

# Applications of laser in the field of chemical solubility determination

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A novel experiment method for chemical solubility determination was brought forward, in which optics and chemistry principles are united and the change of laser intensity indicates the process of chemical dissolving. The more undissolved solid exists in the mixture of solute and solvent, the less transmitted laser intensity is detected. Only when the transmitted laser intensity in stirring state and that in static state comes into equalization, the dissolving process stops. Under the help of laser intensity judgement, measurements turn to be more feasible and objective, especially at high pressure. The average relative errors for the solubility data determined in this paper are 2.3% for those in the minor value scope and 1.7% for those in the high value scope respectively. Comparison of the experimental solubility data with the literature ones demonstrates that the laser-aid solubility determination apparatus is stable and reliable.

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Chemical solubility data are of great technical interest for their essential position in chemical engineering and chemistry researches. For long, solubility data are measured by using two kinds of traditional methods, that is, equilibrium method and synthetic method<sup>[1]</sup>. Generally, when using equilibrium method, suitable analysis methods should be chosen as assistant approaches in order to get accurate solubility data. Therefore, when the aim system only presents minor solubility, this kind of determination method will meet difficulty and synthetic method will exert itself to help. However, for most of the synthetic methods, the processes of dissolving must be observed and judged by eyes. These kinds of visual method will inevitably involve subjective error and be helpless facing solubilities at high pressure furthermore. In this paper, a brand new experiment method for solubility determination was brought forward, in which optics and chemistry principles were united. In the whole course of data collection, the dissolving processes were indirectly observed by laser instead of by eyes. Due to laser's exclusive characteristics, intensive, steady, and all in one specific direction, it will be much helpful in getting minor solubility data usually necessary in environment pollution because laser in use will play the role of man's eyes during the judgement of equilibria, which makes this method much objective and feasible, especially at high pressure.

The main apparatus is shown in Fig. 1.

It is worth of pointing out that there are two quartz windows symmetrically laid near the bottom and in the wall of the solubility cell. It is the necessary route way for laser. Steady laser is emitted out by a semiconductor laser device of 5 mW, 650 nm in wavelength. After passing through aperture and adjustable lens, laser beam goes through the solvent-solute mixture in solubility cell. If there is still solid in the way of laser, it will be scattered and the transmitted intensity will go down. The more solid exists, the less laser transmitted. The intensity of transmitted laser is taken down by computer in terms of

photovoltage. Suppose we get two transmitted intensity values at same temperature, one is at static state that is without stirring and another is under dynamic state during stirring. The time the two transmitted intensities mentioned above eventually turn to be equal to each other, the dissolving point of solid-liquid equilibria can be determined.

Signal can be magnified or minified according to the size of solute crystal and the numerical value of solubility limit. When the solid crystal is relatively large and the solubility is also fairly acceptable, for example, sodium chloride dissolving in water, then the change of photovoltage should be remarkable. In this case, it is unnecessary to magnify the signal and adequate sensitivity that could be achieved. In another case, the solid crystal is quite small and has minor solubility in the solvent. This time, signal should be properly magnified in order to get accurate solubility data.

The solubility equilibrium cell is of approximately 100 cm<sup>3</sup> and is made of titanium to avoid the certain corrosion caused by chemicals, especially at high temperature and high pressure. The magnetic stir works well enough to ensure the mass transfer from the solid phase to the

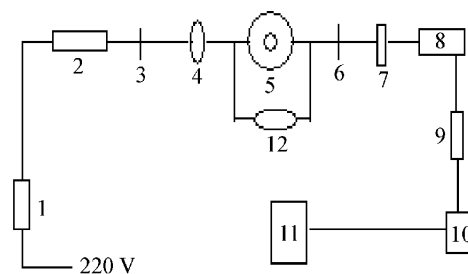


Fig. 1. Schematic diagram of measurement of solubility data using laser. 1: power; 2: semiconductor laser device; 3: aperture; 4: lens; 5: titanium solubility cell and thermostat; 6: filter; 7: photocell; 8: photomultiplier; 9: amplifier; 10: digital voltmeter; 11: computer; 12: magnetic stir.

liquid phase. Using the apparatus, measurements between 300 and 445 K can be performed. In order to guarantee the degree of confidence of data, besides the high precision electronic balance was used to weigh the solvent and solute, other matters were concerned. First, thermocouples used in the experiment were checked in Tianjin Metering Institute and the deviation of the temperature was determined to be  $\pm 0.05$  K. Second, the rate of temperature rising was controlled, especially near the solid-liquid equilibrium point, in less than 0.1 K every 10 min. The accuracy of the given composition is assumed to be within  $\pm 0.0001$ .

According to our experimental method, the weights of solvent and solute are decided in advance, so is the solubility. The key point of this method is step by step rising the mixture's temperature and finding at which temperature the solute in the mixture will just dissolve. This temperature is the corresponding point matching the solubility. The judgement of the dissolving temperature is under the help of a graph plot of photovoltage versus temperature, as shown in Fig. 2. The point of intersection of the two trendlines is just the dissolving point matching certain solubility.

To verify the reliability of the experimental apparatus in both large and minor solubility ranges, solubilities of sodium chloride in water and terephthalic acid in acetic acid were measured seriatim. The experimental data for the above mentioned two systems were compared with those of Ref. [2]. The agreement, as shown in Figs. 3 and 4, is very good, which means the equipment is suitable for the measurement of solubility in both low and high scopes, the average relative errors of which are 2.3% and 1.7% respectively.

However, in order to make the measurement more efficient and get more accurate result, it is better to do some pre-experiment among the normal temperature and normal pressure range, which will provide the solubility data approximately, together with the rough tendency of the dissolution. It will be helpful in terms of arranging the experiment under the condition of higher temperature and higher pressure.

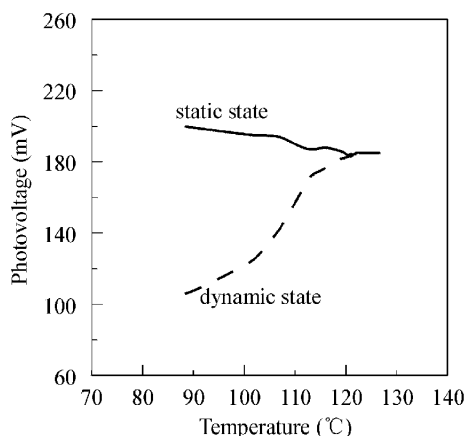


Fig. 2. Dissolving temperature determination.

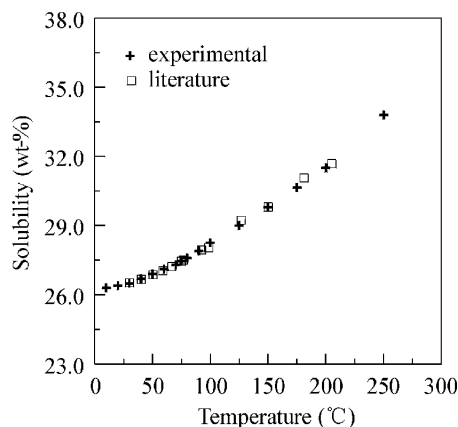


Fig. 3. Experimental solubility of sodium chloride in water compared with literature data.

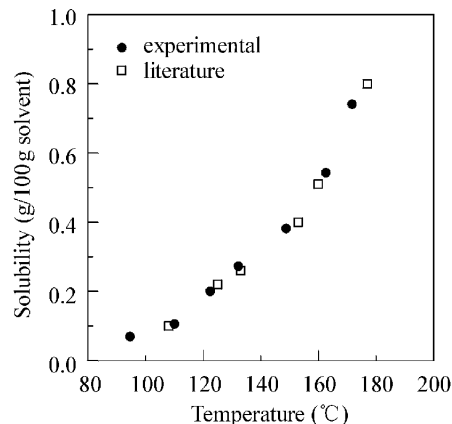


Fig. 4. Experimental solubility of terephthalic acid in acetic acid compared with literature data.

In conclusion, laser-aided solubility determination apparatus proposed in this letter is stable and reliable. It performs more objectively and has little subjective error during measurements. Attributed to its inside adjustable device, the apparatus meets simultaneously high and minor solubility determination's needs. The range of application is much wide, so that it is not only suitable for the systems at high temperature and high pressure but also capable to handle systems containing corrosive chemicals.

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## References

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