The Synthesis and  
Analysis of Aspirin

before you begin

* This experiment is compatible with the Go Direct UV-VIS Spectrophotometer (order code: GDX-SPEC-UV) and the Vernier UV-VIS Spectrophotometer (VSP-UV).
* This experiment requires the most recent version of the data-collection software you are using (e.g., Logger *Pro*, LabQuest 2 App, LabQuest 3 App, or Spectral Analysis). Download the latest version of your software at <www.vernier.com/downloads>

Experiment Notes

1. It is recommended that you read the Melt Station user guide before beginning this, or any, experiment with the instrument.

2. It is recommended that you read the UV-VIS spectrophotometer user guide before beginning this, or any, experiment with the instrument.

3. Each lab team will use about 2 g of salicylic acid.

4. The yield for this reaction will be lower than your students’ expectations. A low yield may not always be the result of sloppy work, but poor lab technique will certainly result in a disappointing yield.

5. You may choose to shorten Part III by providing your students with Beer’s law standards.

6. It is critical for your students to complete the spectrophotometric analysis of their samples (Part III) in one lab period because of the instability of the prepped samples.

7. After students complete the Part I synthesis, they will need to dry the sample. You may choose to have them wait until the next lab period, to ensure that the sample is dry. Or, if you wish to have them complete Parts II and III in the same lab period, you will need to provide an air-flow supply (low flow) to fairly quickly air dry their aspirin sample.

COMPOUND INFORMATION

|  |  |  |  |
| --- | --- | --- | --- |
| Compound | Chemical formula | Melting temperature  (°C) | Molar mass  (g/mol) |
| Salicylic acid | C7H6O3 | 158-161 | 138.12 |
| Acetylsalicylic acid | C9H8O4 | 134-136 | 180.16 |

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Compound | Chemical formula | Boiling temperature (°C) | Molar mass  (g/mol) | Density (g/mL)  at 25°C |
| Acetic anhydride | C4H6O3 | 138–140 | 102.09 | 1.08 |
| Phosphoric acid (85%) | H3PO4 in H2O | 158 | 98.00 | 1.685 |
| Ethanol | C2H6O | 78.3 | 46.07 | 0.789 |

SAMPLE DATA

Part I Synthesis of Aspirin

|  |  |
| --- | --- |
| Mass of salicylic acid used (g) | 2.01 |
| Volume of acetic anhydride used (mL) | 5.0 |
| Mass of acetic anhydride (1.08 g/mL) used (g) | 5.40 |
| Mass of aspirin and filter paper (g) | 2.52 |
| Mass of filter paper (g) | 0.56 |
| Mass of aspirin synthesized (g) | 1.96 |

Part II Melting Temperature Data

|  |  |
| --- | --- |
| Melting temperature range (°C) | 132.6–133.9 |

Part III Salicylic Acid Standard Stock Solution

|  |  |
| --- | --- |
| Initial mass of salicylic acid (g) | 0.201 |
| Initial molarity of salicylic acid (mol/L) | 0.00584 |

Part III Beer’s Law Data for Salicylic Acid Standard Solutions

|  |  |  |
| --- | --- | --- |
| Solution | Concentration (mol/L) | Absorbance |
| 1 | 1.46 × 10–3 | 0.986 |
| 2 | 1.16 × 10–3 | 0.764 |
| 3 | 8.73 × 10–4 | 0.565 |
| 4 | 5.82 × 10–4 | 0.378 |
| 5 | 4.66 × 10–4 | 0.298 |

Part III Test of the Purity of the Synthesized Aspirin

|  |  |
| --- | --- |
| Initial mass of aspirin sample (g) | 0.200 |
| Absorbance of aspirin sample | 0.477 |
| Concentration of salicylic acid (mol/L) | 7.09 × 10–4 |
| Moles of salicylic acid in aspirin sample (mol) | 1.77 × 10–4 |
| Mass of salicylic acid in aspirin sample (g) | 0.024 |
| Mass of aspirin in sample (g) | 0.032 |
| Percent aspirin in sample (%) | 75 |

ANSWERS TO THE DATA ANALYSIS QUESTIONS

1. For the sample data, the theoretical yield of aspirin is 2.63 g. The calculation is based on salicylic acid (the limiting reactant),

mol of salicylic acid used = 2.01 g ÷ 138.13 g/mol = 0.0146 mol  
mol of salicylic acid = mol of aspirin produced (theoretical)  
theoretical mass of aspirin produced = 0.0146 mol × 180.16 g/mol = 2.63 g

2. The experimentally determined melting temperature of 132.6°C compares well with the accepted melting temperature of pure acetylsalicylic acid, 135°C

3. The results of the spectrophotometric test show that the percent purity of the synthesized aspirin is 75%. This compares reasonably well with the melting temperature test.

4. Answers will vary. For the sample data, the mass of synthesized aspirin is 1.96 g. Corrected for purity, is: (1.96 × 0.75) = 1.47 g. The percent yield is: (1.47 g ÷ 2.63 g) × 100 = 55.9%.

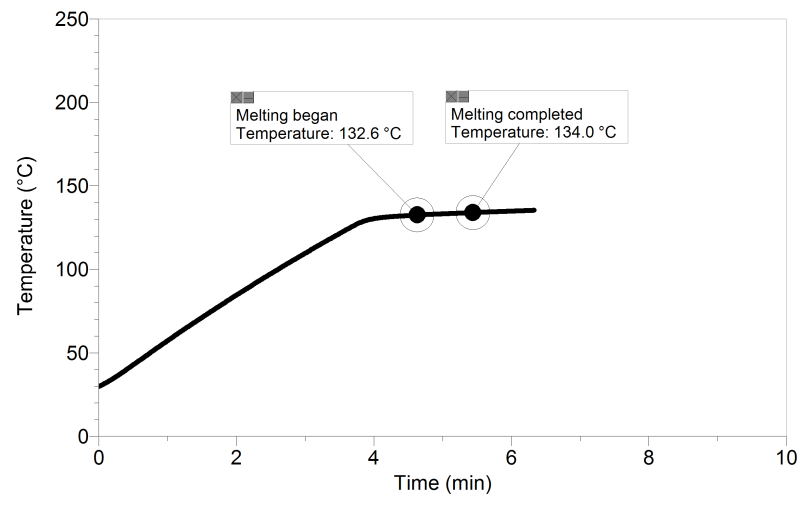
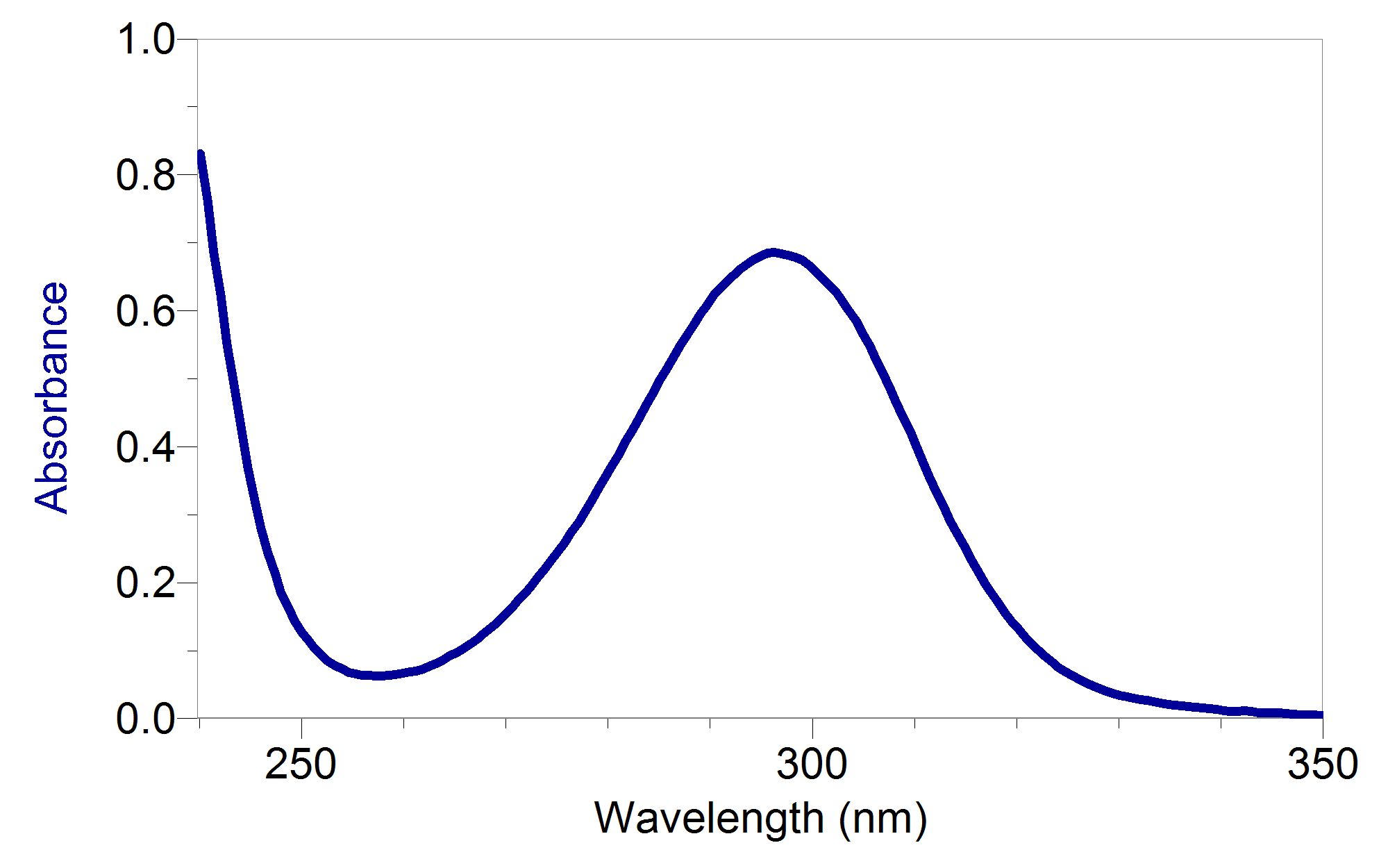
SAMPLE GRAPHS

Figure 1 Melting temperature determination for aspirin sample



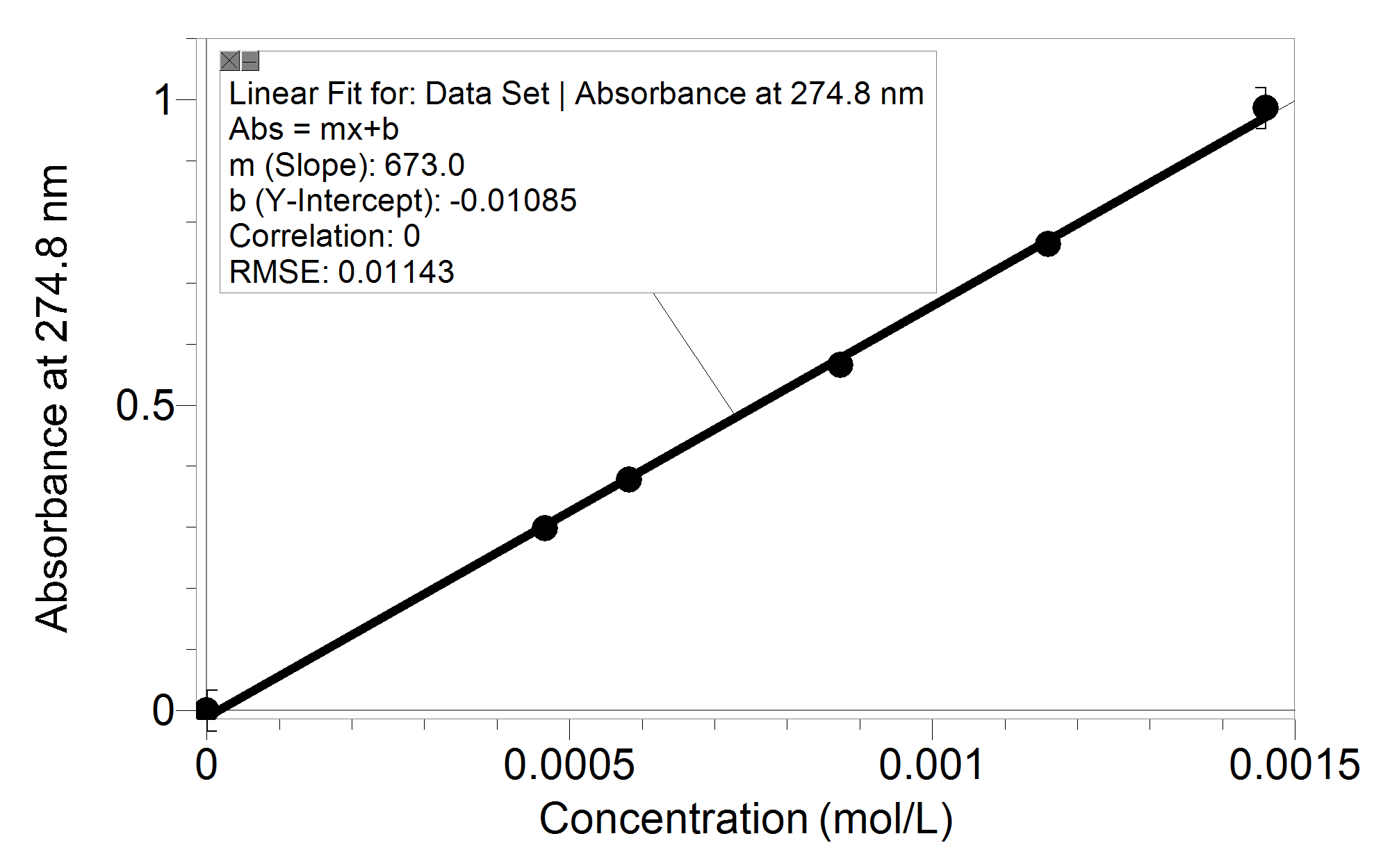
Figure 2 UV-Vis Spectrum for salicylic acid

Figure 3 Absorbance vs. concentration for salicylic acid