Introductory Extraction Investigation for 4 students (2 pairs of students)

Pair 1

Student 1

Weigh 0.050 grams aspirin on a weigh boat. Transfer the aspirin to a small test tube. Add 2 mL ethyl acetate to the aspirin sample, swirl vigorously to dissolve. You may dip the test tube in a beaker of warm water to hasten dissolving. Once the aspirin is dissolved add 1 mL of 1 M potassium hydroxide, KOH (in water) to the small test tube. Record whether the KOH solution dissolves in the ethyl acetate. If two layers are present, which layer is ethyl acetate and which is water? If two layers are present, use a pipet to squirt the lower layer through the upper layer several times. If two layers are present allow the layers to separate.

Once any layers have separated, use the pipet to transfer the bottom layer to another small test tube. For this qualitative study, you do not need to transfer all of the bottom layer, but try not to transfer the top layer to the second test tube. You should now have the ethyl acetate layer in one test tube and the water layer in another test tube. You can keep tract of which is which by remembering how much of each solvent, ethyl acetate, or KOH solution (water) you used.

Add 6 M hydrochloric acid, HCl, dropwise (2 to 6 drops) to the test tube with water as the solvent. Record any changes that would indicate a chemical reaction is occurring as the HCl is added. Record your information in the Table below and prepare to discuss your results with your group.

Student 2

Weigh 0.050 grams aspirin on a weigh boat. Transfer the aspirin to a small test tube. Add 2 mL ethyl acetate to the aspirin sample, swirl vigorously to dissolve. You may dip the test tube in a beaker of warm water to hasten dissolving. Once the aspirin is dissolved add 1 mL of 1 M K_2 HPO₄/KH₂PO₄ 19/1 buffer solution (in water) to the small test tube. Record whether the phosphate buffer solution dissolves in the ethyl acetate. If two layers are present, which layer is ethyl acetate and which is water? If two layers are present, use a pipet to squirt the lower layer through the upper layer several times. If two layers are present allow the layers to separate.

Once any layers have separated, use the pipet to transfer the bottom layer to another small test tube. For this qualitative study, you do not need to transfer all of the bottom layer, but try not to transfer the top layer to the second test tube. You should now have the ethyl acetate layer in one test tube and the water layer in another test tube. You can tract of which is which by remembering how much of each solvent, ethyl acetate, or phosphate buffer solution (water) you used.

Add 6 M hydrochloric acid, HCl, dropwise (2 to 6 drops) to the test tube with water as the solvent. Record any changes that would indicate a chemical reaction is occurring as the HCl is added. Record your information in the Table below and prepare to discuss your results with your group.

Student 3

Weigh 0.050 grams phenyl salicylate on a weigh boat. Transfer the phenyl salicylate to a small test tube. Add 2 mL ethyl acetate to the phenyl salicylate sample, swirl to dissolve. Once the phenyl salicylate is dissolved add 1 mL of 1 M potassium hydroxide, KOH (in water) to the small test tube. Record whether the KOH solution dissolves in the ethyl acetate. If two layers are present, which layer is ethyl acetate and which is water? If two layers are present, use a pipet to squirt the lower layer through the upper layer several times. If two layers are present allow the layers to separate.

Once any layers have separated, use the pipet to transfer the bottom layer to another small test tube. For this qualitative study, you do not need to transfer "all" the bottom layer, but try not to transfer the top layer to the second test tube. You should now have the ethyl acetate layer in one test tube and the water layer in another test tube. You can keep tract of which is which by remembering how much of each solvent, ethyl acetate, or KOH solution (water) you used.

Add 6 M hydrochloric acid, HCl, dropwise (2 to 6 drops) to the test tube with water as the solvent. Record any changes that would indicate a chemical reaction is occurring as the HCl is added. Record your information in the Table below and prepare to discuss your results with your group.

Student 4

Weigh 0.050 grams phenyl salicylate on a weigh boat. Transfer the phenyl salicylate to a small test tube. Add 2 mL ethyl acetate to the phenyl salicylate sample, swirl to dissolve. Once the phenyl salicylate is dissolved add 1 mL of 1 mL of 1 M K₂HPO₄/KH₂PO₄ 19/1 buffer solution (in water) to the small test tube. Record whether the phosphate buffer solution dissolves in the ethyl acetate. If two layers are present, which layer is ethyl acetate and which is water? If two layers are present, use a pipet to squirt the lower layer through the upper layer several times. If two layers are present allow the layers to separate.

Once any layers have separated, use the pipet to transfer the bottom layer to another small test tube. For this qualitative study, you do not need to transfer "all" the bottom layer, but try not to transfer the top layer to the second test tube. You should now have the ethyl acetate layer in one test tube and the water layer in another test tube. You can keep tract of which is which by remembering how much of each solvent, ethyl acetate, or phosphate buffer solution (water) you used.

Add 6 M hydrochloric acid, HCl, dropwise (2 to 6 drops) to the test tube with water as the solvent. Record any changes that would indicate a chemical reaction is occurring as the HCl is added. Record your information in the Table below and prepare to discuss your results with your group.

Pair 2

Pair X / Student X	Compound	Functional group	Appearance after addition of KOH	Appearance after addition of phosphate buffer	Appearance after treatment with KOH, followed by HCl	Appearance after treatment with phosphate buffer followed by HCl
Pair 1/ Student 1		Carboxylic Acid				
Pair 1/ Student 2	C C H ₃	Carboxylic Acid				
Pair 2/ Student 3		Phenol				
Pair 2/ Student 4		Phenol				

When either aspirin or phenyl salicylate is dissolved in ethyl acetate and "reacts" with either KOH or phosphate buffer solution what if any physical changes are observed?

When the aqueous solution from aspirin or phenyl salicylate "reacting" with KOH or phosphate buffer solution subsequently "reacts" with added hydrochloric acid what if any physical changes are observed?

As a group of four students you just investigated the reaction of a representative carboxylic acid, aspirin with two different basic aqueous solutions. You also investigated the reaction of a representative phenol, phenyl salicylate with two different basic aqueous solutions. The big question you need to answer is: if you have a mixture of both a carboxylic acid and a phenol how can you use these two reactions to separate the carboxylic acid from the phenol?

(Hints: What will be the result if the mixture of the acid and the phenol are reacted with KOH solution? What will be the result if the mixture of the acid and the phenol are reacted with phosphate buffer solution?)

Describe in as much detail as possible what operations you need to do in order to get both the acid and the phenol separated from each other. When you know how to separate the representative carboxylic acid and the phenol continue the laboratory investigation in a more quantitative manner using the very handy separatory funnel.