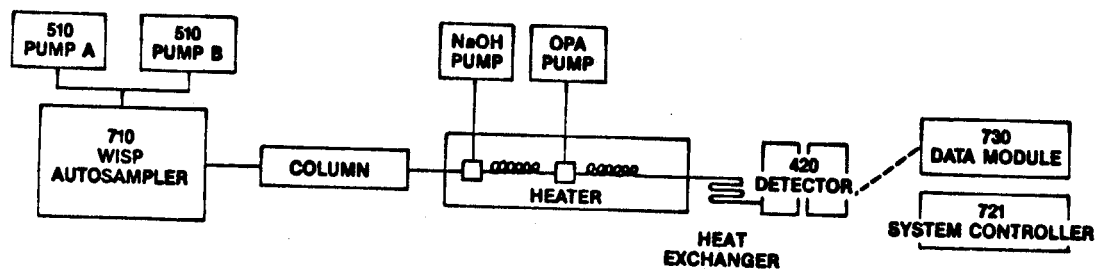


POST-COLUMN DERIVATIZATION OF CARBAMATES II:SYSTEM HARDWARE AND CONDITIONS

The post-column derivatization of carbamates described previously (LAH 0119) can be performed by the addition of existing Waters hardware to any LC system (Figure 1). The Post Column Reaction System (PCRS, WA #07086) contains two low pressure pumps which can be used to deliver the NaOH and OPA solutions to the appropriate reaction coil. A column heater (WA #07011) is used to heat both the hydrolysis and OPA coils to 90°C. A heat exchanger is placed between the OPA coil and the detector. The exchanger drops the effluent temperature from 90°C to near room temperature prior to entry into the detector. This resulted in an improved detector response (peak height) by 5 (carbaryl) to 19% (temik). This heat exchanger is the same one used in the Sugar Analyzer I (WA #07003). It is plumbed so that the column effluent exchanges with the OPA coil effluent and the detector inlet flow is heat exchanged with the detector outlet flow.

FIGURE 1: SYSTEM CONFIGURATION

The hydrolysis and derivatization of carbamates is temperature dependent. The peak heights of various carbamates were measured at several temperatures and 90°C was chosen as the optimum. Methomyl and temik were particularly sensitive to reaction temperature. The reaction times for this system are 31 seconds for the base hydrolysis and 10 seconds for the OPA derivatization. The wavelengths chosen for the M420 AC were optimized for maximum peak height with minimal baseline noise. The emission filter can be either 400 or 425 nm, however, using the 400 nm filter increases peak heights by 15 (carbofuran) to 23% (methomyl). The baseline noise measured at 128X (maximum span) was 2.8% FSD (full scale deflection) or 0.28 mV. The difference in baseline noise between the 400 and 425 nm filter is negligible.

POST-COLUMN REACTION SYSTEM CONDITIONS

● NaOH Hydrolysis:

Reaction Coil: 12 ft. X .020 in. ID Stainless Steel Coil
Reagent: 0.05 N NaOH
Flow Rate: 0.4 ml/min
Temperature: 90°C

● OPA Derivatization:

Reaction Coil: 20 ft. x .009 in. ID Stainless Steel Coil
Reagent: 500 mg o-phthalaldehyde + 1 ml 2-mercaptoethanol per
liter of 0.05 M potassium borate (123.6 gm boric acid +
105.0 gm KOH per liter), pH 10.4
Flow Rate: 0.4 ml/min
Temperature: 90°C

● System Bandspread: 350 μ l.