A LAB EXPERIMENT TO INTRODUCE GAS/LIQUID SOLUBILITY

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The concept of the solubility of a gas in a liquid is familiar in many everyday ways. When we drink a bottle of carbonated beverage, we understand it contains dissolved carbon dioxide, and we know animals can live in oceans and rivers because oxygen dissolves in water and animal life depends on the dissolution of oxygen in blood. For chemical engineers, this everyday phenomenon is important from both practical and theoretical points of view.^[1, 2] From a practical point of view, this concept is used in the design of absorption columns where a gaseous mixture is separated by contact with suitable solvents that dissolve its components differently. Further, knowledge of gas solubility in water is important in the processes that control environmental distribution of contaminants, such as halogenated hydrocarbons. From the theoretical point of view, the solubility of gases in liquids is an excellent tool to investigate solute-solvent intermolecular forces in the liquid state, since solute-solute interactions are almost negligible.

In this paper we present a simplified version recently developed by Fonseca, et al,^[3] for experimental determination of the solubility of a gas in a liquid. This experiment is implemented in the chemical engineering department of Coimbra, and is a part of one of the third-year laboratory courses in phase equilibria domain. The experiments are carried out by groups of a maximum of three students during approximately four hours. The full report of the experiment is completed by the group at home and must be presented to the teacher



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the following week and discussed one week later. The report consists of the following sections: introduction, experiment objectives, background of the experiment, description of the apparatus, experimental procedure, and a discussion/results section. The analysis of uncertainties must also be done using the propagation law of errors, and the relative contribution of each measured variable uncertain in the final result—*i.e.*, the solubility—evaluated. The students must also include in the report a section of conclusions, where they can also present comments and recommendations for improvements.

THEORY

When a gas is in contact with a liquid it tends to dissolve in the liquid and the liquid evaporates until equilibrium is reached. Consider a binary mixture of components 1 and 2 at temperature T and pressure P, represented schematically in Figure 1. The component 2 is near or above its critical temperature, which means this component is a gas at temperature T. (A pure fluid above its critical temperature is called a gas, and a vapor below T_c). When the liquid-phase mole fraction, x_2 , is small and the equilibrium vapor-phase mole fraction, y_2 , is large (near unity), it is conventional to call species 2 a "dissolved gas," and to label the physical situation one of "gas solubility."^[4] Gas/liquid solubility is a particular case of vapor/liquid equilibrium; therefore, the classical treatment of this subject is similar in some important aspects.

Let's consider species 1 the major component of the liquid phase (the solvent) and species 2 the dissolved gas (the solute). The liquid phase mole fraction, x_2 , represents the solubility of gaseous solute 2 in liquid solvent 1. For component 2, the thermodynamic condition of phase equilibrium states:^[5]

$$\mathbf{f}_2^{\mathrm{G}} = \mathbf{f}_2^{\mathrm{L}},\tag{1}$$

where f_2^G and f_2^L are the fugacities of component 2 in the vapor and liquid phases, respectively. f_2^G is given by the expression:

$$\mathbf{f}_{2}^{\mathrm{G}} = \boldsymbol{\varphi}_{2} \mathbf{y}_{2} \mathbf{P}, \qquad (2)$$

where ϕ_2 represents the fugacity coefficient of component 2 in the vapor phase and P is the total pressure. f_2^L is represented by:



Figure 1. Schematic representation of gas/liquid solubility.

$$\mathbf{f}_{2}^{\mathrm{L}} = \gamma_{2} \mathbf{x}_{2} \mathbf{f}_{2}^{0} \,, \tag{3}$$

where γ_2 is the activity coefficient of component 2 in the liquid and \mathbf{f}_2^0 the standard-state fugacity of the same component, which is usually assumed to be the fugacity of the pure liquid at the temperature and pressure of equilibrium $\left(\mathbf{f}_2^0 = \mathbf{f}_2^{*,L}\right)$. The substitution of Eqs. (2) and (3) in Eq. (1) gives:

$$\mathbf{y}_2 \boldsymbol{\varphi}_2 \mathbf{P} = \boldsymbol{\gamma}_2 \mathbf{x}_2 \mathbf{f}_2^{*,\mathsf{L}} \,. \tag{4}$$

The above equation can be simplified if we consider the following assumptions:^[5]

(i) $\gamma_2 = 1$ ($\gamma_1 = 1$), *i.e.*, the liquid phase is an ideal solution;

(ii) $\varphi_2 = 1$ ($\varphi_2 = 1$), *i.e.*, the vapor phase is an ideal mixture;

(iii) $f_2^{*,L} = P_2^* (P_2^* \text{ is the pure vapor pressure of compo$ nent 2 at temperature T)*i.e.*, the effect of pressure on thefugacity of the pure liquid phase is negligible (Poynting $correction =1) at moderate pressures; and <math>\varphi_2^* = 1$, $(\varphi_2^* \text{ is the}$ fugacity coefficient of pure component 2) which is valid if P_2^* is low at temperature T.

Therefore Eq. (4) reduces to Raoult's law:

$$P_{2} = y_{2}P = x_{2}P_{2}^{*}, \qquad (5)$$

where P_2 represents the partial pressure of component 2.

The solubility x_2 , as given by Eq. (5), is called the "ideal" solubility of the gas. This expression states that the solubility is independent of the solvent and for a given gas, at a constant partial pressure, the solubility always decreases with rising temperature, which is not always true. Because of these disadvantages, the ideal solubility expression usually gives no more than a rough estimate of gas solubility.

The solubility of gas in a liquid is often proportional to its fugacity in the vapor phase. This situation is described by a more realistic expression, Henry's law:

$$f_2^{L} = H_{2,1} X_2, (6)$$

where $H_{2,1}$ is a constant that for a given solute and solvent depends only on temperature.

Again the assumptions leading to Eq. (6) can be readily recognized by comparing it with Eq. (4). The more important thing is to consider the constancy of the activity coefficients (independent of the composition), which means that Henry's law is only valid at infinite dilution. This can be stressed writing Eq. (6) in more convenient form:

$$H_{2,1} = \lim_{x_2 \to 0} \frac{f_2^{L}}{x_2},$$
 (7)

which is equivalent to

$$H_{2,1} = \lim_{x_2 \to 0} \frac{\varphi_2 y_2 P}{x_2},$$
 (8)

taking into account Eqs. (1) and (2).

Chemical Engineering Education

EXPERIMENTAL SECTION APPARATUS

The determination of the solubility of gas in a liquid is made using a volumetric method.^[6] The principle of this procedure is to bring a measured volume of liquid into contact with a known volume of gas at a given temperature and pressure. After equilibrium has been attained, the change in the gas volume yields the amount of gas dissolved in the liquid and hence the solubility. The solubility apparatus is shown schematically in Figure 2. The apparatus is housed in a water thermostat where the temperature is maintained constant using a temperature controller, TC. The temperature is measured with a precision thermometer graduated in 0.01 °C. The level of the thermostat bath can be adjusted using an elevator, E, in order to immerse the whole vacuum line.

The main features of the apparatus are: EQ, equilibrium vessel, (~50 cm³) where the dissolution of the gas takes place; GB, gas burette, which consists in a piston-cylinder arrangement; PT, pressure transducer, for total pressure readings; LA, linear actuator, which moves the piston in the gas burette; and PC, pressure controller.

PROCEDURE

The experimental procedure begins with the evacuation of the whole apparatus. After this two fundamental steps in any gas solubility measurement need to be performed:

(i) degassing of the solvent

(ii) dissolution of the gas

To accomplish step (i), the equilibrium vessel is removed from the line, lowering the level of the thermostat bath, and filled with a known amount (~ 6 g) of solvent. After this, it is again connected to the line and the position of the thermostat bath re-established. The stopcocks V2 and V4 are opened to degas the solvent in EQ during about 10 min. The magnetic stirrer must be on. This degassing procedure should be repeated two or three times until the measured pressure equals the solvent vapor pressure at the equilibrium temperature. Then EQ must be removed from the line in order to be weighed. After this procedure, EQ is connected again to the vacuum line.

Step (ii) begins with slowly opening stopcock V1, with V2 opened (V3 and V4 must be closed) to admit the gas to the equilibrium vessel. The total pressure is adjusted to ca 1 atm and, after this, stopcock V1 is closed and V3 is opened. This pressure acts as a reference value for the pressure controller, which commands the linear actuator, LA. As the gas dissolves the pressure decreases and this is detected by PT. The linear actuator, LA, drives the piston down the cylinder to maintain the pressure constant at the reference value. The number of encoder pulses is counted and displayed, and a conversion is made to determine the volume of gas displaced from the precision-bore tube that

comprises that cylinder of the burette. This volume represents the volume of the gas dissolved. The detailed experimental procedure is described in Appendix A.

ADDITIONAL INFORMATION FOR INSTRUCTORS

In this experiment it is important to use gases, such as CO_2 , N_2O or CH_3F , that are highly soluble in water. Methyl fluoride has the disadvantage of being very expensive. Less soluble gases $(O_2, N_2, \text{etc.})$ should not be used in this experiment since the time of dissolution increases substantially. For these kinds of gases an equilibrium vessel with a greater volume should be used.^[3] Other solvents such as primary alcohols (*e.g.*, methanol, ethanol, propan-1-ol, butan-1-ol) can also be used. They must have purities greater than 99.8 percent.

The accuracy of the method can be improved using lecture bottles of gases, which have higher purity (>99.5%) than other commercial gases. A pressure reducer should be used connected to the lecture bottle. The lecture bottles are quite appropriate to classroom experiments and must be used in these experiments. An adequate pressure reducer must be coupled to the lecture bottle. In the present experiments we have used a pressure reducer HSB – 280 – 5 (from PRAXAIR), but other reducers can be adapted depending on the mark of the lecture bottle. This allows a safe use of the lecture bottles. The linear actuator, LA, consists of a permanent magnetic DC motor, which drives a worm screw coupled to an optical encoder. The worm screw moves the piston inside the cylinder of the gas burette and the displacement is proportional to the number of encoder pulses (n_p). The proportionality constant



Figure 2. Solubility apparatus: TB, thermostated bath; TC, temperature controller; PT, pressure transducer; LA, linear actuator; PC, pressure controller; EQ, equilibrium vessel with connector; GB, gas burette; V1,...V4, high vacuum Teflon stopcocks; AGIT, magnetic stirrer; E, elevator.

The students must also include in the report a section of conclusions, where they can also present comments and recommendations for improvements.

indicated in the manual of the linear actuator should be given to the students. In the present apparatus, the displacement is obtained from: $\Delta h/(mm) = 2.20189 \times 10^4 n_p$. In this experiment we have used a pressure transducer from Honeywell, model PPT0015AWN2VA-A.

The thermostat bath can be built using Perspex or glass (71 $\times 26 \times 38$ cm) where a thermostatic control unit is immersed. The whole setup costs about 1,800 euros.

CALCULATIONS

Solubility Calculation

To simplify the treatment of the raw data we consider some assumptions: neglect the volume change of the liquid sample during saturation and ideal solution behavior.

The raw data obtained from experiment are: n_p , the number of encoder pulses from the linear actuator, the equilibrium temperature, T, the reference pressure, P, and m_1 , the mass of solvent. The number of encoder pulses is converted in Δh , the displacement of the piston, using a conversion factor indicated in the user manual of the pressure controller. The change of the volume of gas in the gas burette due to gas dissolution is obtained from:

$$\Delta \mathbf{V} = \pi \mathbf{r}^2 \Delta \mathbf{h},\tag{9}$$

where r represents the internal radius of the gas burette. The quantity of the gas absorbed in the liquid, n_2 (in moles), can be obtained from ΔV using the equation,

$$PV_{mix}^{G} / (RT) = 1 + B_{mix}RT / P,$$
 (10)

where V_{mix}^{G} represents the molar volume in the vapor phase and B_{mix} the second virial coefficient of the binary mixture, given by the expression,

$$\mathbf{B}_{\rm mix} = \mathbf{y}_1^2 \mathbf{B}_{11} + \mathbf{y}_2^2 \mathbf{B}_{22} + 2\mathbf{y}_1 \mathbf{y}_2 \mathbf{B}_{12}, \qquad (11)$$

where B_{11} , B_{22} , and B_{12} are the second virial coefficients of pure components 1 and 2, and the second cross coefficient, respectively.

Substituting, in Eq. (10), $V_{mix}^G = \Delta V / n^G$, where n^G represents the total number of moles contained in the V volume, one obtains,

$$\Delta V/n^{G} = (RT/P) + B_{mix}, \qquad (12)$$

which multiplied by $(1/y_2)$ gives,

$$\mathbf{n}_2 = \mathbf{y}_2 \mathbf{P} \Delta \mathbf{V} / (\mathbf{R} \mathbf{T} + \mathbf{B}_{\text{mix}} \mathbf{P}). \tag{13}$$

The value of the solubility,
$$x_2$$
, is then obtained from:

$$\mathbf{x}_{2} = \mathbf{n}_{2} / (\mathbf{n}_{1} + \mathbf{n}_{2}),$$
 (14)

where n_1 and n_2 represent the amount of solvent and solute in moles in the liquid phase, respectively. The n_1 is obtained directly from $n_1 = m_1 / M_1$. Since we need to know y_2 to obtain n_2 from Eq. (13), this calculation requires an iterative procedure. The calculations begin with estimates of the vapour and liquid phases obtained from Raoult and Dalton laws. In the following iteractions, these compositions are improved using Eqs. (13) and (14) and the following expression:

$$y_{2} = 1 - (1 - x_{2}) \frac{P_{1}^{*} \varphi_{1}^{*}}{P \varphi_{1}}, \qquad (15)$$

which results from the thermodynamic condition of phase equilibrium written for the solvent.

The φ_1 is the fugacity coefficient of solvent in the vapor phase, which is given by,

$$\varphi_1 = \exp\left\{ (P / RT) \left[B_{11} + y_2^2 \left(2B_{12} - B_{22} - B_{11} \right) \right] \right\},$$
 (16)

and ϕ_1^* represents the fugacity coefficient of pure solvent in saturation conditions obtained from,

$$\varphi_1^* = \exp\left[\left(\mathbf{B}_{11}\mathbf{P}_1^* / (\mathbf{RT})\right)\right],\tag{17}$$

where P_1^* is the vapor pressure of pure component 1 at the equilibrium temperature, which can be obtained using a vapor pressure equation.



Figure 3. Solubility of carbon dioxide (•) and nitrous oxide (\blacktriangle) in water. The curves were obtained from Eq.(18).

The calculation ends when convergence is obtained between two consecutive x_2 values. The determination of Henry's constant, $H_{2,1}$, is then straightforward from Eq. (8). All the solubilities found in this work were corrected to 1 atm partial pressure using Henry's law, since the literature values are referred to this pressure.

TABLE 1 Solubility of CO2 and N2O in water, expressed as mole fraction, x2 at a partial pressure P, = 101325 Pa. H2, is the Henry coefficient.					
Solute	T/K	x ₂ /10 ⁻⁴	x _{21it} /10 ⁻⁴	σ (%) ^(a)	H _{2.1} / (MPa)
CO2	290.27	7.70	7.61	1.1	131.7
	291.49	7.36	7.34	0.3	137.7
	292.11	7.22	7.21	0.2	140.4
	293.39	6.90	6.94	0.6	146.8
	294.58	6.65	6.71	0.9	152.3
	295.15	6.66	6.60	0.9	152.1
	296.19	6.36	6.42	0.8	159.2
	297.19	6.19	6.24	0.9	163.8
	298.39	5.98	6.04	1.0	169.4
	299.37	5.89	5.89	0.0	172.0
	300.15	5.80	5.77	0.5	174.8
	301.10	5.61	5.63	0.3	180.5
	302.13	5.43	5.48	1.1	186.7
	302.93	5.35	5.38	0.5	189.5
N ₂ O	290.36	5.49	5.53	0.6	184.4
	291.36	5.38	5.36	0.4	188.4
	292.36	5.17	5.19	0.4	196.1
	293.30	5.03	5.04	0.2	201.5
	294.20	4.93	4.90	0.5	205.5
	295.40	4.71	4.73	0.3	214.9
	296.15	4.66	4.63	0.8	217.2
	297.25	4.49	4.48	0.3	225.6
	298.13	4.38	4.37	0.2	231.5
	299.07	4.28	4.25	0.7	236.6
	300.16	4.13	4.12	0.2	245.2
	301.15	3.97	4.01	1.0	255.0
	302.15	3.84	3.90	1.7	263.9
	303.13	3.81	3.80	0.0	266.3

 $a^{(a)} \sigma(\%) = \begin{vmatrix} x_2 - x_{2lit} \\ x_{2lit} \\ this work and x_{2lit} is obtained from Reference 8. \end{cases}$

TABLE 2Parameters in the equation $R \ln x = A + B/T + C \ln T$					
Solute	A/(J K ⁻¹ mol ⁻¹)	$B/(J \text{ mol}^{-1})$	C/(J K ⁻¹ mol ⁻¹)	AAD(%)	
CO ₂	-2785.2	138287.6	396.617	0.5	
N ₂ O	890.1	-23763.0	-153.526	0.5	

ERROR ANALYSIS

The analysis of uncertainties or "errors" is a vital part of any scientific experiment. The instructor should stress that all measurements, however careful and scientific, are subject to some uncertainties. In this experiment the de-

termination of the uncertainty of the final result, the mole fraction solubility, provides use of the propagation law of errors.^[7] This law gives the relative magnitude of the uncertainties of the measured variables, *i.e.*, the student will be able to tell which of the experimental errors affect the solubility value more.

Appendix B presents an entrance form distributed to each group of students in order to guide them in treating the raw data and analyzing the uncertainties of the results. The data was obtained by one of these groups to determine the solubility of CO_2 in water, at the temperature 298.39 K.

RESULTS AND DISCUSSION

The experimental solubility data and the values reported in the literature for the systems CO_2/H_2O and N_2O/H_2O are shown in Table 1. The accuracy of the experimental method is found to be about 1 percent.

We have also determined the ideal solubilities of both gases at $P_2 = 101325$ Pa using Eq. (5). The vapor pressures of the pure components were obtained from the Wagner equation.^[9] The value determined for CO₂ is $x_2 = 1.57 \times 10^{-2}$ (T = 298.39 K) and for N₂O is $x_2 = 1.91 \times 10^{-2}$ (T = 298.13 K). These values are quite different from the experimental ones, since Raoult's law gives only a rough estimate of the solubility of a gas independent of the solvent.

The second virial coefficients were obtained from polynomial functions fitted to the experimental second virial coefficients taken from the Dymond and Smith compilation.^[10]

The dependence of the solubility on temperature has been represented by:

$$R \ln x_{2} = A + B / T + C \ln T, \qquad (18)$$

with the parameters fitted to the data by a least-squares method. The optimized parameters of Eq. (18) and the average absolute

deviation of x_2 , defined as,

$$AAD = (1/M)\Sigma \left[\left| x_2(exp) - x_2(calc) \right| / x_2(exp) \right] x 100 (19)$$

where M is the number of experimental points, are listed in Table 2 (previous page).

In Figure 3 (p. 150) we have plotted the experimental solubility data and the fitted Eq. (18) for each system. The agreement is good.

CONCLUSIONS

The apparatus used here to measure the solubility of gas in a liquid is simple and clearly illustrates this concept. It combines easy handling with automated data retrieval, leading to experimental results with reasonable accuracy for a pedagogical experiment.

The treatment of the raw data to obtain the solubilities is a good application and demonstration of some gas laws (Dalton's law of partial pressures, Raoult's and Henry's laws). This experiment also gives the opportunity to demonstrate real behavior of a gas (through the fugacity concept) to the students. The analysis of errors will allow evaluation of the relative magnitudes of the uncertainties within the measured variables and how they propagate to the final result, the solubility.

ACKNOWLEDGMENT

This work was carried out under Research Project POCTI/ EQU 44056/2002 financed by FCT—Fundação para a Ciência e Tecnologia (Portugal) and FEDER. We thank Professor Margarida F. Costa Gomes from the Laboratoire de Thermodynamique des Solutions et des Polymères, Université Blaise Pascal, Clermont-Ferrand, for her advice related to the assembly of the solubility apparatus.

NOMENCLATURE

A, B, and C parameters of Eq. (18)

$\mathbf{f}_2^{\mathrm{L}}$	fugacity of component 2 in the liquid phase [Pa]
$\mathbf{f}_{2}^{*,\mathrm{L}}$	fugacity of pure liquid 2 [Pa]
$ ilde{\mathbf{f}}_2^{ ext{G}}$	fugacity of component 2 in the vapor phase [Pa]
\mathbf{f}_{2}^{0}	standard-state fugacity of component 2 [Pa]
H_{21}	Henry constant of component 2 in solvent 1 [MPa]
n ₁	amount of solvent [mol]
n ₂	amount of solute [mol]
n	number of encoder pulses
P	equilibrium pressure [Pa]
P2	partial pressure of component 2 [Pa]
P_2^*	vapor pressure of pure component 2 [Pa]
P_1^*	vapor pressure of pure component 1 [Pa]
R	ideal gas constant [J mol ⁻¹ K ⁻¹]
Т	equilibrium temperature [K]
ΔV	volume of dissolved gas [m ³]
X ₂	mole fraction of component 2 in the liquid phase
y ₂	mole fraction of component 2 in the vapor phase

Greek symbols

- Δh displacement of the piston [m]
- φ_2 fugacity coefficient of component 2 in the vapor phase

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APPENDIX A

The experimental procedure to determine the solubility of a gas in a liquid using the apparatus shown in Figure 2 is as follows:

- 1) Connect the lecture bottle to the solubility apparatus by means of a pressure reducer.
- 2) With the valve of the lecture bottle closed, open the valve of the pressure reducer.
- Switch on the vacuum pump and open stopcocks V1, V3, and V4 with V2 closed. Evacuate the whole apparatus during 1 h.
- Close the stopcocks. Remove the equilibrium vessel from the line, lowering the level of the thermostat bath in order for the connector of the equilibrium vessel to be out of the water.
- 5) Introduce ~ 6 cm^3 of water in the equilibrium vessel.
- 6) Connect EQ again to the vacuum line and re-establish the initial position of the thermostat bath.
- 7) In order to degas the water, open V2 and V4. Switch on the stirrer. This procedure lasts about 30 min until the measured pressure equals the solvent vapor pressure at the equilibrium temperature.
- 8) Close stopcocks V2 and V4 and remove the equilibrium vessel again from the line.
- 9) Weigh the equilibrium vessel and then connect it to the vacuum line, re-establishing the level of water bath using the elevator.
- Open stopcock V2, (with V3 and V4 closed). Then slowly open stopcock V1 to admit the gas to the equilibrium vessel.

- 11) Slightly open the valve of the pressure reducer and then the valve of the lecture bottle in order to adjust the total pressure to ~ 1 atm (reference pressure).
- Close stopcock V1 and open V3. 12)
- 13)Switch on the magnetic stirrer to promote contact between the liquid and vapor phases.
- 14)The dissolution process lasts about 2 h. As the gas dissolves the pressure decreases, which is detected by PT. The piston comes slowly down to re-establish the reference pressure.
- Record the last constant value displayed by PC, which 15)is the number of pulses (n_n) of the linear actuator.
- 16) To convert n in displacement of the piston use the conversion factor indicated in the user manual of the linear actuator. The value obtained must be multiplied by the internal crosssection area of the cylindrical gas burette to obtain the volume of the dissolved gas (ΔV).

APPENDIX B

(The data presented in the following tables was obtained by a group of students.)

Entrance form for G/L solubility data

Gas: CO,

Solvent: water

Raw data: n_n is the number of encoder pulses; T is the equilibrium temperature; P is the reference pressure; m, is the mass of the solvent.

T / K	n _p	P/Pa	m ₁ / kg
298.39	82346	100070	0.007737

Treatment of the experimental data

The value of n_n must be converted in Δh , the dis-

placement of the piston, using the conversion factor indicated in the user manual of PC. Using Eq. (9) calculate ΔV , the volume of gas dissolved in the liquid.

Write a simple computer program to calculate the solubility of the gas and the Henry coefficient, at a partial pressure of 101325 Pa.

Results: x', represents the mole fraction solubility calculated with Eqs. (9-17); φ_1 the fugacity of the solvent in the vapor phase obtained from Eq. (16); φ_1^* the fugacity coefficient of pure solvent in saturation conditions, from Eq. (17); x_2 is the corrected solubility at partial pressure $P_2 = 101325$

T / K	x'2/10-4	ϕ_1	φ*	x ₂ / 10 ⁻⁴	H _{2,1} /(MPa)
298.39	5.88	0.9880	0.9985	5.98	169.4

Pa; $H_{2,1}$ is the Henry coefficient.

Error analysis

- What are the uncertainties of the measured variables?
- Using these uncertainties and the propagation law of errors, determine the uncertainty in the final result, the mole fraction solubility.
- Which uncertainty most affects the solubility value?
- Using your own results of solubility data and some of the results of other groups (at least 6 pairs of experimental data), make a fitting by a least squares method of the equation:

$$R \ln x_2 = A + B/T + C \ln T$$

to represent the temperature dependence of the solubility.

- Calculate the average absolute deviation of the fitting.
- Compare your solubility data with those of the literature. 🗖