Analysis of Unknown Solids

Scientific Method Inquiry Lab Activities

Introduction

Identify five unknown substances by using only distilled water, vinegar, and phenolphthalein. Learn to use a variety of chemical and physical tests to identify these five common household substances, all of which are white solids.

Concepts

- Qualitative analysis
- Chemical reactions
- Physical and chemical properties
- Flow diagrams

Background

The process of determining the identities of unknown substances is called *qualitative analysis*. This can be contrasted to quantitative analysis, which is the process of determining how much of a given component is present in a sample. A qualitative analysis scheme using simple chemical and physical tests is designed, in this laboratory experiment, for the identification of five common household white solids: chalk (calcium carbonate) washing soda (sodium carbonate), baking soda (sodium bicarbonate), citric acid, and Alka-Seltzer® (mixture of citric acid and sodium bicarbonate). Qualitative analysis schemes are generally summarized by a flow diagram. A flow diagram is designed with the procedural steps on the vertical lines, the possible test results on the horizontal lines, and the resulting identifications in the boxes. Qualitative analysis procedures can include physical tests as well as chemical tests. The physical tests in this lab are solubility in water. The chemical reactions or tests in this lab are with vinegar and phenolphthalein. All of these tests involve either a color change, evolution of gas bubbles or no reaction. On the basis of observations, each of the five white household substances is identified.

Materials

Unknown Samples (in alphabetical order):

Approximately 0.50–1.0 g of each of the following unknowns is needed:

Alka-Seltzer® (mixture of citric acid and sodium bicarbonate)
Calcium carbonate, $CaCO_3$
Citric acid, $C_6H_8O_7$
Sodium bicarbonate, $NaHCO_3$
Sodium carbonate, Na_2CO_3

Chemicals/Test Reagents

Phenolphthalein solution, 1%,
Vinegar, white
Water, distilled or deionized

Apparatus

Pipets, Beral-type
Solubility Table
Spot plate
Toothpicks (for stirring)

Safety Precautions

Phenolphthalein solution is a flammable liquid and is toxic by ingestion and inhalation. All other reagents and unknowns are considered non-hazardous; however, all may cause slight irritation to the skin, eyes, or respiratory tract. Avoid contact of all chemicals with eyes and skin. Follow all laboratory safety guidelines. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Please review current Material Safety Data Sheets for additional safety, handling, and disposal information. Remember to wash hands thoroughly with soap and water before leaving the laboratory.

Caution

Since all of the samples are unknown to you, treat each and every one of the white solids as if it were a hazard. Follow all safety precautions; be sure to properly label all unknowns so the identity may be determined in the case of an emergency.

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Procedure

1. Place a very small amount of each of the 5 unknown substances, into the appropriate spot well.
2. Add approximately a half spot well full of distilled or deionized water to each sample.
3. Stir the contents of each tube gently with a separate wooden toothpick to attempt to dissolve the solids.
4. Record observations of which substances are soluble (dissolve). which are insoluble (do not dissolve), or any other reaction regarding water in the data table.
5. Add 1 drop of phenolphthalein into each of the samples.
6. Record the observations in the data table.
7. Add 2 drops of vinegar into each of the samples.
8. Record the observations in the data table.
9. Reference Solubility Table during data analysis.

Disposal

Please consult your current *Flinn Scientific Catalog/Reference Manual* for general guidelines on specific procedures governing the disposal of laboratory waste. Remaining amounts of the five white solids can be saved for later use or disposed of in the solid waste according to Flinn Suggested Disposal Method #26a. The solutions in the spot plates can be rinsed down the drain with water according to Flinn Suggested Disposal Method #26b. Excess test reagents can be saved for later use or disposed of according to the appropriate Flinn Suggested Disposal Methods.

Tips

- Note that the 5 substances to be identified by the students have not been assigned unknown letters. We have left this up to the individual instructor who may want to vary the identity of the unknowns for different groups in one class and for different classes throughout the day. Many teachers label the unknown samples randomly with no evident patterning with numbers or letters A to Z, AA to ZZ etc and have a key for grading purposes.
- This is a qualitative lab; therefore, exact amounts used in the tests are not crucial.
- Stress to students that a small amount is less than about 0.2 g, or less than half the size of a small pea. Using too much of the solids may create unclear test results, especially for the solubility test. It may be helpful to weigh 0.20 g out once before beginning the lab to show the students how little the amount of a small scoop is and to stress that more is not always better.

— Grinding the solids to a fine powder with a mortar and pestle will help make them indistinguishable.

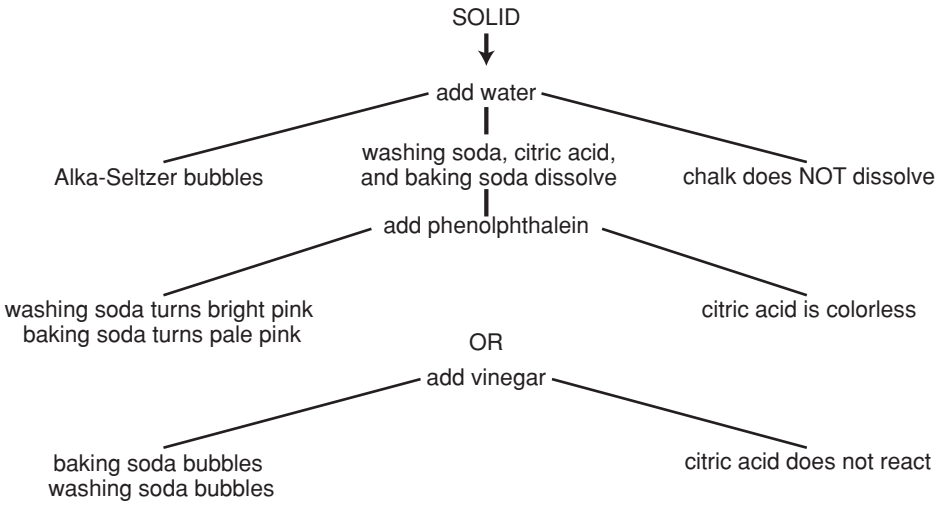
— This experiment was developed and conducted as a lab assessment with "open notes". The students had the results of a hydrolysis demonstration of baking soda and washing soda with Universal Indicator. These results show a higher pH for the washing soda. Students could be shown this test, or make educated assessments on K_a values, etc.

- This experiment can be adapted for various levels of instruction. The lab can be performed as written for general chemistry classes. For more advanced classes, consider not providing the step-by-step procedure or data table. Allow the students to design their own identification scheme given only the names of the five white solids. Known samples of each of the solids can be provided to students for testing; then the unknowns can be tested and their identity determined.
- All of the compounds used as the unknowns are white solids and are common household substances found in drugstores or supermarkets.

Answers to Worksheet

Substance (not in any order)	**Add Water**	**Add Phenolphthalein**	**Add Vinegar**
Washing soda	*dissolves*	*bright magenta*	*bubbles*
Baking soda	*dissolves (not as well as washing soda)*	*pale pink*	*bubbles*
Alka-Seltzer	*bubbles*	*colorless*	*bubbles*
Citric Acid	*dissolves*	*colorless*	*no reaction*
Chalk	*does not dissolve*	*colorless*	*bubbles*

Flow Chart for Analysis of Unknown Solids



Connecting to the National Standards

This laboratory activity relates to the following National Science Education Standards (1996):

Unifying Concepts and Processes: Grades K–12
Evidence, models, and explanation

Content Standards: Grades 5–8
Content Standard A: Science as Inquiry
Content Standard B: Physical Science, properties and changes of properties in matter

Content Standards: Grades 9–12
Content Standard A: Science as Inquiry
Content Standard B: Physical Science, structure and properties of matter, chemical reactions

Flinn Scientific—Teaching Chemistry™ eLearning Video Series

A video of the *Analysis of Unknown Solids* activity, presented by Kathleen Dombrink, is available in *Inquiry Lab Activities* and in *Scientific Method Inquiry Lab Activities*, part of the Flinn Scientific—Teaching Chemistry eLearning Video Series.

Materials for *Analysis of Unknown Solids* are available from Flinn Scientific, Inc.

Catalog No.	Description
A0111	Alka-Seltzer® Tablets, Pkg/24
C0347	Calcium Carbonate, 100 g
C0135	Citric Acid, Anhydrous, 100 g
S0051	Sodium Carbonate, 500 g
S0042	Sodium Bicarbonate, 500 g
P0019	Phenolphthalein, 1%, 100 mL
V0005	Vinegar, 3.78 L
AP1725	Reaction Plates, 6-well

Consult your *Flinn Scientific Catalog/Reference Manual* for current prices.

Analysis of Unknown Solids Worksheet

Data Table

Substance	Add Water	Add Phenolphthalein	Add Vinegar
A			
B			
C			
D			
E			

Flow Chart for Analysis of Unknown Solids

Draw a possible flowchart to determine the unknown solids using the above data table.