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5. Cannibinol is only slightly soluble in methyl alcohol because the large hydrocarbon component of cannibinol negates the fact that they belong to the same family.

Experiment 2

CRYSTALLIZATION

TIME ESTIMATE: Parts A and B (3 hours), Parts C (about 1hour)

CHEMICALS AND SUPPLIES PER 10 STUDENTS:

Part A

Impure sulfanilamide (5% fluorenone as the impurity) Grind thoroughly to make homogeneous.	10 g
95% Ethyl alcohol	250 mL
Filter paper for Büchner funnel	
Melting point capillary tubes	
Waste container for non-halogenated organic wastes.	

Part B

The appropriate solvent for crystallizing the impure fluorene is methyl alcohol. Fluorene is too soluble in toluene and insoluble in water at all temperatures.

Impure fluorene (5% fluorenone as the impurity) Grind thoroughly to make homogeneous.	10 g
Methyl alcohol	300 mL

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Toluene	25 mL

Waste container for non-halogenated organic wastes.

Part C

Acetylsalicylic acid	5 g
Benzoic acid	5 g
Benzoin	5 g
Dibenzoyl ethylene	5 g
Succinimide	5 g
o-Toluic acid	5 g

Prepare unknowns consisting of pure samples of the above 6 compounds; about 0.2 g for each unknown sample.

Part D (Answers)

1. Phenanthrene
   95% ethyl alcohol - best solvent
   water - not soluble
   toluene - too soluble

2. Cholesterol
   ether - too soluble
   95% ethyl alcohol - best solvent
   water - not soluble

3. Acetaminophen
   95% ethyl alcohol - too soluble
   water - best solvent
   toluene - not very soluble

4. Urea
   Water - too soluble
   95% ethyl alcohol - best solvent
   Hexane - not very soluble

CAS Registry numbers:

Sulfanilamide	63-74-1

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Acetanilide	103-84-4
95% Ethyl alcohol (Ethanol)	64-17-5
Fluorene	86-73-7
Fluorenone	486-25-9
Methyl alcohol (methanol)	67-56-1
Toluene	108-88-3
Acetylsalicylic acid	50-78-2
Benzoic acid	65-85-0
Benzoin	119-53-9
Dibenzoyl ethylene	4070-75-1
Succinimide	123-56-8
o-toluic acid	118-90-1

SPECIAL NOTES

In the Pre-lab Calculations for Part A, students calculate the amount of sulfanilamide which will remain in the mother liquor. If they perform the Optional Exercise in Part A, they determine the weight of solid in the mother liquor. However, the actual weight of solid in the mother liquor is usually much greater than the amount calculated in the Pre-lab Calculations. This is because the calculation does not take into account the impurity, which ends up in the mother liquor. Also, the calculation assumes that a minimum amount of solvent is used to dissolve the impure sulfanilamide at 78 ºC. It is likely that most students use more than the minimum amount.

ANSWERS TO QUESTIONS

1. Too much solvent was added. Since 10 mL of 95% ethyl alcohol will dissolve 0.14 g of sulfanilamide at 0 ºC, none of the 0.1 g of sulfanilamide will crystallize when the solution is cooled. To make the crystallization work, the excess solvent must be evaporated.

2. The boiling point of the solvent is higher than the melting point of fluorenol. While performing this crystallization, it is possible that the fluorenol would melt rather than dissolve, thus forming an oil which could be difficult to crystallize.

3. Biphenyl is highly soluble in both hot and cold benzene. The solubility curve would like **C** in Figure 11.1 on page 665 of the Textbook.

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