



Determination of Sulfide and Cyanide Using Integrated Pulsed Amperometric Detection on a Disposable Silver Electrode

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Because of their toxicity, the determination of free sulfide and cyanide is increasingly important. Liquid chromatography with amperometric detection on silver electrodes offers excellent sensitivity for this application. The development of disposable silver electrodes ushers in a new era for reproducibility and ease of use.

Liquid chromatography with dc amperometry is a sensitive method for the separation and detection of sulfide and cyanide. However, when sulfide and cyanide are detected in real samples by dc amperometry on a silver working electrode, the electrode surface is frequently fouled (1). Although a fouled electrode can be reconditioned, the process is cumbersome and time consuming. Recently, a method was published showing that the application of a pulsed waveform is an effective method of preventing electrode fouling (1). In this note, we show that the combination of an optimized waveform with disposable silver electrodes provides a rugged, simple, and stable method for the determination of sulfide and cyanide.

Experimental

Dionex ICS-2500 consisting of a GP50 gradient pump with online degas, an ED50A electrochemical detector, and an AS50 autosampler. The working electrode is a disposable silver working electrode. The separation was achieved using a strong anion-exchange column (2 × 250 mm), with a capacity of 25 μ equiv/column, operated at 0.25 mL/min. All instrument control, data acquisition, and data analysis was performed using Dionex Chromeleon® software.

Results

Figure 1a shows the separation of 50 ppb sulfide and 50 ppb cyanide in 50,000 ppb chloride with exceptional resolution when compared with existing methods, and no apparent interference from the chloride matrix. Since there was no evidence of chloride in trace 1a, 50,000 ppb chloride was injected in the absence of sulfide and cyanide to determine if there was a coelution. Figure 1b confirms that there is no interference of chloride with sulfide or cyanide. Under these conditions, as little as 1 ppb cyanide and sulfide can be detected in real-world matrices, such as soil and wastewater.

Conclusions

This method demonstrates the determination of sulfide and cyanide, simultaneously, using anion-exchange chromatography with integrated pulsed amperometry on a disposable silver electrodes. Disposable silver electrodes can be used to produce equivalent or better results to nondisposable electrodes, while improving the ease of use and ruggedness of the method.

References

- (1) C. Giuriati, S. Cavalli, A. Gorni, D. Badocco, and P.J. Pastore., *J. Chromatogr., A* **1023**, 105–112 (2004).

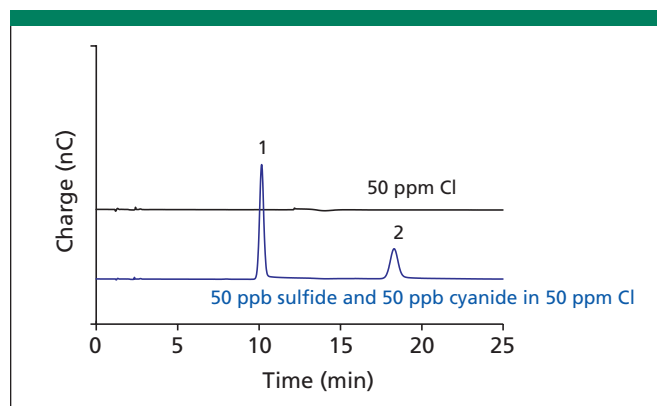


Figure 1: Separation of sulfide and cyanide in the presence of chloride using a disposable silver electrode. Eluent: 75 mM NaOH/100 mM NaOAc; flow rate: 0.25 mL/min; injection volume: 10 μ L; detection: integrated pulsed amperometric detection; disposable silver working electrode with Ag/AgCl, KCl_(sat) reference electrode; sample: 1 = sulfide (50 ppb); 2 = cyanide (50 ppb) in 50,000 ppb chloride.

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