

preliminary notes and applications from Bioanalytical Systems, Inc.

Inhibitors in Monomer Formulations

p-Methoxyphenol (PMP) is widely used as an inhibitor for various monomer formulations and as a stabilizer for chlorinated hydrocarbons. One specific application involved identifying and quantitating PMP in hydroxyethyl methacrylate (HEMA), a polymer used in making contact lenses. Normal concentrations of PMP in HEMA are approximately 30 ppm. The main reason for turning to LCEC in this case was improved selectivity. HEMA is not electroactive, therefore PMP may be detected by direct injection with little need for pre-LC cleanup.

F2 and F3 illustrate qualitative information which may be obtained by dual electrodes placed in parallel. By comparing the retention times to those of PMP standards, preliminary identification of PMP in HEMA was made. Due to the very low detection limits of LCEC, solutions of HEMA (containing PMP) had to be diluted with mobile phase before injections could be made. Positive identification was made by comparison of peak current ratios (i1/i2). Quantitative information was found by comparing the peak response of PMP in HEMA with that of PMP standard. The PMP concentration in HEMA was found to be 41.4 ppm. Sample analysis time was 2-4 minutes depending upon flow rate.

Conditions

Liquid Chromatograph: LC-154 (Bioanalytical Systems Inc.) with dual LC-4B detection.

Mobile Phase: 60% 0.1 M sodium citrate, 0.2 M sodium perchlorate, 250 mg/L EDTA (pH 6.2)/40% CH₃CN.

Stationary Phase: Biophase ODS 3μ column (100 x 4.6 mm)

Detector: Dual LC-4B controllers with dual glassy carbon electrodes operated in parallel.

Applied Potential: $W_1 = +850 \text{ mV}$, $W_2 = +700 \text{ mV}$,

vs Ag/AgCl.

Flow Rate: 0.5 mL/min. or 1.0 mL/min.

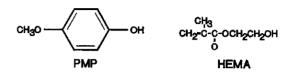


Figure 1. Structures of p-methoxyphenol (PMP), hydroxyethyl methacrylate (HEMA)

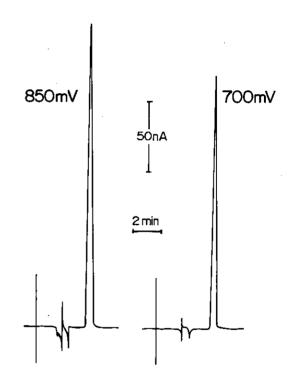


Figure 2. 1:1000 dilution of HEMA monomer (containing PMP) at +700, +850 mV using dual LC-4B controllers and parallel glassy carbon electrodes. Peak height ratio = 0.89. Flow rate = 0.5 mL/min.

Amount Injected: 20 μL Temperature: Ambient



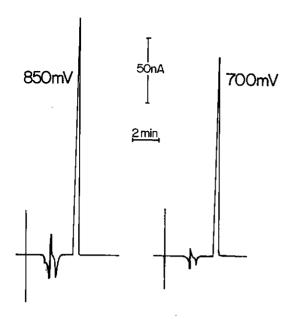


Figure 3. 1 ppm PMP standard, peak height ratio = 0.86, same conditions as F2.

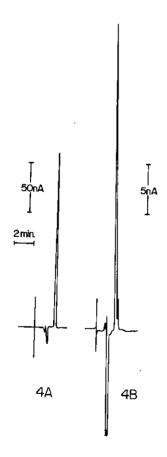


Figure 4. (A) 1 μ g/mL PMP standard, +850 mV, 1.0 mL/min. (B) 1:250 dilution of HEMA monomer. PMP concentration found: 41 ppm.

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