Practical experiments

Course on "Chemical risk", labeling, MSDS, toxicity and safety behavior in the laboratory

Experiment 1. To determine the general expression for pressure drop in a linear tube.

Objective: This experiment will allow you to study pressure drop through different pipes fed with water

In an experimental part, you will measure pressure in the entrance pipe and the exit pipe at different flow rates to get pressure drop

In a calculation part, you will apply the non-dimensional analysis, a simple method fairly accurate for a wide variety of compounds, flow-rates and pipes

- Measure the pressure drop in smooth linear tubes of different diameters using the appropriate manometers for different flow rates

- Determine the two non-dimensional numbers, Reynolds number and Pressure Drop Coefficient, characteristic of fluid flow

- Point the data obtained on a Moody chart. Discuss about these results and this predictive method

- Carry out the same study using a rough sided tube. Use the moody chart to estimate the roughness

Experiment 2. Stirring. The determination of the relationship between the power number N_p and the Reynolds number Re.

Objective: The measurements carried out on this pilot stirrer allow the relationship linking the power number N_p to the Reynolds number Re to be determined.

It can be deduced that certain impellers are much more 'energy consuming' than others and that, depending on the phenomenon that one wishes to favorize, it is better to choose one type of impeller rather than another. This also allows one to optimize energy consumption.

- establish a protocol that will enable the measurement of the power number N_p to be made as a function of the Reynolds number.

- choose two sizes from the large groups of impellers provided (Rushton turbine or marine propeller). For each impeller, observe the behaviour of the fluid within the stirred tank for different values of speed. Measure the different values of power, speed and torque to determine the two non-dimensional numbers (Reynolds number and power number Np). What conclusions can you make?

Experiment 3. Ebulliometry. Liquid-vapor equilibrium.

Objective: This experiment will allow you to illustrate a liquid-vapor equilibria.

In an experimental part, you will work on an ebulliometer to find experimental datas (T, x,y) at atmospheric pressure or low pressure.

In a calculation part, from data to be found in the literature, you will be able to compute activity coefficients and adjust the parameters of Wilson's excess model for the binary system considered.

- Get data (T,x,y) for the liquid-vapor equilibrium of the binary water-methanol mixture with an ebulliometric experiment

- Using experimental data (T,x,y), and the equilibrium criterion : $f_i^l(T,P,x) = f_i^v(T,P,y)$, calculate the activity coefficients of water and methanol over the whole composition range

- From these coefficient values ,estimate the infinite dilution activity coefficient γ_i^∞

- Using this information, calculate the parameters of Wilson's excess model

- Verify its validity for the binary system considered

Experiment 4. Distillation of the binary water-methanol mixture at atmospheric pressure

- How to start the column and to choose parameters?

- Determination of the number of trays in total reflux

- Material and thermal balance

- Distillation of the binary mixture, respecting the specifications.

- optimize the separation of a methanol/water mixture by distillation to reach the purities required while minimizing energy expenditure

Experiment 5. Precipitation and filtration of adipic acid

- Prepare an aqueous solution of adipic acid

- Precipitation by adding sulphuric acid

- Put the mixture in a filtration cell

- perform the filtration at a constant pressure

- Determine the resistance of the cake and of the support using the filtration law and experimental values.

- Is the column well sized?

- Compare a precipitation and a crystallization

- Purify industrially a solid

Experiment 6. Kinetics

This practical involves 2 days in the laboratory for practical work and calculations:

1st day: t-butyl-chloride hydrolysis: acid quantification as kinetics measurement tool and its use to prove that the reaction is 1st order.

Ref : K.M. Doxsee, J.E. Hutchinson in Green Organic Chemistry , Brooks/cole Ed. 2004, p.167 (url : themalloryfamily.net/...%20**Hydrolysis**%20of%20t-Butyl%20**Chloride**.… for a pdf document 02/04/2014)

2nd day: Heck arylation of but-3-en-2-ol catalyzed by palladium: Use of quantitative GC analysis of the reaction medium to follow the conversion, to control the global order of the reaction, to prove that the two observed reactions are parallel, initiation to TON concept.

Ref : C. Gozzi, N. Bouzidi, J. Chem. Educ., 85 (2008) 1126-1128 and supporting infos.

Experiment 7. Chemical analysis of water for quality control.

Determination of main parameters in drinking water:

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Bâtiment Hubert Curien
B.P. 82077 - 69616 Villeurbanne cedex - France
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- Determination of pH and alkalinity (pHmetric titration); HCO_3^- ,

-Hardness : Ca^{2+} and Mg^{2+} (automatic titrator and calcium selective electrode) ,

-Conductivity,

- Flame photometry for the determination of Na+ and K+

- Ionic chromatography for Cl^- , NO_3^-, SO_4^{--}

Synthesis of all the results : Control of the ionic balance. Comparison of mineralization determined by addition of the values determined by each specific method, global approach by conductivity and dry residue.

Conclusion about drinkability or not of the sample.

Experiment 8. Quantification of polyaromatic hydrocarbons by UV spectroscopy

Spectrum, calibration curve, dilution of the sample. Sensitivity of the method. Results expressed in different units.

Quantification of polyaromatic hydrocarbons by UV spectroscopy Spectrum measurement, construction of the calibration curve, dilution of the sample in order to predict the analyte concentration using the calibration curve. Sensitivity of the method. Results expressed in different units (mg/l, mol/L, ppm).

Experiment 9. Project

The aim of this project is to study the kinetics of a saponification reaction in the laboratory scale and then to calculate and design the scale-up of the reaction to a 5L reactor.

This project is designed to help students become aware of the interconnection of the different chemistry domains. The objective is to successfully define the problem and the solutions of the scale-up of a chemical reaction.