

Equipment & Chemical Preparation

Preparation of Triphenylmethanol: The Grignard Reaction

In this two-week experiment, students will work *individually* to prepare triphenylmethanol from phenyl magnesium bromide (generated from bromobenzene and magnesium) and benzophenone. Students will prepare the product the first week (2 steps in the reaction sequence) and purify and analyze the triphenylmethanol the second week.

In the first step of the reaction, students will generate phenyl magnesium bromide from bromobenzene and magnesium metal (aka magnesium turnings). The procedure requires that the magnesium turnings (~0.5g per group) are placed in a 100 ml rb flask and placed in the drying oven (~110C) for 30 minutes. After the flask is allowed to cool, bromobenzene (3.5g) and ether is added. **Since ether is so volatile, it evaporates rapidly. It is likely we will go through a lot of ether and the ether will need to be replenished frequently. Therefore, a significant excess of ether is needed and should be ordered.** Students should be reminded repeatedly and frequently to cap the ether bottles and to keep the ether in the hood. Once the Grignard reagent is prepared, it is added to a solution of benzophenone in ether. Aqueous acid (5% HCl) is used to quench the reaction. The reaction mixture is transferred to a separatory funnel and more ether is added. The ether layer (containing the product) is separated from the aqueous layer and washed with 5% sodium bicarbonate and saturated sodium chloride solution. The product is triturated in hexane and analyzed in the second week of the experiment by melting point determination, TLC analysis and IR spectroscopy.

Glassware/Equipment
24 Hot Plate/Stirrers (one in each hood)**
24 Ring Stands with a ring and clamp**(2 in each hood)
24 Magnetic Stir Bars
24 100ml round bottomed flasks**
24 125ml separatory funnels
24 condenser tubes
24 drying tubes w/ drying agent
24 water baths (glass)
24 drying tubes
24 100ml heating mantles
24 powder funnels (glass)
24 50ml Erlenmeyer flasks
24 200 ml beakers
24 vacuum filtration apparatus (Buchner funnel, 125 ml vacuum flask)
24 watch glasses
24 TLC set ups (400ml beaker, watch glass)
12 I ₂ chambers; 12 UV lamps

Chemicals/Supplies
Bromobenzene (3.5g per student)
Magnesium turnings (0.5g per student)
Diethylether (~80ml per student)
Benzophenone (3.7g)
5% HCl (15 ml per student)
Large filter paper for gravity filtration (1 /student)
Small filter paper for vacuum filtration (1/student)
5% Sodium bicarbonate(15ml/student)
Saturated sodium chloride (15ml/student)
Hexane (10ml/student)
Ethanol (5ml per student; 100ml for TLC standards)
70:30 hexane:ethyl acetate (20ml per student)
Triphenylmethanol (~10g for TLC standard)
Vials (1 per student)
Disposable pipets with bulbs (2 for each student)
Melting Point cover slips/tubes (1 per student)
Capillary tubes for TLC (4 per student)
TLC plates (1 per student)

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For S2009 (261 students)

Chemicals	per student	for 261
bromobenzene	3.5	1 kg
magnesium	0.5	200 g
ether	80	21 liters
5% HCl	15	4 liters
5% sodium bicarbonate	15	4 liters
Saturated sodium chloride	15	4 liters
hexane	10	3 liters
ethanol	5	1.5 liters
70:30 hexane:ethyl acetate	20	5.5 liters
triphenylmethanol		10 g
benzophenone	3.7	1 kg
Supplies	per student	for 261
Lg Filter paper	1	4 boxes/100
Sm Filter paper	1	4 boxes/100
Glass pipets	2	1 box/1000
TLC plates	1	261
		12
TLC capillaries	4	boxes/100
MP tubes/slips	1	2 boxes/100
Vials	1	261