

# Recrystallization and melting points of benzoic acid



**ASSIGN  
BUSTER**

## Experiment 1: Recrystallization and Melting Points of Benzoic Acid

**Purpose:** The purpose of this experiment was to use different methods of recrystallization in order to purify the sample of benzoic acid that is contaminated 5% salicylic acid. The class collectively tested four different methods of recrystallization to determine the most efficient method. The most efficient method was determined by measuring the melting point of the final products.

**Results:**

**Observations:**

The initial sample mixture of benzoic acid and salicylic acid was a white powder. About 1.0g of the sample was dissolved in minimum amount of boiling water. The dissolved solution was a clear solution with no impurities. After the solution was cooled to room temperature and cooled in an ice bath, precipitation was observed and the solution was not clear then. I performed the same experience for part A and B. A conical funnel was used to collect the crystals by vacuum filtration. After the vacuum filtration was completed, some solid particles were stuck to the funnel and also on the filter paper. The final product's mass, physical appearances, and melting points were collected in order to analyze the purity of the benzoic acid.

**Calculations:**

Table 1. Raw Data from Part A and B

Part	Initial Mass	Initial	Final Mass	Product Appearance	Melting
------	--------------	---------	------------	--------------------	---------

	(g)	Appearance	(g)		Point(°C)
A	1.01	Fine White Powder	0.97	White, sharp, shiny crystals	120.4 - 124.7
B	0.98	Fine White Powder	0.96	White, sharp, shiny crystals	101.3 - 107.8

Theoretical yield: Initial mass \* 95 % = Initial mass \* 0.95.

Part A:  $1.01\text{g} * 0.95 = 0.960\text{g}$

Part B:  $0.98\text{g} * 0.95 = 0.931\text{g}$

Percent Yield =  $\frac{\text{actual yield}}{\text{theoretical yield}} * 100$

%

Part A:  $\frac{0.97\text{g}}{0.96\text{g}} * 100\% = 101.04\%$

Part B:  $\frac{0.96\text{g}}{0.931\text{g}} * 100\% = 103.11\%$

Discussion:

Crystals that were collected from part A had a percent yield of 101.04% and a melting point range of 120.4 °C - 124.7 °C. Although the range of 4.3°C is not ideal, the melting point range that includes the literary value of benzoic acid of 122.4

°C [ 1 ]

suggests that the product is relatively pure in content. The white crystal-like product also matches the physical description of benzoic acid. The high percent yield over 100% might be result of the crystals not being dried for long enough, still retaining water in the final product. The exact same procedure was carried on for part B. In part B, the crystals collected had a percent yield of 103.11% and a melting point range of 101.3-107.8. The lower melting point range suggests that the second batch was more impure compared to the first batch. The final product of B still had the white crystal-like appearance with no visible impurities. The lower impurity of the second job might have been caused by having some of the impure mixture left over from part A in the laboratory equipment such as the beakers.

The first modification followed the same procedure as part A and produced the greatest amount of solids compared to other modifications. Dissolving the mixture in minimum volume of water allowed the solution to slowly cool down and increased the amount of benzoic that could be recrystallized into a solid. After the boiling water was added and the solution was cooled in the ice bath, the difference in solubility in benzoic acid and salicylic acid, 3.44g/L<sup>[1]</sup> and 2.48g/L<sup>[2]</sup> respectively, allowed only the benzoic acid to recrystallize and not the salicylic acid.

The second modification followed similar procedure as modification (i). The only difference between the two was in the beginning, 100mL of boiling water was added onto the mixture instead of minimum volume of boiling water. The large volume of boiling water cause the solution to be undersaturated. For a unsaturated solution, the rate of dissolution is much

greater than the rate of crystallization. <sup>[3]</sup> From this in this modification, in the same amount of time, the amount of benzoic solution that was crystallized was comparatively smaller than the first modification. However, with the solution being undersaturated, the melting points were closer to the actual melting points of benzoic acid, showing that it was more pure than the first modification.

Third modification was opposite of what happened for modification (ii). With reducing the amount of boiling water to half the original about, the solution was supersaturated. For a supersaturated solution, the rate of crystallization is much greater than the rate of dissolution. Because the process of crystallization is happening so fast, when the benzoic acid is crystallizing some of salicylic acid might also crystallize alongside. With this theory, the percent yield for modification (iii) should be the highest. However, it is shown as that the average percent yield is 65.41. This could be explained by the fact that since the original volume was so small, when transferring the solutions from one place to another, a large amount of the benzoic acid and/or salicylic acid might have been discarded.

The fourth modification reduced the cooling time in room temperature to one minute instead of 20 minutes. By putting the solution through a sudden drop without time at room temperature, it wouldn't allow the benzoic acid and salicylic acid to separate according to their different solubility at room temperature. So, with not enough time to separate, the percent yield will contain both benzoic acid and salicylic acid, explaining the high percent yield of 76.44%.

At last, modification (i) yielded the highest percent yield with a melting range that was close to the literature value of benzoic acid's melting point, meaning that it produced the most pure benzoic acid out of the four methods.

## Appendix

Table A1. Class data for modification (i) [4]

Trial	Melting Point (°C)	Range (°C)	Percent Yield (%)
1	115.2 – 117.8	2.6	98
2	118 – 121	3	91
3	112 – 118	6	138.30
4	101.3 – 107.8	6.5	103.11
5	121.8 – 124.1	2.3	84.00
6	92 – 95	3	136
Average		3.85	108.40

Table A2. Class data for modification (ii) [4]

Trial	Melting Point (°C)	Range (°C)	Percent Yield (%)
-------	-----------------------	---------------	----------------------

1	117 - 122	5	50.40
2	119.2 - 122.6	3.4	70
3	121.4 - 125.7	4.3	49.50
4	119.7 - 122.3	6.5	46
5	117.5 - 120.6	3.1	64
Average		4.46	55.98

Table A3. Class data for modification (iii) [4]

Trial	Melting Point (°C)	Range (°C)	Percent Yield (%)
1	116 - 120	4	62
2	120.5 - 122.8	2.3	62
3	122.4 - 123.6	1.2	63.80
4	120 - 123	3	52
5	106.8 - 107.5	0.7	76.85
6	118.9 - 120.3	1.4	75.80
Average		2.1	65.41

Table A1. Class data for modification (iv) [4]

Trial	Melting Point (°C)	Range (°C)	Percent Yield (%)
1	120.5 - 121.5	1.0	83.20
2	118.3 - 122.5	4.2	68.42
3	117.9 - 122.4	4.5	77.70
Average		3.2	76.44

## References

[1] Properties of Benzoic Acid

PubChem <https://pubchem.ncbi.nlm.nih.gov/compound/Benzoic-acid>

[2] Properties of Salicylic Acid

PubChem <https://pubchem.ncbi.nlm.nih.gov/compound/338>

[3] Types of saturation

LibreTexts [https://chem.libretexts.org/Bookshelves/Physical\\_and\\_Theoretical\\_Chemistry\\_Textbook\\_Maps/Supplemental\\_Modules\\_\(Physical\\_and\\_Theoretical\\_Chemistry\)/Equilibria/Solubility/Types\\_of\\_Saturation](https://chem.libretexts.org/Bookshelves/Physical_and_Theoretical_Chemistry_Textbook_Maps/Supplemental_Modules_(Physical_and_Theoretical_Chemistry)/Equilibria/Solubility/Types_of_Saturation)

[4] Yiye Lu class' data compilation

<https://assignbuster.com/recrystallization-and-melting-points-of-benzoic-acid/>



Google Sheets[https://docs.google.](https://docs.google.com/spreadsheets/d/1BedzY0nyUN65t8rtDNjYH6hZkx-jDJ1oODAqj4bNQol/edit#gid=1485732538)

[com/spreadsheets/d/1BedzY0nyUN65t8rtDNjYH6hZkx-jDJ1oODAqj4bNQol/edi](https://docs.google.com/spreadsheets/d/1BedzY0nyUN65t8rtDNjYH6hZkx-jDJ1oODAqj4bNQol/edit#gid=1485732538)

[t#gid= 1485732538](https://docs.google.com/spreadsheets/d/1BedzY0nyUN65t8rtDNjYH6hZkx-jDJ1oODAqj4bNQol/edit#gid=1485732538)