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Bonnie Harkins[a][b]; Tom L. Boehm[a]; David J. Wilson[a]
[a] DEPARTMENT OF CHEMISTRY, VANDERBILT UNIVERSITY, NASHVILLE, TENNESSEE [b] Hume-Fogg Academic High School, Nashville, Tennessee

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Removal of Refractory Organics by Aeration. VIII. Air Stripping of Benzene Derivatives

BONNIE HARKINS,* TOM L. BOEHM, and DAVID J. WILSON†

DEPARTMENT OF CHEMISTRY
VANDERBILT UNIVERSITY
NASHVILLE, TENNESSEE 37235

Abstract

The removal of several benzene derivatives from water was carried out by bubble column aeration. The presence of NaCl enhanced removal rates, while the presence of alcohols decreased them. Mixtures of two hydrophobic solutes are removed by aeration as if each were present alone. The use of the equilibrium assumption for mass transport between phases seems to be reasonably (but not perfectly) satisfactory. Henry's law constants calculated from aeration data are in fairly good agreement with those calculated from vapor pressure and solubility data. Toluene, ethylbenzene, *p*-xylene, chlorobenzene, *p*-dichlorobenzene, styrene, benzene, and 3-pentanone were studied. As predicted, the ketone is not removed by aeration at a significant rate. Removal rates of the other compounds are reasonably rapid, with Henry's law constants in the range of roughly 0.1 to 0.3 (dimensionless).

INTRODUCTION

The EPA's National Organics Reconnaissance Survey, prepared in 1974, concluded that trihalomethanes were common in chlorinated waters and that a large number of organic compounds from industry and agriculture were present in the raw and finished drinking waters of the 80 American cities included in the study (*1*). Since that time the problem has been found to be even more extensive and complex, with widespread

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*Present address: Hume-Fogg Academic High School, Nashville, Tennessee.
†To whom correspondence should be addressed.

contamination of groundwater from leaking underground storage tanks and from chemical waste disposal sites (*2*, *3*). In some areas, cleanup and reinjection of contaminated groundwater has been mandated (*4*).

The removal of volatile organics from water by sparging with air has been practiced for many years; at bench scale, it is the heart of the "purge and trap" method introduced by Bellar and Lichtenberg for determining trace levels of volatile organics (*5*). It is also the basis for closed-loop stripping (*6*, *7*).

Aeration techniques are by no means universally applicable to the removal of volatile organics; if the solubility of the compound to be removed is high, then its removal will be inefficient, even though it may be extremely volatile. For this reason EPA has concluded that granular activated carbon (GAC) treatment is preferable to aeration for the removal of trihalomethanes (THM's) (*8*); a National Research Council report (*9*) agreed that substantial air:water ratios were required, but concluded that aeration did appear to be a technically feasible method of THM control. Trussell and Trussell (*10*) subsequently noted that stripping in packed towers was found to be quite efficient, and that reasonable minimum air:water ratios were required for the effective removal of chloroform, 1,2-dichloroethane, and *cis*-1,2-dichloroethylene.

Quite extensive studies on the removal of volatile chlorinated organics were carried out by Roberts and his students. Their study of mass transfer and phase equilibria of a number of violatile chlorinated solvents in a bubble column is of particular relevance to our work here (*11*). They found a slight but significant dependence of Henry's law constants on turbulence in the column, indicating that the assumption of equilibrium-controlled mass transport is not perfect. They also found that Henry's law constants calculated for chloroform and for 1,1,1-trichloroethane aeration data show substantial deviations from those estimated from vapor pressure and solubility data. Much of the work of this group has been concerned mainly with the removal of chlorinated volatile organics by surface aeration (*12–17*).

A number of other workers published on the removal of volatile organics by aeration (*18–21*); our list of references is representative, not complete. Two recent papers address the design of cost-efficient air stripping installations (*22*, *23*).

We have investigated the removal of 1,1,1-trichloroethane (*24*), naphthalene and phenanthrene (*25*), and ammonia (*26*) by aeration in bubble columns. The removal rate of naphthalene and phenanthrene is enhanced by the presence of added salt, but decreases slightly by the presence of acetone or ethanol.

In the present work we investigate the removal by aeration of several benzene derivatives and of 3-pentanone. The effects of added NaCl and of alcohols are determined, and the removal of mixtures of two volatile hydrophobic organic solvents is studied. Henry's law constants are estimated from vapor pressure and solubility data; these are compared with Henry's law constants obtained from the aeration results.

THEORY

An apparently adequate analysis of batch scale aeration is quite simple; we proceed as follows. Let us assume that the aeration vessel can be described as a perfectly stirred tank, and that the contact time between the liquid and the air bubbles from the sparger is sufficiently long that the liquid and vapor phases are equilibrated with respect to volatile solute transport.

Let $m(t)$ = mass of solute at time t in the solution being treated, g
V_l = volume of solution being treated, L
v_a = airflow rate, L/min
c_l = solute concentration in the solution, g/L
c_a = solute concentration in the vapor phase in equilibrium with the solution, g/L
K = Henry's law constant for the solute, defined by $c_a = Kc_l$

Then

$$c_l V_l = m \tag{1}$$

and

$$-dm = v_a K c_l dt \tag{2}$$

which yield

$$\frac{dm}{m} = \frac{-Kv_a}{V_l} dt \tag{3}$$

from which

$$m(t) = m_0 \exp(-Kv_a t/V_l) \tag{4}$$

or

$$c_l(t) = c_l(0) \exp(-Kv_a t/V_l) \tag{5}$$

For a continuous-flow apparatus operating in steady-state conditions, we obtain

$$v_{\mathrm{infl}}c_{\mathrm{infl}} = v_{\mathrm{infl}}c_{\mathrm{effl}} + v_a c_a \tag{6}$$

where v_{infl} = influent flow rate
c_{infl} = influent solute concentration
c_{effl} = effluent solute concentration

Equation (6) and Henry's law lead to an expression for the effluent solute concentration,

$$c_{\mathrm{effl}} = \frac{c_{\mathrm{infl}}}{1 + V_a K / v_{\mathrm{infl}}} \tag{7}$$

If the apparatus is not operating in the steady-state mode, the governing equation can readily be shown to be

$$v_l(t)\frac{dc_{\mathrm{effl}}}{dt} = -(v_{\mathrm{infl}} + v_a K)c_{\mathrm{effl}} + v_{\mathrm{infl}}c_{\mathrm{infl}} \tag{8}$$

where the flow rates of air and water and the influent solute concentration may vary with time, and were

$$v_l(t) = v_l(0) + \int_0^t (v_{\mathrm{infl}} - v_{\mathrm{effl}})dt \tag{9}$$

If $v_{\mathrm{infl}} = v_{\mathrm{effl}}$ = constant, and v_a and c_{infl} are also held constant, then the solution to Eq. (8) is

$$c_{\mathrm{effl}}(t) = \left[c_{\mathrm{effl}}(0) - \frac{v_{\mathrm{infl}}c_{\mathrm{infl}}}{v_{\mathrm{infl}} + v_a K}\right]\exp\left(\frac{-v_{\mathrm{infl}} + v_a K}{V_l}t\right) + \frac{v_{\mathrm{infl}}c_{\mathrm{infl}}}{v_{\mathrm{infl}} + v_a K} \tag{10}$$

For many volatile solutes it is possible to estimate Henry's law constants from vapor pressure and solubility data; the equation is

$$K = \frac{0.01603P_e}{c_s T} \tag{11}$$

where P_e = equilibrium vapor pressure (mmHg) at temperature T

T = temperature, °K
c_s = solute solubility in water (mol/L) at temperature T

The results of such calculations for a number of benzene derivatives and for 3-pentanone are given in Table 1. In order to remove x percent of a given volatile solute, the ratio of the volume of air required to the volume of water to be treated must be

$$V_{\text{air}}/V_l = K^{-1} \log_e \left(\frac{100}{100 - x} \right) \tag{12}$$

Thus, if Henry's law constant is 0.3 and 95% removal is desired, the air-to-water volume ratio needed is 10.

In order for these calculations to be relevent to actual industrial wastes, the presence of other solutes (such as salts, alcohols, etc.) must not change the Henry's law constants significantly. One of the points to be explored here is the extent to which this is true. A second is the validity of the assumption that local equilibrium exists with respect to mass transfer between phases and that the aerator may be regarded as perfectly stirred.

TABLE 1
Henry's Law Constants for Several Volatile Organic Compounds in Aqueous Solution at 25°C

Compound	Molecular weight	Vapor pressure (mmHg)[a]	Solubility (g/L)[b]	K
Benzene	78.11	95.07	1.80	.222
Toluene	92.13	28.38	0.50	.281
m-Xylene	106.16	8.34	0.196	.243
p-Xylene	106.16	8.77	0.198	.253
Ethylbenzene	106.16	9.66	0.18	.307
Styrene	104.14	7.37	—	—
Chlorobenzene	112.56	11.64	0.38	.186
p-Dichlorobenzene	147.01	1.99	0.079	.199
3-Pentanone	86.13	16.78	48.1	.0016

[a] From C. D. Hodgman (ed.), *Handbook of Chemistry and Physics*, 42nd ed., CRC Press, Boca Raton, Florida, 1960.

[b] From H. Stephen and T. Stephen, *Solubilities of Inorganic and Organic Compounds*, Macmillan, New York, 1963.

EXPERIMENTAL

The aerator consisted of a glass column 55 cm high by 6.5 cm inside diameter with a fine glass frit fused into the bottom through which air was dispersed. A drain just above the frit permitted sampling. After a run was started, the top of the column was closed with a large rubber stopper and the exhaust gas from the column could be sent to a soap-film flowmeter or to a small burner in which the organic vapors were destroyed. House air was used at a pressure of about 5 psig; it was passed through a glass wool-packed column and a humidifier, and its flow was regulated with a micrometer needle valve.

Organic solvents were obtained from Fisher (lab grade) or Eastman, NaCl was from Fisher, and ethanol was 200 proof from Aaper. Stock solutions of the volatile organic compounds were made up at concentrations of 100 or 400 mg/L, depending on the solubility of the compound, by pipetting the required quantity into 3.00 L of water, capping, and stirring magnetically for several hours.

Runs were made by adding the required volume(s) of stock solution(s) to a 1-L cylinder, adding any desired additional solute (NaCl or alcohol), and diluting to 1.00 L. The air flow rate was adjusted by the desired value, the solution to be treated was added to the column, and the rubber stopper at the top of the column firmly inserted. Samples were taken, after the sampling drain was purged, at 5-min intervals.

Analyses were done spectrophotometrically on a Cary Model 14 recording vis-UV spectrophotometer; spectra were recorded over the range 200–350 nm. Five-centimeter cells were used for most of the runs; 1-cm cells were used for one of the styrene runs. Calibration curves were made for each compound at the wavelengths of two or more of its absorption maxima, and Beer's law was found to be followed. Least-squares straight lines were fitted to the data, and these were used to calculate concentrations from absorbancies where a single absorbing solute was present.

In cases where mixtures were used, the absorption spectra overlapped and concentrations were calculated by the following procedure.

$$I_1 = \varepsilon_{1a} l c_a + \varepsilon_{1b} l c_b \tag{13}$$

$$I_2 = \varepsilon_{2a} l c_a + \varepsilon_{2b} l c_b \tag{14}$$

where I_i = corrected absorbancy at wavelength λ_b, $i = 1, 2$
ε_{ia} = extinction coefficient of Compound *a* at wavelength λ_i
ε_{ib} = extinction coefficient of Compound *b* at wavelength λ_i

l = cell path length (5.00 cm)
c_a = concentration of Compound a
c_b = concentration of Compound b

The concentrations are then given by

$$c_a = (\varepsilon_{2b}I_1 - \varepsilon_{1b}I_2)/D \tag{15}$$

$$c_b = (\varepsilon_{1a}I_2 - \varepsilon_{2a}I_1)/D \tag{16}$$

$$D = l(\varepsilon_{1a}\varepsilon_{2b} - \varepsilon_{2a}\varepsilon_{1b}) \tag{17}$$

In calculating concentrations by this procedure, the absorbancies were corrected by subtracting the absorbancy of a solvent blank from the measured absorbancy of the sample at each of the two wavelengths used. This corrects for a slight mismatch in optical properties of the two cells used in this double-beam instrument.

No interference resulted from the presence of either alcohols or NaCl, since the absorption bands of these occur at substantially lower wavelengths than were used to monitor the aromatic compounds studied here.

The mathematical model used predicts that the removal rates of these volatile organics should be first-order with time, so that plots of $\log_{10} c_1(t)/c_1(0)$ should be linear.

$$\log_{10}[c_1(t)/c_1(0)] = -(Kv_a/2.3026V_l)t \tag{18}$$

from which it is evident that removal rates should be proportional to v_a and to K. The experimental data were fitted by linear least-squares to Eq. (18) to obtain values of $Kv_a/2.3026V_l$, from which values of the Henry's law constants could be calculated for comparison with those given in Table 1.

RESULTS

A typical plot of log 10 $c_1(t)$ versus t is shown in Fig. 1; the solution initially contained 100 mg/L of benzene. The plot indicates that the removal is indeed first order. This was found to be the case in all of the runs made except those for 3-pentanone, for which the removal rate was too slow be measured accurately, as expected from the extremely small Henry's law constant given for this compound in Table 1.

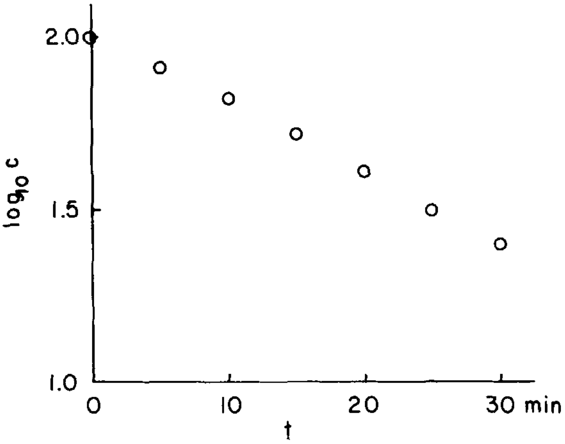

FIG. 1. Log$_{10}$ $c(t)$ versus time (min) for the removal of benzene from water by aeration. Initial benzene concentration is 100 mg/L; airflow rate = 0.1935 L/min; charge volume = 1.00 L; T = 26°C. The points are fitted by the equation $\log_{10} c(t) = -(0.0202 \pm 0.0003)t + (2.012 \pm 0.006)$.

The results of the various runs were summarized by calculating the effective Henry's law constants obtained from Eq. (18); if the slope of a plot of $\log_{10} c_l(t)$ is defined as $-b$, then one has

$$K = 2.3026 V_l b / v_a \quad (19)$$

The results for all of the runs made are listed in Table 2.

The dependence of removal rate for toluene on airflow rate is shown in Fig. 2; the results indicate that removal rates are proportional to the airflow rate, as required by the model. The data are fitted by the equation

$$b = (0.1224 \pm 0.0028 L^{-1}) v_a + (2.9 \pm 6.3 \text{ min}^{-1}) \times 10^{-4} \quad (20)$$

Examination of the Henry's law constants calculated for these data, however, indicate a slight decrease of K values with increasing airflow rates; this may represent a slight breakdown in the equilibrium assumption as the bubbles increase in size with increasing airflow rate. This result appears to be consistent with Roberts's earlier findings for chlorinated organic solvents (*11*).

The effect of increasing salt concentration is displayed in Fig. 3; the

TABLE 2

Results of Aeration Runs Made. V_l = 1.00 L, T = 25°C in all cases

Solute(s)	v_a (L/min)	Conditions	b (min^{-1})	K
Toluene	.122	100 mg/L toluene	.0175	.330
Toluene	.193	100 mg/L toluene	.0232	.277
Toluene	.194	100 mg/L toluene	.0222	.265
Toluene	.0896	100 mg/L toluene	.0111	.287
Toluene	.322	100 mg/L toluene	.0404	.289
Toluene	.201	100 mg/L toluene, 5% ethanol	.0216	.247
Toluene	.205	100 mg/L toluene, 10% ethanol	.0204	.229
Toluene	.217	100 mg/L toluene, 5% methanol	.0245	.259
Toluene	.218	100 mg/L toluene, 10% methanol	.0215	.227
Toluene	.217	100 mg/L toluene, 5% *n*-propanol	.0214	.226
Toluene	.216	100 mg/L toluene, 10% *n*-propanol	.0182	.194
Toluene	.194	100 mg/L toluene, 2.5% NaCl	.0293	.349
Toluene	.226	100 mg/L toluene, 5% NaCl	.0364	.371
Toluene	.194	100 mg/L toluene, 5% NaCl	.0327	.390
Toluene	.200	100 mg/L toluene, 7.5% NaCl	.0409	.471
Toluene	.230	100 mg/L toluene, 7.5% NaCl	.0471	.471
Toluene, 3-pentanone	.204	100 mg/L of both solutes	.0255 (toluene) 0 (3-pentanone)	.287 0
p-Xylene	.199	100 mg/L	.0279	.323
Ethylbenzene	.199	100 mg/L	.0308	.357
Toluene, *p*-xylene	.210	100 mg/L toluene, 25 mg/L *p*-xylene	.0276 (toluene) .0377 (*p*-xylene)	.302 .370
Toluene, ethylbenzene	.216	100 mg/L toluene, 75 mg/L ethylbenzene	.0255[a]	.272[a]
3-Pentanone	.216	100 mg/L 3-pentanone	0	0
Benzene	.194	100 mg/L benzene	.0202	.240
Chlorobenzene	.185	100 mg/L chlorobenzene	.0112	.139
p-Dichlorobenzene	.287	79 mg/L *p*-dichlorobenzene (sat.)	.0126	.101
Styrene	.313	100 mg/L styrene	.0169	.124
Styrene	.307	20 mg/L styrene	.0159	.119
Styrene	.312	4 mg/L styrene	.0156	.115
Styrene	.303	4 mg/L styrene	.0150	.114
Styrene	.303	4 mg/L styrene, 5% (v/v) ethanol	.0145	.111
Styrene	.303	4 mg/L styrene 10% (v/v) ethanol	.0125	.095

[a] The spectra of toluene and ethylbenzene are so similar that it is not possible to resolve the compounds.

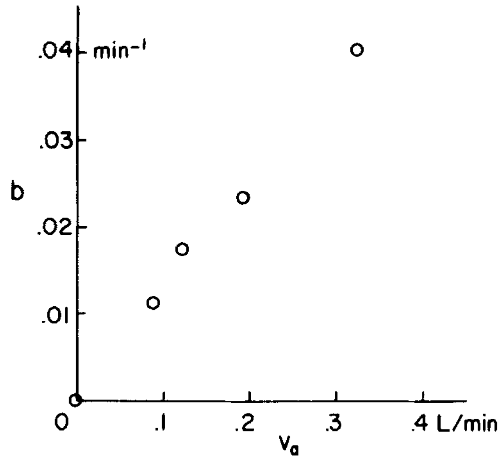

FIG. 2. The parameter *b* versus airflow rate. Charge volume = 1.00 L; initially the solutions contained 100 mg/L of toluene; T = 26°C. The removal rate appears to be proportional to the airflow rate, in agreement with the simple theory.

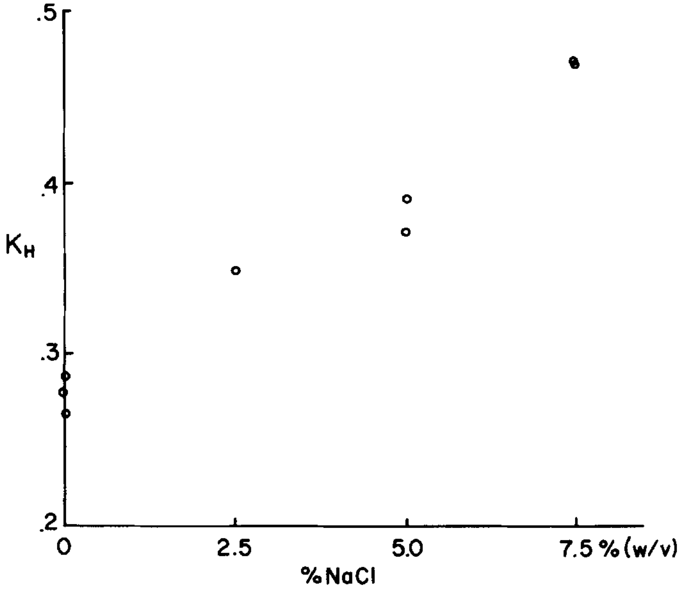

FIG. 3. Henry's law constants K versus NaCl concentration (% by weight) for toluene. Initial toluene concentration = 100 mg/L; airflow rates were approximately 200 mL/min; charge volume = 1.00 L; T = 26°C.

Henry's law constant for toluene increases very markedly with increasing NaCl concentration. The plot is fitted by the equation

$$K = (0.275 \pm 0.005) + (0.0248 \pm 0.0012\%^{-1}) \times (\%\mathrm{NaCl})$$

Evidently there is a "salting out" effect here, as was seen earlier with the solvent sublation of naphthalene (*25*). Presumably the binding of water molecules in the ion hydration shells makes them unavailable for dissolving toluene. This effect has long been used by chemists to recover organic compounds from aqueous solution.

Addition of alcohols (methanol, ethanol, *n*-propanol) to the solutions decreases the removal rate of toluene, as one might expect. As seen in Fig. 4, the effect is not so great as to render aeration techniques useless at alcohol concentrations below 10% (v/v), but it is sufficient so that it should be taken into account in considering the use of aeration. *n*-Propanol appears to be more effective than methanol or ethanol in reducing removal rates.

Most of the runs were made using an initial concentration of the hydrophobic organic of 100 mg/L. The very high molar absorptivity of styrene at 237 nm made it possible to make runs with initial styrene concentrations of 20 and 4 mg/L as well. The Henry's law constants calculated at initial styrene concentrations of 100, 20, and 4 mg/L were found not to be significantly different, as was expected. This suggests that one is justified in using the Henry's law constants for the other solvents, as well, at concentrations far below those used in this study. The data for aeration of 4 mg/L styrene in the presence of 5 and 10% ethanol indicate that ethanol decreases the Henry's law constant somewhat; a least squares fit of the data gives

$$K = (0.116 \pm 0.001) - (1.8 \pm 0.2) \times 10^{-3} \times (\%\mathrm{EtOH})$$

Comparison of Henry's law constants calculated from vapor pressure and solubility data with those calculated from the aeration data is made in Table 3. There do not appear to be major differences except for the two chlorobenzenes; one is probably not justified in attempting to interpret the discrepancies in view of the probable uncertainties in the solubility data and the uncertainties of roughly 10% in our Henry's law constants calculated from the aeration data. The results indicate that use of vapor pressure and solubility data for compounds of this type should yield reasonably good values for the Henry's law constants.

The data on mixtures of volatile organics in Table 2 indicate that one can estimate removal rates for components in such mixtures as if each

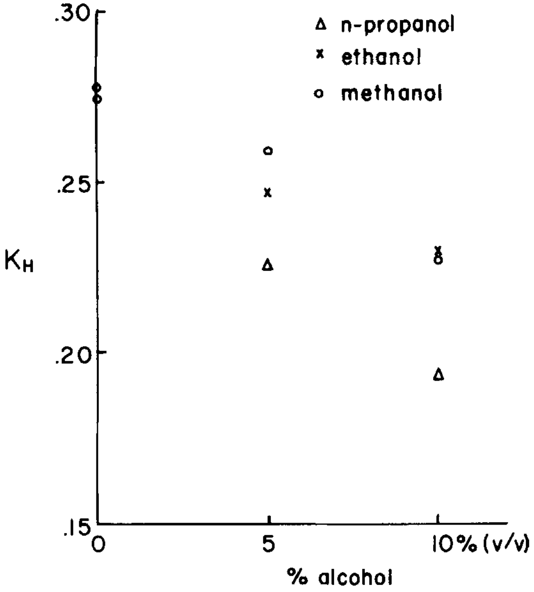

FIG. 4. Henry's law constants K versus alcohol concentrations (% by volume) for toluene. Alcohols used were methanol, ethanol, and *n*-propanol. Initial toluene concentration = 100 mg/L; airflow rates were approximately 200 mL/min; charge volume = 1.00 L; T = 26°C. Least squares straight lines were fitted to these points with the following results. For methanol:

$$K = (2.73 \pm 0.004) - (4.22 \pm 0.6) \times 10^{-3} \times \%\text{MeOH}$$

For ethanol:

$$K = (2.70 \pm 0.004) - (4.25 \pm 0.5) \times 10^{-3} \times \%\text{EtOH}$$

For *n*-propanol:

$$K = (0.270 \pm 0.004) - (7.82 \pm 0.5) \times 10^{-3} \times \%\ n\text{-PrOH}$$

The coefficients of determination were .894, .941, and .976, respectively.

TABLE 3
Comparison of Henry's Law Constants

Compound	K from Table 1 (vapor pressure, solubility)	K from Table 2 (aeration at ~.2 L/min)[a]
Toluene	.281	.277
Ethylbenzene	.307	.357
p-Xylene	.253	.323
Benzene	.222	.240
Chlorobenzene	.186	.139
p-Dichlorobenzene	.199	.101
3-Pentanone	.0016	0
Styrene	—	.119

[a] Uncertainties in these figures are approximately ±10%.

compound were being removed independently. The Henry's law constants calculated for the components in the mixtures appear to be comparable to those for the same components taken singly.

We conclude that these benzene derivatives can be removed from aqueous systems by aeration, with air/water volume ratios of roughly 10 being required to achieve 95% removal for most of the compounds. Salt enhances removal rates; alcohols decrease removal rates somewhat.

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