Beckfoot

Subject: Chemistry	Topic: Acid derivatives & Acylation 3.3.9.2	Year Group: 13

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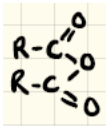
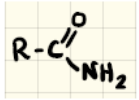
Carboxylic acid derivatives

1	Acyl chlorides	The functional group is RCOCl 
		Named as **– anoyl chloride**, e.g. CH_3COCl is called ethanoyl chloride
2	Acid anhydrides	The functional group is: 
		Named as **–anoic anhydride**. For example, $(CH_3CO)_2O$ is ethanoic anhydride
3	Amides	The functional group is $RCONH_2$ 
		Named as **–anamide**. For example, CH_3CONH_2 is ethanamide

Equations for acylation / nucleophilic addition-elimination

1	Acyl chloride + water example	$CH_3COCl + H_2O \rightarrow CH_3COOH + HCl$
2	Acyl chloride + alcohol example	$CH_3COCl + CH_3OH \rightarrow CH_3COOCH_3 + HCl$
3	Acyl chloride + ammonia example	$CH_3COCl + 2NH_3 \rightarrow CH_3CONH_2 + NH_4Cl$
4	Acyl chloride + primary amine example	$CH_3COCl + 2CH_3NH_2 \rightarrow CH_3CONHCH_3 + CH_3\dot{N}H_2Cl^-$

Equations for acylation / nucleophilic addition-elimination

1	Acid anhydride + water example	$(CH_3CO)_2O + H_2O \rightarrow 2CH_3COOH$
2	Acid anhydride + alcohol example	$(CH_3CO)_2O + CH_3OH \rightarrow CH_3COOCH_3 + CH_3COOH$
3	Acid anhydride + ammonia example	$(CH_3CO)_2O + 2NH_3 \rightarrow CH_3CONH_2 + CH_3COO^-\dot{N}H_4$
4	Acid anhydride + primary amine example	$(CH_3CO)_2O + 2CH_3NH_2 \rightarrow CH_3CONHCH_3 + CH_3COO^-\dot{N}H_2CH_3$

Key Vocabulary

1	Acid derivatives	Compounds that are related to carboxylic acids; the OH group has been replaced by something else
2	Acylation	The process of replacing a hydrogen atom in certain molecules by an acyl group (RCO-)
3	Acylating agents	Compounds that carry out acylation by introducing the acyl group into another compound. *Acyl chloride & acid anhydrides* are examples
4	Recrystallisation	Practical method used to **purify an organic solid**
5	Refluxing	A method of heating a reaction so that you can increase the temperature of an organic reaction to boiling without losing volatile substances. Any vaporized compounds are cooled, condensed & drip back into the reaction mixture

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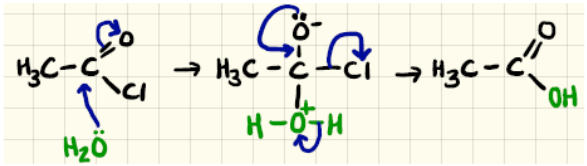
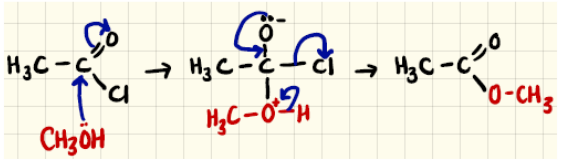
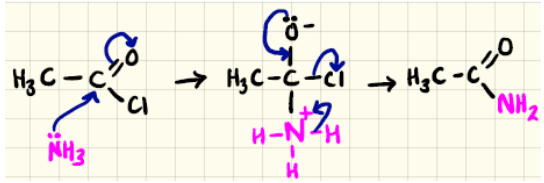
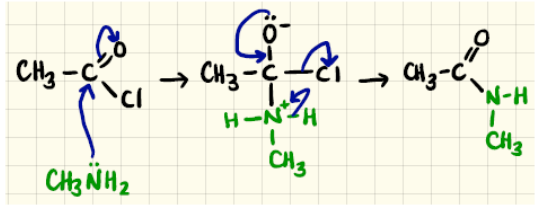
Nucleophilic addition mechanism

1	Ethanoyl chloride + water example	
2	Ethanoyl chloride + methanol example	
3	Ethanoyl chloride + ammonia example	
4	Ethanoyl chloride + methylamine example	

Acylation reactions: what you need to know

1	You need to write equations for the reaction of acyl chlorides and acid anhydrides, separately with water, alcohols, ammonia and primary amines
2	You need to draw mechanisms for the reaction of acyl chlorides with water, alcohols, ammonia and primary amines. These are all **nucleophilic addition mechanisms**.

Industrial advantages of ethanoic anhydride in making aspirin

1	Safer as no corrosive HCl formed	3	Less hazardous to use as gives a less violent reaction
2	Cheaper than ethanoyl chloride	4	Less vulnerable to hydrolysis unlike ethanoyl chloride

Key Vocabulary

6	Solvent extraction	A form of separation, where the product is shaken vigorously with an immiscible solvent
7	Separating funnel	Equipment used to separate water-soluble impurities out of an organic mixture. The aqueous & organic solutions can be separated as they are immiscible, & separate out into two distinct layers due to their different densities
8	Washing	A method of purifying a product by washing it with chemicals, such as washing with $NaHCO_3$ solution to remove acids
9	Distillation	A method of separating liquids with different boiling points by gently heating them

Acylation reactions: practical based Questions and Answers

1	Give 2 reasons why the % **yield from recrystallisation may be < 100%**	Product left in glassware / sample lost in solvent / not all of the product recrystallises / lost in transfer between equipment
2	Give **2 reasons why the % yield from the purification of a liquid ester may be < 100%**	Side reactions may occur / reagents may be impure / reaction incomplete / loss in washing & transfer
3	Give **1 reason why the % yield from recrystallisation may be > 100%**	Sample contains solvent (i.e. not dried completely) or impurities
4	Describe how you would **determine the purity of your organic solid**	Take the melting point by putting sample in a capillary tube & inserting into a melting point apparatus. Heat slowly towards the expected melting point

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Practical Techniques RP10a: Recrystallisation steps (making a pure organic solid)		
1	**Choose an appropriate SOLVENT to dissolve your impure solid**	Makes saturated solution so highest yield of product obtained as maximum amount crystallises out when temp of solvent falls
2	**Dissolve the impure solid in a MINIMUM volume of HOT SOLVENT**	One in which solid is highly soluble in hot solvent but only sparingly soluble at room temperature so desired solid can crystallise out when solution cools (**impurities remain dissolved**)
3	**Carry out HOT FILTRATION of your saturated solution**	Removes insoluble impurities and filtrate will contain your desired product & any soluble impurities. Keep apparatus hot so prevents desired product crystallising out
4	**Allow the FILTRATE to cool slowly**	Allows the desired product to crystallise. If done in an ice bath or too quickly some impurities may crystallise out too
5	**Carry out REDUCED PRESSURE / SUCTION FILTRATION**	Faster than gravity filtration & gives a drier solid. Allows the desired crystals to be separated from solvent (formed as residue). Removes soluble impurities
6	**Wash RESIDUE (recrystallized crystals) with a small amount of COLD SOLVENT**	This washes away the contaminated solvent covering the crystals & any soluble impurities they may contain. Use cold solvent so crystals aren't dissolved & washed away
7	**DRY the crystals between 2 sheets of filter paper**	Since solvent is pure it leaves no residue apart from the product
8	**CHECK the purity of the crystals by determining the MELTING POINT**	A pure substance has a fixed melting point. The smaller the range & closer to the correct value, the purer the product

Practical Techniques RP 10b: making a pure organic liquid		
1	Practical steps involved	Reflux, separation (with separating funnel) and distillation
2	When making the liquid ester, why is the concentrated sulfuric acid added slowly with swirling?	It is an exothermic reaction so reduces splashing of mixture & prevents any part of it getting too hot
3	Why are anti-bumping granules added to the flask?	To prevent large bubbles from forming so that the mixture boils smoothly
4	Why is the crude liquid ester added to water in the separating funnel?	To remove water-soluble impurities
5	How are acidic impurities removed from the mixture?	By shaking crude liquid ester with sodium carbonate in a separating funnel
6	When removing acidic impurities by inverting the separating funnel, why is it important to open the tap regularly?	To remove carbon dioxide gas & prevent pressure build-up
7	What step is carried out to remove any water left in the organic ester?	Dry it by adding anhydrous sodium sulfate (VI) or another drying agent
8	How do you know when the drying agent has removed all the water from the organic ester?	The liquid becomes clear and stops clumping
9	Why is the yield less than 100%?	Side-reactions occur, product lost in washing & transfer between apparatus; reaction may be incomplete; some product lost in distillation

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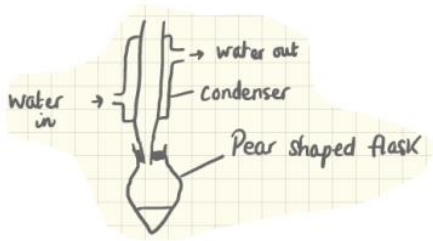
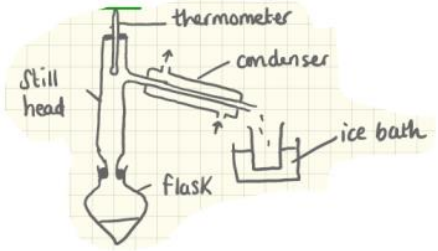
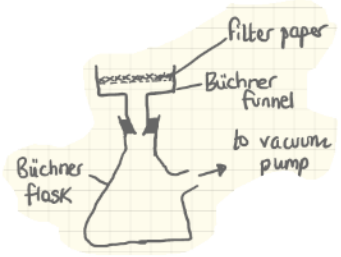
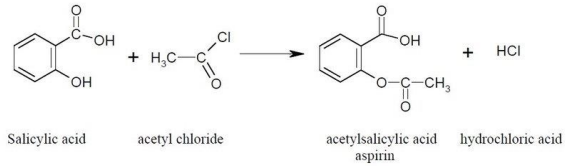
Apparatus Set-ups / Diagrams

1	Reflux	
2	Distillation	
3	Reduced pressure Filtration	

Making aspirin equations

1	With an acyl chloride	 Salicylic acid acetyl chloride acetylsalicylic acid aspirin hydrochloric acid
2	With an acid anhydride (preferred method)