

## Degradation of Aspirin Lab Key

### Lab Prep

Equipment and glassware (each group will need what is listed)

- UV spectrometer
- (3) 1000 mL flasks
- 100 -1000  $\mu$ L eppendorf
- 1000 – 5000  $\mu$ L eppendorf
- 24 15 mL graduated test tubes
- (3) 100 mL volumetric flasks
- (3) 200 mL flasks
- Parafilm
- Labeling tape
- (3) Hot plates
- Thermometer
- Stopwatch
- Cuvets

Chemicals (the totals are based on 5 groups of three and some extra for mistakes. The amount in parenthesis shows the exact amount per group)

- 50 ml of 1mg/ml Salicylic Acid (6ml per group)
- 5g of Acetylsalicylic Acid (.6g per group)
- 40ml of Spectrophotometric grade Methanol (6ml per group)
- 2000ml of Phosphate buffered solution (360ml per group)
- 2000ml of DI H<sub>2</sub>O (180ml per group)

The students according to the following table should dilute the SA.

Conc. Of SA (mg/ml)	Amt of DI H <sub>2</sub> O (ml)	Amt of 1mg/ml soln of SA (ml)
.133	13.005	1.995
.100	13.500	1.500
.0667	14.000	1.000
.0333	14.500	.500
.0133	14.800	.200
.00667	14.900	.100

Table 5. Amounts needed to make the correct concentration of SA.

The tables should be close to the following ones. The absorbance will vary by spectrophotometer, but the amounts of SA and ASA should be close.

<b>Conc. Of SA (mg/mL)</b>	<b>Absorbance</b>
.133	2.27760
.100	1.73160
.0667	1.13210
.0333	.54758
.0133	.20349
.00667	.10918

Table 1. Absorbance of SA in certain concentrations

<b>Time</b>	<b>Absorbance</b>	<b>Concentration SA (mg/mL)</b>	<b>ASA degraded (mg)</b>	<b>ASA remaining (mg)</b>
0	0.01358	0.001906086	3.716867	196.2831
15	0.06975	0.005146526	10.03573	189.9643
30	0.13651	0.008997913	17.54593	182.4541
45	0.19744	0.012513019	24.40039	175.5996
60	0.26012	0.016129085	31.45172	168.5483
75	0.30673	0.018818058	36.69521	163.3048

Table 2. Absorbance and ASA remaining at 50°C.

<b>Time</b>	<b>Absorbance</b>	<b>Concentration SA (mg/mL)</b>	<b>ASA degraded (mg)</b>	<b>ASA remaining (mg)</b>
0	0.018869	0.002211097	4.31164	195.6884
15	0.29171	0.017951541	35.0055	164.9945
30	0.48655	0.029192036	56.92447	143.0755
45	0.65711	0.039031796	76.112	123.888
60	0.77066	0.045582598	88.88607	111.1139
75	0.87995	0.051887637	101.1809	98.81911

Table 3. Absorbance and ASA remaining at 70°C.

<b>Time</b>	<b>Absorbance</b>	<b>Concentration SA (mg/mL)</b>	<b>ASA degraded (mg)</b>	<b>ASA remaining (mg)</b>
0	0.035533	0.003172459	6.186294	193.8137
15	0.53657	0.032077735	62.55158	137.4484
30	0.90165	0.05313953	103.6221	96.37792
45	1.1224	0.065874796	128.4559	71.54415
60	1.2557	0.073564994	143.4517	56.54826
75	1.3468	0.078820635	153.7002	46.29976

Table 4. Absorbance and ASA remaining at 80°C.

There should be 5 graphs as follows:

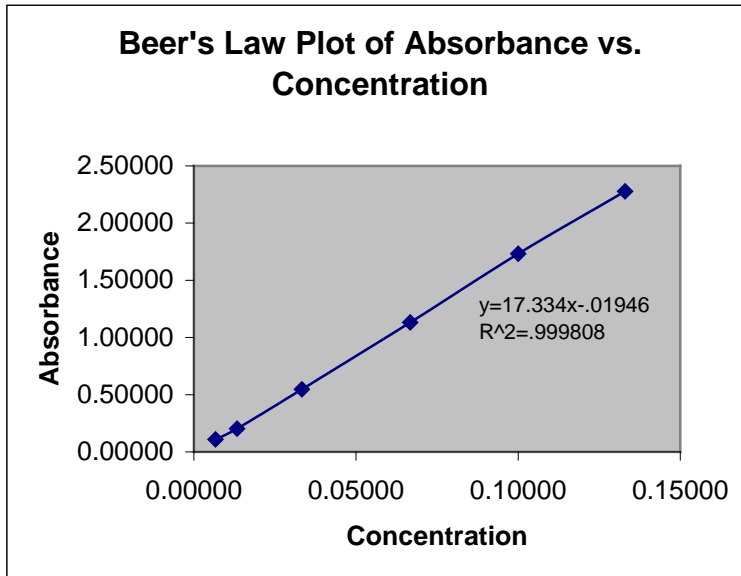


Figure 1. Beer's Law Plot.

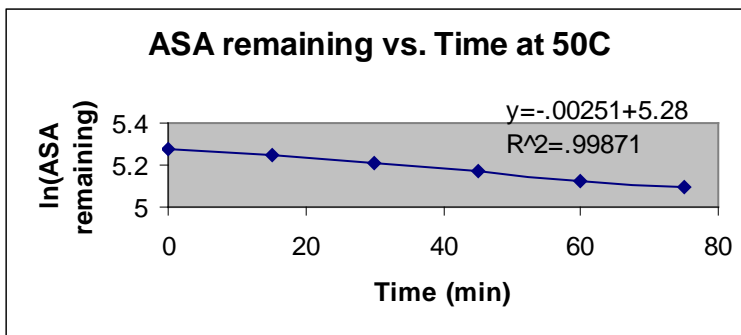


Figure 2. ASA remaining vs. Time at 50°C.

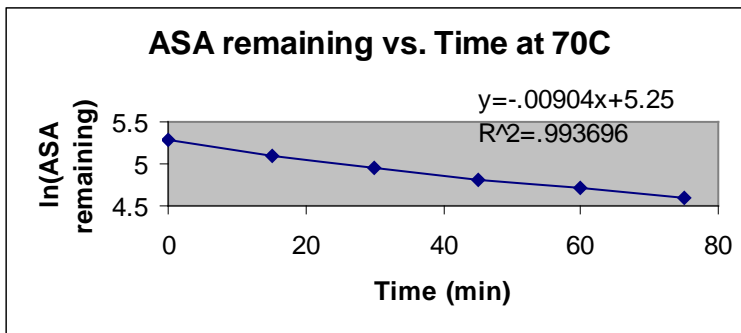


Figure 3. ASA remaining vs. Time at 70°C.

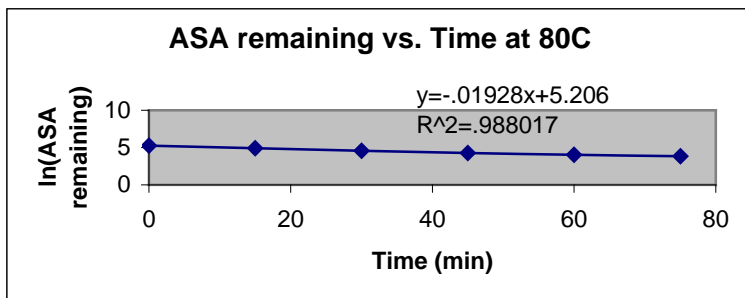


Figure 4. ASA remaining vs. Time at 80°C.

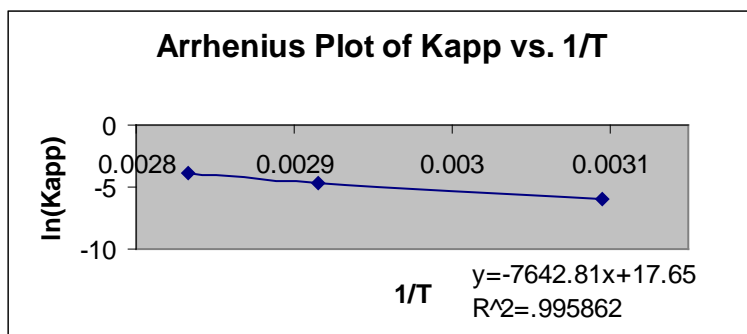


Figure 5. Arrhenius Plot of  $K_{app}$ .

This table should be filled out.

Temp (°C)	Temp (°K)	1/T (°K)	$K_{app}$ (min <sup>-1</sup> )	K (min <sup>-1</sup> )
50	323	.003096	.00251	7943
70	343	.002915	.00904	28608
80	353	.002833	.01928	61013

Table 6. Data Analysis

The  $E_a$  should be 15186 cal/mol.

### Sample Calculations

The sample calculations were done using the numbers from 50°C at 60 min.

$$\text{Conc.ofSA} = \frac{\text{absorbance} + .01946}{17.334}$$

$$\frac{.26012 + .01946}{17.334} = .01613$$

$$\text{ASAdgrded} = \text{concofSA} \cdot 15 \cdot 100\text{ml} \cdot 1.30$$

$$.01613 \frac{\text{mg}}{\text{ml}} \cdot 15 \cdot 100\text{ml} \cdot 1.30 = 31.45\text{mg}$$

$$\text{ASAremaing} = \text{InitialASA} - \text{ASAdgrded}$$

$$200\text{mg} - 31.45\text{mg} = 168.55\text{mg}$$

$$\ln(K_{app}) = \frac{-E_a}{R} \cdot \frac{1}{T} + \ln(A)$$

$$\ln(K_{app}) = -7642.81 * \frac{1}{298} + 17.65$$

$$\ln(K_{app}) = -7.997$$

$$K_{app} = e^{-7.997}$$

$$K_{app} = .000336$$

$$t_{90} = \frac{\ln\left(\frac{200}{180}\right)}{K_{app}}$$

$$t_{90} = 313.14 \text{ min}$$

$$t_{90} = 5.22 \text{ hrs}$$