ammonia-N, 0.05–5 mg/L for nitrite-N and 0.1–10 mg/L for nitrate-N. The three N species were determined in 12 min for each water sample. The system has had limited trials in river waters containing ammonia and nitrate. Good precision and recoveries were obtained for the two species in the samples tested.

*Keywords:* Nitrogen speciation; Disinfection; Chloramination; Microstill; Flow injection

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