box 9

DETERMINATION OF RESIDUAL VINYL CHLORIDE MONOMER IN VINYL RESINS

1 PURPOSE

A procedure for determining the amount of residual vinyl chloride present in vinyl resins is described. The resin is dissolved in a solvent and the vinyl chloride analyzed using a gas chromatographic technique. The method is applicable for use with poly (vinyl chloride), vinyl chloride-vinyl acetate copolymers and other vinyl resins in which there are no volatiles which interf re in the determination.

2 EQUIPMENT AND REAGENTS

Gas Chromatograph, Hewlett-Packard (F and M) Model 5750 or equivalent, equipped with a hydrogen flame ionization detector.

Tetrahydrofuran, reagent grade.

Balance with accuracy to 0.10 gram.

3 SAMPLE PREPARATION

Prepare the sample for chromatograph injection by dissolving th test resin in tetrahydrofuran (THF). Weigh 9 grams of THF into a suitable vial. Add 1 gram of the resin to be tested to the THF. Resin addition should be made as quickly as possible. Cap th bottle and place it on a can-roller to attain complete solution. When complete solution has occurred, the sample is ready for injection into the chromatograph.

4 INSTRUMENT PARAMETERS

Instrument	Hewlett-Packard(F/M) Model 5750 (or equivalent) equipped with hydrogen flame ionization detector.
Column	6-ft by 1/8-in. stainless steel tubing packed with Chromosorb 102, 60/80 me h.
Temperatures:	
Column	100°C for 4 minutes, manually increased to 250°C
Injector	100°C (maximum)
Detector	300°C (flame ionization)
Sample Size	4 microliters
Flows:	
Helium Carrier Gas	25 cc/minute
Hydrogen	20 psi at cylinder head
Compressed Air	40 psi at cylinder head

Elution Times

Vinyl Chloride	2.8 minutes
Tetrahydrofuran	6.3 minutes

5 CALIBRATION

Prepare solution mixtures of varying amounts of vinyl chlorid in tetrahydrofuran to cover the expected range of concentration. Obtain from the chromatograph scan the area per cent of vinyl chloride for each sample of known monomer content. Prepare a chart plotting the area per cent vinyl chloride versus the known weight per cent vinyl chloride to establish the relative det ctor response of the vinyl chloride with respect to tetrahydrofuran.

6 CALCULATION

Determine the area per cent vinyl chloride from the chromatograph scan for the resin solution to be characterized. Multiply this figure by 10, which converts the data to area per cent vinyl chloride based on resin weight. Using the chart developed with the calibration samples, read off the corresponding weight p r cent of vinyl chloride.

TYPICAL CALIBRATION CURVE

Residual Vinyl Chloride Monomer in Vinyl Resin

Area Percent Conversion to Weight Percent

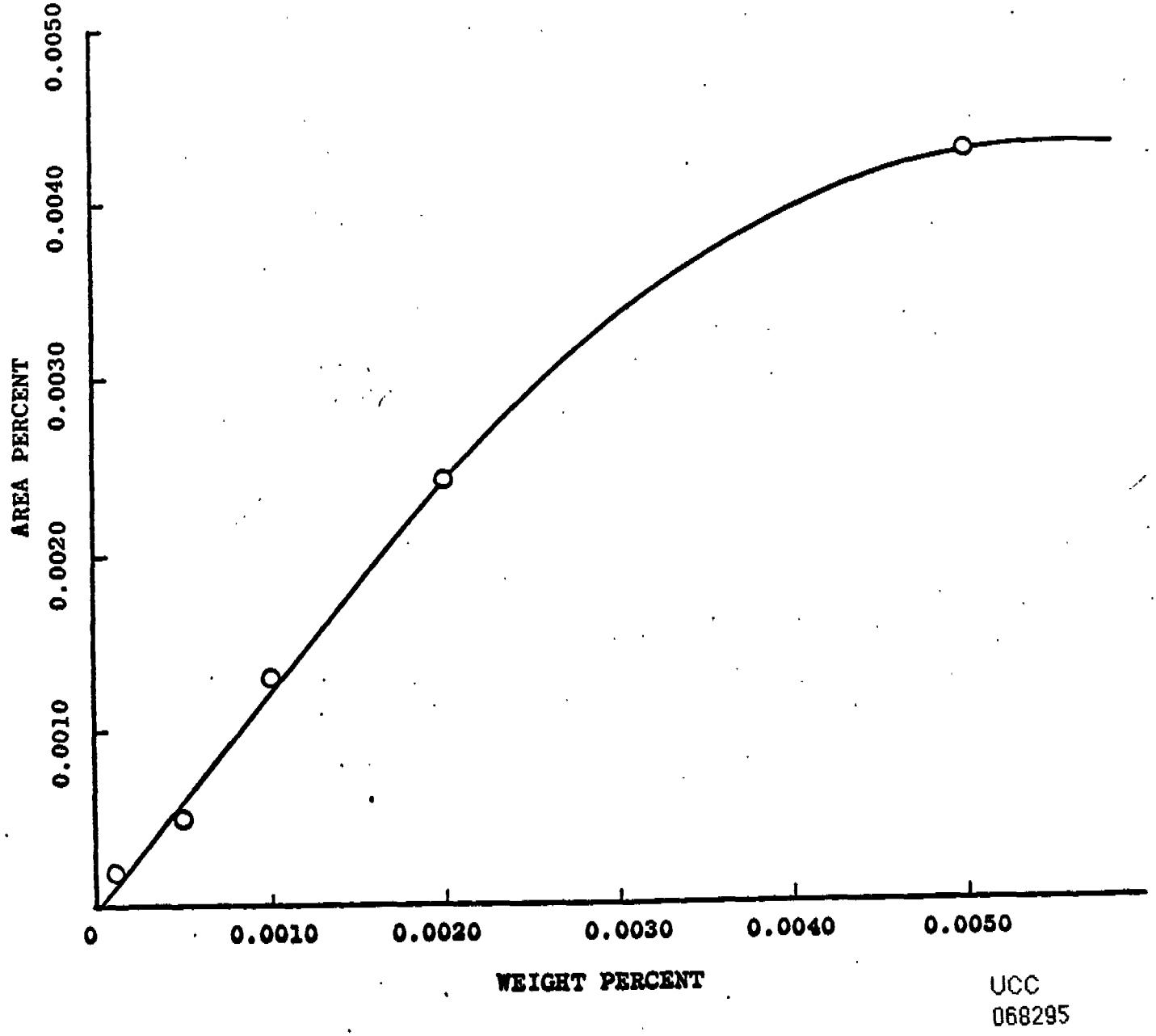

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FIGURE 1

SYNTHETIC SAMPLE OF 1 PPM VINYL CHLORIDE IN THE SOLVENT
(Sample Volume = 2 Microliters)

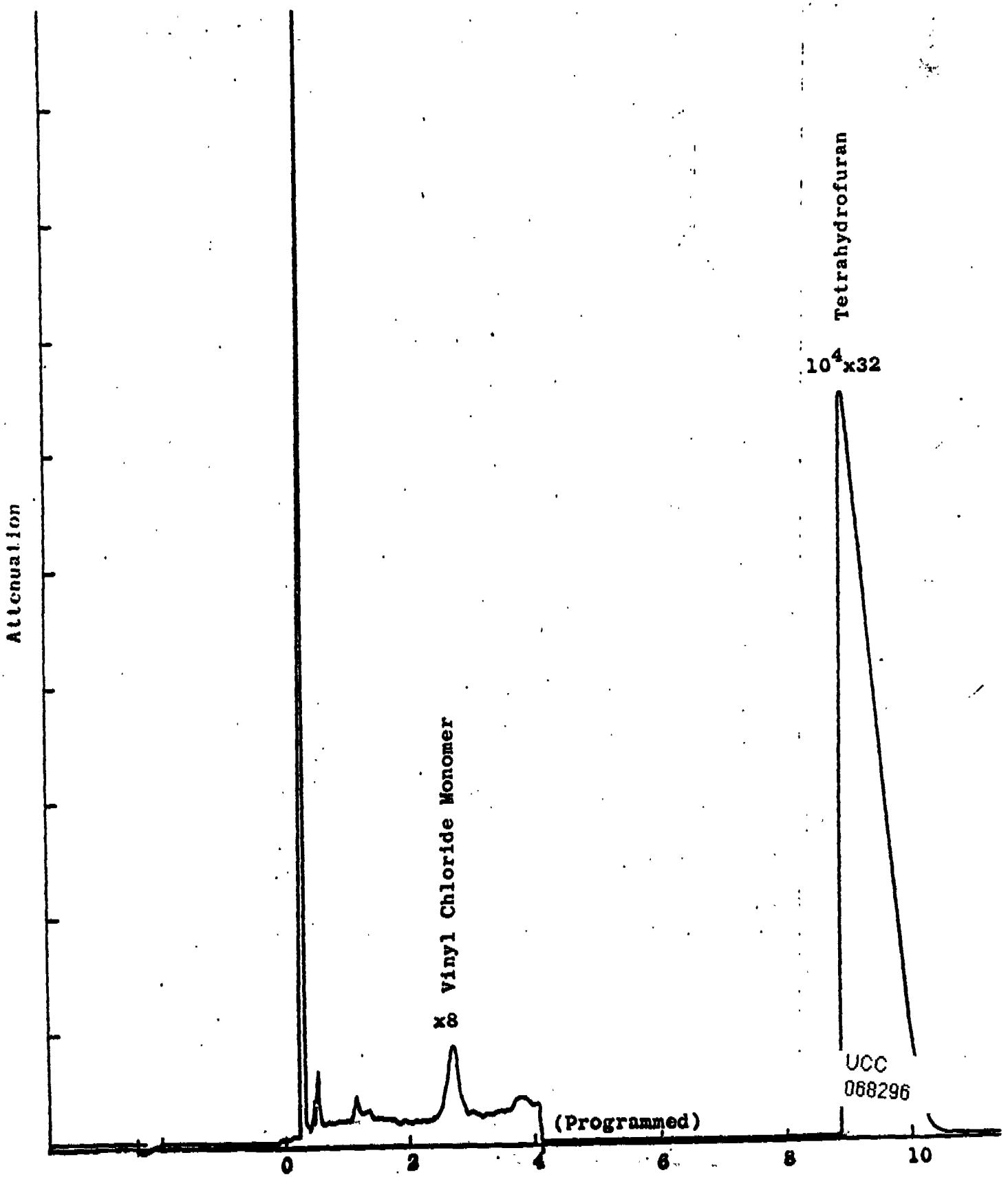


FIGURE 2

SYNTHETIC SAMPLE OF 10 PPM VINYL CHLORIDE IN THE SOLVENT

(Sample Volume = 2 Microliters)

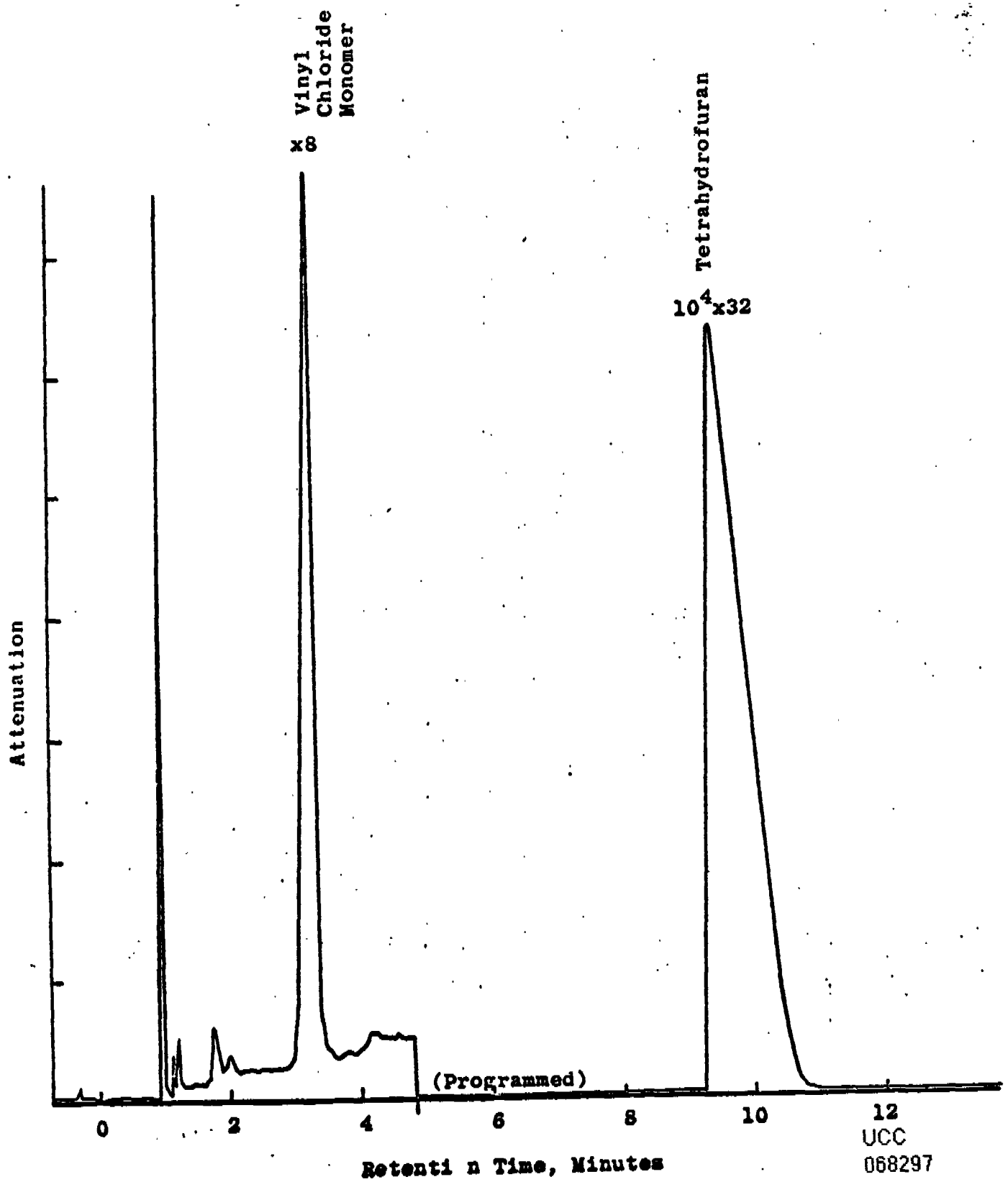
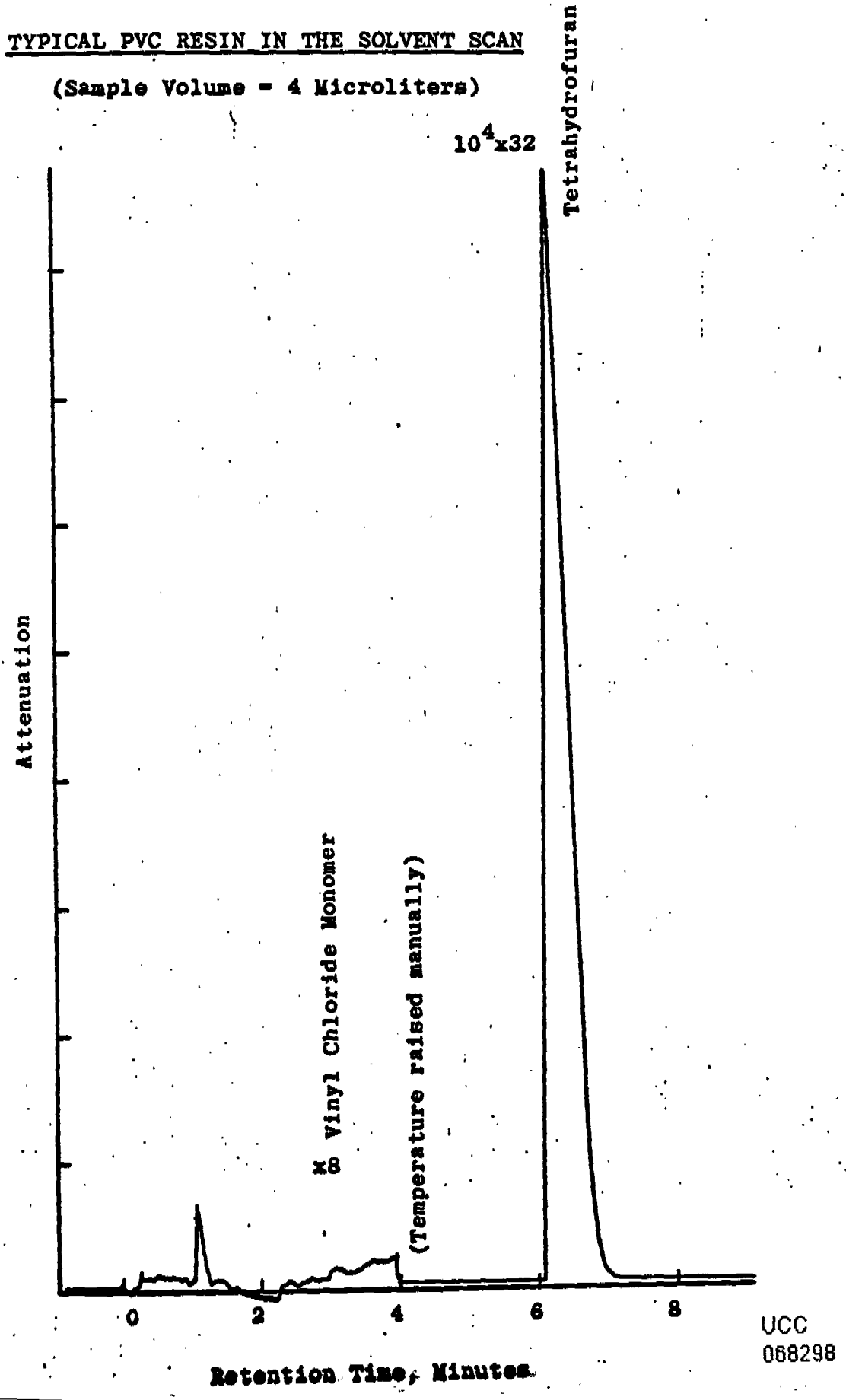


FIGURE 3

TYPICAL PVC RESIN IN THE SOLVENT SCAN

(Sample Volume = 4 Microliters)



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