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Letter to the Editor

Sensitive monitoring of phenols after liquid chromatography

Sir,

We are pleased to find others adapting to their problems the cerate oxidative–fluorescence detector for liquid chromatographic monitoring that we described earlier[1,2]. However, "A highly sensitive technique for the liquid chromatographic analysis of phenols and other environmental pollutants"[3] describes some operating conditions which, in our judgment, are less desirable, and could produce unsatisfactory and discouraging results for other investigators. The following comments are based on over four years of study of the applications of the cerate oxidative monitor and on the experience of routine operation of six separate chromatographs using that monitor.

(1) Boiling of the reagent solution is unnecessary and results in significant levels of undesirable cerium(III). Stabilization of the reagent as cerium(IV) with sodium bismuthate proceeds rapidly at 40–50° with an insignificant yield of cerium(III).

(2) As discussed in our later paper[4], 10–30 mg sodium bismuthate per liter are adequate where the reservoir had been previously stabilized to the presence of cerium (IV). Quantities of sodium bismuthate in excess of 75 mg per liter reduce the detector sensitivity.

(3) We find no troublesome particulate formation when the sulfuric acid concentration in the prepared reagent is 1.5 M even when mixed with column eluent of 6 M of ammonium acetate. Including the sulfuric acid with the reagent solution eliminates a number of homogeneity, flow, pumping, and stability problems. No time limit to reagent stability has been noted.

(4) We found the peristaltic pumping of reagent solution to be less satisfactory than the gas pressuring system described earlier[1,2]. In our tests of peristaltic pumping, all flexible tubing that was used reacted to a measurable extent with the reagent to produce the undesired cerium(III).

(5) Systems are available for separating phenols that are more compatible with the cerate oxidative detector, including carrying out the oxidation reaction at 100°. One such is the acetate-buffered anion-exchange chromatographic system, as described earlier[1,2], that provides good separation of phenolic and other organic compounds. Because of the temperature dependence of the rate of many of the cerate

oxidation reactions, we have found a large improvement in sensitivity at the higher reaction temperature, particularly for the more difficultly oxidizable compounds.

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