The Use of an Open Tubular Cadmium Reactor for the Determination of Nitrate-Nitrogen in Sugarbeet Extracts

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ABSTRACT

Cadmium reduction as a means for measuring nitrate-nitrogen has been shown to be less subject to interference from other ionic species than ion selective electrodes. The effectiveness of an open tubular cadmium reactor in an automated flow analyzer was evaluated for the measurement of nitrate-nitrogen in sugarbeet brei extracts. The reduction results strongly correlated (r^2> 0.99) with the results obtained from an ion selective electrode.

Key Words: Nitrate-Nitrogen, Cadmium Reduction, Ion Selective Electrode (ISE), Flow Injection Analyzer (FIA)

INTRODUCTION

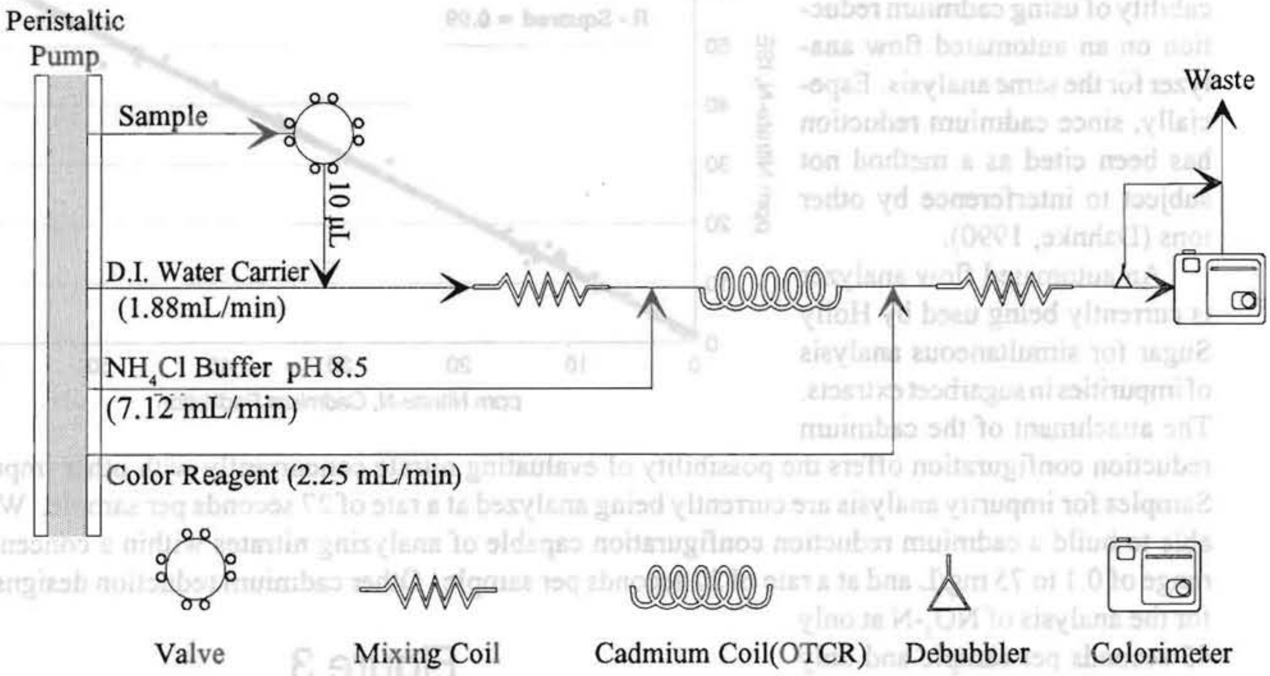
Many different methods are available for the determination of nitrate-nitrogen content in sugarbeet extracts. For rapid analysis of nitrate-nitrogen, many tare laboratories are using ion selective electrode technology. However, ion selective electrodes are subject to interference from ionic species of greater concentrations that are commonly found in sugarbeet extracts. The effects of the interfering ions can be quite large, and the removal of the interference can be complex and time consuming.

Automated flow instrumentation has led to the uncomplicated use of cadmium as a reducing agent for the colorimetric determination of nitrate-nitrogen. Realizing that this technology has potential for the analysis of sugarbeet extracts, we evaluated the effectiveness of using an open tubular cadmium reactor for the analysis of nitrate-nitrogen.

MATERIAL AND METHODS

Sugarbeet brei samples, extracted with 0.3% aluminum sulfate [$Al_2(SO_4)_3 \cdot 18H_2O$], were analyzed for nitrate-nitrogen [NO_3-N]. The NO_3-N concentration in the extracts ranged from <1 mg/L to over 50 mg/L. NO_3-N was determined directly from the extract using an ion selective electrode (ISE) attached to an Orion EA 940 using recommended techniques (Carlson, 1971). The same extract was then analyzed on the same day using an automated flow analyzer with flow injection capability and a modified cadmium reduction technique described by Griess-Ilosvay (EPA-600/4-79-020, 1984). In this method, nitrate is reduced quantitatively to nitrite by cadmium metal in the form of a cadmium coil (Open Tubular Cadmium Reactor (OTCR)). The nitrite formed is determined as an azo dye at 540 nm following diazotization with N-1-naphthylethylenediamine dihydrochloride. A schematic of the configuration used for this method is shown in Figure 1. Using this setup, we were able to determine NO_3-N in the extracts at a rate of 27 seconds per sample and at concentrations ranging from 0.1 mg/L to 75 mg/L. The use of an open tubular cadmium reactor, as opposed to a packed cadmium column, greatly eased the analysis.

Figure 1
Nitrate-Nitrogen in Sugarbeet Extracts
(0.1 - 75 ppm)



Fifty sugarbeet brei samples extracted with 0.3% aluminum sulfate and with varying concentrations of nitrate-nitrogen (NO_3-N) were selected for method validation. The NO_3-N concentration in the extracts ranged from <1 mg/L to over 50 mg/L. NO_3-N was determined directly from the extracts using an ion selective electrode(ISE) and cadmium reduction. The results were compared to each other to show precision.

Twenty sugarbeet brei extracts with varying concentrations of nitrate-nitrogen were spiked with different amounts of potassium nitrate [KNO_3] to determine accuracy. NO_3-N was determined directly from the extract using ISE and cadmium reduction. Spike recovery was then used to show the accuracy of the two methods.

RESULTS

The results obtained from the fifty samples comparing ISE and cadmium reduction analysis for NO_3-N are shown in Figure 2. The two methods strongly correlated (r^2= 0.99). Although the readings from the ISE were generally higher than cadmium reduction, for practical purposes, the differences are not important. Further evaluation of over 10,000 brei samples extracted during the 1992 campaign at a factory supported the premise that both methods are precise (r^2=0.90). The samples were analyzed by the tare laboratory using ISE and the Sheridan laboratory using cadmium reduction.

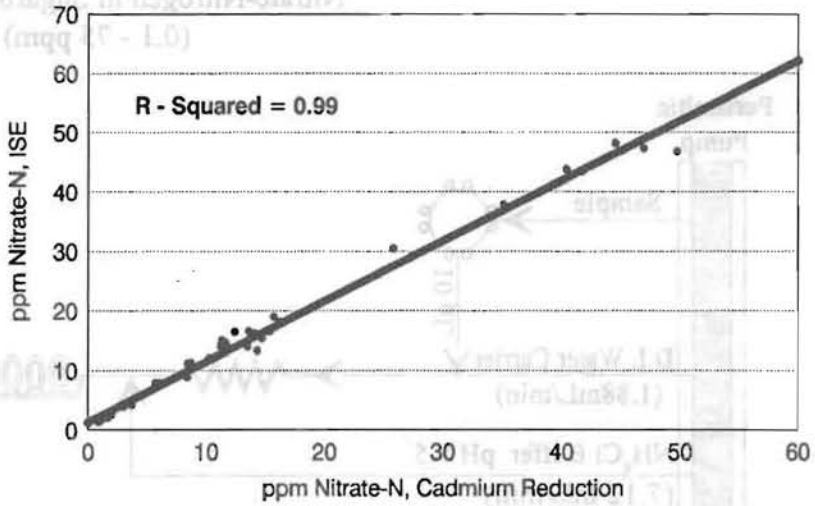
Accuracy was shown by spiking twenty brei extracts (Figure 3) with a known amount of potassium nitrate. The ISE recovered 103% (SD ± 3%) and cadmium reduction recovered 97% (SD ± 3%) of the standard indicating that both methods are relatively accurate.

DISCUSSION

Ion specific electrodes are being used on a routine basis for the analysis of NO_3-N in sugarbeets produced by growers. We wished to evaluate the applicability of using cadmium reduction on an automated flow analyzer for the same analysis. Especially, since cadmium reduction has been cited as a method not subject to interference by other ions (Dahnke, 1990).

Figure 2

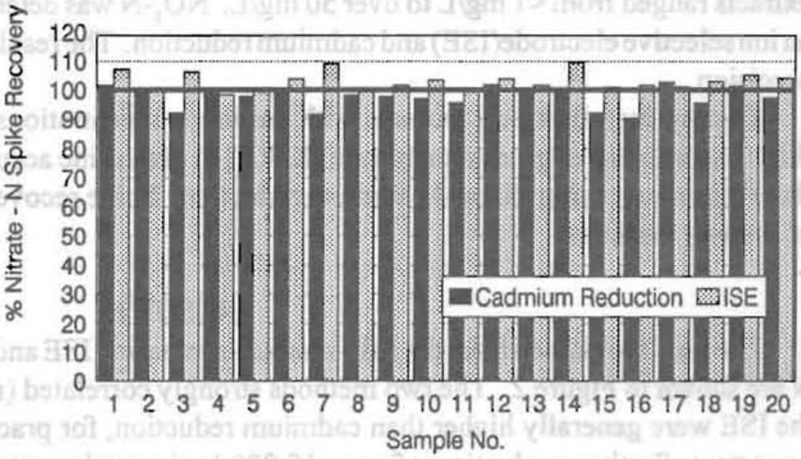
ISE/Cadmium Reduction Comparison



An automated flow analyzer is currently being used by Holly Sugar for simultaneous analysis of impurities in sugarbeet extracts. The attachment of the cadmium reduction configuration offers the possibility of evaluating nitrate concurrently with other impurities. Samples for impurity analysis are currently being analyzed at a rate of 27 seconds per sample. We were able to build a cadmium reduction configuration capable of analyzing nitrates within a concentration range of 0.1 to 75 mg/L and at a rate of 27 seconds per sample. Other cadmium reduction designs allow for the analysis of NO_3-N at only 45 seconds per sample and only within a concentration range of 0.1 to 10 mg/L. Sugarbeet extracts often contain much higher levels of nitrates. To make this possible, it was necessary to use an OTCR coupled with a Flow Injection Analyzer (FIA). High speed analysis is achieved with FIA because it is possible to use very small sample volumes and reduce sample to sample carryover.

Figure 3

Nitrate - N Spike Recovery



ISE vs Cadmium Reduction

CONCLUSION

NO_3-N can be precisely and accurately determined in sugarbeet extracts using either ISE or cadmium reduction. By using an OTCR for cadmium reduction and an automated flow system, determinations over a large range of concentrations can be made rapidly (>2 samples per minute). Cadmium reduction has been used extensively in soil, plant, and water analysis for a number of years. This technique provides the advantage of simultaneously measuring nitrates rapidly and accurately with other impurities in sugarbeet extracts.

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