

1 Crystallization

Aug. 30, 2007

01

Name

~~XXXXXXXXXX~~

Course

321 L

Section

V07

Lab partner

T.A.

Jackie

Purpose: Take M.P. to identify unknown compounds, once known, attempt to purify it by recrystallization.

NAME

Table: Name	Molecular Structure	Molecular Weights	M.P.	B.P.	Density (@ 28°C)
Salicylic Acid ($C_7H_6O_3$)		138.123 g/mol	159°C	211°C	1.44 g/mL
Benzoic Acid (C_6H_5COOH)		122.12 g/mol	122.4°C	249°C	1.32 g/mL
Phthalic Acid ($C_6H_4(COOH)_2$)		166.14 g/mol	210°C	—	1.59 g/mL
Naphthalene ($C_{10}H_8$)		128.17 g/mol	80.2°C	218°C	1.14 g/mL
Biphenyl ($C_{12}H_{10}$)		154.21 g/mol	68.9°C	256°C	1.04 g/mL
Water (H_2O)		18.02 g/mol	0°C	100°C	1.0 g/mL
Ethanol (C_2H_5OH)		46.07 g/mol	-114.3°C	78.4°C	0.789 g/mL

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Crystallization #1

8-31-16

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~~Crystallization #1~~

32L

104

GTA-Solomon

Hazards Hazards

lactic Acid - harmful to eyes, skin & through swallowing or inhalation affects CNS, kidney & pancreas

nitric Acid - irritation to eyes, skin & respiratory tract

nitric Acid - irritation to eyes, skin & respiratory

naphthalene - flammable, harmful if inhaled or swallowed, irritant to skin & eyes

phenyl - same as naphthalene

thanol - flammable, skin & eye irritation

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T.A. Jackie

Procedure Part A.

- ① place 200 mg of Salicylic Acid into a 50 mL Erlenmeyer flask
- ② Add 2 mL of HOT H₂O (dropwise) ~ (5 mL - 10 mL)
- ③ Add boiling stick, Heat to B.P.
- ④ Check solid, if not all dissolved, repeat steps 2 & 3
- ⑤ Mark solvent line with sharpie, let cool to room Temp.
- ⑥ Place flask in ice-bath for 10 min and vacuum filter
- ⑦ Wash with cold H₂O to remove solid residue
- ⑧ Determine volume of H₂O used by filling flask to sharpie line, transfer H₂O to graduated cylinder
- ⑨ weigh recrystallized solid, record M.P. if assigned.
- ⑩ Repeat procedure for both benzoic acid and Phthalic acid.

Part B.

- ① place 200 mg of Naphthalene into 50 mL Erlenmeyer flask
- ② Dissolve (dropwise) into boiling Ethanol.
- ③ Remove from hot plate, add H₂O (dropwise) while swirling until solution remains slightly cloudy.
- ④ Reheat to B.P., if solution isn't clear, add ethanol until clear
- ⑤ Let cool to room temp., place in ice-bath for 10 min, then vacuum filter
- ⑥ rinse with cold ethanol to remove and solid residue
- ⑦ weigh the recrystallized solid, record M.P. if assigned.
- ⑧ Repeat procedure for biphenyl.

Observations

- ① once Phthalic Acid was removed from heat and set on table, solution turned cloudy immediately.
- ② Adding H₂O to hot ethanol - naphthalene acted like a titration.

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Bxbe

321 L

v 07

T.A. Jackie

Date:
(Results)

Initial weight of Salicylic Acid .20 g

Final weight of Salicylic Acid .17 g

Volume of H₂O used to Recrystallize 6.4 ml

Initial weight of benzoic acid .19g

Final weight of benzoic Acid .12g

Volume of H₂O used to Recrystallize 6.0 ml

Initial weight of Phthalic Acid .19g

Final weight of Phthalic Acid .13g

Volume of H₂O used to Recrystallize 5.8 ml

Compound: Salicylic Acid → M.P. 159.1°C - 162.5°C

Initial weight of Naphthalene .19g

Final weight of Naphthalene .12g

Initial weight of biphenyl .21g

Final weight of biphenyl .11g

Compound: Naphthalene → M.P. 77.1°C - 78.2°C

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POSTLABS EASIER!