**Water Determination by Refractive Index**

**Background**

The basic understanding of refractive index (RI) is to remember that light travels at a different rate in air compared to traveling through a condensed medium (solids and liquids). The definition of *refractive index* (n) is the ratio of light’s velocity in air (*c*) to light’s velocity in the medium being analyzed (v). Characterization of pure compounds, assessment of compound purity, and composition of a mixture can all be completed via refractive index measurements.1

Refractive index is dependent on both temperature and wavelength. Density of a solution changes with temperature, therefore the speed with which light passes through the medium also changes. Furthermore, light of different wavelength energies is refracted differently through the same medium being analyzed. Different refractive indices are the result. Therefore, consistency & reliability of the light source for refractive index measurements is key.1

The instrument used to measure refractive index is called a *refractometer*. There are both bench-top and hand-held refractometers. A commonly used refractometer called the Abbé refractometer has both a temperature control and built-in thermometer. The light source is the sodium D-line (589.3 nanometers).1 Twenty degrees Celsius (20°C) is the reference temperature of refractometer measurements, as that is considered to be room temperature. If a refractive index measurement is taken at a temperature other than 20°C, a correction factor is applied to the measured refractive index value.1 For measurements above the reference temperature, the following correction equation is used: nD20 = nD20 + x + x(0.00045). The term nD20 + x is the refractive index value at the non-reference temperature, “x” is the difference between the non-reference temperature and 20°C, and nD20 is the [calculated] corrected refractive index. The value 0.00045 represents how much a refractive index measurement decreases for each degree Celsius above 20°C.1

1Padías, A.B. *Making the Connections3: A How-To Guide for Organic Chemistry Lab Techniques, 3rd Ed.*; Hayden-McNeil Publishing: Plymouth, MI, 2015; pp 60–64.

**Synopsis**

Standard solutions with different concentrations of 1,3-Butanediol will be prepared using Nanopure H2O as the solvent. The refractive index of each solution will be measured, recorded, and plotted. An unknown sample containing 1,3-Butanediol with then be obtained allowing for the determination of water content based on the calibration curve created with the standards prepared with Nanopure water as the solvent. See the next page for specific procedure instructions.

What you will want to note in this experiment aside from superb technique in standards preparation, instrument use, and consistency of sample analysis (equipment operator), is lab efficiency & consistent monitoring of experimental conditions. Remember, RI measurements are temperature dependent.

**Procedure**

1. Using four appropriately labeled 4 dram glass vials, prepare standard solutions having between 0% and 100% 1,3-Butanediol using Nanopure H2O as the solvent. The concentrations of all prepared standard solutions are to be determined based on weight, having a total mass of about 3 grams for each solution. Use Pasteur pipettes to transfer the 1,3-Butanediol and Nanopure H2O into the appropriate vials. Record the masses of the 1,3-Butanediol used in each vial as well as the final mass of each of the four solutions prepared. Note you do NOT need to prepare a 100 % 1,3-Butanediol standard as you already have one [the stock solution]!
2. Note the brand [or RI#] of refractometer utilized in the experiment. First, measure the refractive index (RI) of an aliquot of Nanopure H2O. Then, measure RI for all of the four standard solutions prepared. Lastly, measure the RI of an aliquot of pure 1,3-Butanediol. Record the refractive index measurement and the temperature at which each measurement is taken. It is good practice to complete the analysis work in the order of most dilute to most concentrated 1,3-Butanediol standard solutions. *Think about why.*
3. To measure the RI, first make sure the stage (prism) is clean. Then, add a few drops of the solution to the stage of the refractometer, close the upper door, and raise the light attached to the sidearm until it meets the upper door. Carefully adjust the focus of the instrument using the dials on the side and front of the refractometer. The goal is to get the “horizon” as sharp as possible and in the middle of the crosshairs as viewed through the eyepiece. When the “horizon” is properly focused, press the toggle switch on the left of the refractometer, read the scale through the eyepiece, and record the value to the fourth decimal place (ex., 1.3326 was measured for a random water sample). Do not put your eye directly on the optics.
4. After all standard solutions are measured, carefully clean the stage with Nanopure H2O and a Kimwipe. Be careful: Do NOT scratch the stage surface!
5. Using the same refractometer, measure the RI of an unknown 1,3-Butanediol sample. Record the measurement and the temperature at which the unknown RI is taken.
6. Once finished, plot the calibration curve for the set of standard solutions. Evaluate the linearity of the plot by applying a linear regression. If a particular data point for the 1,3-Butanediol standard solutions does not fit the linearity of the curve, a standard solution may be re-analyzed, or re-made and then re-analyzed via the *same* refractometer used. Determine the % H2O in the unknown solution using the calibration curve for the 1,3-Butanediol solutions. Be mindful of the species being determined versus the species represented by the calibration curve when completing the calculations.