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Short communication

Simple multipurpose apparatus for solubility measurement of solid solutes in liquids

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ABSTRACT

Solubility is one of the most basic chemical phenomena. It has a key role in understanding of major unit operations such as crystallization, solid–liquid extraction, and dissolution. In addition, solubility data is crucial for design, development, and operation of these processes. Therefore, solubility measurement of solid solutes in different solvents or solvent mixtures as a function of temperature can be of great significance to the university students and researchers. In this article, comparatively inexpensive apparatus for solubility measurement of solid solutes in pure or mixture of solvents, which can be easily assembled by using readily available equipment in the laboratory, is demonstrated. The proposed apparatus uses classical isothermal technique for measurement of solubility. Solubility of the naturally occurring antimalarial drug artemisinin was measured in *n*-hexane-ethyl acetate mixtures of varying composition at different temperatures to demonstrate the suitability and reliability of the proposed solubility measurement apparatus.

The proposed apparatus has been used to conduct laboratory exercises in the course "Industrial Separation Technology" offered to undergraduate students of chemical engineering program at University of Southern Denmark. The exercises included solubility measurement and cooling crystallization of salicylic acid from five different organic solvents and extraction of artemisinin from the leaves of the plant *Artemisia annua* by using different solvents. Performance of the apparatus during laboratory exercises was evaluated through the survey among students. The results showed the general positive feedback from students and revealed enhanced interest among students to understand the exercises through the simple design of the apparatus.

1. Introduction

The solubility phenomenon is very common to the daily life and it is frequently witnessed through the activities such as sweetening of tea with sugar, distribution and bioavailability of medicines in our body, removal of salt deposited at the bottom of kettle with hot water, and so forth. It also dictates several processes in nature such as weathering of rocks and contamination of water bodies with pollutants (Tomlinson, 1985). What dissolves what, to what extent, and at what temperature and pressure are pertinent questions that has great significance to the science and industrial applications? Ritonavir is an example of antiviral drug, which is used for the treatment of AIDS and illustrates the importance of solubility in the pharmaceutical industry. A batch of this drug had to be withdrawn from the market due

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to its conversion into a conformational polymorph, which had significantly lower solubility as compared to the desired polymorph (Bauer et al., 2001). There is a plentiful of literature reporting the solubility data for variety of solid solutes (IUPAC-NIST SOLUBILITY DATA SERIES, 2015). However, solubility data is mostly not available for the solute at the conditions of interest. This is especially true for mixed and non-aqueous solvents, and for systems involving impurities. Therefore, solubility measurement of solid solutes at required conditions becomes necessary and should be part of any chemical laboratory due to its widespread applications.

The measurement of solubility mainly concerns the determination of solute concentration in the solution at equilibrium. Thus, the procedure for measurement of solubility can be considered of two parts; firstly, the attainment of equilibrium and secondly, determination of solute concentration in the equilibrated solution. There are several methods mentioned in the literature for attainment of equilibrium (e.g., isothermal, polythermal techniques etc.) as well as determination of concentration of solute in the equilibrated solution (e.g., gravimetry, conductivity, turbidity, transmittance measurement etc.). Some of these techniques have been comprehensively summarized in a special volume by IUPAC dedicated to the experimental measurement of solubility (Hefter and Tomkins, 2003; Zimmerman, 1952). Most commonly used and recommended technique, classical isothermal technique, involves suspension of excess amount of solute in a solvent at constant temperature until the equilibrium is reached (Myerson, 2001). The procedure appears very simple but can be easily done wrongly resulting into large errors. The accuracy of solubility data obtained depends on several factors such as equilibration time, agitation, temperature control, interaction of solute–solvent system with ambient atmosphere, and analysis of saturated solution to determine the concentration of solute. Pressure is not considered here as it does not affect the solubility of solid solutes significantly. The effect of all parameters on solubility measurement can be controlled through the proper design of apparatus except the equilibration time. Equilibration time is system specific and varies according to the solute–solvent system.

Many automatic and semi-automatic devices have been proposed for the measurement of solubility according to the range of temperature, pressure, or the nature of components (Barrett and Glennon, 2002; Mohan et al., 2002; Solubility Measurements, 2015). However, most of them are too expensive to have as standard equipment in all laboratories. Literature survey shows that inexpensive simple apparatus for solubility measurement have been reported decades ago and had major limitations in terms of accuracy, throughput, and required large amount of solute (Butter, 1974; Harle et al., 2003; Knox, 1938; Schmitt and Grove, 1960). Moreover, most of the recent articles publishing solubility data measured by using classical isothermal technique lack detailed schematic illustration of the equipment used for the measurements (Cheuk et al., 2015; Deng et al., 2015; Farjami and Jouyban, 2015; Shakeel et al., 2015; Zhang et al., 2015b). Therefore, in this article we demonstrate comparatively inexpensive, multipurpose, and easy to assemble solubility measurement apparatus, which can be useful for university students as well as researchers unfamiliar with solubility measurements.

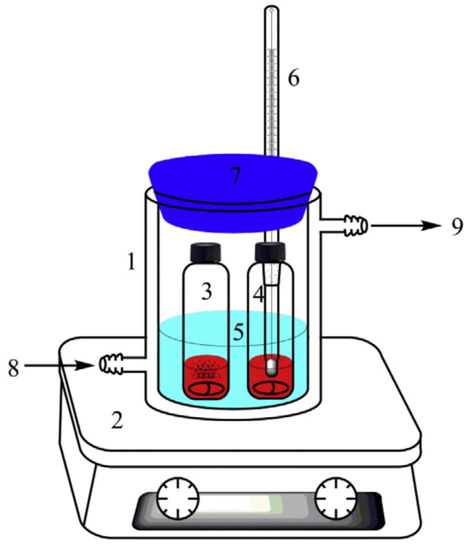

Fig. 1 – Schematic illustration of an apparatus for solubility measurement of solid solutes in liquids. (1) Jacketed glass beaker, (2) magnetic stirrer plate, (3) glass vial containing suspension of solute–solvent and PTFE coated magnetic stirrer, (4) control vial, (5) water, (6) thermometer/Pt100 thermocouple, (7) rubber cork, (8) thermostatic fluid inlet, and (9) thermostatic fluid outlet.

2. Apparatus for solubility measurement

A schematic illustration of the proposed solubility measurement apparatus is depicted in Fig. 1. It mainly consists of a jacketed glass beaker (250 ml) connected to the circulating water bath. The beaker is placed over the magnetic stirrer plate to provide agitation. Simultaneous measurement of solubility at constant temperature for numerous samples is possible by connecting multiple jacketed beakers in a series placed on a multi position magnetic stirrer plate as shown in Fig. 2. The sample consisting of excess amount of solute suspended in solvent is contained in a 10 ml glass vial along with polytetrafluoroethylene (PTFE) coated small magnetic stirrer bar. Measurements can be done with the volume of suspension as low as 1 ml. The vial can be sealed so that the loss of solvent is prevented, especially in case of measurements above

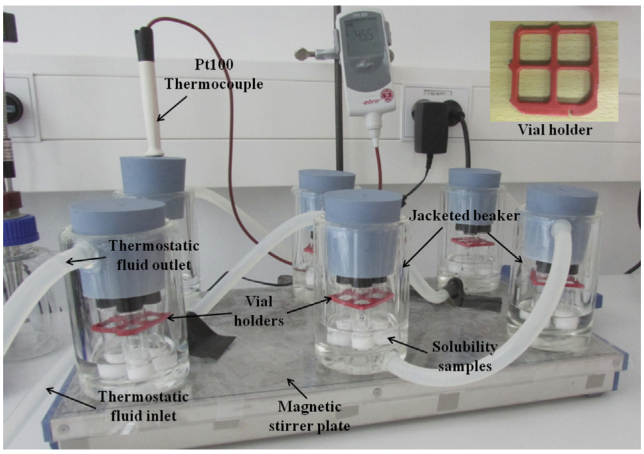

Fig. 2 – Picture of the solubility measurement apparatus assembled from parts. As an aid to assemble the proposed solubility measurement apparatus, different parts of the apparatus and their prices are mentioned in Table 1.