

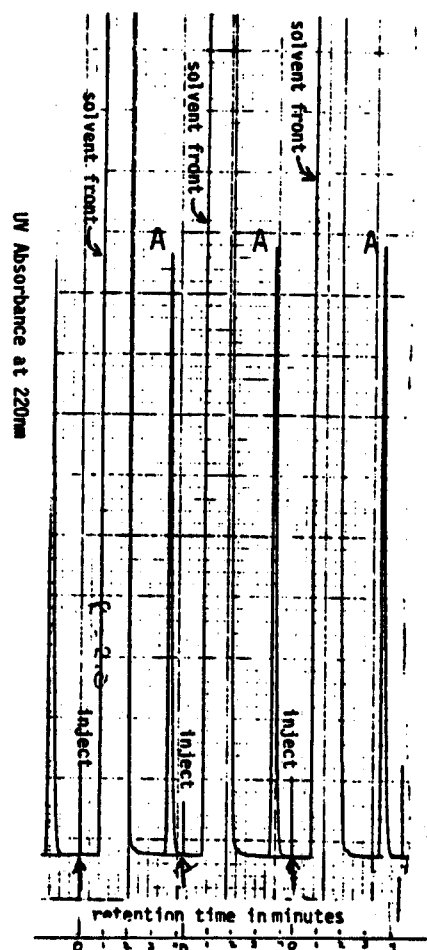
## USING CHEMICAL REACTIONS TO DETECT ALIPHATIC EPOXIDES

Aliphatic epoxides are very difficult to detect even with lower wavelength detectors (<214 nm). Very few LC applications have been described for such underivatized compounds. Using silver picrate as a solution reagent it was possible for researchers at Dr. Krull's lab to do off-line derivatizations of epoxides such as cyclohexene oxide. This derivatization was performed by mixing an acetonitrile solution of cyclohexene oxide with silver picrate, and then heating this resultant solution at 70°C for about 2 hours. The final reaction mixture was then diluted to a total volume of 3 ml with the LC mobile phase and 250  $\mu$ l of this solution was injected onto a Waters  $\mu$ BONDAPAK™ CN packing with an in-line Guard-Pak™ CN Precolumn Insert.

Figure 1 shows 3 replicate injections of the post-reaction mixture. The major peak eluting in the solvent front is excess silver picrate and the second peak (A) is the desired product (1-hydroxy-2-picryl cyclohexane) of the ring-opening reaction of this reagent with cyclohexene oxide. These reactions have now been shown to lead to 50-65% conversion of the starting epoxide to the desired single derivative under a variety of reaction conditions. The reaction demonstrated here (as a typical example) involves a starting compound which has no UV absorbance while the product, which now contains a picryl moiety within its structure, has a good UV absorbance.

FIGURE 1

Waters  $\mu$ BONDAPAK™ CN 10  $\mu$ m column (3.9 X 30 cm)  
with Guard-PAK™ cartridge  
Mobile Phase: H<sub>2</sub>O/acetonitrile (55/45 v/v)  
Flow Rate: 2 ml/min, 1500 psi back pressure  
Waters Lambda Max Model 480 Spectrometer  
at 220 nm, 10 mV full scale, 250  $\mu$ l injections.



Literature references on back.

1. I. S. Krull, K.-H. Xie, S. Colgan, U. Neue, T. Izod, R. King and B. Bidlingmeyer, J. Liquid Chrom., 6(4), 605 (1983).
2. K.-H. Xie, C. T. Santasania, I. S. Krull, U. Neue, B. Bidlingmeyer, A. Newhart, J. Liquid Chrom., 6(11), 2109 (1983).
3. K.-H. Xie, S. Colgan and I. S. Krull, J. Liquid Chrom., 6(S-2), 125 (1983)
4. I. S. Krull, S. Colgan, K.-H. Xie, U. Neue, R. King and B. Bidlingmeyer, J. Liquid Chrom., 6(6), 1015 (1983).
5. I. S. Krull, X.-D. Ding, C. Selavka, K. Bratin and G. Forcier, J. Forensic Sci., 29(2), 449 (1984).
6. X.-D. Ding and I. S. Krull, J. Agric. & Food Chem., 32, 622 (1984).