


AN ABSTRACT OF THE THESIS OF

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in Water

Abstract Approved: 

Groundwater pollution with petroleum hydrocarbons is a widespread problem. Two of the most toxic and soluble are benzene and toluene. Their volatility also makes accurate analysis difficult by the commonly used gas chromatography method. In this study, reverse phase high performance liquid chromatography was evaluated for determination of their concentrations in mixtures simulating contaminated groundwater. The contaminants, in concentrations of 2-5 mg/L, were first extracted from the water by siphoning it through 15 cm³ of Altech XAD-4 resin contained in a buret. The resin was found to remove 99% of the contaminants. Various rinse agents, combinations of rinses, and techniques were tested and compared to determine the most efficient way to recover the benzene and toluene from the resin. The highest recoveries, 50%, and most reproducible results were obtained by using 90 mL of HPLC grade methanol in a series of 10 mL elutions through the resin. A rinse with methylene chloride followed by 6-10 rinses with methanol was found to be a satisfactory method of cleaning the resin.

EXTRACTION AND HPLC ANALYSIS
OF BENZENE AND TOLUENE IN WATER

A Thesis

Presented to
the Division of Physical Sciences
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INTRODUCTION

Water quality is of increasing concern. Pollutants which are particularly dangerous to health are the volatile organic compounds which can be toxic and are classified as carcinogens [1]. The most common source of these hydrocarbons is petroleum, which is a complex mixture of hundreds of chemicals [1]. Of particular concern are benzene, toluene, and the xylenes (BTX) because of their high toxicity and their high solubility which make them more likely to reach groundwater [2]. In fact, BTX and ethylbenzene (BTEX) are found in the highest concentrations of all aromatic components when water becomes contaminated with gasoline (3).

The specific composition of gasoline, including the concentrations of the BTX compounds, may vary widely depending on the source of the petroleum and the production method. In addition, gasoline may contain any of several additives: dyes, antiknock agents, lead scavengers, corrosion inhibitors, metal deactivators, and/or octane enhancers [3]. Benzene, toluene, and xylene are the most likely to be in all gasolines regardless of the maker [2,4]; therefore they more likely to be good indicators of petroleum pollution. Also, lead-free gasoline has a higher fraction of aromatic hydrocarbons and is almost universal in its use [3].

The most common source of BTX in groundwater is from leaking underground storage tanks [5]; other sources include spills of gasoline, fuel oil, kerosene, or diesel fuel [1], poor disposal practices of cleaning solvents [6], and manufacturing [7].

Concentrations of about one part per million can cause water to have objectionable taste and odor; however, many locations have set maximum allowable levels as low as one part per billion [1].

In Kansas, both the action level and the clean-up level are 5 ppb for benzene and 1000 ppb for toluene [8]. Two sites in Emporia, service stations with monitoring wells, have been reporting to Kansas Department of Health and Environment since 1991. Levels of from 1,000 to 10,000 micrograms per liter have been found [9]; the underground storage tanks at one site had no visible holes but had BTX concentrations up to 55.3 ppm in the water nearby [10]. Figure 1 is taken from a report by a waste consultant firm to the Kansas Department of Health and Environment. The map illustrates combined concentration levels of 1-10 mg/L of benzene, toluene, ethylbenzene, and xylene (BTEX) in the groundwater near a service station in Emporia [10].

All BTX/groundwater studies found in the search of recent literature done for this research employed gas chromatography as the analytical method. High performance liquid chromatography (HPLC) offers improved precision because of the fixed sample size provided for by the injection loop [11]. In addition less sample preparation is required so analysis can proceed more rapidly and reduce the possible sample degradation. On-site HPLC has been used successfully for other organic compounds and reveals that information is lost to alteration over time. In these studies the only sample preparation necessary was centrifugation or filtration to remove particulates [12].

A considerable amount of current research is being done on these compounds in soil and water to determine how they are degraded by microbes, aquifer transfer patterns, and methods of clean-up. This research focuses on use of a resin to extract and concentrate the analytes from water, methods for their removal from the resin, and improving the technique for quantitative analysis by using HPLC.

EXPERIMENTAL

The first part of the process was the extraction procedure. One gallon, 3.8 L, of distilled, deionized or tap water was used for each extraction process. A known mass of gasoline, or volume of benzene and toluene from standard solutions prepared for this research, was added to the water. "Blanks" containing only tap water or distilled, deionized water with no added chemicals were often run for comparison. Figure 2 illustrates the extraction apparatus. The water was siphoned through a "U"-shaped, 7 mm glass tube and into a 50 mL buret containing 15 cm³ of XAD-4 Altech resin saturated with water. A cotton plug separated the resin from the bottom buret stopcock. Another stopcock was inverted in the top of the buret to hold the end of the glass tubing which had been flared slightly to provide a better fit. Contamination problems in early trials led to the addition of a charcoal filter to the extraction apparatus and later to using N₂ to replace the volume lost in the jug as it emptied. The water passed through the resin at a rate of about 22 mL per minute; the last few milliliters were poured directly from the jug into the buret.

In earlier trials the gasoline was added to a large volumetric flask partly filled with water, mixed, and decanted to the gallon jug. Later the analytes were added directly to the gallon jug and the contents mixed by repeated inversions of the capped jug.

The analytes were removed from the resin with the rinse apparatus, Figure 3. In most cases pressure tubing was attached to the top of the buret which now had a buret tip in the inverted stopcock. Suction was applied to draw rinse solutions into the resin from below in order to provide the best possible contact with the resin and to eliminate problems of air pockets in the resin. The rinse solutions were contained in 50 mL graduated cylinders fitted with one-hole stoppers and glass tubing. Rinse agents included methanol (MeOH), hexane, acetonitrile (ACN), dichloromethane (CH_2Cl_2), and some combinations of these. All rinse agents which were to be analyzed were HPLC grade. In most cases the amount of the solvent added was recorded. Exceptions in the last set of analyses are noted; in these the volume of solvent eluted from the resin is used.

The instrument used for the analyses was a Varian HPLC system equipped with a model 2010 pump, a model 2050 variable wavelength UV detector, and a Rheodyne injector. The column was a Alltech Echonosphere C-18, 5 micrometer particle diameter, reverse phase with dimensions of 250 mm by 4.6 inside diameter. A stripchart recorder was used for output. The wavelength was set at 208 nm, the range was adjusted as needed, the flow rate was 1.0 mL per minute, and the chart speed was set at 1.0 cm per min.

The mobile phase was a solution of HPLC grade methanol and distilled, deionized water. The two solvents were measured by graduated cylinder and filtered through a nylon-66 filter. The volume compositions ranged from 80/20 to 70/30 methanol/water. The heights and retention times produced on the elution chromatograms

were compared with those of prepared standards for quantitative analysis. The standard solutions used were of a concentration close to that predicted for the results of the analyses.

A McPherson/GCA EU-707-D spectrometer was also used to help determine the efficacy of the extraction of benzene and toluene by the resin. The instrument was set at 208 nanometers. Matched quartz cuvettes were used to contain the reference solution, the rinse solution without the analytes; and the sample solution, the rinse solution with benzene or toluene.

RESULTS

Calibration curves for benzene and toluene are shown in Figures 4 and 5 for a range of concentrations from less than 2.0 mg/L to about 25 mg/L. This range included all analyzed solutions; benzene response was linear between 2.5 and 24 mg/L. Toluene linearity occurred between 2.5 and 21 mg/L.

Retention times varied with mobile phase and column condition. Typical values for 70/30 methanol/water were water, 1.8 minutes; methanol, 3.0 minutes; dichloromethane, 3.5 minutes; benzene, 4.7 minutes; and toluene, 5.9 minutes. Acetonitrile and hexane absorption peaks did not appear at 208 nanometers.

Checks of the reproducibility of peak heights for the duration of one extraction run were made. Examples of these heights are shown in Table I. Two injections were made of the benzene standard and two of the toluene standard. The differences of the two heights is given as the range. The average range for benzene is 0.24 cm and for toluene, 0.1 cm.

Several determinations were made of the amounts of benzene and toluene present in gasoline. One old sample of gasoline tested 1.34 and 1.44 % benzene and 7.87 and 7.72 % toluene. A different, fresh, sample tested a year later showed 8.3% benzene and 14% toluene; ten days later the results were 6.4 and 10.2%, respectively. The literature gave ranges of 2-5% for benzene and 6-7% toluene [7].

Tests of the resin were made to determine if it was effective for removing benzene and toluene from water. Spectrometer analysis data is given in Table II. Other checks were made by comparing elution chromatograms from an HPLC analysis of tap water, gasoline-spiked tap water, and the spiked water after it passed through the resin. An example of these chromatograms is shown in Figure 6. Peak heights for benzene and toluene in spiked water were 5.29 and 11.05 cm; 0.25 and 0.70 cm in the water before spiking; and four tests of the eluent at different points in the extraction run averaged 0.26 and 0.76 cm.

An attempt was made to calculate the breakthrough point of the resin using only 2.5 cm³ of the cleaned resin and concentrations of benzene and toluene in the same range as other extraction procedures. A normal extraction process was started. Samples were drawn as the mixture was eluted from the buret, injected into the HPLC, and compared with the standards. The apparent concentrations of benzene and toluene in the eluent were 0.41 and 0.32 mg/L for the first sample and nearly the same concentrations were seen in samples drawn after 300 and then 500 mL were run through the buret.

Different solvents were used to remove the adsorbed benzene and toluene from the resin as explained in the procedure section. The percent recovery of single rinses using different amounts of methanol is given in Table III.

Consecutive rinses of the resin with methanol were tested. Three trials were made using three successive 25 mL rinses which were combined and analyzed with the HPLC. The results are given in Table IV. The values ranged from 13 to 40% for both benzene and

toluene. In another test, four trials were made where three 30 mL portions of methanol were rinsed through the resin and analyzed separately. The results are presented in Table V and Figures 7 and 8. The percent recoveries ranged from 29.7 to 52.4% for benzene and 26.0 to 50.9% for toluene.

The most detailed analysis of benzene and toluene recovery with methanol is shown in Table VI and VII and Figures 9 and 10. The methanol was added 30 mL at a time to the top of the extraction buret which held the adsorbed analytes. Ten mL aliquots were drained from the buret and analyzed. The time elapsed between elutions of the methanol was kept constant for each of the three extraction runs. The total recovery averaged 50.4% for benzene and 48.8% for toluene.

Various other rinse agents and combinations were used to wash the resin. Table VIII lists average recoveries from four trials when a methanol-hexane combination was used. In this case KHCO_3 was added to the gasoline-water mixture. The average recoveries were 52% for benzene and 53% for toluene. A fifth trial in which the KHCO_3 was omitted had recoveries of 54 and 53%, respectively. Table IX is the set of results when methanol, acetonitrile, hexane, and dichloromethane are used as rinse agents; recoveries are 30-35%. Results from methanol, hexane, and dichloromethane rinses are in Table X; the total percents recovered are 21.9 for benzene and 62.1 for toluene.

Methanol was rinsed through the jug, tubing, and volumetric flask in order to determine how much benzene and toluene were left

in the apparatus. Twenty-five mL of methanol was used to rinse the volumetric flask and another 25 mL to rinse the gallon jug and glass tubing. In three trials, the average percentages recovered from the volumetric flask were 3.1 for benzene and 3.8 for toluene. For the jug and tubing combined, the percentages were 1.0 and 1.5, respectively.

Analyses were also done on the spiked water soon after the benzene and toluene were added and at various other times as the extraction process progressed. The concentrations of benzene and toluene in these samples were determined. Examples of these concentrations are presented in Table XI. The apparent concentrations of benzene and toluene in the water at the beginning average 3.49 and 3.23%, respectively; at the end, 2.28 and 2.90%. Three of the trials had tests done of the spiked water near the midpoint of the extraction run. These concentrations were intermediate between those at the beginning and end.

Gradually increasing peaks in the benzene and toluene areas began appearing on the chromatograms of water and the spiked water after it was eluted from the resin during the last several trials when the mobile phase was being recycled. An example near the beginning of this period is on p. 1 of Figure 10 and one from a month later, or 12 lab days, is given on the second page of figure 10. Also during this time the peak heights of the standard solutions gradually decreased. The peak height of the benzene standard solution decreased from 7.41 to 3.62 cm and the toluene peak height went from 12.52 to 8.31 cm. These can also be compared with Figure 5 which was done midway between these two. midway

Figure 12 is an example of the chromatograms used to determine the best procedure for cleaning the resin between extraction processes. Chromatogram segment a. illustrates pure methanol, b. is a methanol rinse of the resin after completion of an extraction and rinsing procedure, and d. is the sixth 30 mL methanol rinse of the resin after 25 mL of CH_2Cl_2 was rinsed through. The pure methanol and sixth methanol rinse produce nearly identical chromatograms.

DISCUSSION

Observations of the chromatograms typified by Figure 6 of tap water and of benzene, toluene-spiked tap water after it had passed through the resin were used to determine that the resin is effective for the removal of these species at a flow rate of 20-25 mL per minute. Calculating the decrease in peak heights of benzene and toluene in the spiked sample to the extracted sample as a percentage of the decrease in peak heights in the spiked water to the tap water gives these results:

$$(5.29-0.26)/(5.29-0.25)\times 100\% = 99.8\% \text{ for benzene, and}$$

$$(11.05-0/76)/(11.04-0.70)\times 100\% = 99.4\% \text{ for toluene.}$$

The absorbance data from UV analysis, Table II, reinforces this conclusion and indicates that another absorbing species is also removed from the tap water. This data and the presence of interfering peaks in the later chromatograms would make it necessary to run blanks and to get a baseline chromatogram of the water without the analytes before any quantitative analysis could be done.

Although the plot of absorbance versus concentration is linear for concentrations of 2-25 mg/L for both benzene and toluene, results from other procedures tested in this research indicate a need for further revisions in the procedure. Peak heights on chromatograms of the standard solutions show some variation as can be seen Table I. Variations from day to day could be explained by column deterioration, breakdown of the standards, or slight variations in the wavelength setting which can occur as the instrument was adjusted after use by different operators. The first explanation is most consistent with the observations that the peak widths, taken at one-half the peak

height, increased as the peak heights decreased; examples of this are visible in Figures 6 and 11.

Analysis of the composition of the spiked water in the gallon jug give a very wide range of concentrations for benzene and toluene (Table XI). This is possibly due to evaporation while the extraction is in progress as the concentrations decreased without exception during the roughly three hours necessary to siphon the water through the resin. After these declines were first noted, more inversions of the jug were made in an effort to mix the benzene and toluene into the water. The beginning and ending concentrations remained roughly the same as before.

Another problem is the low recovery rate. However, as the recoveries were based on the starting concentrations of the analytes instead of what may have actually been present in the water for the duration of the extraction run, the true percentages may have been 10-20% higher. The highest and most consistent recoveries were achieved when 90 mL of methanol were rinsed through the resin and eluted 10 mL at a time for a total of 80 mL of recovered methanol. This procedure recovered about 50% of the starting amount of each analyte. This rinsing process took slightly over an hour. A mechanized approach to taking the rinse samples and analyzing them would probably increase the reproducibility of the results, as would finding a way to solve the problem of the air bubbles which appeared in the resin every time methanol was added to the buret at the conclusion of the siphoning process.

The percent recoveries shown in Figures 9 and 10 and Tables VI and VII indicate that it is definitely better to measure the volume of

the recovery rinses eluted instead of the volume added. Pouring the rinses into the top of the buret instead of vacuuming them up also led to more consistent results. Although this sometimes necessitated making repeated inversions of the buret manually to rid the resin of air bubbles, it seemed that less air contacted the adsorbed analytes than when the rinses were vacuumed up through the resin. Attempts to determine how well and how long the analytes were held by the resin before rinsing would be a logical extension of this work and could lead to a procedure for collecting the benzene and toluene in the field and transferring them to the lab for analysis.

Little difference was noted when the potassium bicarbonate was added to "salt-out" the analytes to the resin. As some later studies indicated that the resin seemed to absorb the benzene and toluene effectively, this process can be eliminated. If evaporation is the cause of the low recoveries and of the declining concentrations of benzene and toluene in the jug of spiked water, then studies could be made to find a substance which would hold the analytes in the tap water and release them to the resin, a floating "lid" on the spiked water, or use of a collapsible jug.

No conclusions could be drawn with regard to the relationship of the amount of time a rinse is left in the resin with the amount of benzene and toluene recovered. Efforts to reduce contamination such as the charcoal filter and replacement of room air with nitrogen gas as the incoming gas cannot be substantiated as effective. Chromatograms did, as a general trend, show fewer interfering peaks as the research continued, but comparisons made from summer to summer

were of little value since water, resin, HPLC equipment, columns, techniques, and contaminants in the air changed.

An effective cleaning process for the resin between extraction procedures was found, as described in the results and in Figure 12. Dichloromethane was effective for removing adsorbed species from the resin and then 6 to 10 rinses of the resin with methanol were used to remove the dichloromethane.

No theories were tested about the identity and size increase of the interfering peaks which appeared on the chromatograms during the last month of analyses. The size decrease of the peaks for the standard benzene and toluene standard solutions could be due to a change in the composition of the standard solutions. However, since the water was spiked from these same solutions, the comparisons of recovery rates from one extraction run to another over that time would still be appropriate.

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Table I: REPRODUCIBILITY OF PEAK HEIGHT
in centimeters
OF 2.5 mg/L STANDARD SOLUTIONS
70/30 methanol/water mobile phase

		BENZENE		
Injection:		1	2	range
Date	tr's(min)			
6/24/93	4.6	7.76	7.22	0.54
7/06/93	4.5	6.03	6.20	0.17
7/08/93	4.8	3.83	3.65	0.18
7/09/93	4.5	3.70	3.75	0.05
7/19/93	4.5	3.69	3.97	0.28
		avg		0.24

		TOLUENE		
Injection		1	2	range
Date	tr's(min)			
6/24/93	5.9	13.9	13.52	0.4
7/06/93	5.6	11.6	11.57	0.0
7/08/93	5.8	6.41	6.44	0.03
7/09/93	5.6	6.59	6.56	0.03
7/19/93	5.7	9.02	9.10	0.08
		avg		0.1

RESIN EFFICIENCY
UV SPECTROMETER ANALYSIS
BENZENE (B) AND TOLUENE (T) REMOVAL
from 6-8 mg each analyte
per gal. tap water

AVERAGE ABSORBANCES AT 208 nm

	before extraction	after extraction
B/ACN/WATER	0.097	-0.040
T/ACN/WATER	0.126	-0.029
ACN/WATER	-0.006	-0.034

Table II

Table III: BENZENE AND TOLUENE RECOVERY
 SINGLE MEOH RINSES
 from 50-70 mg/L gasoline in water

rinse volume mL	%B	%T
50.0	*	29
30.0	8.14	4.56
25.0	19.3	*
50.0	14.7	10.8

* not tested

Table IV: BENZENE AND TOLUENE RECOVERY
THREE 25 mL MEOH RINSES, COMBINED
from 10-15 mg/L gasoline in tap water

Trial	%B	%T
1	21.4	17.8
2*	40.0	40.0
3**	15	13

*backed up with spectrometer
analysis

**spectrometer analysis only

Table V: BENZENE AND TOLUENE RECOVERY
 THREE 30 mL MEOH RINSES
 from 2-5 mg/L each analyte in tap water

BENZENE RECOVERY (%)				
fraction	trial			
	1	2	3	4
1	4.82	11.5	5.57	2.92
2	20.7	26.6	22.7	18.0
3	10.6	14.3	13.3	8.76
total %	36.12	52.4	41.6	29.7

TOLUENE RECOVERY (%)				
1	1.66	7.55	6.14	0.81
2	25.0	24.9	20.4	15.7
3	8.44	18.4	13.1	9.47
total %	35.1	50.9	39.6	26.0

Table VI: BENZENE RECOVERY
 EIGHT 10 mL MeOH RINSES
 from 2-5 mg/L benzene in tap water

fraction	trial	percent recovered		
		1	2	3
1		0.00	0.00	0.00
2		8.96	10.	10.
3		11.	11.	12.
4		11.	10.	12.
5		10.	8.65	9.83
6		5.62	4.43	5.00
7		2.91	2.11	1.72
8		1.09	0.77	0.52
total % benzene recovered:		51.	47.	51.

Table VII: TOLUENE RECOVERY
 EIGHT 10 mL MeOH RINSES
 from 2-5 mg/L toluene in tap water

fraction:	percent recovered		
	1	2	3
1	0.00	0.00	0.00
2	6.81	7.56	8.17
3	8.87	9.41	10.2
4	10.0	9.90	11.0
5	9.80	8.69	10.2
6	6.68	5.72	6.64
7	4.45	3.47	3.46
8	2.22	1.64	1.52
total % toluene recovered:	48.9	46.39	51.2

Table VIII: BENZENE AND TOLUENE RECOVERY
 MeOH AND HEXANE RINSES
 from 10-18 mg/L gasoline in 0.01 M KHCO₃/tap water
 results from 4 trials

RINSE	Benzene		Toluene	
	mean %	std dev	mean %	std dev
40 mL MeOH	6	2.6	2	0.8
40 mL hexane ' + 120 mL MeOH	46	8.3	51	6.0
avg % recovered:	52		53	

Table IX: BENZENE AND TOLUENE RECOVERY
 WITH MEOH, ACN, HEXANE, CH₂CL₂ RINSES
 Different Standing Times
 from 2-5 mg/L each analyte in tap water

Trial 1

solvent	standing time	solvent recovered (mL)	% B	% T
MeOH	none	28.7	3.80	2.40
ACN	none	32.0	18.0	18.0
MeOH	18 hrs	25.0	4.10	6.30
ACN	1.5 hrs	32.4	3.80	5.00
Hexane +MeCl ₂	1 hr	25.0	1.40	3.70
total % recovered:			31.0	35.4

Trial 2

MeOH	none	23.0	3.50	1.03
ACN	none	24.7	17.1	18.0
MeOH	18 hrs	29.0	10.1	11.8
ACN	1.5 hrs	30.8	3.69	5.02
Hexane +MeCl ₂	1 hr	31.2	*	*
total % recovered:			34.4	35.8

Table X: BENZENE AND TOLUENE RECOVERY
 MEOH, CH₂CL₂, HEXANE RINSES
 from 20-25 mg/L gasoline in tap water
 results from 3 trials

	Benzene		Toluene	
	mean %	std	mean %	std
40 mL MeOH	2.2	0.63	4.47	1.25
40 mL hexane +120mL MeOH	11.6	0.16	45.97	4.66
50 mL dichloro- methane	4.5	1.30	8.26	4.24
40 mL MeOH	3.6	0.47	3.44	2.18
total	21.9		62.14	

Table XI: VARIATIONS IN BENZENE AND TOLUENE CONCENTRATIONS
IN SPIKED WATER
AS THE EXTRACTION RUN PROGRESSES

in mg/L benzene or toluene in tap water

trial	before extraction B, T	during B, T	near end B, T
1	2.87, *	3.46, *	2.86, *
2	3.20, 3.11	2.97, 2.87	2.16, 2.18
3	3.34, 3.34	*	2.30, 2.30
4	* , 3.27	3.47, 3.06	2.36, 2.21
5	3.23, 3.22	*	2.00, 2.15
6	3.40, 3.06	*	2.28, 2.00
7	3.90, 3.37	*	1.62, 1.46**
8	4.48, 3.56	*	2.63, 2.39

(1.98, 1.91 after standing)

*not calculated

**instrumentation problems

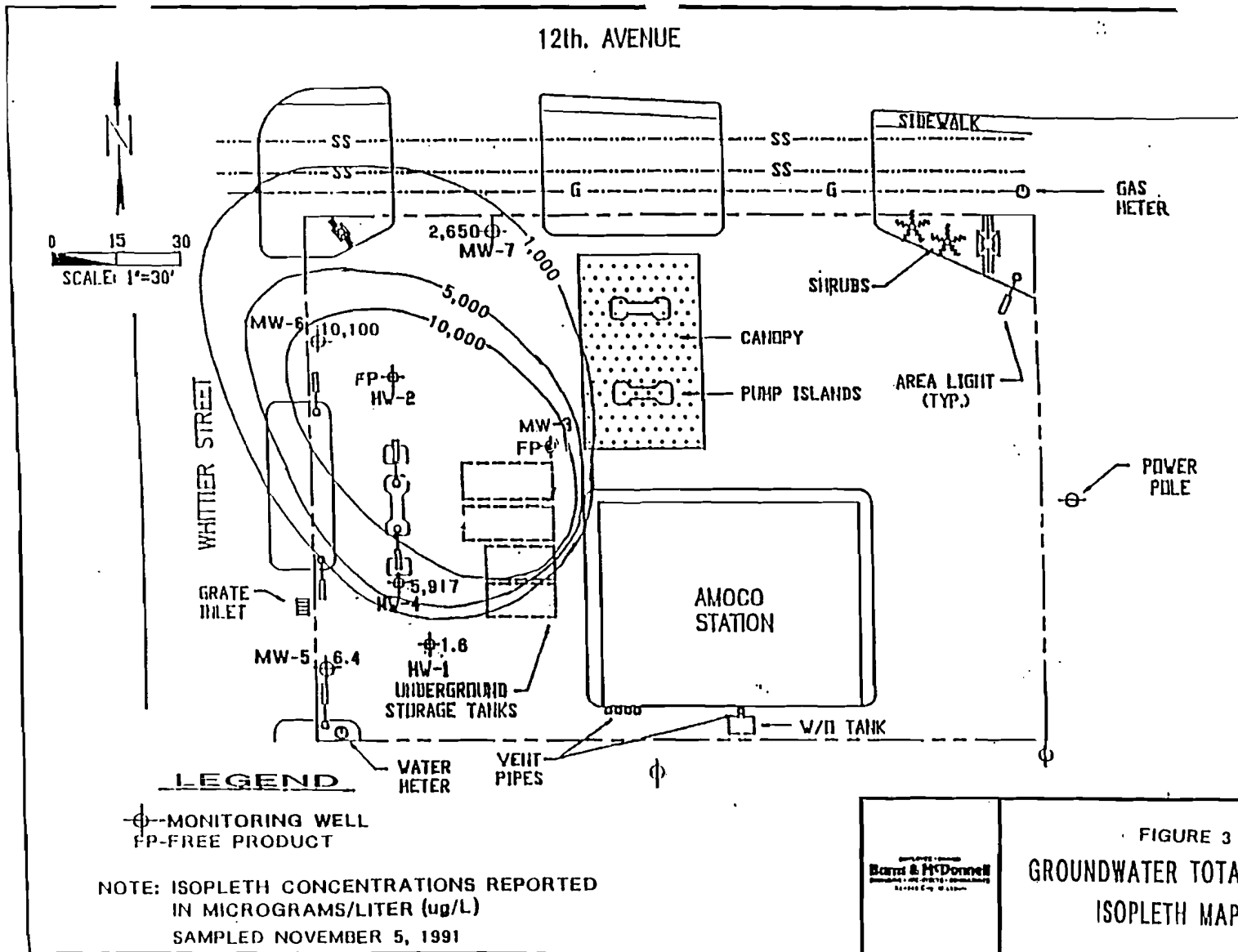


Figure 1
 GROUNDWATER CONCENTRATIONS
 OF BTEX
 AT AMOCO SERVICE STATION
 EMPORIA, KS

CONSULTING ENGINEERS
Baird & McDonnell
 1000 EAST 10TH AVENUE
 EMPORIA, KANSAS 66848

FIGURE 3
 GROUNDWATER TOTAL BTEX
 ISOPLETH MAP

Figure 2
EXTRACTION APPARATUS

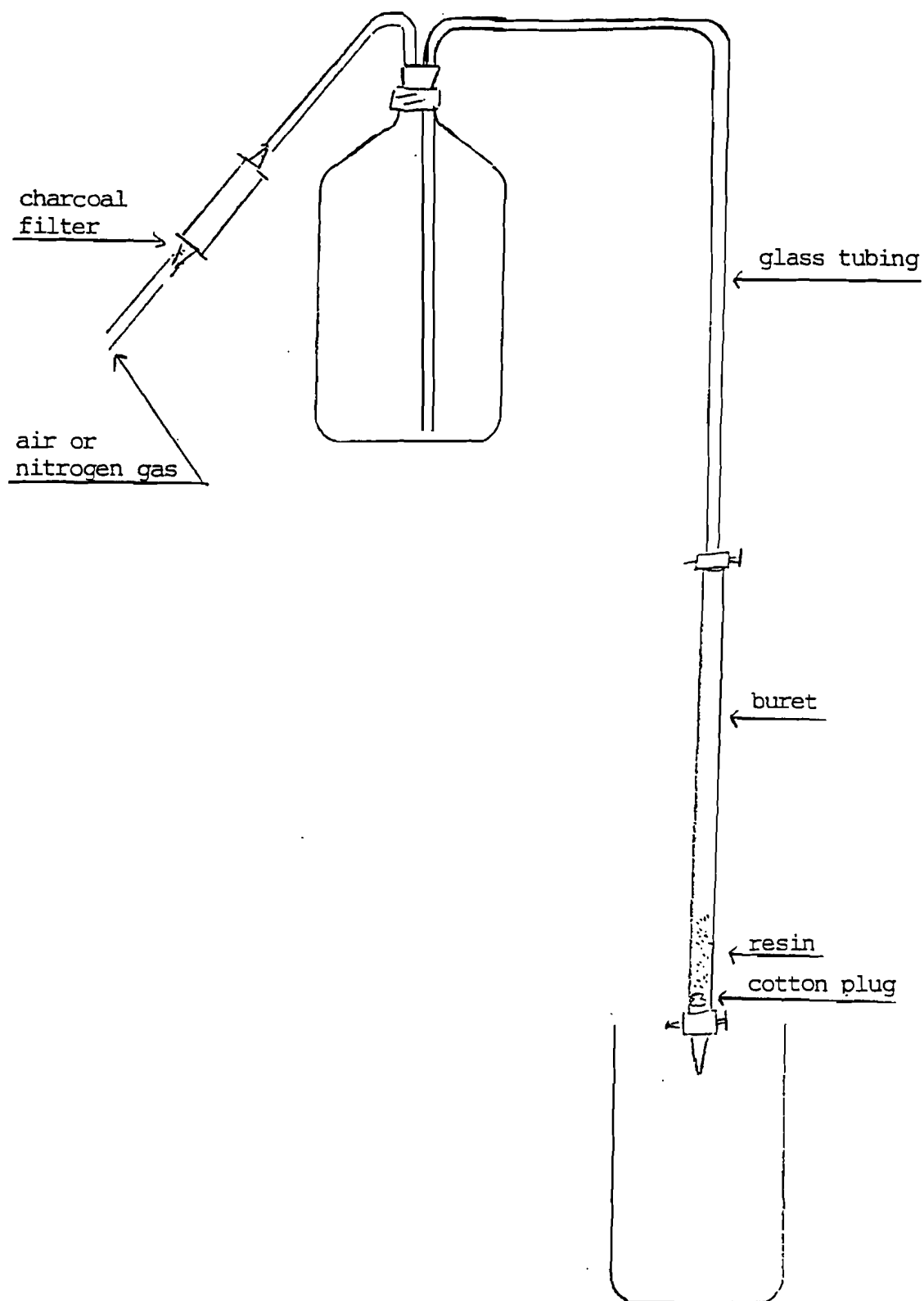


Figure 3
RINSE APPARATUS

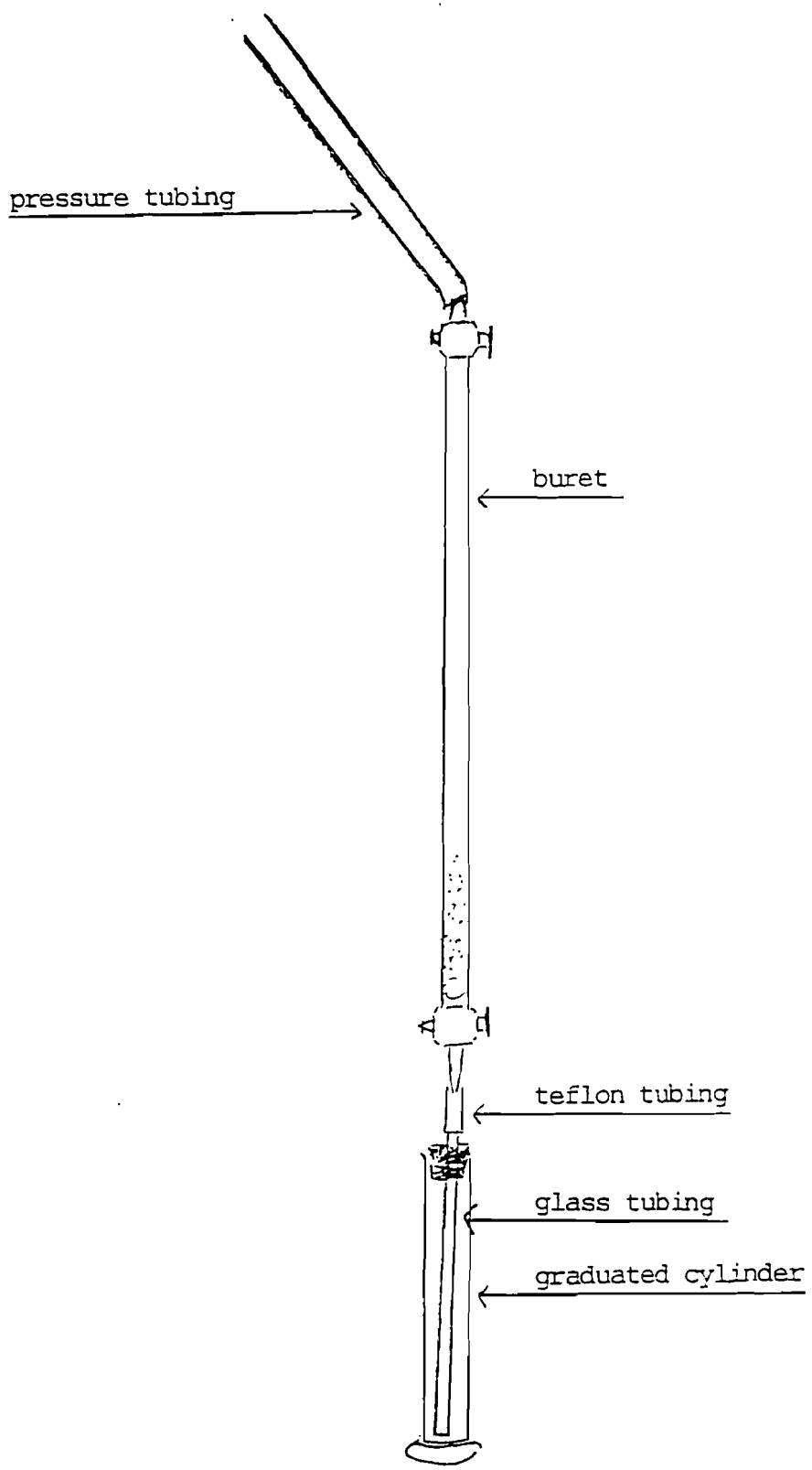
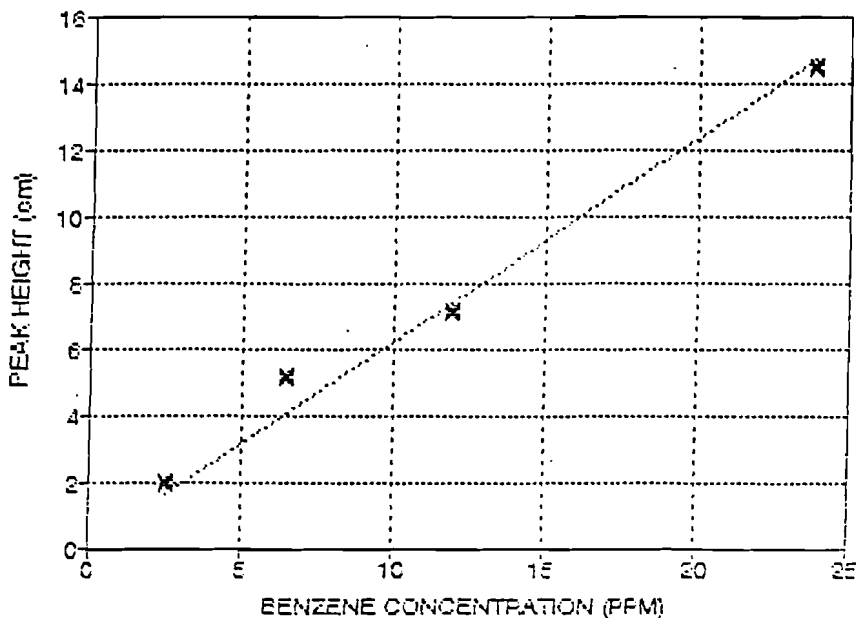


Figure 4
 CALIBRATION CURVE
 FOR BENZENE

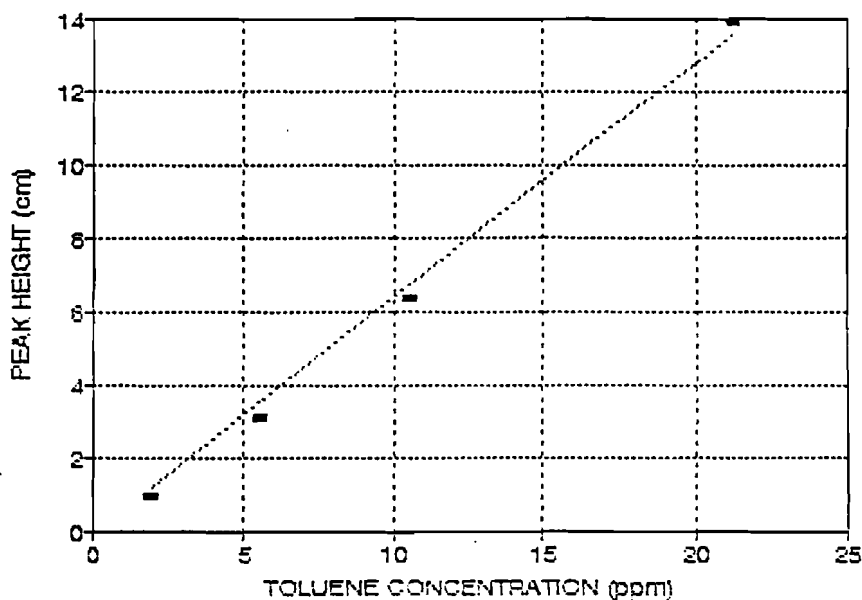


conc (ppm)	peak ht (cm)	calc ht
23.84	14.52	14.76
11.91	7.14	7.37
6.48	5.18	4.01
2.59	1.95	1.60

Regression Output

Constant	0
Std Err of Y Est	0.729531
R Squared	0.981266
No. of Observations	4
Degrees of Freedom	3
X Coefficient(s)	0.61907
Std Err of Coef.	0.02648
Rel std dev of slope	4.27%

Figure 5
CALIBRATION CURVE
FOR TOLUENE

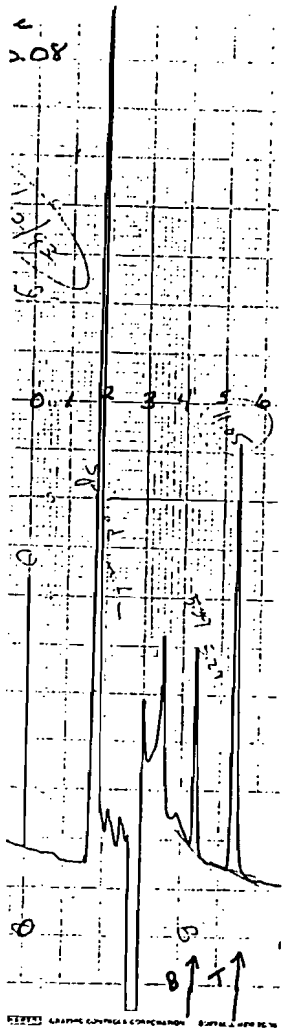


conc (ppm)	peak ht (cm)	calc ht
21.2	13.89	13.56351
10.5	6.35	6.717776
5.56	3.09	3.557222
1.91	0.98	1.221995

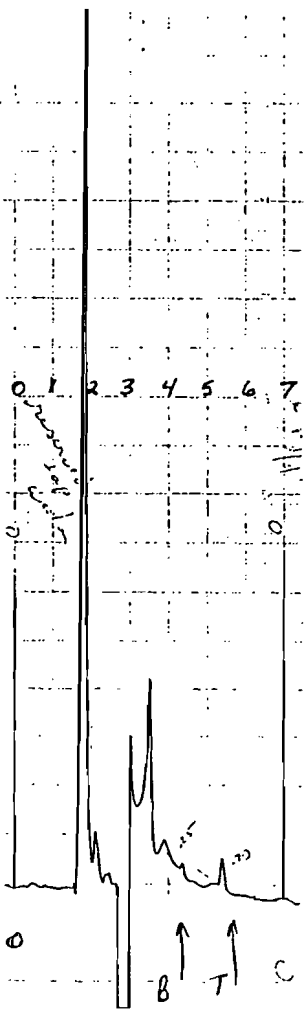
Regression Output:

Constant		0
Std Err of Y Est		0.415818
R Squared		0.994598
No. of Observations		4
Degrees of Freedom		3
X Coefficient(s)	0.639788	
Std Err of Coef.	0.017058	
Rel std dev of slope	2.67%	

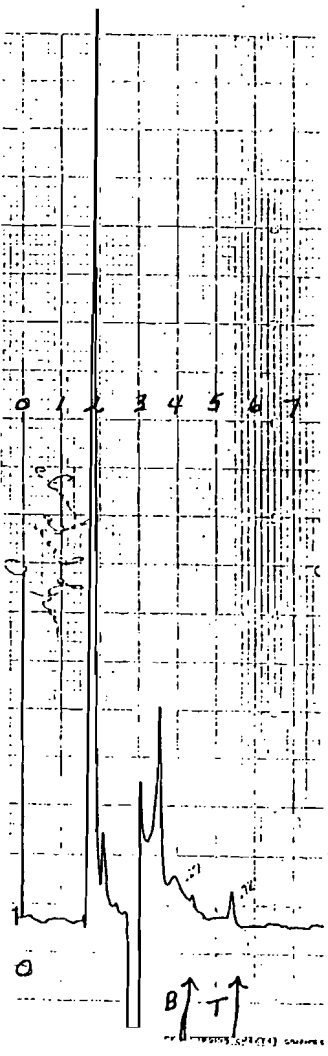
Figure 6
 TEST OF RESIN EFFICIENCY
 FOR REMOVAL OF BENZENE
 AND TOLUENE



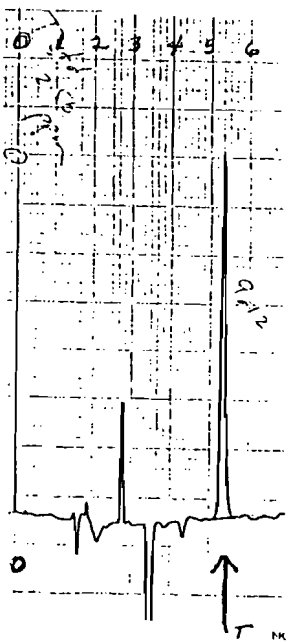
e. spiked tap water



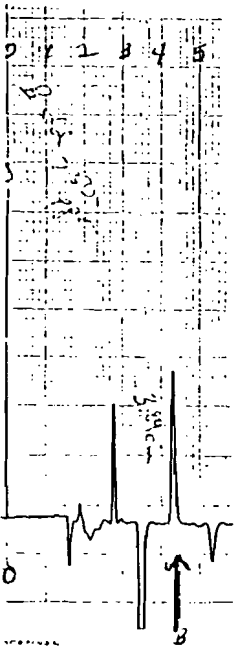
d. tap water before spiking



c. spiked tap water after extraction



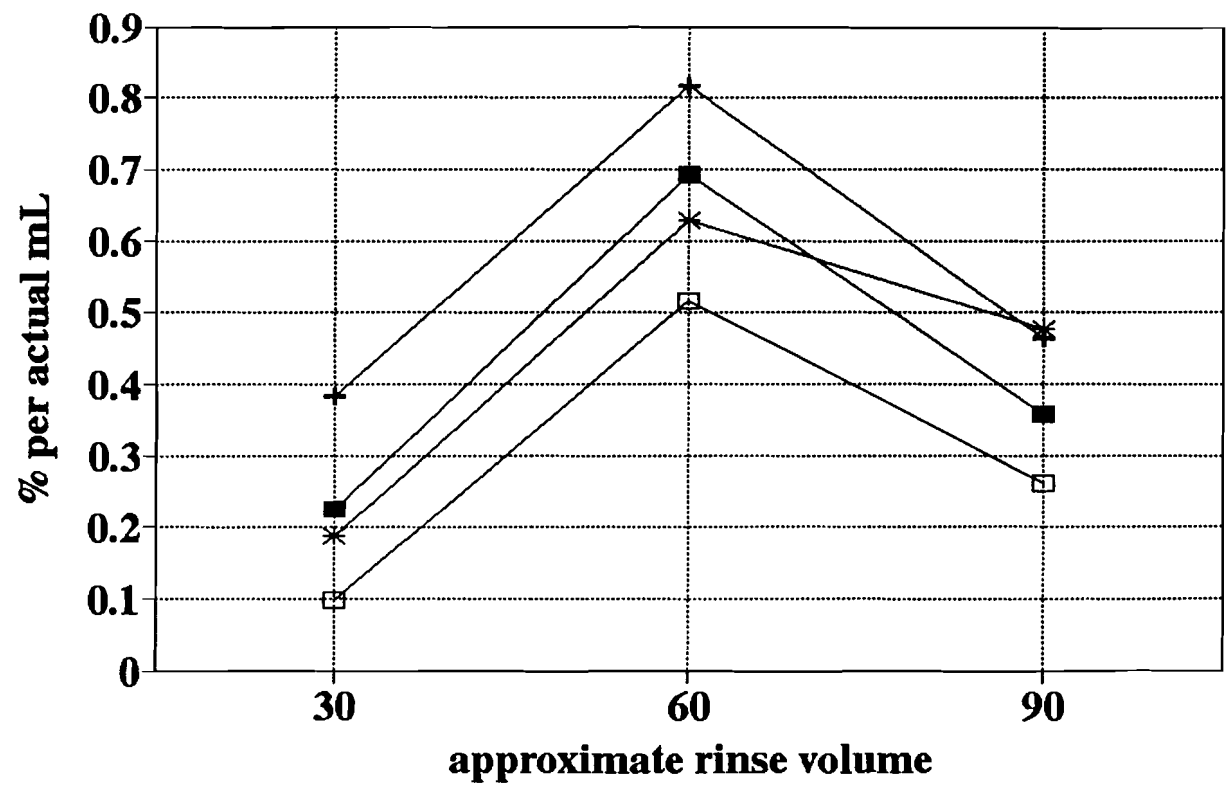
b. toluene standard



a. benzene standard

Chromatograms reduced to 1/2 original size)

**Figure 7: BENZENE RECOVERY
in successive 30 mL rinses of MeOH**



■ July 8, 1993 + July 9, 1993 * July 19, 1993 □ July 20, 1993

**Figure 8: TOLUENE RECOVERY
in successive 30 mL rinses of MeOH**

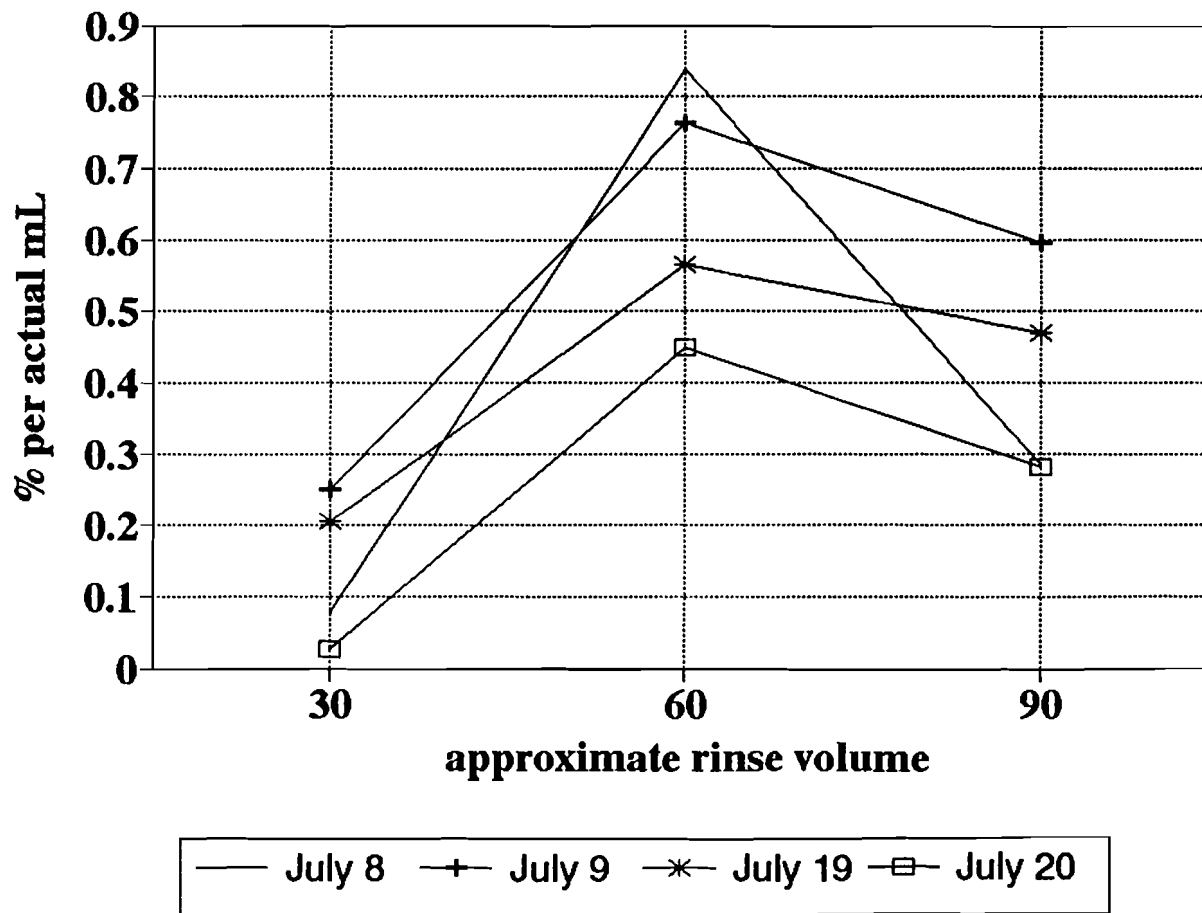
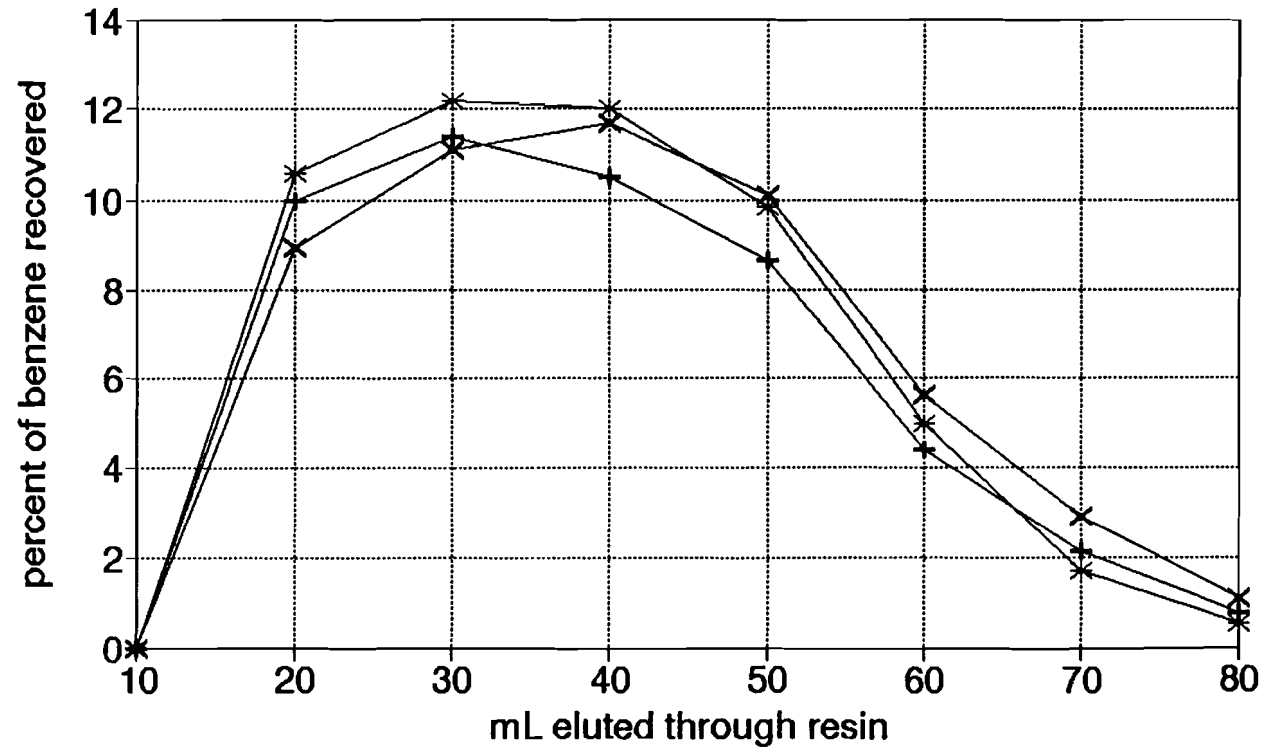
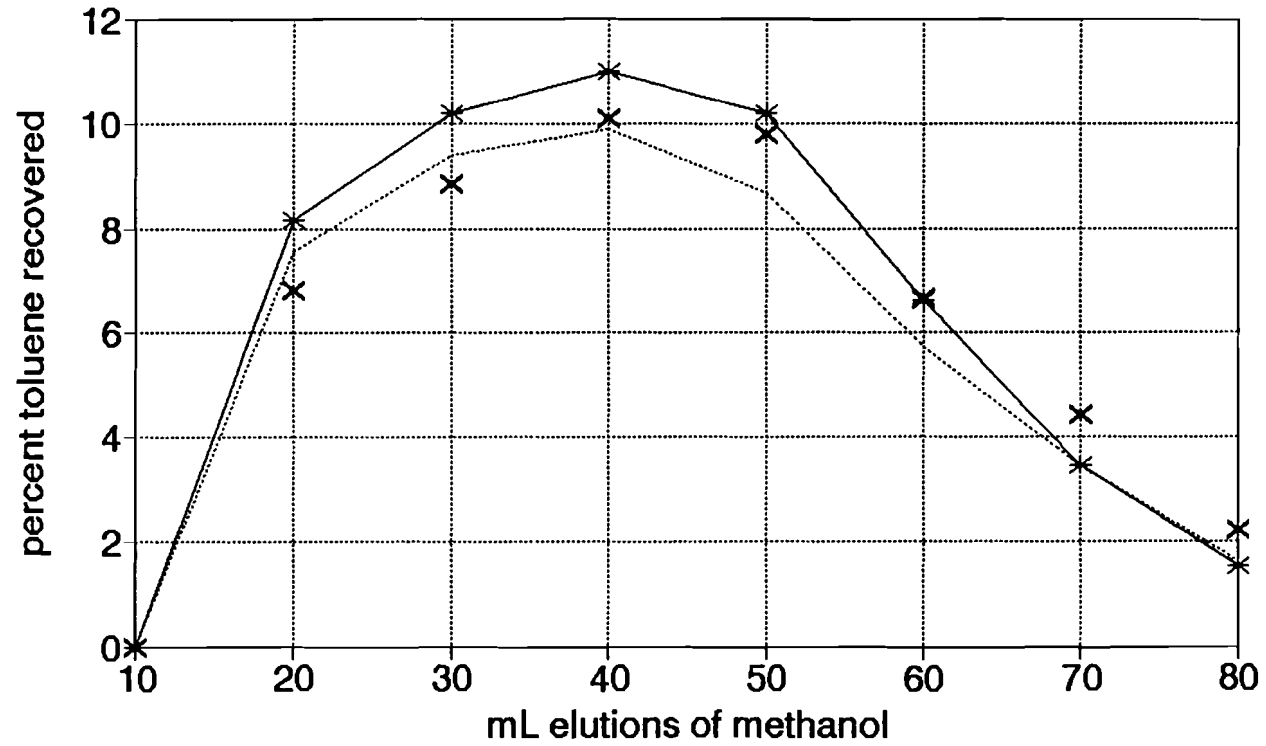


Figure 9: BENZENE RECOVERY
with 10 mL elutions of MeOH



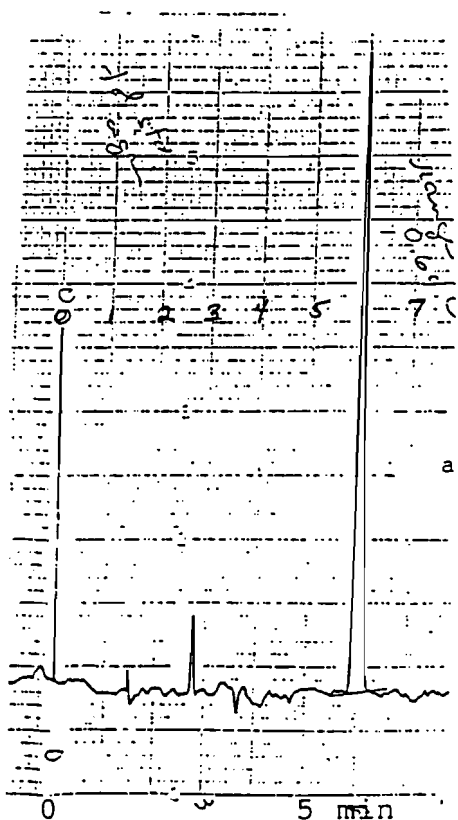
—x— July 27 —+— July 28 —*— July 29

Figure 10: TOLUENE RECOVERY
with 10 mL elutions of MeOH

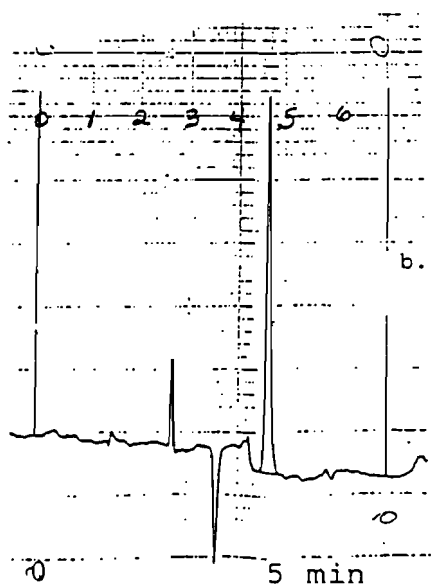


x July 27 * July 28 * July 29

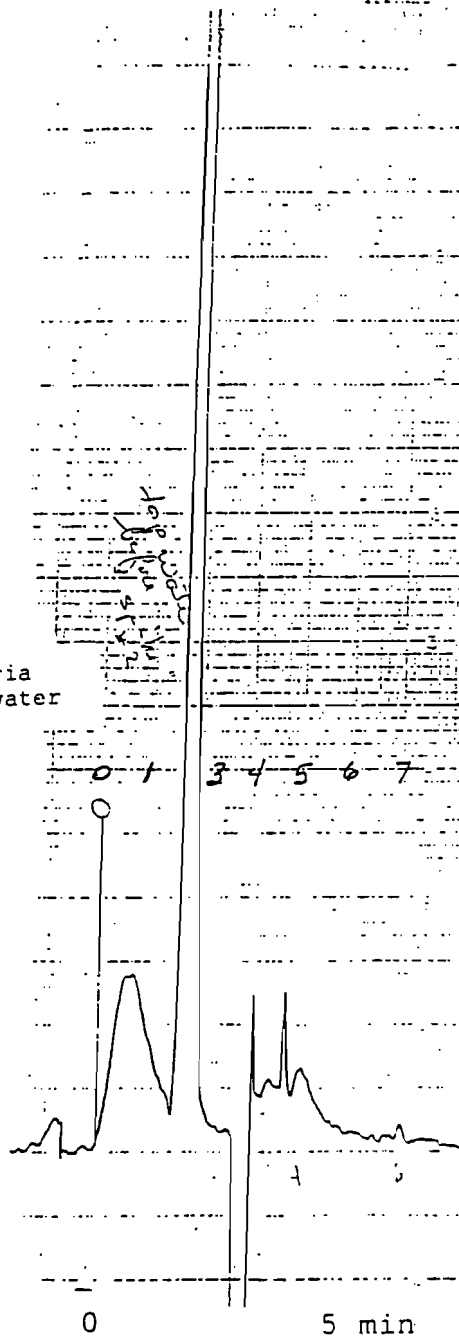
Figure 11
TAP WATER CHROMATOGRAMS
WITH INCREASING INTERFERING PEAKS



a. toluene standard



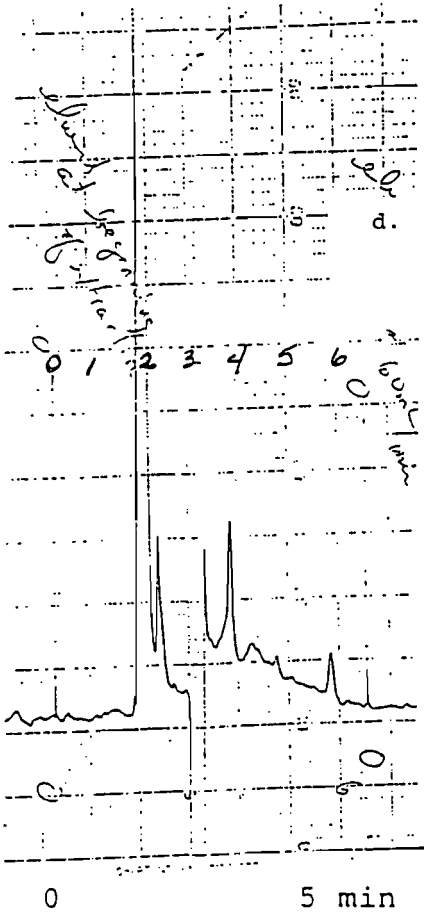
b. benzene standard



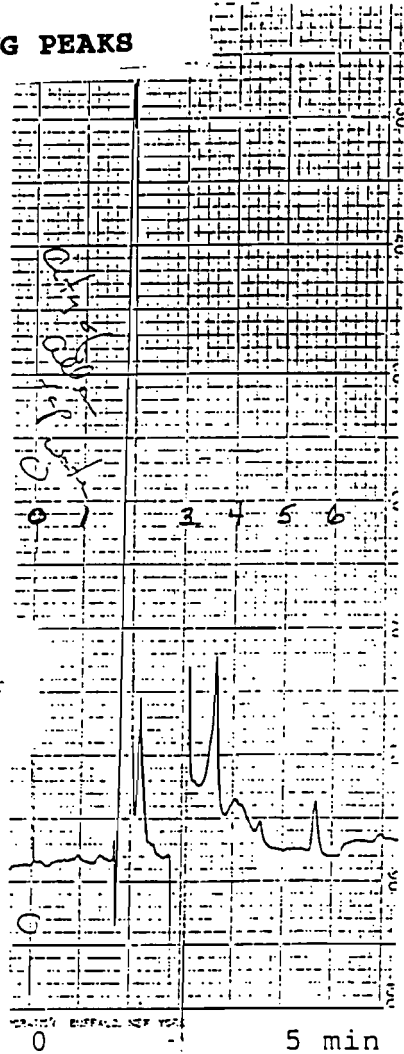
c. Emporia tap water

(Chromatograms reduced to 2/3 original size.)

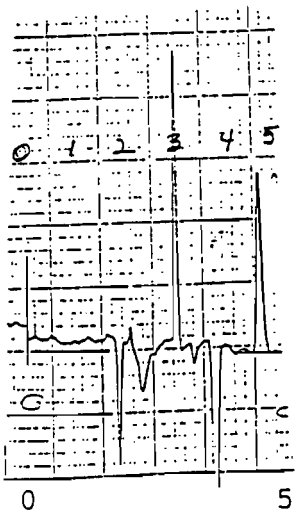
Figure 11
TAP WATER CHROMATOGRAMS
WITH INCREASING INTERFERING PEAKS



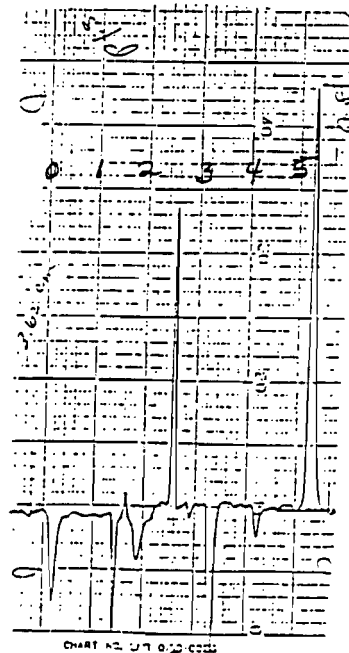
d. B,T-spiked tap water after extraction



f. Allen city water



e. Benzene standard

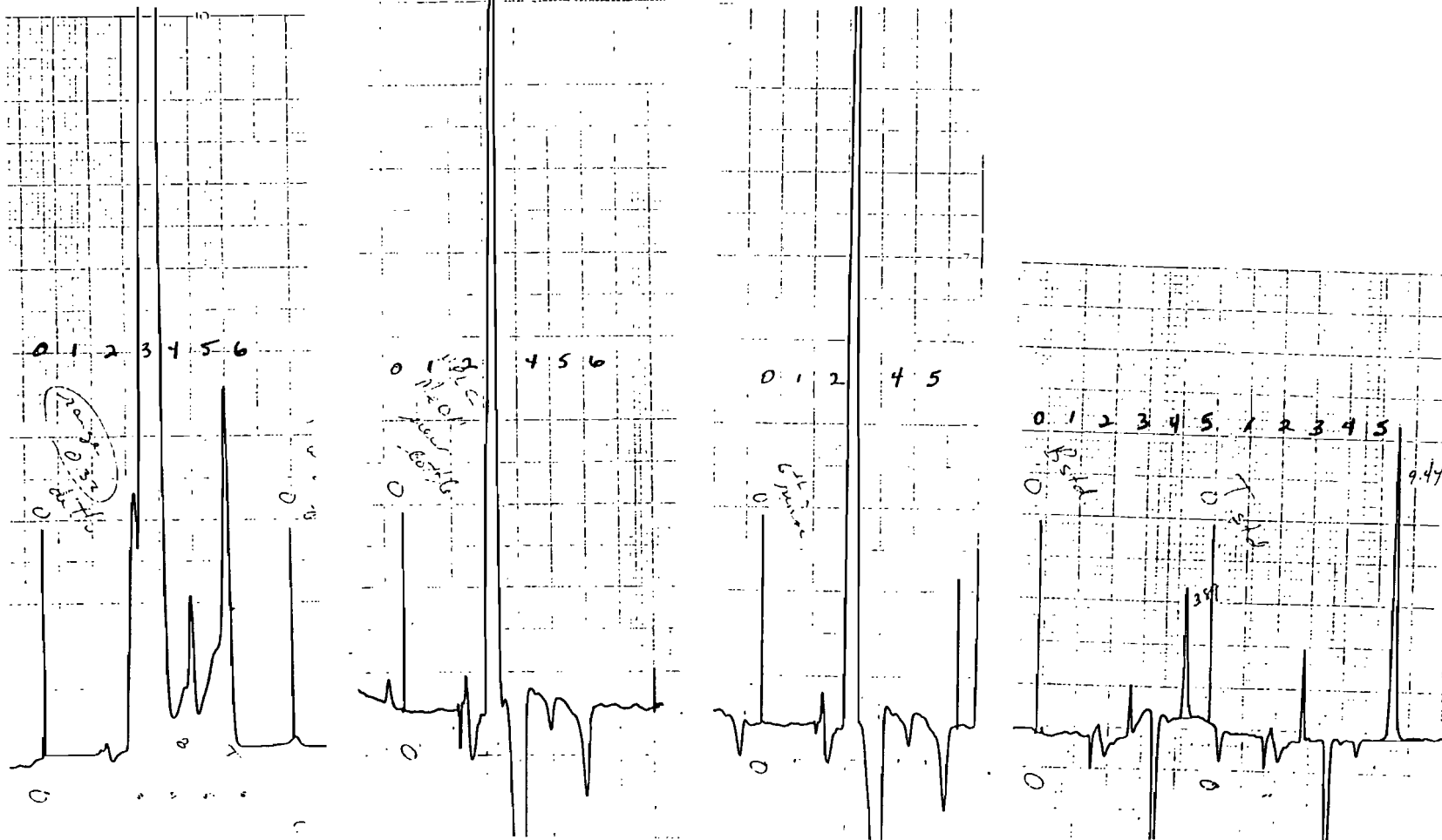


g. toluene standard

(Chromatograms reduced to 2/3 original size.)

5 min

Figure 12
CHROMATOGRAMS OF RESIN CLEANING



a. first MeOH
rinse after
 CH_2Cl_2
range 0.32

b. HPLC grade
MeOH
range 0.08

c. 6th MeOH
rinse
range 0.08

d. benzene
standard
range 0.08

e. toluene
standard
range 0.08

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Janet Hesterman
Signature of Author

May 12, 1994
Date

Extraction and HPLC Analysis of
Benzene and Toluene in Water

Nancy Cooper
Signature of Graduate Office Staff Member

May 13, 1994
Date Received