edited by
GEORGE L. GILBERT
Denison University
Granville, OH 43023

A Simple, Colorful Demonstration of Solubility and Acid/Base Extraction Using a Separatory Funnel

Submitted by
T. Ross Kelly
Boston College
Chestnut Hill, MA 02167

Checked by
Kathryn R. Williams
University of Florida
Gainesville, FL 32611

The use of a separatory funnel is a common operation in organic chemistry. In the course of separating a mixture (or working up a reaction) chemists frequently take advantage of the acidic or basic properties of a solute to partition it between an organic phase and an aqueous phase. Because most solutes are colorless, beginning students often find it difficult to understand exactly what is happening inside a separatory funnel as the pH of the aqueous layer is changed and the separatory funnel is shaken. We herein describe a demonstration suitable for lecture (or laboratory) use that provides a visual record of events occurring within a separatory funnel. It also can be used to illustrate pH concepts in general and other chemistry courses. The demonstration is also effective as a videotape presentation.

The demonstration takes advantage of the color and solubility properties of the indicator 2,6–dichloroindophenol (HA), as shown in eq 1 (the pKa of 2,6–dichloroindophenol is 5.9 (*1*)). A^-, the conjugate base of HA, is blue and, by virtue of its ionic nature, water soluble. HA itself is red and, being neutral, organic soluble.

$$\underset{\text{blue, water soluble}}{A^-} \underset{OH^-}{\overset{H^+}{\rightleftharpoons}} \underset{\text{red, organic soluble}}{HA} \qquad (1)$$

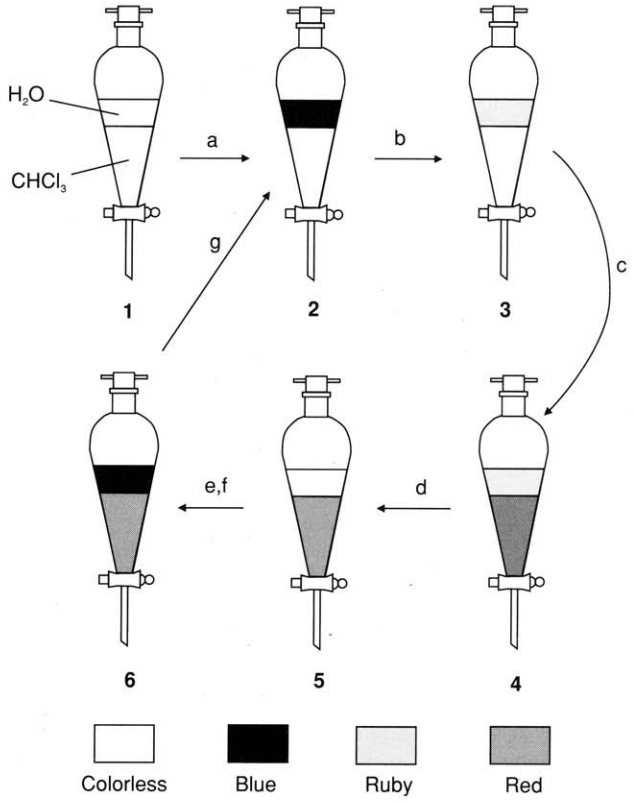
The accompanying flowchart (see figure) illustrates the results of pH and solubility changes and the consequences of partial versus complete mixing of layers as one proceeds through the demonstration. In particular, the appearance of the separatory funnel (**1** to **6**) at various times, and the operations (*a–g*) that produce the changes are highlighted in the figure and detailed below.

Illustrative Procedure

1: In a 2-L separatory funnel (a Squibb type shows off the colored layers best) are placed 1 L of water and 500 mL of $CHCl_3$ (to give **1**). [Note: this demonstration has been designed for use in a relatively large (150-seat) lecture hall. If desired, it can be scaled down easily.]

- ***Caution:*** **Chloroform is toxic, and it is a suspect cancer agent. It should be disposed of properly.**

1 to 2: To **1** is added (*a*) a solution of A^- [prepared, in advance (but preferably the same day, because the solution does not appear indefinitely stable) by mixing 25 mg of 2,6-dichloroindophenol, sodium salt (Sigma, Aldrich, etc.) with 50 mL of water and 1 mL of 1M NaOH]. The upper (H_2O) layer takes on the dark blue color of A^-, while the $CHCl_3$ layer remains colorless. Shaking the separatory funnel (and allowing the layers to separate) produces no change (i.e., still **2**), indicating that it is the solubility properties of ionic A^- that keeps the $CHCl_3$ layer colorless, not merely that upon addition A^- encounters the upper H_2O layer first.



Effect of various operations on the appearance of the separatory funnel. In those operations where the separatory funnel is shaken, it is implicit that the layers are then allowed to separate. (a) Add A^-; (b) Add 1 M HCl and stir aqueous layer; (c) Shake briefly; (d) Shake vigorously; (e) Add 1 M NaOH; (f) Shake gently; (g) Shake vigorously. This sequence of reactions is also shown in a color photograph on the cover of this issue.

2 to 3: Five milliliters of 1M HCl is added (*b*) to **2**. The upper layer is stirred (stirring rod) without mixing the H_2O and $CHCl_3$ layers. A^- (blue) is converted to HA (red) which is preferentially soluble in $CHCl_3$ but, perforce, remains in the upper aqueous layer until the two phases are mixed.

3 to 4: *Brief* mixing (*c*) of the phases (followed by layer separation) provides the opportunity for partial extraction of the neutral HA into the organic layer. Note that the exact color of HA, although essentially red in both H_2O and $CHCl_3$, is solvent dependent.

4 to 5: Thorough mixing (*d*) of the layers in **4** completes the extraction of neutral HA from the water to the $CHCl_3$ layer. There is an inconsequential, slight (and nearly invis-

ible) residual red color in the aqueous layer unless the layers are given ample time to separate completely.

5** to **6: Ten milliliters of 1M NaOH is added (*e*) to **5**, but nothing appears to happen, even though in actuality the aqueous layer has become alkaline, because the two layers are not mixed. "One shake" mixing (*f*) of the layers allows partial conversion of HA (red) to A^- (blue). Because neutralization of HA occurs only when the HA comes in intimate contact with the OH^- continuing aqueous phase, the A^- that is formed is extracted simultaneously into the aqueous phase. The upper aqueous layer in **6** contains blue A^-, the lower organic layer retains unreacted, red HA.

***6** to **2**:* Vigorous mixing (*g*) of the two layers in **6** results in complete neutralization of HA to A^- and extraction of A^- into the aqueous layer, regenerating **2**. The **2** to **6** sequence can then be repeated, if desired.

Conclusion

The demonstration described above clearly, yet simply, illustrates a number of concepts encountered in introductory Organic Chemistry and other lecture and laboratory courses. Those concepts include the underlying principles of a separatory funnel, the solubility behavior of neutral and ionic species, and the consequences of acidity and basicity. Judging from the audience's rapt attention and smiles, the demonstration serves to communicate both the concepts and the beauty of chemistry. A similar partitioning of a different indicator using benzene, ether, and water as solvents was briefly described previously by Silversmith (*2*).

Literature Cited

1. Armstrong, J. *M. Biochim. Biophys. Acta* **1964**, *86*, 194–197.
2. Silversmith, E. F. *J. Chem. Educ.* **1972**, *49*, A694.