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Author

Benner, W.H.

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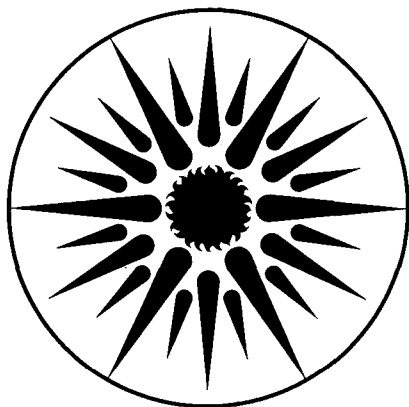
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April 1984

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For "Aids to Analytical Chemists," Analytical Chemistry

A RETROFITTED TEMPERATURE-STABILIZED CHAMBER FOR
ION CHROMATOGRAPHY CONDUCTIVITY DETECTORS

W. H. Benner

Applied Science Division
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

Ion chromatography (IC) is a powerful tool for quantitating low concentrations of ionic species (1); however, these determinations can be severely hindered by temperature fluctuations in the analysis system. Temperature fluctuations cause increasingly greater problems as the analyst tries to lower the limit of detection. In the practice of IC, an analyte is loaded onto the upstream side of a separator column and as eluent is pumped through the column, constituent ions become time resolved and elute in a reproducible order. If suppressor-type IC is performed, these time-resolved ions, which are traveling in a high-conductivity eluent, are pumped through a suppressor column that reduces the conductivity of the eluent and exchanges M^+ with H^+ in anion analysis or exchanges X^- with OH^- in cation analysis. The time-resolved species are detected conductimetrically as they elute. Temperature fluctuations in the separator column, the suppressor column, or in the detector create stability problems, particularly when $\leq 1 \mu\text{mho}$ is full-scale deflection on a chart recorder. During automatic sample injec-

tion, daytime-nighttime room temperature cycles can cause the baseline to drift offscale with the resulting loss of data. Others (2) have solved some of these problems electronically. We present a simple way to stabilize the temperature of the detector and columns of an ion chromatograph.

Experimental

An ion chromatograph (Dionex Corp., Model 12) was programmed to inject samples automatically. One milliliter of sample was loaded onto a concentrator column, and a 3-mM $\text{CO}_3^{=}$ /3-mM HCO_3^- eluent (prefiltered through a 1.2- μm filter, type RA, Millipore Corp.) eluted the sample ions through a precolumn, separator column, and a regenerable suppressor column. The concentrator, precolumn, and separator columns were type AS-1 (anion separation) and were supplied by Dionex Corp. Eluent flow rate was 2.7 ml/min. The output from the conductivity detector was traced with a strip chart recorder. Standard solutions of NO_3^- and $\text{SO}_4^{=}$ were prepared from their dessicated sodium salts (Mallinckrodt, Inc., reagent grade).

Figure 1 shows a stabilized temperature jacket that was constructed from acrylic plastic and aluminum sheeting. A 3/16"-deep channel (No. 1 in Fig. 1) was milled into the 1/2"-thick end plates, using a 1/4"-diameter mill cutter. Two Swagelok fittings (No. 2, 1/8" MPT x 1/4" tubing, S.S.) were connected to the ends of the channel so that automotive antifreeze could be circulated through the channel via a constant temperature water bath circulation pump. Antifreeze was chosen to reduce corrosion of the aluminum. A thin sheet of aluminum (No. 3, 4" x 4" x 1/64") was glued to the channel side of the end caps with Silicone

RTV sealer (General Electric Co.) after the surfaces were pretreated with silicone primer (SS4004, General Electric Co.). RTV by itself did not seal the aluminum properly to the acrylic, but leak-free operation has been possible for more than 2 years of operation with the temperature-stabilized chamber since the primer was used.

The conductivity detector block (Dionex Corp., No. 4) was attached to a second (No. 5) aluminum sheet (4" x 4" x 1/32") with a flat-head screw, using the tapped hole in the detector block that is normally used to mount the detector block in the IC. Several feet of Teflon tubing (1.5-mm o.d.) were wrapped around the detector block and held in place between two aluminum sheets (Nos. 5 and 6). The third aluminum sheet (No. 6) is 3" x 4" x 1/32" with a centered 2"-diameter hole. Heat sink compound (Dow Corning Co., type 340) was placed between 4 and 5 to provide good heat transfer to the Teflon tubing and ultimately to the eluent. As shown in Fig. 1, a second channel plate and four side panels (1/4" thick) were constructed into a detector box. A hole (No. 7) was provided in one of the side plates to accommodate wires and tubing (No. 8).

Antifreeze/water (1:1) from a constant temperature bath was circulated through the channels in the detector box and plates using a Yellow Springs Instrument Co. Model 72 proportional temperature controller to control the temperature of antifreeze in a dewar and an external circulating pump (several liters per minute). It was not necessary to insulate the tubes that carried the circulation solution.

Results and Discussion

Using the modified system, the plots in Fig. 2 were generated to show the need for temperature control and the detector stability afforded by this retrofitted device. The plots in Fig. 2 for NO_3^- and $\text{SO}_4^{=}$ peak heights were obtained by placing the separator and suppressor columns in a large dewar filled with water of the indicated temperature, and the detector temperature was adjusted to the same temperature. The upper curve in Fig. 2 shows the change in suppressed eluent conductivity as a function of temperature. The linear slope between 20°C and 25°C is $0.08 \mu\text{mho}/\text{C}^\circ$, which means that a 1°C temperature change, when operating at $1 \mu\text{mho}$ fullscale, causes an 8% fullscale deflection. It should be noted that this suppressed eluent conductivity change occurs in spite of electronic temperature compensation in the detector circuit. Daytime/nighttime laboratory temperature changes can easily cause the baseline to drift offscale during automatic operation. Autozeroing could solve this problem but will not solve the more serious problem indicated by the other curves in Fig. 2.

A combination of effects, i.e., temperature-induced changes in the partition coefficient of the ion exchange resin and conductivity of the sample ion, causes a change in sensitivity of the ions. The middle and lower curves in Fig. 2 show the change in peak height for NO_3^- and $\text{SO}_4^{=}$ as a function of column and detector temperature. For sulfate, the peak height changes $0.2 \mu\text{mho}/\text{C}^\circ$, or $0.02 \mu\text{mho}/\text{ppm}\cdot\text{C}^\circ$. For NO_3^- , the peak height changes $0.4 \mu\text{mho}/\text{C}^\circ$ or $0.04 \mu\text{mho}/\text{ppm}\cdot\text{C}^\circ$. This corresponds to a $1.1\%/C^\circ$ and a $2.2\%/C^\circ$ change in analytical sensitivity for $\text{SO}_4^{=}$ and NO_3^- , respectively. Depending on the constancy of laboratory air tem-

perature, these temperature-induced sensitivity changes can cause significant errors. In addition, short-term fluctuations in the temperature of a detector without temperature stabilization cause baseline fluctuations that sometimes occur on top of eluted peaks. Regardless of the type of temperature problem (detector temperature changes and/or column temperature changes), operation with the retrofitted temperature-stabilized detector chamber along with immersion of the columns (except the concentrator column) in a dewar filled with room-temperature water greatly extended the operating range to lower concentrations.

Figure 3 shows a typical chromatogram of sulfate in 18-megohm/cm water. The peak corresponds to 0.021 ppm (21 ng $\text{SO}_4^{=}$ absolute). The noise is 0.003 μmho . Finally, the rising baseline in Fig. 3 was attributed to the slow degeneration of the packed suppressor column and indicates that with time the suppressor is losing efficiency.

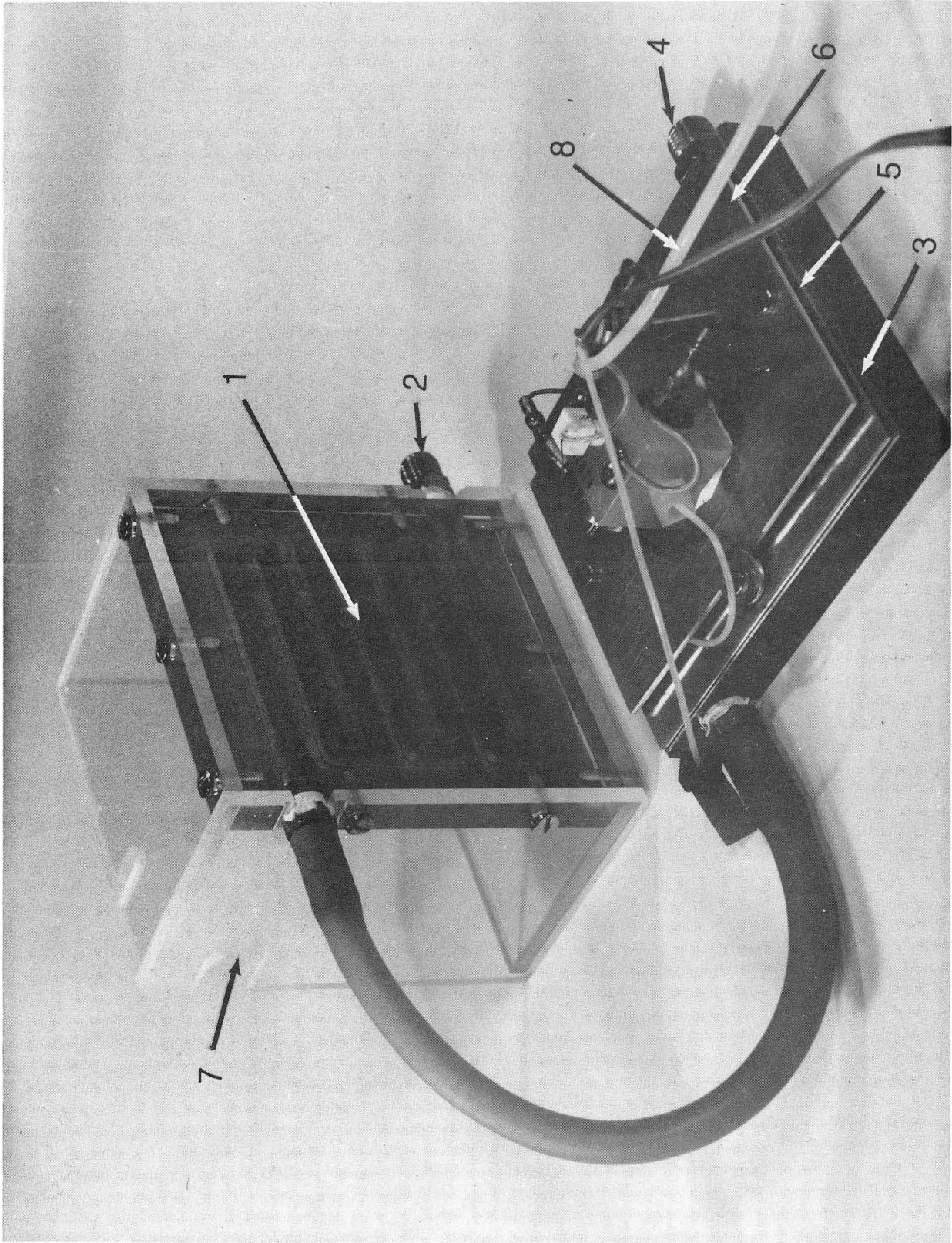
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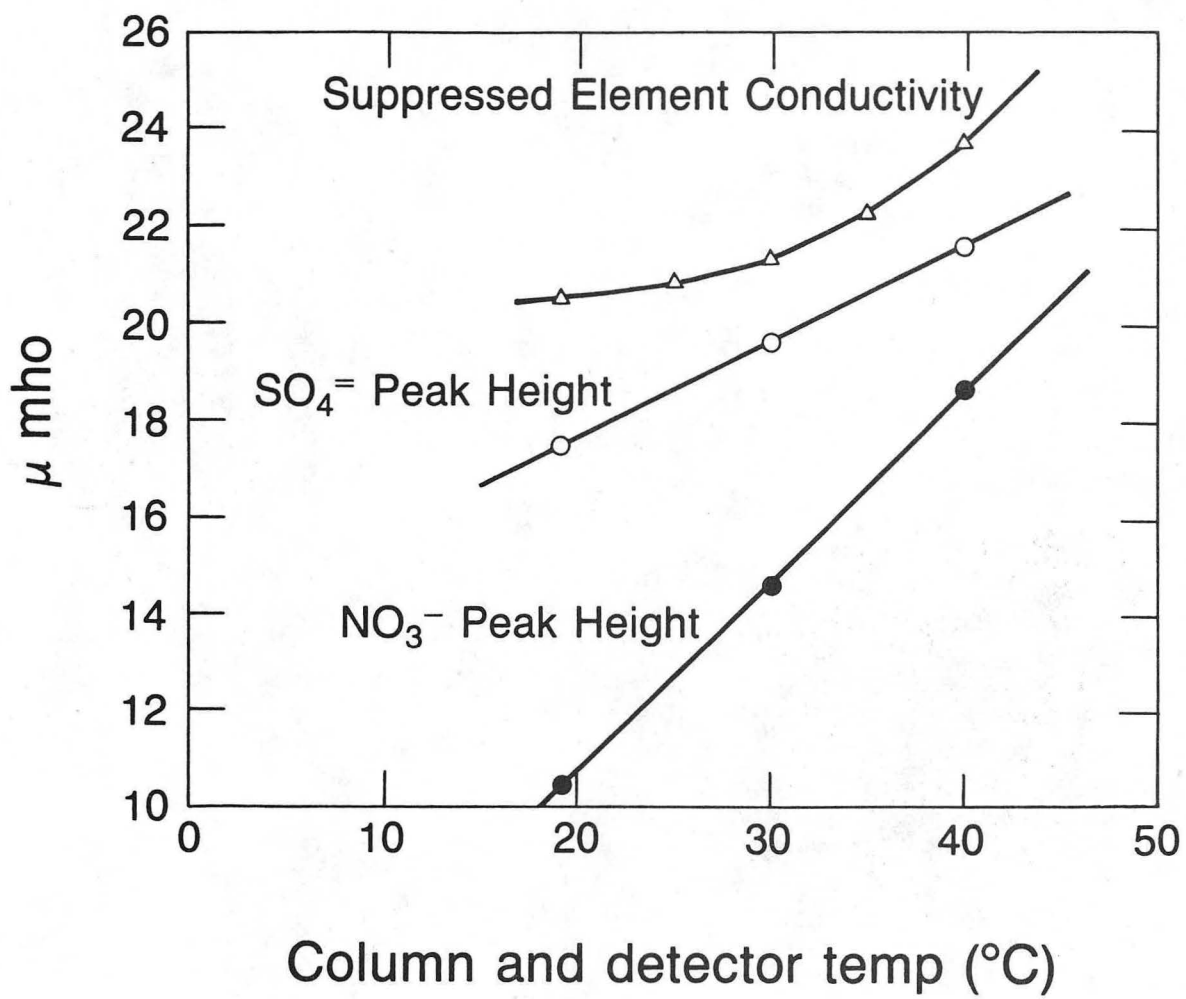
Figure Captions

- Figure 1. Photograph of temperature-stabilized IC detector chamber. See text for explanation of numbers.
- Figure 2. A plot of detector signal vs. operating temperature. (XBL 843-10181)
- Figure 3. Actual example of a typical chromatogram after detector temperature stabilization. (XBL 843-10182)



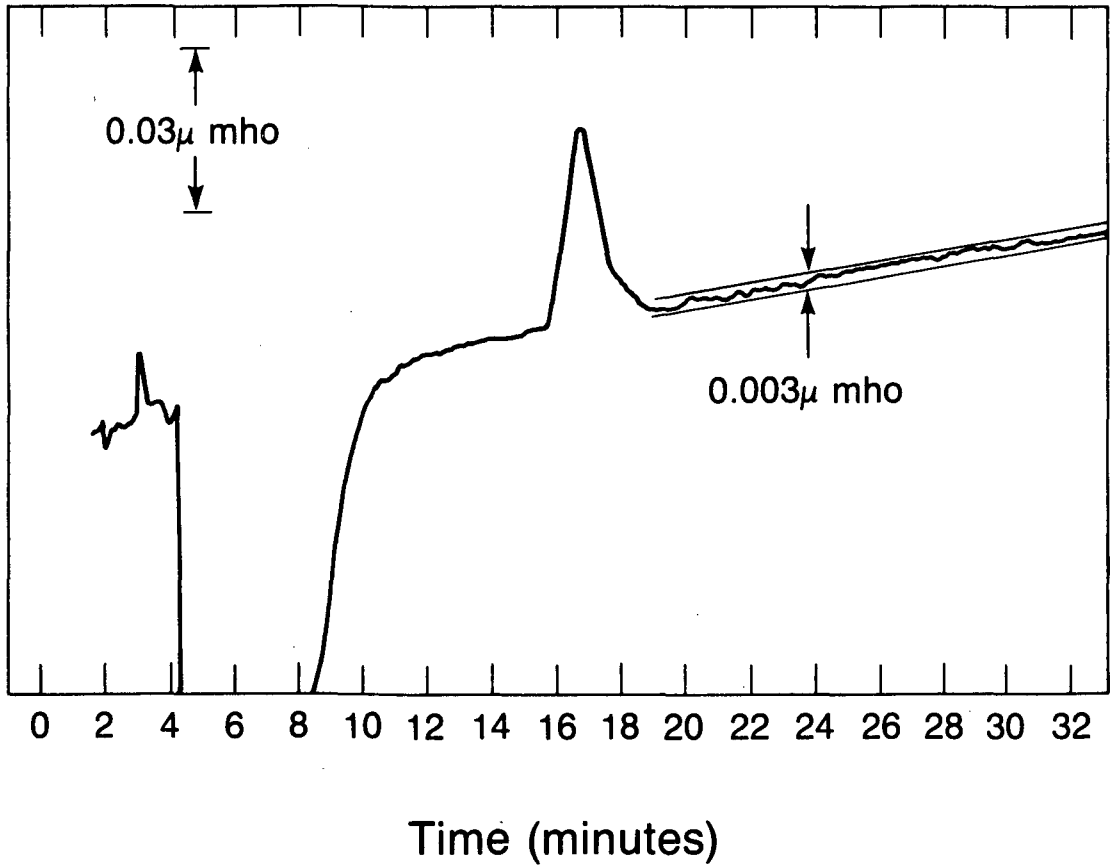
XBB 843-1865A

Figure 1



XBL 843-10181

Figure 2



XBL 843-10182

Figure 3

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