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THE CONCENTRATION OF HIGH BOILING IMPURITIES FROM LOW BOILING COMPOUNDS BY REVERSE FLOW GAS CHROMATOGRAPHY.*

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December 1971

ABSTRACT

The characteristics of gas chromatography peaks arising from various compounds when the column flow is reversed has been studied. It is shown that all compounds are eluted after reversal of the flow with the peak maximum at a time equal to or less than the time of flow reversal. The half width of the reverse peak is determined by a constant related to the number of plates in the column, and the square root of twice the time of flow reversal multiplied by the square root of the time of emergence of the compound when run in the forward direction. The method appears applicable to the concentration of trace high boiling impurities in any matrix in which the bulk components can be rapidly eluted through a suitable column.

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INTRODUCTION

It has been suggested that one method of concentrating high boiling impurities in air or breath is by reverse flow gas chromatography.[1] In this method the sample is introduced into a GLC column with characteristics such that the major components of air, e.g., O_2, N_2, A, CO_2 and H_2O, are rapidly eluted. The flow is then reversed in the column and the higher boiling impurities (actually those compounds with emergence times greater than the time of emergence of the last eluted principal component in the forward direction; they may or may not be higher boiling) are eluted in a short time by reversing the flow through the column and can be collected with a suitable trap for analysis by another method, e.g., mass spectrometry. This method is not limited to air but is applicable to trace impurities in any system in which the major constituent can be flushed through a suitable column and emerge while the constituents of interest are still held on the column.

While a complete search of the voluminous literature on gas chromatography has not been made, a limited survey showed no detailed studies of the reverse elution process. The process is widely used to rid columns of high boiling components which are not of interest in the analysis under consideration. The usual method is to use reverse flow for about the same length of time as the column was run in the forward direction. In order to evaluate the possibilities of this method, a few experiments have been made in which known concentrations of a variety of compounds with forward elution times from 4.2 min to > 1000 min have been studied. Because of the difficulty of obtaining reproducible concentrations in air or N_2 of compounds with such a wide variance in volatility, the compounds were dissolved in methanol. On a Porapak Q column, methanol and water (in small amounts) are eluted quite rapidly and present no problems in the study of the

reverse flow peaks of the compounds under consideration. A few experiments were made with vapors of the more volatile compounds in N_2 gas. The behavior of the reverse peaks of compounds injected as vapors in N_2 showed no differences from those made with solutions of the same compounds in methanol.

EXPERIMENTAL

The experimental column consisted of a 30 inch length of 1/4 inch stainless steel tubing packed with 80-100 mesh Porapak Q.[2] The column was enclosed in a laboratory oven the temperature of which was only roughly controlled with a variable transformer. Temperature drifts were slow and over any 8 hour day were less than 2°C. The detector was a four filament thermal conductivity cell operated at 150°C. This temperature was controlled to better than ±0.01°C with a resistance thermometer and proportional controller. The filament current to the T. C. cell was controlled to ±0.01%. The normal current to the cell was 150 ma. The helium flow through the column was directed forward or reverse by a six way valve. The valve ports are selected by a series of "O"-rings on a push-pull shaft in the valve. These "O"-rings were Viton-A and could be operated at 150°C continuously or up to 200°C for short periods. Most work was done at a column temperature of 150°C. Helium flow was controlled by a Miniflow controller and the helium flow rate was independent of the pressure drop in the column. Injection of the one µl sample was with a 10 µl syringe through a Silicone rubber injection gasket. When 1 µl samples are introduced, the reproducibility of this procedure is ±5%. Within this accuracy the peak areas of well behaved peaks were the same in both the forward and reverse flow modes of operation. The determination of peak areas for compounds with long (> 100 min) elution times is less certain owing to their large half width and low peak intensity. Even under these conditions the peak areas of forward and reverse peaks were found equal

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to within ±10% and this is probably representative of the determination of peak areas on such broad peaks when this area is determined by triangulation (area = width at 1/2 peak height multiplied by the peak height).

Preliminary experiments established that injections of 1 µl of methanol solutions of these compounds yielded peaks, both forward and reverse, the areas of which were linear with concentration and the peak half widths and elution times were independent of concentration in the range 1% to 10% by volume. Samples of this size do not overload the column or the thermal conductivity detector.

1) Back flushing of various compounds.

Experiments have been made to determine whether various compounds, each with its own emergence time from a Porapak Q column, can each be collected with reasonable efficiency when the flow through the column is reversed at a given time after introduction and collection of the reverse peak made for a defined time after reversal. The compounds were introduced as 5% solutions in methanol. Pure methanol has an emergence time, t_e, of ~0.6 minutes on this column and under the conditions of operation, and after 4 minutes, the detector shows no appreciable response (the recorder is back to the baseline). The column flow was reversed at 4 minutes after injection and the emergence time after reversal, t_{er}, and the area of the reverse peak measured as a function of time after reversal of the flow. This area was also compared to the area and characteristics of the peak produced when the sample was run in the normal forward direction. As shown in Fig. 1, the time of emergence in each case was taken as the time the peak maximum occurred. The half width of the peak is the width in minutes at one-half the peak maximum intensity.

The data in Table I and Fig. 2 shows the recovery possible after each of various compounds have been injected, run forward for 4 minutes (n-propanol for

3.6 minutes) and then reversed. The recovery observed for collection periods of 4 minutes after reversal and 5 minutes after reversal are shown. For well behaved compounds with emergence times from the column of less than 1/2 hour, the recovery after 4 minutes is $\geq 90\%$ of the total sample injected and after 5 minutes the recovery is virtually 100%. Acetic acid is an exception and shows poor recovery owing to tailing of the peak in the reverse mode. This behavior is not unexpected because of the equilibrium between the acid dimer and monomer in the column. For compounds emerging one hour or longer in the forward direction, the reversal peak becomes quite broad and the recovery after 4 minutes (and even 5 minutes) decreases rapidly with increasing forward emergence time. This means that components with emergence times will be discriminated against in the reverse collection procedure unless long reverse collection times are used.

The half width of the reverse peak at a given time of reversal is a function of the emergence time in the forward direction. With t_r constant, the half-widths of the peaks in the forward and reverse directions follow the following relations;

$$(hw)_f = k_1(t_e) \qquad (1)$$

$$(hw)_r = k_2(t_e)^{1/2} \qquad (2)$$

where hw is the half width in minutes and t_e is the emergence time in minutes of the compound when run in the forward direction.

In Eq. 1 the value of k_1 is related to the number of theoretical plates in the column.[3] For the 30 inch, 1/4 inch OD column used here, k_1 was close to 0.100. (This corresponds to $\sim$ 500 theoretical plates.) For the same column and reversal of flow 4 minutes after injection, k_2 was close to 0.29. (Note that k_2 is a function of $(\text{time})^{1/2}$.) The solid line drawn in Fig. 1 for

the half width of the reverse peak was calculated from Eq. 2 and $k_2 = 0.294$. The experimental points fit very well except for isoamyl acetate which contained ∿ 40% faster emerging impurities and for dodecane where t_e was based on the injection of 10 µl of pure liquid, which amount saturated the column. The emergence time is shortened under saturation conditions. The collection efficiency for reverse flow of peaks with long forward emergence times is determined by the half width of the reverse peak. Since Eq. 2 appears valid, there does not appear to be any way of avoiding this obvious discrimination against the efficient collection of the reverse peaks of very high boiling compounds except by the use of longer collection times for the reverse peak.

In order to determine the generalized form of Eq. 2, a series of determinations of the half width of the toluene reverse peak was made as a function of the time at which the flow was reversed. Various characteristics of the reverse peaks as a function of time of reversal of the flow are shown in Table II and Fig. 3. The emergence time of the reverse peak, t_{er}, is always slightly less than t_r and for $t_r < 1/2\ t_e$ follows the linear relation shown in Eq. 3.

$$t_{er} = 0.81\ t_r \qquad (3)$$

At times $t_r > 1/2\ t_e$, t_{er} becomes larger than the value given in Eq. 3. When t_r approaches t_e, t_{er} approaches t_r.

An analysis of the data in Table II shows that the half width of the reverse peak follows the relation shown in Eq. 4.

$$(hw)_r = k_1\ (2t_r)^{1/2}\ t_e^{1/2} \qquad (4)$$

In the case of toluene, with an emergence time of 28.7 minutes and a half width of 2.76 minutes, $k_1 = 0.096$. Using these values and t_r, the calculated

half width of the toluene reverse peak at various times of flow reversal are shown in Table II. The agreement is satisfactory, though at quite short times of reversal the peak is slightly wider than calculated from Eq. 4.

In order to check the general applicability of Eq. 4, a variety of compounds, all of which are well behaved on a Porapak Q column, were investigated with variation of the reversal time. The forward emergence times of these compounds at 150°C varied from 9 minutes for ethylene glycol to 407 minutes for n-butyl-benzene. Using the average k_1 of 0.100, the half widths of the reverse peaks agree well in general with the values calculated from Eq. 4. The data is shown in Table III and Fig. 4. For the compounds phenol and cumene (isopropyl benzene) the observed reverse peak half widths at $t_r > 14$ minutes are considerably less than calculated. No explanation of this behavior is obvious.

Also shown in Table III and Fig. 4 are the results with a 5% solution di-isobutyl ketone (DIBK) in n-pentane on a 6 foot column packed with 20% di-nonylphthalate on 60-80 mesh Chromosorb. This column has the equivalent of about 4000 theoretical plates and exhibits a value of k_1 equal to 0.054. The column was operated at 120°C with a helium flow rate of 40 ml/min. The reverse peaks with $t_r = 4$ to $t_r = 20$ minutes show that at low values of t_r the observed peak is somewhat narrower than the calculated peak while at longer times the agreement is satisfactory. This is not a general behavior however because ethylene glycol, diethyl ketone, glycerine and phenol showed excellent agreement between calculated and observed values of $(hw)_r$ at the lowest times of reversal as shown in Table III and Fig. 2.

2) Effect of large amounts of water on reverse gas chromatography peaks.

Because samples of air or breath may contain fairly large amounts of

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water, the effect of water on the concentration of trace components by reverse flow gas chromatography was investigated.

As an example, one liter of air saturated with water vapor at 35°C contains 0.0395 moles of total gas of which 0.0022 moles is water vapor. This is equivalent to 40 µl of liquid water.

As a test procedure, 40 µl of 0.2% by volume solution of ethylene glycol in water was injected onto the 30 inch Porapak Q column and the behavior observed. The water peak with this overloading of the column tails drastically and the tail extends out to 10 minutes after injection. When the flow is reversed 4 minutes after injection, the collected ethylene glycol peak (collected for 4 minutes after reversal) contains 88% of the initial glycol injected and the collected fraction is about 25% water. Since the actual amount of water collected should be independent of whether or not the ethylene glycol is present, one may calculate the concentration factor by this procedure at various levels of impurities in the original air sample. In a 40 µl sample of 0.2% by volume glycol there are 0.00222 moles of water and 1.44 µmoles of glycol. If the ratio of glycol to water in the collected fraction is 3:1, then the collected sample will contain 0.48 µmoles of water. This amount of water collected is independent of the initial concentration of the minor high boiling component, whether it be ethylene glycol or other high boiling organic compounds which emerge from the column with elution times greater than the 4 minutes time at which the flow is reversed.

The 1 liter of air saturated with water at 35°C contains 0.0395 moles of gas including 0.00222 moles of water. A component present in concentration equal to 1 ppm will represent 0.0395 µmoles of material. In the collected fraction this will be diluted by 0.48 µmoles of water, and the concentration of the trace component in the collected fraction will be 7.6 mole %. This is an

acceptable range for mass spectrometer analysis of the collected fraction. If the minor component is present at the level of 1 part in 10^9, then the concentration in the collected fraction will be 0.0082%. Unless the impurities were components of a very simple mixture, this is a concentration at which identification or analysis by the usual mass spectrometer methods is of questionable validity. The limit at which reliable information might be achieved for a fairly complex mixture under these conditions is at the concentration level of about 1 part in 10^8 parts of air. The situation can be improved somewhat by reducing the water load in the collected sample by allowing a longer time for water elution before reversal of the flow. This procedure would also necessitate a concomitant increase in the collection time as well as decrease the effectiveness of the analysis by the loss of compounds with emergence times between t_r = 4 minutes and the new value of t_r chosen. For the system described, if t_r was increased to 8 minutes, such compounds as acetone, acetic acid, methyl ethyl ketone and ethylene glycol would be lost and not included (or be only partially included) in the collected material. The analysis might be improved by changing columns assuming one could be found on which the compounds of interest were held up for a longer time compared to water. For a complex mixture of contaminants in the sample this procedure may result in the substitution of one group of lost components for another. Each problem would require knowledge of the characteristics of the column for the bulk material and the specific compounds of interest in the analysis. Variations are also possible in which the collected material is re-injected on the same column for a second reverse flow concentration step. In the example cited this smaller amount of water in the second injection would result in little if any tailing and the concentration of water in the collected material could be reduced by another factor of several thousand. Such variations

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of the procedure have not been investigated nor has the present apparatus the capability of physical collection of the back-flushed material.

DISCUSSION

The results of this short and preliminary investigation suggest the method of flow reversal after elution of the major component of a mixture is a promising method of concentrating high boiling trace components in a mixture.

It is obvious that the method can be used in any situation where the major components can be rapidly eluted on a suitable column before the compounds of interest are eluted. Such problems are the determination of trace organics in water and the determination of compounds in biological materials where these compounds can be extracted into a high purity low boiling solvent such as hexane, pentane or ether.

The concentration and collection of larger amounts can also be achieved by use of two column operation. The first may be a preparatory type column where samples as large as several ml can be injected and the reverse flow impurities collected. If this is followed by further concentration on a smaller column a second concentration step can result in a sample of the high boiling components freed from essentially all of the initial solvent. Avenues of identification and analysis of these concentrated materials are possible at initial concentration levels well below the level possible by direct methods without concentration.

The results presented show that the behavior of peaks on reversal of flow and elution in the backward direction is determined by the characteristics of the column, i.e., number of theoretical plates, the behavior of the compound on the particular column used as manifested in the time of elution when run

forward, and the time of reversal of the flow. The equations

$$(hw)_f = k_1(t_e) \qquad (1)$$

and

$$(hw)_r = k_1 (2t_r)^{1/2} (t_e)^{1/2}, \qquad (4)$$

govern the spreading of the peak in time for each type of operation. Obviously these two equations can be combined to give Eq. 5.

$$(hw)_r = (hw)_f (2)^{1/2}(t_r/t_e)^{1/2} \qquad (5)$$

In Eq. 4 the term $2t_r$ is only approximately equal to the total time spent in the column when the flow is reversed at t_r. It is more nearly equal to the total time in the column for long times before reversal than for short. We have shown that $t_{er} = 0.81\ t_r$ for $t_r < 1/2\ t_e$ in the case of toluene elution. If this factor is used, i.e., $1.81\ t_r$ instead of $2\ t_r$, in calculating the half widths from Eq. 4, the agreement becomes even less satisfactory at the shorter times of reversal in some cases, e.g., toluene, but is improved in some others such as di-isobutyl ketone. It does not appear that the present data can be better fitted by any simple modification of Eq. 4.

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FOOTNOTES AND REFERENCES

*Work performed under the auspices of the U. S. Atomic Energy Commission.

1. Private communication of Nabil Amer and Walter Perkins, Lawrence Berkeley Laboratory.

2. Porapak Q is the trade name of a proprietary copolymer of styrene and divinyl benzene.

3. A. I. M. Keulemans, "Gas Chromatography", Reinhold Pub. Corp., New York, 113 (1957).

TABLE I. Characteristics of Forward and Reverse Elution Peaks of Various Compounds on a Porapak Q Column.[a)]

Compound	B.P. °C	Forward Peak			Reverse Peak (t_r=4 min)				%Reverse Peak Collected	
		t_e	$(hw)_f$	$(hw)_f/t_e$	t_{er}	$(hw)_r$	$\Sigma t=t_r+t_{er}$	$(hw)_r/\Sigma t$	4 min	5 min
n-Propyl Alcohol[b)]	97.8	4.20	0.45	0.107	3.55	0.66	7.15	0.092	95.2	100.0
Acetic Acid	117.0	4.62	0.48	0.104	3.87[d)]	0.71	7.87	0.090	∿60	∿95
Methyl Ethyl Ketone	79.6	6.85	0.72	0.105	3.60	0.77	7.60	0.101	91.0	>99.9
Ethylene Glycol	190.5	7.65	0.84	0.110	3.50	0.83	7.50	0.110	91.1	98.9
Diethyl Ketone	101.7	13.87	1.42	0.102	3.38	1.02	7.38	0.138	91.2	>99.9
Toluene	110.6	28.7	2.76	0.096	3.20	1.38	7.20	0.197	90.4	99.5
Glycerine	>200 (dec)	58	5.6	0.096	3.05	2.23	7.05	0.316	78	94.2
Phenol	181.4	72.5	7.6	0.105	3.20	2.43	7.20	0.338	75	93
i-Amyl Acetate[c)]	141	81.9	7.9	0.096	3.35	1.80	7.35	0.245	81	94
i-Propyl Benzene	152.9	112.6	10.3	0.091	3.10	3.30	7.10	0.465	67	85
n-Decane[e)]	174	249	33.6	0.135	3.20[d)]	4.6	7.2	0.64	57	71
n-Butyl Benzene	183.3	286	30.2	0.106	2.8	4.9	6.8	0.72	55	74
n-Dodecane[e)]	209	>900[f)]	∿240	∿0.3	2.5[d)]	10	6.5	∿1.5	∿26	∿35

a) t_r = 4 minutes, 30 inch Porapak Q column, 155°C, He = 40 ml/min, all times in minutes.

b) reversed n-propyl alcohol at 3.6 minutes forward.

c) contains ∿40% lower boiling impurities before main component.

d) tails badly.

e) n-decane and n-dodecane may be mixed isomers.

f) t_e forward determined with 10 µl pure liquid sample - column is overloaded.

TABLE II. Characteristics of Reverse Flow Toluene Peak as a Function of Time in the Column Before Reversal.[a]

t_r	t_{er}	t_{er}/t_r	$\frac{(hw)_r}{obs}$	$\frac{(hw)_r}{calc}$[b]	$\Sigma t = t_r + t_{er}$	$(hw)_r/\Sigma t$[c]
1.5	1.25	0.83	1.08	0.89	2.75	0.392
2	1.62	0.81	1.17	1.03	3.62	0.323
3	2.50	0.83	1.35	1.26	5.50	0.245
4	3.23	0.81	1.50	1.45	7.23	0.208
5	4.00	0.80	1.65	1.63	9.00	0.183
6	4.85	0.81	1.82	1.78	10.85	0.167
8	6.55	0.82	2.03	2.06	14.55	0.139
11	9.00	0.82	2.36	2.41	20.00	0.118
15	12.85	0.86	2.77	2.82	27.85	0.099
20	19.00	0.95	3.48	3.25	39.00	0.089

a) 30 inch Porapak Q column, 155°C, 40 ml He/min. All times in minutes.

b) Calculated from Eq. 4 with $k_1 = 0.096$; $(hw)_f = 2.76$ min; $t_e = 28.7$ min.

c) $(hw)_r$ divided by total time in column (forward + reverse).

TABLE III. Reverse Elution Peaks of Various Compounds as a Function of Time of Flow Reversal.[a)]

Compound	t_e	$(hw)_f$	k_l	t_r	t_{er}	$(hw)_r$ obs	$(hw)_r$ calc[b)]
Ethylene Glycol	9.03	0.93	0.103	2	1.72	0.63	0.60
				4	3.59	0.89	0.85
				7	6.72	1.31	1.12
Diethy Ketone	17.80	1.78	0.100	2	1.69	0.84	0.84
				4	3.35	1.18	1.19
				7	6.04	1.60	1.58
				10	8.85	1.91	1.89
				14	13.28	2.43	2.23
Glycerine	78.38	7.00	0.089	2	1.40	1.79	1.77
				4	2.99	2.52	2.50
				7	5.43	3.22	3.31
				10	7.58	3.75	3.95
				14	11.08	4.58	4.68
				20	15.79	5.20	5.59
				30	24.90	7.09	6.85
Phenol	94.85	9.7	0.103	2	1.40	2.03	1.95
				4	3.19	2.76	2.76
				7	5.55	3.47	3.64
				10	8.05	4.11	4.36
				14	11.08	5.09	5.16
				20	16.04	5.56	6.16
				30	24.45	6.80	7.54
Cumene	156	15.5	0.100	2	1.69	2.43	2.50
				4	3.15	3.46	3.53
				7	5.60	5.10	4.67
				10	8.00	5.57	5.58
				14	11.22	6.40	6.60
				20	15.85	7.05	7.90
				30	23.42	7.99	9.67
n-Butyl Benzene	407	43.5	0.107	2	1.26	3.63	4.03
				4	3.14	5.68	5.71
				7	5.3	7.19	7.55
				10	8.0	9.00	9.02
				14	10.8	10.66	10.68
				20	16.1	11.90	12.76
				30	26.0	15.15	15.63
Di-isobutyl Ketone[c)] (DIBK)	36.65	1.98	0.054	4	3.22	0.75	0.92[d)]
				5	4.05	0.87	1.03
				10	8.35	1.37	1.46
				12	9.8	1.64	1.60
				20	18.2	2.08	2.07

(Continued)

TABLE III. (continued)

a) 30 inch Porapak Q column, 150°C, 40 ml He/min, all times in minutes.

b) Calc. from Eq. 4 with $k_1 = 0.100$.

c) DIBK on 72 inch column packed with 20% di-nonyl phthalate on 60-80 mesh Chromosorb, 120°C, 40 ml He/min.

d) Calc. from Eq. 4 with $k_1 = 0.054$.

FIGURE CAPTIONS

Fig. 1. Definitions of terms used in characterizing gas chromatographic peaks when: A) the chromatographic column is operated in the normal manner, and B) the helium flow through the column is reversed at a time, t_r.

Fig. 2. Collection efficiency of various compounds when column flow is reversed after 4 minutes and the reverse peak collected for 4 minutes (curve A) and 5 minutes (curve B) as a function of the emergence time of the compound in the forward direction. Curve C is the half width of the reverse peak after reversal at 4 minutes, points experimental, line calculated from Eq. 2.

Fig. 3. Characteristics of toluene peak on reversal of helium flow.

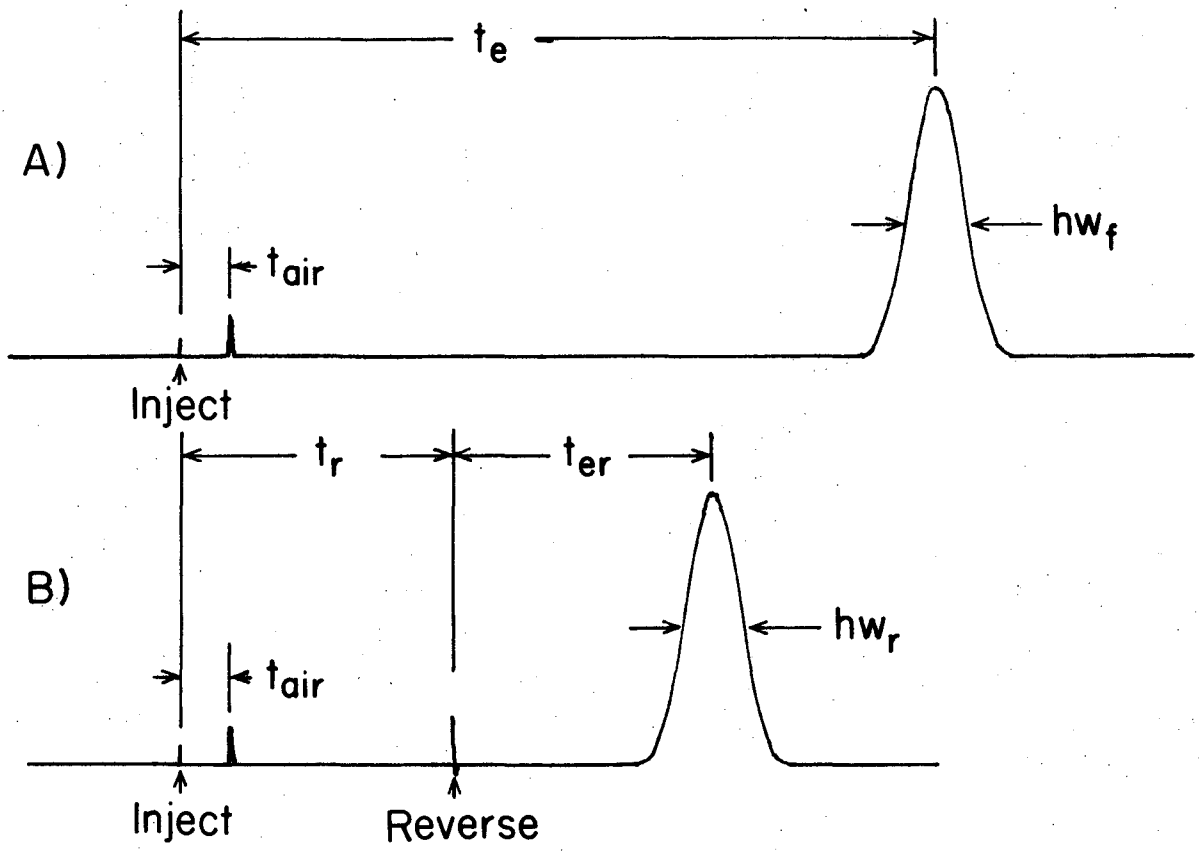
1) ▲ - half width of reverse peak, $(hw)_r$.

2) □ - $\left[(hw)_r/(t_r + t_{er})\right] \times$ 1/10.

3) ○ - elution time after reversal, t_{er}.

Fig. 4. Half width of reverse peaks of various compounds as a function of time of reversal of the helium flow rate. Lines calculated from Eq. 4. Points experimental.

All except di-isobutyl ketone determined with Porapak Q column, 30 inch length, 150°C, 40 ml He/min, $k_1 = 0.100$. Di-isobutyl ketone determined with 6 foot column packed with 20% di-nonyl phthalate on 60-80 mesh Chromosorb, 120°C, 40 ml He/min, $k_1 = 0.054$.



XBL 7112-1796

Fig. 1

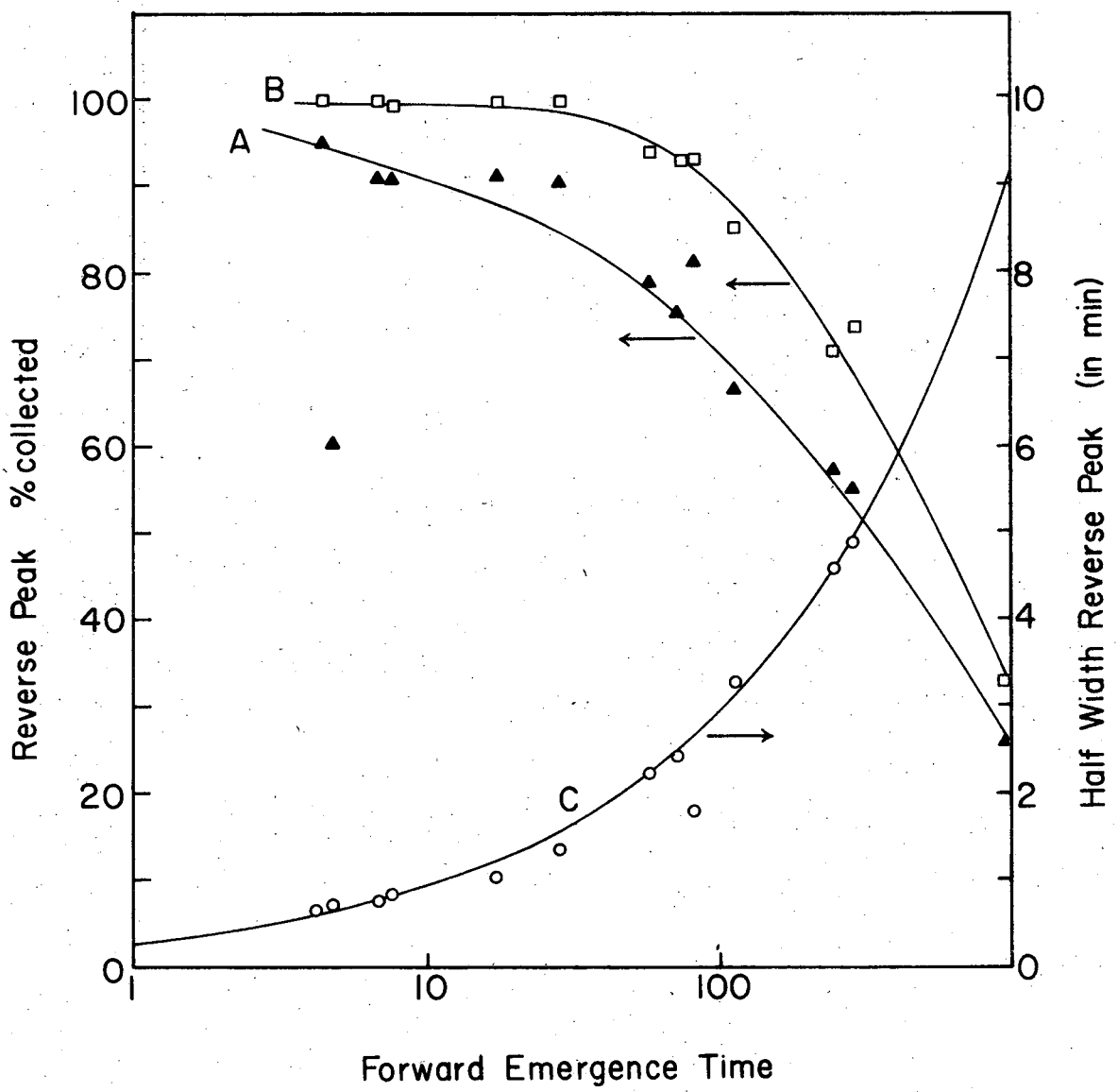


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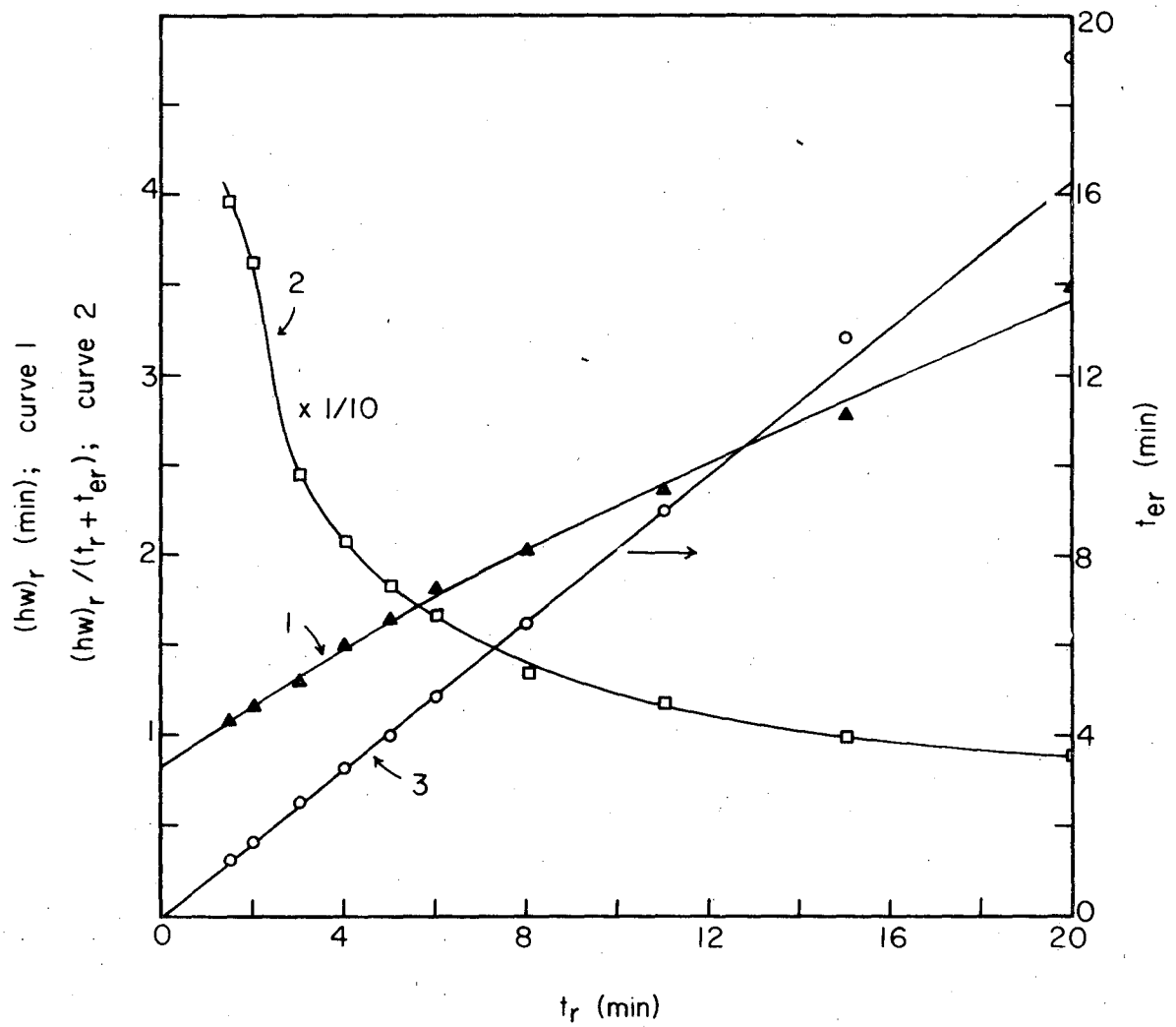
Fig. 2



XBL 7112-1798

Fig. 3

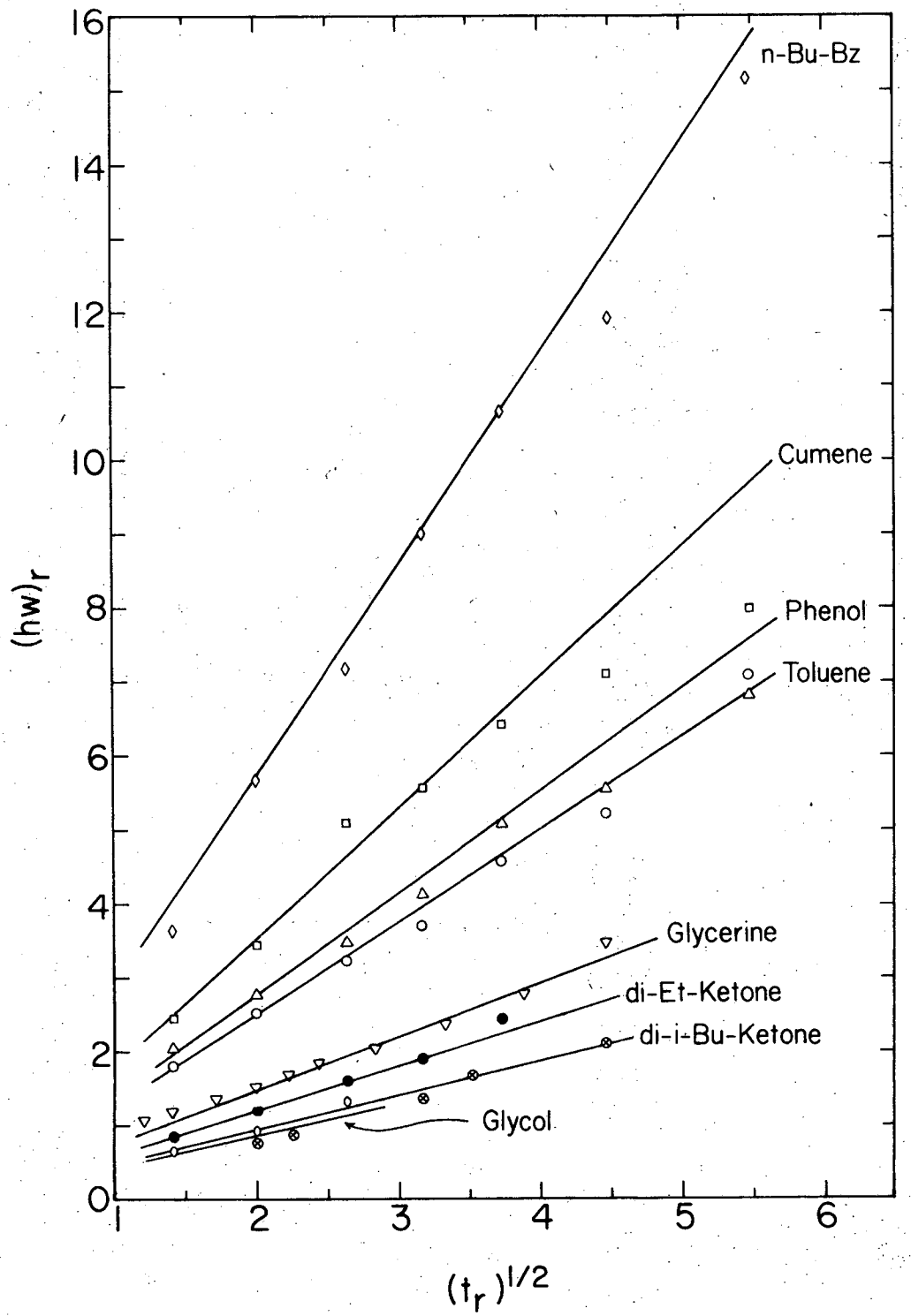


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Fig. 4

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