The application of chemical and professional development

应用化工与其专业化发展

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图书在版编目（CIP）数据

应用化工与其专业化发展 ：英文 / 冯德日著. —
成都：电子科技大学出版社，2014.7

ISBN 978-7-5647-2502-0

Ⅰ. ①应… Ⅱ. ①冯… Ⅲ. ①应用化学－化学工业－
研究－英文 Ⅳ. ①TQ

中国版本图书馆 CIP 数据核字（2014）第 150217 号

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出　　版： 电子科技大学出版社（成都市一环路东一段 159 号电子信息产业大厦　邮编：610051）
策划编辑： 谭炜麟
责任编辑： 谭炜麟
主　　页： www.uestcp.com.cn
电子邮箱： uestcp@uestcp.com.cn
发　　行： 新华书店经销
印　　刷： 北京九州迅驰传媒文化有限公司
成品尺寸： 185mm×260mm　**印张** 9.75　**字数** 228 千字
版　　次： 2014 年 9 月第一版
印　　次： 2014 年 9 月第一次印刷
书　　号： ISBN 978-7-5647-2502-0
定　　价： 48.00 元

Foreword

With the improvement of society chemical quality requirements of professional and technical personnel, with specialized knowledge, skills and mastery of chemical chemical professional and technical personnel of English has been gaining popularity in the community. "Applied Chemical their professional development" is a very important chemical engineering professional subject, focusing on capacity-reader in English reading and writing. To meet the requirements of the new era of professional readers the ability of chemical training objectives and the overall quality of our professional readers for chemical analyzes and compares a number of similar materials, absorbing, combined with the authors' University of Chemical professional teaching practice, the preparation of the book.

When writing the book closely chemical expertise and professional considerations from the applicable terms, highlighting the practical enough and easy to use features. Each chapter covers the basics of chemical engineering and frontier areas. A wide range of materials, vocabulary comprehensive selection adaptability, wide coverage, illustrations, rich in content, suitable for chemical and related professional personnel as a self-selection book for the work.

Since the editor is limited, the book is nothing wrong with the inevitable, please use the correct book reader, be grateful.

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Chapter 1 Applied Chemical Technology Overview

Seldom do we encounter pure materials. Instead, many materials are mixtures made up of two or more chemically different substances. In order to isolate pure components of a mixture, chemists have developed a variety of techniques for the separation of one component from another, taking advantage of the differences in physical properties of the components. Recrystallization is one of the important laboratory processes frequently used for this.

Recrystallization is used to purify a solid substance at the temperature of the experiment. It is a basic purification technique based on different solubilities of solids. Insoluble impurities can be easily removed by filtration after dissolution of the solid that needs to be purified, while small amount of soluble impurities remains in the solution. Increasing the temperature produces a supersaturated solution which can be used to obtain crystals of the pure solid. When slowly cooling the solution down to room temperature, crystals form and crash out, with the impurities in the solution. Sometimes it is easier to conduct recrystallization using two solvents, one good solvent for the compound and one poor solvent.

Single-Solvent Approach.

A single-solvent recrystallization includes the following steps: selecting the solvent; dissolving the solid; cooling the solution; filtering and drying the crystals.

Selecting the Solvent

Choosing an appropriate solvent is the first step in a recrystallization. Water, hexane, methanol, and ethyl acetate are frequently used. Ideally, the solid is virtually insoluble in the solvent at room temperature, yet is completely soluble at higher temperatures at or near the boiling point of the solvent. To find a suitable solvent, it is necessary to test the solubility of the desired compound in different solvents. Test

tubes and a rack, a test tube clamp, pipets and bulbs, a spatula, a beaker, and a hot plate are required, in addition to the compound, water, and the solvents.

Load a small amount of solid into a test tube, followed by adding about one milliliter of the test solvent. If the solid dissolves immediately at room temperature, the solvent is not suitable for recrystallization. Repeat this process with another test solvent using a clean test tube. If the solid does not dissolve, heat up the test tube using a hot water bath whose temperature is set at the boiling point of the test solvent. If the solid still remains, then this solvent is not good, either. Repeat with other solvents until the solid remains at room temperature, but dissolves in the solvent with the temperature - at the boiling point, indicating a good recrystallization solvent.

Dissolving the Solid

In the second step, the solid to be recrystallized is dissolved in the hot suitable solvent. Two Erlenmeyer flasks (one for the solvent and the other for the crystals), a hot plate, a disposable pipet and bulb, finger cots, and some boiling stones are needed in this step. Place two boiling stones in each flask to ensure smooth boiling during heating. A small amount of solvent is added to a flask containing the impure solid, and then the suspension in the flask is heated to the boiling point of the solvent until the complete dissolution of the solid. If the solid does not dissolve, add more hot solvent drop-wise continually until the solid is fully dissolved. A hot filtration is required if the solution contains visible solid impurities other than boiling stones. If the solution appears colored, the hot saturated solution is boiled for a short period of time with the addition of activated carbon to remove colored impurities, followed by a hot filtration to get rid of the activated carbon.

Cooling the Solution

Next, the solution is cooled for the desired compound to crystallize. A more pure solid precipitates out from the solution, leaving soluble impurities in the solvent. The Nobel laureate, the late Professor Robert Bums Woodward stated that crystallization is one of the most beautiful processes known, and no true chemist fails to experience a thrill when he brings a new form of matter into the crystalline state for the first time.

In most cases, crystals grow as the solution cools down. Leave the solution undisturbed until the temperature decreases, and crystals begin to form on the bottom

of the flask. Usually slower cooling leads to a more pure product. The size of crystals that form also depends on the cooling rate. Very small crystals tend to form upon rapid cooling and the impurities may also precipitate out of the solution along with the small crystals. Therefore, it is quite common to allow the solution to cool to room temperature first before cooling it further by setting the flask in an ice-water bath. Wait for the majority of crystals to form at room temperature and then place the flask in an ice-water bath.

However, sometimes crystallization needs to be induced by nucleation. One method is to scratch the flask with a glass rod at the air-solvent meniscus. The scratch increases the surface area of the glass, resulting in a roughened surface on which the solid can nucleate and crystallize. Another technique is to add a small crystal of the desired pure solid as the "seed" into the cooled solution if such a crystal is available. The "seed" crystal serves as the nucleating site for the crystal to grow. Make sure that the solution is cool; otherwise, the added small crystal would dissolve.

If there are no crystals falling out of the solution, it is possible that too much solvent has been used. The solution should be concentrated further by allowing some of the solvent to evaporate. If crystals do not immediately form, reheat and then cool the solution.

Filtering and Drying the Crystals

After crystals have formed, it is time to separate them from the solution. Vacuum filtration is frequently used to isolate and dry the purified solid, sometimes washing the purified solid with chilled solvent. Use the smallest possible amount of cold solvent when washing the product to avoid dissolving some of the sample.

Vacuum is supplied by a pump and applied to the filter flask through a rubber tubing. Add filter paper to the funnel which is placed on the filter vacuum adapter in the neck of the filter flask. Use a small amount of the recrystallization solvent to moisten the filter paper and then turn on the pump. Pour and transfer the crystals and solution to the center of the filter paper. Add cold solvent to the flask and swirl the remaining crystals into the funnel.

Once the liquid is all sucked through, turn off the pump to release the vacuum. Then add a small amount of cold, clean solvent to wash the crystals and apply a

gentle suction to allow the fresh solvent passing through the crystals at a slower rate. Note that suction should not be applied while washing. In order to dry the crystals as thoroughly as possible, full suction is applied for a few minutes. Drying the product via vacuum filtration should remove much of the solvent. Depending on the volatility of the solvent, sometimes open-air drying is used as well.

After filtering and drying, the final step is to remove the crystals from the filter fiinnel. Use a spatula to transfer the crystals to a watch glass. Physically separate any remaining boiling stones from the crystals in this step. In some cases, the recrystallization process is repeated to further purify the substance.

Two-Solvent Approach

When it is not possible to find a single recrystallization solvent, a two-solvent recrystallization method has to be used. In such a process, the primary solvent (Solvent A) can dissolve the desired compound at the boiling point, and the second solvent (Solvent B) should induce crystallization when added to the saturated solution of the compound in Solvent A. The same four steps are involved in a two-solvent recrystallization: selecting the solvents; dissolving the solid; cooling the solution; filtering and drying the crystals.

Selecting the Solvents

Similar to the single-solvent approach, the first step is also to select suitable solvents. As mentioned above, two solvents are needed, with one being a very good solvent for the compound and the other extremely poor at room temperature. And these two solvents must be miscible.

A glass plate, a spatula, and several clean Pasteur pipets and bulbs are needed in this step along with a range of candidate solvents and the compound to be purified. To select the solvents, only a small amount of solid compound is needed. On a glass plate a tiny amount of the compound is placed, then about four centimeters away another sample is added. In a similar fashion place more solid samples on the glass plate until there are enough samples for the number of solvents to be chosen from. Select solvents with different polarity such as water, methanol, ethyl acetate, and hexane. Take three or four drops of one test solvent and add them to one solid sample. And repeat this process for the remaining solvents. Check the solubility results

and evaluate whether the compound dissolves completely, partially, or not at all. Again, the perfect combination of solvents means that one solvent (Solvent A) easily dissolves the compound and the second solvent (Solvent B) does not dissolve the compound.

Dissolving the Solid

In this recrystallization approach, the two solvents A and B should also be hot. Add each solvent in an Erlenmeyer flask along with boiling stones. Then heat up the solvents until near their respective boiling points. Load the impure compound in a tared test tube that is no more than one quarter full of solid.

Add the first recrystallization Solvent A, to dissolve the crystals. Add just enough hot Solvent A with a Pasteur pipet to the test tube that contains the compound. During additions of Solvent A, heat and shake the test tube to help dissolve the compound. Minimum amount of hot Solvent A should be used and the volume of Solvent A should not exceed one third of that of the test tube. The second Solvent B, is then added to the solution until the solution becomes cloudy. Generally, no more than twenty drops of Solvent B is needed.

Alternatively, the solid can be suspended in the second Solvent B. Then hot Solvent A is added until the solid just dissolves.

The last two steps (cooling the solution and filtering and drying the crystals) in two-solvent recrystallization are similar to those in the single-solvent method, although here to wash the crystals in the last step, use a mixture of the solvent system in about the same ratio used to obtain a saturated solution.

Chapter 2 Acid-base Titration Chemical Technology

A volumetric quantitative analytical technique that is often used to measure how much acid or base is present in a solution is called a titration. Acid-base titrations are based on neutralization reactions. If a solution is acidic, a titration is to add a base to it until the base neutralizes all the acid.

Acid-base titrations can be used for most acids and bases, including hydrochloric acid, sulfuric acid, acetic acid, sodium hydroxide, ammonia, and so on. In particular, it is even possible to determine in one titration the composition of a mixture containing acids or bases of different strengths, such as sodium hydroxide and sodium hydrogen carbonate. Hydrochloric acid and sodium hydroxide are two most commonly used reagents in acid-base titrations.

The reaction follows a stoichiometric relationship. The stoichiometric point in an acid-base titration may be visually determined by use of an indicator which tells us when the titration is completed. Visual detection of completion of the reaction is a key factor in maintaining the simplicity of titration. A visual indicator is an organic compound that changes color when the pH of the solution changes. Such pH-dependent color changes are the result of chemical changes in the indicator with its chemical environment caused by the addition of H_3O^+ or OH^-. An example of these changes in the functional moieties of phenolphthalein, a commonly used indicator which changes from colorless to a pink hue at pH 8.0-9.0.

Ideally, the observation of a sudden change in the color of the solution with the addition of a few drops of indicator tells us the completion of the titration. Sometimes color change seems like instant, with a very small drop of the titrant completely changing the color of the solution. However, depending on the concentration of titrant, titrated substance, and the selected indicator, sometimes we have to add even several

milliliters of titrant before we see a color change. This confusion makes it difficult for us to determine when we should stop the titration. In some cases, we should look for a completely different indicator if the one selected fails to guarantee accuracy in the measurement. In order to choose a suitable indicator for an acid-base titration, we need to know the pH of the end point before using standard indicator tables. At the end point of the titration the pH of the solution suddenly changes. The end-point pH can be calculated with the aid of the titration equation.

As an example to show the general procedures in an acid-base titration, sodium hydroxide solution is used to titrate a solid acid dissolved in deionized water. The end point is determined by the color change of an indictor.Shows the schematic of the titration setup.

Designed for classical quantitative volumetric analysis, this experiment serves as a good example of conducting a quantitative experiment with the combination of several quantitative techniques. It is one of the most accurate procedures yet one of the simplest in chemistry lab work. In general, a solution of the acid A is added to an Erlenmeyer flask. A buret is filled with titrant, the solution of base B, at the beginning of the titration to its maximum capacity. The volume of the base solution is read before the beginning of the titration. Solution B is then added drop-wise from the buret to Solution A in the Erlenmeyer flask. The titration is completed when the indicator exhibits a permanent color change. The buret is read again to obtain the volume of Solution B added. With the known concentration of B in the titrant, the titrant volume that reacts with all of A in the flask can be used to calculate how much A is present based on stoichiometry. Some of the chemicals and equipment involved in this experiment include a buret, a buret clamp, a pipet, a small funnel, a standardized sodium hydroxide solution, and phenolphthalein indicator.

The beginning of the acid-base titration starts with the dissolution of the solid acid sample in deionized water. Add the end-point indicator, which is phenolphthalein, with two drops to each flask containing the acid sample and deionized water. Properly label the flasks. Be consistent in all of the samples when adding the indicator. Swirl the flasks until the solid acid is completely dissolved. Finally, rinse with deionized water three times around, which is critical to ensure that all solid acid has been

removed from the flask walls and dissolved in the solution. All solid particles must be dissolved prior to the titration.

The buret needs to be checked for if it is quantitatively clean, both to avoid contamination and to be sure that titrant volumes are accurately read. Make sure the buret stopcock is closed. Fill the buret with water and then drain it to check the buret, making sure that its walls drain cleanly. Before checking for drainage, wait a minute or two after completely draining the buret. Sometimes droplets appear on the inner walls of the buret after some time, indicating that the buret is not quantitatively clean. In this case, it is necessary to use standard cleaning procedures to clean the buret. If the buret is droplet free, then it is quantitatively clean and can be used for titration.

After the buret is cleaned, it is necessary to rinse it with the titrant, sodium hydroxide solution. Use a clean and dry funnel to add titrant to the buret. Titrant can also be poured into the buret directly with the buret removed from its holder. Small portions of titrant are used to rinse the buret in order to conserve the titrant. Hold the buret on its side and roll it to rinse the internal buret walls thoroughly. The buret tip is rinsed with the buret held over a waste container or sink and all the liquid being allowed to pass through the tip. Remove the last drop of titrant and continue with rinsing. Usually three times of rinsing is needed to remove any deionized water left in the buret.

Titrant can then be filled in the well-rinsed buret. Still use a funnel to add titrant to the buret. Carefully lift the funnel for smooth delivery and to avoid overfilling of the titrant. Similar to the cleaning of the buret, an alternative is to remove the buret from the buret holder, and directly pour the titrant from the titrant bottle. Let some titrant run through the buret tip into the waste container and check whether there are any air bubbles in the tip. The bubbles will cause difficulty in obtaining accurate values of volume if they are not removed. The bubbles can be shaken out by opening the stopcock, firmly holding the buret with both hands, and jerking downward a bit. When bubbles are removed, tip off the hanging titrant drop and mount the buret for titration.

With the titrant filled in the buret, the samples and the buret are ready for titration. First, the initial level of the meniscus should be read. Look directly at the

meniscus, and measure the meniscus at eye level from the center of the meniscus. It is critical to use a consistent buret reading procedure throughout the experiment. Use a contrast card to assist in reading the buret consistently. As a standard practice, the reading of the meniscus level should be immediately recorded in a permanent lab book. Taking notes on a scratch paper is not a correct way of recording such a critical observation.

Titration starts with sample flask number 1. Place a white paper beneath the titration flasks to aid in judging the end-point color. The buret is positioned in such a way that its tip is a few centimeters below the flask rim. The sample is titrated, using the disappearance of the indicator color as a guide of the titration rate. At the beginning of the titration, allow the titrant to run full bore into the flask. At the point where the titrant hits the acid solution the color may temporarily turn pink, but this color disappears upon swirling. The color disappearance is very rapid because of the fast production of colorless water by reaction of the base from the buret with the acid in the sample. During the titration process, continuously swirl to ensure proper mixing which leads to fast reaction.

As the rate of color changes slows, titrant can be added more slowly. With more sodium hydroxide from the buret added to the solution in the flask, more acid in the sample is reacted and less acid is available in the solution. When the red indicator color lingers in the flask for a second or two on swirling after addition of titrant, the end point is near and the delivery of titrant should be slowed down. Smaller volumes of titrant should be added carefully into the flask. The rapid addition rate at the start of the titration is consistent with the rapid indicator color change at the start of the titration, so is the slow addition near the end point and slow indicator color change near the equivalence point.

As the end point is approaching, the addition of titrant is reduced to a few drops. It requires patience and skill to locate the correct end point. Carefully watch the rate of color change. The addition should be even smaller if it takes longer for the color to fade away. Continue with ever smaller increment addition of titrant. Rinse the inner flask walls and the buret tip to make sure that no droplets remain in those places and that all the acid is in the solution. The end point is reached when the addition of a final

half-drop leads to a persistent color change. The first appearance of a permanent pink coloration indicates the end point, and the solution should appear extremely pale.

Then read the final level of the meniscus in the buret and also record the reading immediately in the lab book. The difference between the initial volume and the volume left in the buret at the end of titration is the volume of the base consumed. It needs to be pointed out again that consistent reading of the buret is important. Be careful not to add too much titrant. If too much base is added and the indicator in the flask becomes deep pink or purple, an error called overtitration occurs. The entire titration needs to be repeated with a new sample.

After the first sample is done with titration, repeat the procedures for the other samples. The average value of the titrant volumes can then be used to calculate the concentration of acid in the sample with the aid of the titration equation. Note that each titration should be an independent measurement. The first or even a rough titration allows the quick determination of the approximate volume of titrant needed to neutralize the acid. Such knowledge can be used to estimate the end point for each sample. However, the predicted end point is only a guide. It should not be the target of the titration.

Chapter 3 Isolation, Purification, Identification Techniques

The objectives of this experiment are: to isolate caffeine from tea leaves through extraction; to purify the crude material by sublimation; to identify the purified substance by measuring its melting point.

Background

Caffeine has the molecular formula $C_8H_{10}N_4O_2$ with a molecular weight of 194.19 g/mol. Its chemical name is 3,7-dihydro-l,3,7-trimethyl-lH-purine-2,6-dione. Caffeine belongs to a group of compounds called alkaloids, more specifically, a member of the methylxanthines. The caffeine molecule has base characteristics (alkali-like) and the purine ring system, which is an important framework in living systems.

Caffeine is a chemical with a variety of uses. From medicines to beverages to foods, caffeine is one of the most popular natural products used today. It is the most widely used of all the stimulants and acts to stimulate the heart, central nervous system, and the respiratory system. Its usage can increase blood pressure, contraction force, and volume output by increasing heart rate. A small dose of this compound at an amount of 50 to 200 mg increases alertness and reduces drowsiness and fatigue. Caffeine is the main ingredient of many “stay-awake” pills. It is a smooth muscle relaxant and a diuretic. Caffeine is also a food additive. It can be found in popular soft drinks. However, it needs to be pointed out that caffeine has side effects. Large doses in excess of 200 mg can cause insomnia, restlessness, headaches, and muscle tremors. In addition, continued, heavy use of this chemical may lead to addictiveness. Furthermore, some research connects high caffeine consumption in pregnant women with the malformation of their children.

As a natural product, caffeine constitutes as much as 5% by weight of tea and