Measuring the Henry’s law constant for carbon dioxide and water with UV-visible absorption spectroscopy

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Abstract

The Henry’s law constant defines the solubility of a gas in a liquid solution. In this study, a new method for measuring the Henry’s law constant is described. This new colorimetric method is suited for gases which react with water to form acidic or basic solutions when they dissolve, and makes use of measuring the concentration of two forms of a colorimetric pH indicator. By measuring the concentration of the protonated and deprotonated forms of the indicator with UV-visible absorption spectroscopy, the concentration of the hydronium in solution was determined. After determining the hydronium concentration, the equilibrium expression for the dissolved gas reacting with water was solved to determine the concentration of the dissolved gas. The concentration of the dissolved gas and the measured partial pressure of the dissolved gas at equilibrium were then used to calculate the Henry’s law constant for the gas. The efficacy of the method is demonstrated by measuring the Henry’s law constant for carbon dioxide in water over a range of pressures (0.680-5.10 atm). The results obtained with this method are comparable to the value for the Henry’s law constant that have been previously reported via more traditional methods, and yielded values for the Henry’s law constant for carbon dioxide that ranged from $3.45 \times 10^{-2} \text{M atm}^{-1}$ to $3.99 \times 10^{-2} \text{M atm}^{-1}$.

**Keywords:** Henry’s law constant, carbon dioxide solubility, acid-base equilibrium, Absorption spectroscopy, Analytical method development
Introduction

The solubility of gases in liquids has long been an active area of research. An overview of traditional methods for determining the Henry’s law constant, such as measuring the ratio of vapor pressure and solubility of the gas, directly measuring the partial pressure and concentration of the dissolved gas, and measurement of partial pressure or concentration of dissolved gas via an equilibrium air-water exchange process has been previously detailed by Mackay and Shiu. Recently, several different methods such as batch air stripping, equilibrium portioning in closed a system, and the static headspace method have been developed to determine the Henry’s law constant. These recent method developments have tended to focus on determining the Henry’s law constant for hydrophobic compounds that are of interest because they are environmental pollutants.

Of the more recently developed methods, batch air stripping was developed to determine the Henry’s law constant for hydrophobic pollutants. In this method, the hydrophobic species was dissolved in water, and then isothermally removed via an inert gas flow. During the air-stripping process, the concentration of the hydrophobic species was monitored with UV-visible absorption spectroscopy. The Henry’s law constant was then determined utilizing the linear regression of the logarithm of concentration of the hydrophobic species versus time.

Equilibrium portioning in a closed system (EPICS) is a competing method for determining the Henry’s law constant. Equilibrium portioning was also primarily used for hydrophobic solutes. The equilibrium portioning in a closed system requires dissolving the hydrophobic species in water, while leaving a headspace in the sealed container. The UV-visible absorption intensity of the hydrophobic species was monitored over time. The system reached equilibrium when the absorption intensity had leveled off. The Henry’s law constant was then calculated utilizing the following equation:
\[ H = \left( \frac{V_{aq}}{V_{vap}} \right) \left( \frac{C_{A,aq,0} - C_{A,aq,eq}}{C_{A,aq,eq}} \right) \]  

(1)

where \( H \) is the Henry’s law constant, \( V_{aq} \) is the volume of the aqueous phase, \( V_{vap} \) is the volume of the vapor phase, \( C_{A,aq,0} \) is the initial concentration of species A in the aqueous phase, and \( C_{A,aq,eq} \) is the equilibrium concentration of species A in the aqueous phase. Rather than calculate the concentration, the EPICS method utilized the linear relationship between concentration and absorption intensity described by the Beer-Lambert law to rewrite equation 1 in terms of measured absorption intensity, rather than having to calculate concentrations from the absorption data.\(^5\)

Thus, the value of the Henry’s law constant can be calculated by the following equation:\(^5,6\)

\[ H = \left( \frac{V_{aq}}{V_{vap}} \right) \left( \frac{Abs_0 - Abs_{eq}}{Abs_{eq}} \right) \]  

(2)

where the term \( Abs_0 \) is the initial absorption intensity of species A in solution and \( Abs_{eq} \) is the equilibrium absorption intensity of species A in solution. By utilizing equation 2, the EPICS method allows for the calculation of the Henry’s law constant based on four experimentally parameters: volume of the aqueous phase, volume of the vapor phase, initial absorption intensity of the analyte in solution, and the equilibrium absorption intensity of the analyte in solution.\(^5\)

A third method developed for determining the Henry’s law constant was the static headspace method.\(^7,11\) The static headspace method differs from the other methods in that it utilized gas chromatography rather than UV-visible spectroscopy to make measurements. The method was slightly more complicated in that it involved preparing at least three samples in closed vials and allowing them to reach equilibrium with differing headspace to liquid volume ratios.\(^7\)

Samples from the headspace were then analyzed by gas chromatography, and the peak area of the hydrophobic compounds were determined. A plot was then constructed of reciprocal peak area versus the headspace to liquid volume ratio.\(^7\) After performing a linear regression, the Henry’s law constant was determined by dividing the slope of the linear regression by the y-intercept.\(^7\)
The static headspace method has been demonstrated to give values for the Henry’s law constant similar to those obtained via batch airstripping.\textsuperscript{11}

Previously developed methods have focused on determining the Henry’s law constant for hydrophobic compounds, the method described in this report is best suited for determining the Henry’s law constant for gases which react with water. Take for instance carbon dioxide, which reacts with water according to equation 3:\textsuperscript{12,13}

\begin{equation}
H_2O(\text{l}) + CO_2(\text{aq}) \rightleftharpoons H_2CO_3(\text{aq}) \rightleftharpoons H_3O^+(\text{aq}) + HCO_3^- \rightleftharpoons H_3O^+ + CO_3^{2-} \tag{3}
\end{equation}

The concentration of hydronium ions can be determined by addition a colorimetric indicator to the solution, and measuring the concentration of the protonated and deprotonated forms of the indicator with UV-visible absorption spectroscopy. Since the concentration of hydronium ions could be measured, then by solving the equilibrium expressions for equation 3, the concentration of dissolved carbon dioxide could be calculated. The concentration of dissolved carbon dioxide could be calculated from the following equilibrium expressions:

\begin{equation}
K_{a1} = \frac{[H_3O^+][HCO_3^-]}{[CO_2]} \tag{4}
\end{equation}

\begin{equation}
K_{a2} = \frac{[H_3O^+][CO_3^{2-}]}{HCO_3^-} \tag{5}
\end{equation}

Once the concentration of dissolved carbon dioxide was known for a given pressure ($P_{CO_2}$), the Henry’s law constant ($k_H$) could be calculated from the following equation:

\begin{equation}
[CO_2] = P_{CO_2}k_H \tag{6}
\end{equation}

A simple way to determine the hydronium concentration would be with a colorimetric pH indicator. With a colorimetric pH indicator, the concentration of the acidic and basic forms of the indicator can be accurately measured utilizing UV-visible absorption spectroscopy.\textsuperscript{14} While a pH meter could be used to determine the hydronium concentration, absorption spectroscopy provides the possibility to obtain an answer with a greater degree of accuracy as pH meters are often limited to two decimal places due to interference from sodium ions.\textsuperscript{15} The indicator should
have a noticeable color change over a pH range that overlaps with the hydronium ion concentration generated by the reactive gas. In this report, the feasibility of this colorimetric method is demonstrated for carbon dioxide, utilizing bromocresol green as the indicator.

**Experimental**

The bromocresol green indicator solution was prepared by dissolving 40 mg of bromocresol green sodium salt (Alfa Aesar) into 500.0 mL of deionized water. For each pressure that the value of the Henry’s law constant was measured, six trials were loaded into the pressure chamber (Emerald Welding, Greenwood, SC). In each sample, 25.00 mL of deionized water and 5.00 mL of the bromocresol green indicator solution were added to a 30 mL beaker. Each beaker was loaded into the pressure chamber, and the chamber was bolted closed. The chamber was pressurized with carbon dioxide (99%, Airgas) until the desired partial pressure of carbon dioxide was reached. Trials were conducted at partial pressures of carbon dioxide which ranged from 10 psi to 75 psi. In order to allow the samples to reach equilibrium, the chamber remained pressurized for 4 hr for each sample. The four hour time required to reach equilibrium was determined experimentally by measuring the dissolved carbon dioxide concentration. All trials were conducted at 20.0°C.

When the samples had reached equilibrium, the chamber was depressurized and opened. Samples were removed and the absorption spectrum of the bromocresol green solution was measured. All samples were measured in a 1 cm quartz cuvette. All absorption spectra were measured using a Cary 60 UV-visible spectrophotometer (Agilent).

In order to determine the molar extinction coefficient for the protonated (yellow) form of bromocresol green, a 1.00 M HCl solution was prepared. In a 100 mL volumetric flask, 10.00 mL of bromocresol green indicator solution was added, and the flask was diluted to the mark with the 1.00 M HCl solution, this ensured that bromocresol green indicator was fully protonated.
UV-visible absorption spectrum of the resulting yellow solution was measured. The molar extinction coefficient for the deprotonated (blue) form of the indicator was determined in a similar manner. In a 100 mL volumetric flask, 10.00 mL of bromocresol green indicator solution was added and diluted to the mark with a 1.00 M NaOH solution, this ensured that the bromocresol green was fully deprotonated. The UV-visible absorption spectrum of the resulting blue solution was measured.

**Results and Discussion**

Bromocresol green is a weak acid that is a common colorimetric pH indicator that changes color over the pH range of 3.8-5.4.\(^\text{16}\) The equilibrium between the protonated and deprotonated forms of bromocresol green can be generalized by the following equation:

\[
HIn\text{(aq)} + H_2O\text{(l)} \leftrightharpoons In^-\text{(aq)} + H_3O^+\text{(aq)} \quad (7)
\]

where HIn is the protonated form of the indicator and In\(^-\) is the deprotonated form of the indicator. Based on equation 6 the concentration of hydronium ions can be calculated from the following equation:

\[
K_a = \frac{[In^-][H_3O^+]}{[HIn]} \quad (8)
\]

The acid dissociation constant (\(K_a\)) for bromocresol green has been well characterized, and 1.603\(\times\)10\(^{-5}\) was used as the value for the calculation of the concentration of HIn and In\(^-\).\(^\text{14,16-18}\) In order to determine the concentration of the hydronium ion, the concentration of each form of the indicator must be accurately measured. Due to the relatively small equilibrium constants for equations 4 and 5 (4.20\(\times\)10\(^{-7}\) and 4.69\(\times\)10\(^{-11}\) respectively),\(^\text{12,19}\) the dissolved carbon dioxide when reacting with water should produce relatively small amounts of hydronium, bicarbonate,
and carbonate ions, therefore it should not be necessary to consider the activity of any species in solution.

If the peak in the absorption spectrum for each form of the indicator is sufficiently resolved, then it is possible to easily determine the concentration of both species HIn and In⁻ in solution simultaneously.

![Absorption Spectrum](image)

Figure 1. The absorption spectrum for each form of the indicator bromocresol green is shown. The protonated form, indicative of low pH, is shown in red. The deprotonated form, indicative of high pH, is shown in blue.

As can be seen in Figure 1, the peak absorption for the protonated (HIn, red spectrum) and deprotonated (In⁻, blue spectrum) forms of bromocresol green are well resolved. By preparing separate solutions with a pH far from the pKa, the equilibrium can be manipulated to force all of the bromocresol green into either HIn (very acidic conditions) or In⁻ (very basic conditions). This allows for the determination of the molar extinction coefficient, ε, using the Beer-Lambert law:

\[ A = b c \varepsilon \]  

Solving the Beer-Lambert law for a known concentration of bromocresol green yielded the values shown in Table 1 for the molar extinction coefficient of bromocresol green.
Table 1  The molar extinction coefficient for the protonated and deprotonated forms of bromocresol green at the peak wavelength from the absorption spectrum. The values reported represent the mean value for five trials.

| Indicator form and wavelength | Molar extinction coefficient, $\varepsilon$ (M$^{-1}$cm$^{-1}$) |
|------------------------------|-------------------------------------------------------------|
| $\text{HIn}_{444\text{nm}}$  | 14,900 ± 298                                               |
| $\text{In}^-_{444\text{nm}}$ | 2,550 ± 58.7                                               |
| $\text{HIn}_{617\text{nm}}$  | 0                                                          |
| $\text{In}^-_{617\text{nm}}$ | 33,900 ± 610                                               |

The Beer-Lambert law for a mixture of the two forms of bromocresol green can be used to determine the concentration of HIn and $\text{In}^-$ with the following equations:

\[
A_{444\text{nm}} = b_c \text{HIn} \varepsilon_{\text{HIn}_{444\text{nm}}} + b_c \text{In}^- \varepsilon_{\text{In}^-_{444\text{nm}}} \quad (10)
\]

\[
A_{617\text{nm}} = b_c \text{HIn} \varepsilon_{\text{HIn}_{617\text{nm}}} + b_c \text{In}^- \varepsilon_{\text{In}^-_{617\text{nm}}} \quad (11)
\]

Equation 10 describes the contribution of each species which absorbs light at 444 nm. The measured absorption at 444 nm is the result of the concentration of indicator in the form HIn multiplied by the molar extinction coefficient at 444 nm for the indicator in the form HIn multiplied by the path length of the cuvette (1 cm in the case of our measurements). The absorption at 444 nm of the deprotonated form of the indicator ($\text{In}^-$) must be accounted for as well. This was done by multiplying the concentration of the deprotonated indicator by its molar extinction coefficient at 444 nm and multiplying by the path length. These two products were added together to account for the absorption by both the protonated and deprotonated forms of the indicator at 444 nm. Equation 11 describes the contribution of form of the indicator for the absorption peak observed at 617 nm, and was the same as equation 10.
Equation 11 was simplified due to the lack of absorption by the protonated form of bromocresol green (Figure 1, Table 1). This yields the following equation to calculate the concentration of the deprotonated form of bromocresol green:

\[ A_{617\text{nm}} = b c_{\text{In}} \varepsilon_{\text{In}_{617\text{nm}}} \]  \hspace{0.5cm} (12)

Figure 2 shows how the absorption spectrum of bromocresol green changes once the indicator solution has reached equilibrium with pressurized carbon dioxide atmosphere. Prior to pressurization the absorption spectrum is dominated by the peak at 617 nm which represents the deprotonated form of the indicator (In\(^-\)). This is not unexpected because prior to pressurization and reaching equilibrium, the only sources of hydronium ions were the autoionization of water, and a very small amount of carbonic acid due to the carbon dioxide naturally present in the atmosphere. At normal atmospheric pressure, the partial pressure of carbon dioxide was \(4.07 \times 10^{-4}\) atm.\(^2\) Once equilibrium with the pressurized carbon dioxide atmosphere was reached, the intensity of the peak at 617 nm was greatly reduced (red spectrum); simultaneously the intensity at 444 nm (the peak associated with the protonated HIn form of the indicator) increased in intensity. Using the peak intensity values from the equilibrium absorption spectrum (Figure 2 red spectrum), equations 10 and 12 were solved to determine the concentration of the indicator in both the protonated and deprotonated forms.
Figure 2. The absorption spectrum of bromocresol green prior to adding carbon dioxide to the pressure chamber is shown in blue. The red spectrum is the absorption spectrum of bromocresol green after the system has reached equilibrium in a carbon dioxide enriched atmosphere.

With the concentration of each form of bromocresol green having been determined, equation 8 was solved and the concentration of hydronium ions in the solution was determined. By calculating the concentration of hydronium ions, it will be possible to calculate the concentration of dissolved carbon dioxide in the solution using the equilibrium expressions in equations 4 and 5.

The hydronium concentration calculated from equation 8 represents the total hydronium concentration generated by the reaction in equation 3. While carbonic acid is a diprotic acid, the contribution of hydronium from the equilibrium described in equation 5 will be negligible since the solution is obviously acidic, as indicated by the red curve of Figure 2. Thus, the concentration of hydronium generated by the reaction of bicarbonate with water will represent a negligible amount of the total hydronium present in solution. This allows for the concentration of the dissolved carbon dioxide to be calculated from equation 4.

The value of $K_{a1}$ for equation 4 was $4.20 \times 10^{-7}$.\textsuperscript{12,19} Stoichiometry, from equation 4, tells us that the moles of hydronium generated should be equal to the moles of bicarbonate ions.
generated. This simplifies equation 4 and allows for the determination of the concentration of dissolved carbon dioxide, the equation can be rearranged as follows:

$$\left[CO_2\right] = \frac{[H_3O^+]^2}{K_a} \quad (12)$$

With the concentration of dissolved carbon dioxide now determined, it was possible to calculate the Henry’s law constant for carbon dioxide in water by solving the equation for Henry’s law from equation 6.

Where P is the partial pressure of carbon dioxide, and $k_H$ is the Henry’s law constant (given in units of molL$^{-1}$atm$^{-1}$).

![Figure 3](image)

Figure 3. The graph shows the concentration of dissolved carbon dioxide over time for a partial pressure of carbon dioxide of 0.680 atm (10.0 psi). The system reached equilibrium when the concentration ceased to change.

In order to determine the time required for the system to reach equilibrium, the concentration of dissolved carbon dioxide was calculated for each time interval of pressurization. As shown in Figure 3, the concentration of dissolved carbon dioxide increases over time until it reaches a plateau. The plateau indicates that the system has reached equilibrium. For the lowest pressure studied, it took 4 hours for the system to reach equilibrium. While it is reasonable to assume that at higher pressures the system would require less time to reach equilibrium, all trials were held at pressure for four hours in order to ensure that all samples had reached equilibrium.
Table 2  The value of the Henry’s law constant (k_H) at partial pressures of carbon dioxide ranging from 0.680-5.10 atm is shown. The uncertainty in the reported value was +/- 1 standard deviation.

| Partial Pressure of Carbon Dioxide (atm) | k_H (M/atm)       |
|-----------------------------------------|-------------------|
| 0.680                                   | 3.45x10^{-2} ± 9.84x10^{-4} |
| 1.36                                    | 3.82x10^{-2} ± 6.51x10^{-4} |
| 2.04                                    | 3.99x10^{-2} ± 1.41x10^{-3} |
| 2.72                                    | 3.79x10^{-2} ± 9.73x10^{-4} |
| 3.40                                    | 3.77x10^{-2} ± 1.04x10^{-3} |
| 4.08                                    | 3.52x10^{-2} ± 1.21x10^{-3} |
| 4.76                                    | 3.47x10^{-2} ± 7.53x10^{-4} |
| 5.10                                    | 3.68x10^{-2} ± 1.19x10^{-3} |

The National Institute of Standards and Technology has published a range of values for the Henry’s law constant for carbon dioxide from $3.1x10^{-2}$ molL^{-1}atm^{-1} to $4.5x10^{-2}$molL^{-1}atm^{-1}, depending on the method used to make the measurement. In this study the partial pressure of carbon dioxide was varied between 0.680-5.10 atm (10.0-75.0 psi), and the value of the Henry’s law constant was calculated based on the process described above. The results of the calculation of Henry’s law constant based on spectroscopic measurements are shown in Table 2. For each partial pressure a minimum of 12 trials were conducted. As shown in Table 2, the measured value of the Henry’s law constant does not change over the range of pressures that was studied. The average value over all pressures measured was $3.69x10^{-2} ± 1.03x10^{-3}$ molL^{-1}atm^{-1}. It should be
noted that the standard deviation of the Henry’s Law constant is low, which indicated that the method of measurement was highly reproducible. The most significant source of error in the measurements resulted from the formation of bubbles in the cuvette, which have the effect of scattering the incident radiation in the spectrophotometer. This source of error was more likely to occur at higher partial pressures of carbon dioxide. Utilizing this method could also yield a more accurate estimation for the Henry’s law constant if the acid dissociation constant of the indicator (equation 8) and the equilibrium constant for the dissolved gas reacting with water (equation 4) were known more accurately.

Conclusions

By measuring the intensity of two forms of a colorimetric pH indicator, the Henry’s law constant was calculated. The colorimetric method yielded a value that is within the range published by the National Institute of Standards and Technology. The method was demonstrated with carbon dioxide and water, using bromocresol green as the indicator because bromocresol green has a Ka similar to the $K_a$ of carbonic acid. The colorimetric method would be applicable for any gas which reacted with water upon dissolution, such as ammonia, hydrogen sulfide, or sulfur dioxide. The appropriate indicator would differ for each gas, but the indicator should have a Ka value similar to the Ka value of the weak acid formed when the gas reacts with water. This colorimetric method also has the potential to yield a more accurate measurement of the Henry’s Law constant if more accurate data for equilibrium constants of the indicator and dissolution reaction could be collected.

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Figure Captions

Fig. 1  The absorption spectrum for each form of the indicator bromocresol green is shown.  The protonated form, indicative of low pH, is shown in red.  The deprotonated form, indicative of high pH, is shown in blue.

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Graphical Index
