Waters Lab Highlights
An Internal Communication of Applications and Techniques
LAH 0423 3/90
AN/EF/PM/IC/AX

Application of WISE Software: Optimization of Phthalate Concentration for Anion Separations

Previous Lab Highlights[1-5] have demonstrated that a wide range of eluent variables can be optimized using the WISE™ strategy. For the separation of inorganic anions, WISE software turns out to offer an extremely efficient approach for the optimization of eluent ion concentration. For ion exchange separations, Haddad and Cowie[6,7] showed that log k' for inorganic ions is directly proportional to the log of concentration of the competing eluent ion(s). This applies to eluents where a single eluent ion (e.g. hydroxide) controls retention and to the case where multiple eluent ions (e.g. phthalate/biphthalate) control retention.

The significance of the linear log-log relationship between retention and eluent composition is this: only two experiments will be required to determine the optimal eluent ion concentration. Note that the two experiments needed are for the lowest and highest concentrations to be considered. Accurate retention values for each mixture component can then be calculated at any intermediate value of the eluent concentration.

Haddad and Cowie used a phthalate eluent at pH 5.3 as an example. At this pH, phthalic acid (H_2P) is fully ionized and exists as a nearly even mixture of P^{2-} and HP^-. Total phthalate concentration was allowed to range between 2 and 10 mM and pH was held constant at 5.3. To test the linear log-log relationship, five eluent concentrations were tested. The resulting plot (Figure 1) confirms the expected linearity and indicates the feasibility of accurate interpolation based on lines joining just the retention values corresponding to the extremes of phthalate concentration (i.e. 2 and 10 mM).

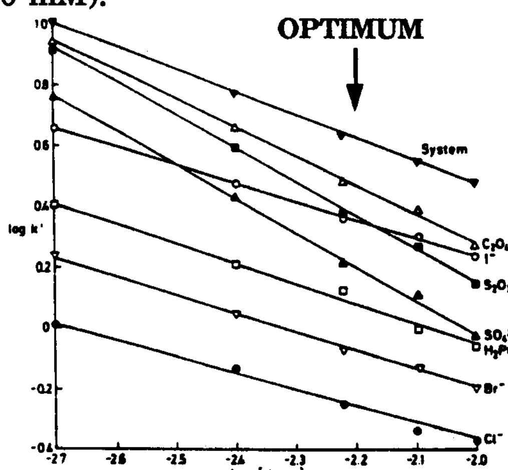

Figure 1: Linear relationship between log k' and log phthalate concentration (M). Column: low capacity silica-based anion exchanger. Eluent: 0.002 to 0.010 M aqueous phthalate at pH 5.3.

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From Figure 1, Haddad and Cowie determined an optimal concentration of 4.7mM phthalate (See Figure 2). To confirm that WISE software would determine the same optimum on the basis of just two experiments, Haddad and Cowie's retention data for the 2 and 10 mM phthalate eluents were entered into the single variable optimization module of WISE software. Log [phthalate] was the eluent variable. As expected, the optimum of 4.7 mM was predicted immediately (see arrow on Figure 1). Thus, a total of just three experiments (two initial experiments plus confirmation of the optimum) were needed for the optimization of the eluent for this ion exchange separation of anions. The efficiency of WISE software for optimizing eluent ion concentration for ion exchange separations should extend to other similar separations.

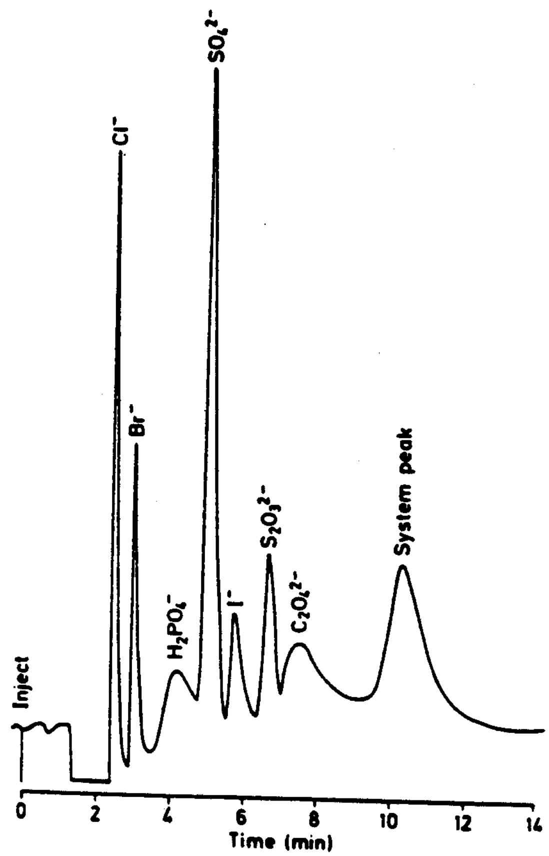

Figure 2: Chromatogram obtained at the optimal phthalate composition of 4.7 mM.

References:

1. LAH 0360 2/88.
2. LAH 0369 6/88.
3. LAH 0389 2/89.
4. LAH 0393 4/89.
5. LAH 0413 6/89.
6. P.R. Haddad and C.E. Cowie, *J. Chromatogr.*, **303** (1984) 321.
7. P.R. Haddad and A.D. Sosimenko, *J. Chromatogr. Sci.*, **27** (1989) 456.

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