SYNTHESES OF YOUTUBE

SELECTED PROCEDURES FROM 42 CHANNELS

Abstract

A compiled document consisting of journal-style procedures taken from a wide range of YouTube channels with the aim of preserving and condensing the essential information from nearly 400 videos.

12thealchemist

Contact me via U2U on sciencemadness if you have any burning comments

Abbreviated Table of Contents

Detailed Table of Contents

12thealchemist

Preparation of antimony

$Sb_2S_3 + 2\ Al \rightarrow 2\ Sb + Al_2S_3$

Aluminium powder (2 g) was mixed thoroughly with antimony trisulphide (18 g) and ignited with a magnesium ribbon. After allowing to cool to room temperature, the result was digested with hydrochloric acid (2 M) and the precipitate filtered off and washed well with water. The final yield was 12 g (93%) of black powder.

Preparation of silicon

$SiO_2 + 2\ Mg \rightarrow Si + 2\ MgO$

Sand (10 g) was mixed thoroughly with magnesium powder (8 g). The mixture was piled onto a steel plate and ignited with a blowtorch. When the resulting friable mass had cooled down, it was digested with hydrochloric acid (50 mL, 31%) and allowed to react overnight. The black precipitate was filtered off, washed with water, and air-dried.

Preparation of manganese

$7\ MnO_2 + C_6H_4O_7 + 7\ H_2SO_4 \rightarrow 7\ MnSO_4 + 6\ CO_2 + 9\ H_2O$

$MnSO_4 + FeSO_4 + 5\ NaOH \rightarrow Mn(OH)_2 + Na[Fe(OH)_4] + 2\ Na_2SO_4$

$2\ Mn(OH)_2 + O_2 \rightarrow 2\ MnOOH + 2\ H_2O$

$2\ MnOOH \rightarrow Mn_2O_3 + H_2O$

$Mn_2O_3 + 2\ Al \rightarrow 2\ Mn + Al_2O_3$

To a 600 mL beaker was added water (500 mL) and crude manganese dioxide (100 g) and stirred thoroughly. The solids were filtered off and washed well with water. To a 600 mL beaker was added citric acid (12 g), water (100 mL), and sulphuric acid (65 mL, 98%) and stirred until all the solids had dissolved. Then the manganese dioxide was continually added until the solution stopped bubbling (~100 g). The precipitate was filtered off, and to the filtrate was added excess sodium hydroxide. The precipitate was filtered off, and washed with water, then allowed to air dry. The precipitate was then heated to ~300°C to decompose the oxyhydroxide to manganic oxide. After completely drying and oxidising, the soft dark brown powder was crushed up and mixed with a stoichiometric quantity of aluminium powder, then ignited with magnesium powder. After allowing to cool, the reaction mixture was digested with sulphuric acid (2 M) and the large clumps of manganese metal retrieved.

Preparation of yttrium chloride

$Y_2O_3 + 6\ HCl \rightarrow 2\ YCl_3 + 3\ H_2O$

A fluorescent tube that had ceased to work was carefully broken at one end, and a small amount of water added. Using a large bottlebrush, the powder on the inside of the tube was washed off and suspended in the water. The suspension of phosphor was then poured out, and hydrochloric acid (20 mL, 31%) added to dissolve the yttrium oxide. The insoluble material (consisting mainly of lanthanum phosphate) was filtered off and washed well. The filtrate was then evaporated to dryness.

Preparation of mercury

$6\ NaOH + 7\ S = 2\ Na_2S_3 + Na_2SO_3 + 3\ H_2O$

$Na_2S_3 + HgS = Na_2HgS_4$

$8\ NaOH + 2\ Al + 3\ Na_2HgS_4 = 2\ NaAl(OH)_4 + 3\ Na_2S_3 + 3\ Na_2S + 3\ Hg$

To a 250 mL beaker was added sodium hydroxide (7.2 g), sulphur powder (0.96 g), and water (100 mL) and the reaction mixture stirred and heated until all the solids had dissolved. Cinnabar (4.9 g) was then added to the reaction mixture and stirred well for 30 minutes. Aluminium foil was then added in small portions[1] until no more dissolved. The aqueous layer is decanted, and the residue washed well with water. The mercury metal was removed with a pipette and filtered through a pinhole. The final yield was 3.8 g.

(1) Hydrogen sulphide is evolved at this point.

Synthesis of tetraethyl orthosilicate

$Si + S \rightarrow SiS_2$

$2\ Al + 3\ S \rightarrow Al_2S_3$

$SiS_2 + 4\ EtOH \rightarrow Si(OEt)_4 + 2\ H_2S$

Synthesis of silicon sulphide

Silicon (3 g), aluminium (3 g), and sulphur (12 g) were mixed thoroughly and heated in a steel crucible (mistake). Upon ignition of the mixture, it reached a glowing white-heat (no way of measuring temperature) and melted through the crucible with great ease. This was allowed to cool, and the whole mixture of reaction product and crucible was put into azeotropic ethanol.

Synthesis of tetraethyl orthosilicate

The reaction mixture was allowed to sit and evolve gases outside overnight, and the following day the remains of the crucible was removed and the mixture filtered to remove unreacted particulate. The ethanol was distilled off, leaving a high boiling residue that was considered to be the final product. It was a rich orange-brown liquid that produced a white precipitate when a little was added to water. The resulting aqueous solution reduced acidified dichromate.

Synthesis of potassium cuprate(III)

$K + O_2 \rightarrow KO_2$

$KO_2 + CuO \rightarrow KCuO_3$

A glass jar with a previously-burned steel lid was filled with oxygen and inverted. A small piece of potassium metal was placed in the centre of the lid and covered with copper(II) oxide. The lid was fitted to the jar, and the potassium heated strongly to ignite it. When the flames had died down, it was found that a blue-grey solid had formed on the inside of the lid, surrounded by a yellow-orange crust and unreacted copper oxide. It was concluded that the blue-grey material was potassium cuprate(III).

Preparation of boron

$Na_2B_4O_7 + 2\ HCl + 5\ H_2O \rightarrow 2\ NaCl + 4\ H_3BO_3$

$2\ H_3BO_3 \rightarrow B_2O_3 + 3\ H_2O$

$B_2O_3 + 3\ Mg \rightarrow 2\ B + 3\ MgO$

Synthesis of boric acid

To a 250 mL beaker was added hydrochloric acid (2 M) and the maximum amount of borax dissolved, heating the solution to boiling. The reaction mixture was allowed to cool to 0°C and the precipitate filtered off and washed with ice-water.

Synthesis of boron

To a large crucible was added boric acid (15 g) and was heated strongly (450-500°C) until steam ceased to be evolved. The glassy material was allowed to cool and was chipped out of the crucible. The resulting hard material was crushed with difficulty to a powder and mixed with magnesium powder (9 g) and ignited. The reaction mixture was allowed to cool to room temperature, and the result digested with dilute hydrochloric acid, and the precipitate filtered off.

AlchemicalGarden

Synthesis of potassium trichromate

$3K_2Cr_2O_7 + 2HNO_3 \rightarrow 2K_2Cr_3O_{10} + H_2O + 2KNO_3$

To a large test tube was added potassium dichromate (5 g) and nitric acid (10 mL, 69%), and the reaction mixture heated strongly until all the solids had dissolved, then allowed to cool to room temperature, and allowed to crystallise in the refrigerator for several days. The precipitate was filtered off, and dried in a desiccator over sodium hydroxide. The yield was of blood-red crystals. The compound is unstable in aqueous solution; it decomposes to potassium dichromate.

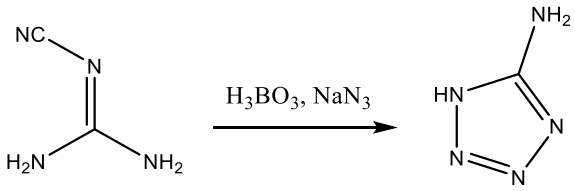
Synthesis of potassium tetraperoxochromate

$2K_2CrO_4 + 9H_2O_2 + 2KOH \rightarrow 2K_3CrO_8 + 10H_2O + O_2$

To a beaker was added potassium dichromate (5 g), potassium hydroxide (5 g), and water (30 mL), then stirred to dissolve and chilled to 0°C. Chilled (0°C) hydrogen peroxide (40 mL, 15%) was then added dropwise to the chromate solution with stirring, then allowed to stir in the ice bath for 2.5 hours. The precipitate was then filtered off, and washed with ice-water, then sucked dry. The final yield was of fine black needles.

AllChemystery

Synthesis of 5-aminotetrazole



To a 1000 mL flat bottom flask fitted with a reflux condenser cooled with ice-water was added boric acid (55 g), dicyandiamide or 2-cyanoguanidine (50 g), sodium azide (38.71 g), and distilled water (500 mL). Stirring was started and the flask heated to reflux for 24 hours. At the end of this time hydrochloric acid (75 mL, 31%) was added while the flask was still hot and then allowed to cool before being placed in the refrigerator for 2 days to fully precipitate the product. This was filtered and recrystallized from hot water yielding snow-white plate 5-aminotetrazole.

Synthesis of calcium cyanamide

$Na_2C_3N_3HO_3 + CaCl_2 \rightarrow CaC_3N_3HO_3 \rightarrow CaCN_2$

To a 3000 mL Erlenmeyer flask was added boiling distilled water (1800 mL), cyanuric acid (82.5 g), sodium hydroxide solution (40 g in 100 mL water), then stirred for 15 minutes. Calcium chloride (55 g) dissolved in water (250 mL) was then added and stirred for a further 30 minutes. The precipitate was filtered off and washed thoroughly with water. The resulting dry powder was then heated to 700°C for 2 hours when the resulting gases were no longer flammable. The final yield was 71 g.

Preparation of bromine

$Cl_2 + 2NaBr \rightarrow 2NaCl + Br_2$

To a 1000 mL two-necked round bottomed flask fitted with a pressure-equalising addition funnel set up for distillation was added a solution of sodium bromide (150 g) in distilled water (350 mL), TCCA (75 g). Hydrochloric acid (65 mL, 15%) was added to the addition funnel, and then slowly added. The reaction mixture was distilled, water removed from the distillate and dried over sulphuric acid (98%). The final yield was 85 g.

Synthesis of hydrazine sulphate

$NaOCl + CO(NH_2)_2 + H_2SO_4 \rightarrow N_2H_5{\bullet}HSO_4 + NaCl + CO_2 + H_2O$

To a 2000 mL Erlenmeyer flask was added sodium hypochlorite (885 mL, 10%) that had been chilled in a freezer overnight, then placed in an ice bath. Sodium hydroxide (119 g) was then added slowly, maintaining the temperature below 25°C. Urea (85.25 g) was then dissolved in boiling distilled water (50 mL), and gelatine (1.2 g) dissolved in hot water (40 mL) was added. The urea-gelatine solution was then added slowly with very strong stirring to the sodium hypochlorite solution. After the foam had died down, the reaction mixture was heated gently to complete evolution of gas. Hydrochloric acid (305 mL, 31%) was then added slowly, followed by sulphuric acid (200 mL, 50%). The reaction mixture was then cooled in an ice bath, and the precipitate filtered off. The final yield was 94.68 g.

Synthesis of benzene

$PhCOONa + NaOH \rightarrow C_6H_6 + Na_2CO_3$

To a paint can fitted with a ground glass joint was added powdered sodium benzoate (200 g) and sodium hydroxide (120 g), and then set up for distillation. The reaction mixture was heated strongly until no further distillate collected. The distillate was washed twice with an equal volume of water, then dried over calcium chloride. The product was distilled, yielding 96 mL of clear colourless liquid.

Synthesis of isopropyl nitrite

$^{i}PrOH + NaNO_2 + HCl \rightarrow {}^{i}PrONO + NaCl + H_2O$

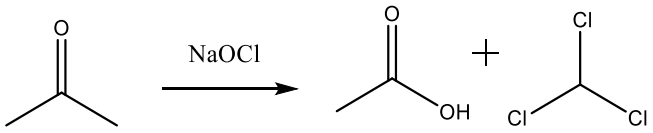
To a 500 mL Erlenmeyer flask was added sodium nitrite (72.3 g), distilled water (125 mL) and chilled in an ice-and-salt bath. A mixture of hydrochloric acid (135 mL, 31.5%) and anhydrous isopropanol (90 mL) chilled to 0°C was then added slowly. The upper organic layer was separated, and washed with an equal volume of saturated sodium bicarbonate solution, then dried over anhydrous magnesium sulphate. The final product was stored over anhydrous magnesium sulphate in an amber glass bottle.

Synthesis of potassium azide

$N_2H_4 + KOH + {}^{i}PrONO \rightarrow KN_3 + H_2O + {}^{i}PrOH$

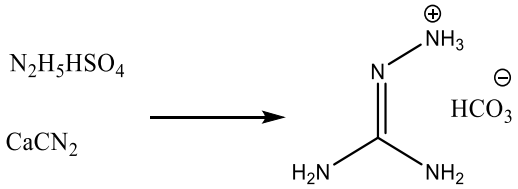
To a 100 mL beaker was added hydrazine sulphate (11.8 g), sodium hydroxide (3.6 g), and water (5 mL), followed by absolute ethanol (20 mL) upon initiation of the reaction, and stirred thoroughly. A second portion of sodium hydroxide (3.7 g) was then added and stirred well. The supernatant was then decanted, and the solids rinsed with ethanol (7-8 mL). A 100 mL two-neck round bottomed flask was then set up for reflux, and the ethanolic hydrazine solution added, along with potassium hydroxide (5.1 g). Isopropyl nitrite (13 mL) was then added slowly over the course of 25 minutes. The reaction mixture was then refluxed for 20 minutes and allowed to cool in the freezer overnight. The precipitate was filtered off and dried on the pump. The final yield was 3.93 g.

Synthesis of chloroform



To a 2000 mL Erlenmeyer flask was added sodium hypochlorite (2500 mL, 12.5%) and chilled to 0°C in an ice bath. Acetone (40 mL) was then added with strong stirring, and then allowed to cool back to below 5°C. A second portion of acetone (40 mL) was then added with stirring and allowed to cool. The lower organic layer was separated and dried over molecular sieves, then distilled. The final yield was ~50 mL.

Synthesis of aminoguanidine bicarbonate



To a 500 mL beaker were added hydrazine sulphate (55 g), calcium cyanamide (40 g), and water (200 mL) over a period of 30 minutes, maintaining the pH at 9 with sulphuric acid (50%) and the temperature at 40-45°C, and stirring was continued for a further 15 minutes after the last addition, maintaining the pH at 9. The reaction mixture was then neutralized and heated to 80°C for 1 hour, adding water as necessary to maintain volume. The reaction mixture was then filtered, and the filtrate acidified to pH 6.5. Sodium bicarbonate (36.9 g) was then added slowly with stirring, and the reaction mixture chilled. The precipitate was filtered off, and washed with water, then recrystallised from water.

Preparation of white phosphorus

$$12\ NaPO_3 + 6\ SiO_2 + 20\ Al \rightarrow 3\ P_4 + 6\ Na_2SiO_3 + 10\ Al_2O_3$$

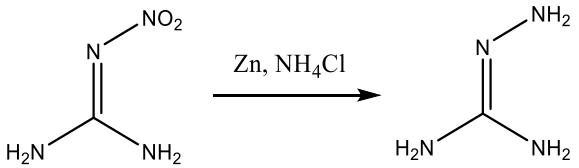
Sodium metaphosphate (34 g), silicon dioxide (20 g), and sodium chloride (4 g) were intimately ground to a fine powder, then dried at 200°C for 1.5 hours. Fine aluminium powder (15 g, German Dark) was combined with the other reagents, and loaded into a paint can retort and flushed with argon. The reaction mixture was then heated in a furnace to 500-800°C, condensing the exhaust in water. The product was transferred to a fresh beaker of water, and warmed to just molten. Acidified potassium dichromate solution was then added with stirring and allowed to stir until the product was judged clean. The supernatant was decanted, and the product washed with water and allowed to coalesce. The yield was variable; the first run yielded 1.24g, the second 2.42 g.

Synthesis of calcium cyanamide

$$CO(NH_2)_2 + CaO \rightarrow CaCN_2 + 2H_2O$$

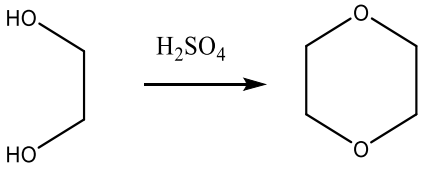
Urea (180 g) was dried in an oven at 150°C for 1 hour, then ground to a fine powder and mixed thoroughly with calcium oxide (58 g). The mixture was then heated until it had resolidified, then allowed to cool. The result was then pulverised and heated to 800°C for 1 hour with exclusion of air. The final yield was 66.21 g (85%).

Synthesis of aminoguanidine bicarbonate



Nitroguanidine (30 g) and zinc dust (102 g) were made into a paste with distilled water (20 mL) and added slowly to acetic acid (36 mL, 50%) chilled to 0°C, maintaining the temperature at 5-15°C. The mixture was then allowed to sit for a further hour at 0°C, then very slowly warmed to 40°C and held at 40°C for 15 minutes. The reaction mixture was then filtered, and ammonium chloride (27.3 g) added and stirred to dissolve. Sodium bicarbonate (30.3 g) was then added, and the solution chilled overnight. The precipitate was then filtered off, yielding 21 g.

Synthesis of dioxane



To a 1000 mL round bottomed flask was added sulphuric acid (50 mL, 93%), ethylene glycol (450 mL), and set up for distillation. To the distillate was then added sulphuric acid (25 mL, 93%) and the solution fractionally distilled, collecting the fraction at 88-90°C. Potassium hydroxide (40 g) was then added to the distillate, and the mixture stirred thoroughly, then allowed to sit overnight. The mixture was filtered, and lower aqueous layer discarded. The organic layer was then dried over sodium overnight, and the solution was then filtered. The filtrate was distilled and stored over sodium.

Preparation of caesium

$2\ CsCl + Ca \rightarrow CaCl_2 + 2\ Cs$

To a steel retort was added dry caesium chloride (25 g), calcium granules (3.00 g) and stirred well. A portion of stainless steel wool was placed in the top of the retort, and the retort evacuated and flushed with argon three times before being returned to vacuum. The reaction mixture was then distilled until no more distillate was collected. The final yield was 2.3 g. The product was stored double-tubed under argon.

Synthesis of Prussian blue

$3\ K_4Fe(CN)_6 + 4\ FeCl_3 \rightarrow Fe_7(CN)_{18} + 12\ KCl$

Potassium ferrocyanide (1.50 g) dissolved in water (10 mL) was added to ferric chloride (5.0 g) dissolved in water (40 mL), and the precipitate filtered off.

Preparation of mercury

$6\ NaOH + 7\ S = 2\ Na_2S_3 + Na_2SO_3 + 3\ H_2O$

$Na_2S_3 + HgS = Na_2HgS_4$

$8\ NaOH + 2\ Al + 3\ Na_2HgS_4 = 2\ NaAl(OH)_4 + 3\ Na_2S_3 + 3\ Na_2S + 3\ Hg$

To a 400 mL beaker was added sodium hydroxide (72 g), sulphur powder (9.6 g), and water (200 mL) and the reaction mixture stirred and heated until all the solids had dissolved. Cinnabar (48.8 g) was then added to the reaction mixture and stirred well for 30 minutes. Aluminium foil was then added in small portions[1] until no more dissolved. The aqueous layer is decanted, and the residue washed well with water. The mercury metal was removed with a pipette, and filtered through a pinhole. The final yield was 37.62 g.

(2) Hydrogen sulphide is evolved at this point.

Preparation of potassium

$2\ KOH + 2\ Mg \rightarrow 2\ K + H_2 + 2\ MgO$

To a 100 mL Erlenmeyer flask was added lamp oil (60 mL), potassium hydroxide (7 g), and magnesium powder (1.5 g), and a reflux condenser fitted with aluminium foil over the opening to restrict the influx of air. When hydrogen evolution slowed, magnesium powder (2.0 g) was added and any lumps of potassium hydroxide crushed. The reflux was continued for 4 hours 30 minutes, and then allowed to cool before recovering the potassium metal.

Applied Science

Synthesis of yttrium barium cuprate

$2\ Ba(NO_3)_2 + 6\ Cu(NO_3)_2 + 8\ Y(NO_3)_3 + 2\ C_6H_8O_7 = 2\ BaO + 6\ CuO + 4\ Y_2O_3 + 12\ CO_2 + 40\ NO_2 + 8\ H_2O + O_2$

$BaO + Y_2O_3 + 3\ CuO \rightarrow BaY_2Cu_3O_{7-x}$

Barium nitrate (26.13 g), copper nitrate trihydrate (72.48 g), yttrium oxide (45.16 g), nitric acid (18.0 g, 70%), citric acid (), were dissolved in water (mL) and evaporated to dryness. When the solution becomes a sludge, the mixture ignites and burns to an ashy material. The brown ash was then heated to 960°C in a furnace for 2 hours, then allowed to cool at 1°C per minute until cold, with a constant stream of oxygen at a flow rate of 10 SECM. The result is a single block of superconducting ceramic.

Artisan man

Synthesis of sodium ethyl sulphate

$EtOH + H_2SO_4 \rightarrow EtHSO_4$

$2\ EtHSO_4 + Na_2CO_3 \rightarrow EtNaSO_4 + H_2O + CO_2$

To a 100 mL round bottomed flask was added anhydrous ethanol (14 g), chilled in an ice bath and sulphuric acid (30 g, 98%) added slowly over a period of 35 minutes. The reaction mixture was then heated to 50°C for 30 minutes. The reaction mixture was then poured into distilled water (200 mL), and calcium carbonate (26 g) added ensuring a neutral pH, then warmed to 60°C and the precipitate filtered off. Saturated sodium carbonate solution (35 g) was then added to the filtrate, and the precipitate filtered off. The solution was evaporated to dryness, and ethanol (40 mL) added to dissolve the solids. The precipitate was filtered off, and the filtrate cooled in the freezer to obtain a second batch. The final yield was 23.4 g (52%).

Synthesis of nitric acid

$NaNO_3 + H_3PO_4 \rightarrow HNO_3 + NaH_2PO_4$

To a 1000 mL flask set for distillation was added phosphoric acid (250 g, 85%) and sodium nitrate (160 g) and mixed well. The reaction mixture was distilled at 120°C until large volumes of nitrogen dioxide were generated. The final yield was 191 g, 143 mL (88%) of 57% nitric acid with a density of 1.35 g cm^{-3}.

Astral Chemistry

Synthesis of hydrogen peroxide

$Ba(NO_3)_2 \rightarrow BaO + 2\ NO_2 + O_2$

$2\ BaO + O_2 \rightarrow 2\ BaO_2$

$BaO_2 + H_2SO_4 \rightarrow BaSO_4 + H_2O_2$

To a porcelain crucible was added barium nitrate (20 g) and heated strongly. Air scrubbed of carbon dioxide and water was then passed over the heated barium oxide product to form barium peroxide. The barium peroxide was then added to ice-cold concentrated sulphuric acid, and the precipitate filtered off.

Synthesis of aniline black

To a 250 mL beaker was added sulphuric acid (100 mL, 1%), potassium dichromate (1 g), followed by a solution of aniline (0.5 mL) in hydrochloric acid (1 mL, 31%). The reaction mixture was then heated strongly for 5 minutes, then allowed to cool. The precipitate was filtered off and rinsed with water.

Preparation of a platinum catalyst for the Ostwald process

Platinum (20 mg) was dissolved in aqua regia (10 mL) with the aid of a little heat. The excess nitric acid was removed by boiling the liquid to a syrup, then adding hydrochloric acid (30%) and repeating until no more oxides of nitrogen are evolved. The final syrup was then dissolved in the minimum of water, and sodium chloride (1 g) was added and the solution evaporated to dryness. The solid was resuspended in ethanol (95%), filtered, and evaporated to dryness. The product was then recrystallised from water.
Sodium hexachloroplatinate (10 mg) was then dissolved in water (20 mL), and a very clean portion of quartz wool (0.5 g) was soaked in the solution. Ascorbic acid solution (10 mL, 5%) was then added, and allowed to stand overnight. The quartz wool was then removed, and washed with water, then allowed to dry. The wool was then heated to red-heat for a few seconds to fuse the platinum to the wool.
An alternative way of preparing platinised wool is to dissolve the hexachloroplatinic acid in ethanol, then soak the glass wool in the solution. The wet wool is then ignited and heated briefly to red-heat. The resulting catalyst is too active for the Ostwald process however.

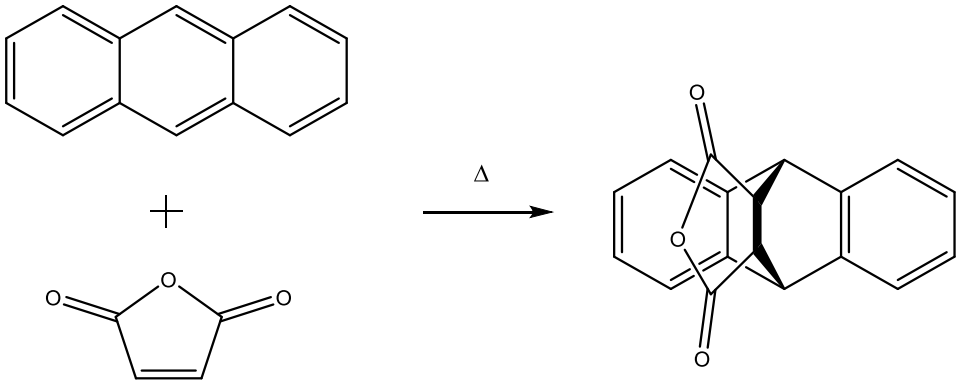
Synthesis of sodium azide (Wislicenus process)

$N_2O + NaNH_2 \rightarrow NaN_3 + H_2O$

Nitrous oxide is produced by thermally decomposing ammonium nitrate, then dried over calcium chloride and sulphuric acid. The dried gas is then passed over sodamide heated in a porcelain boat. The resulting solids were digested with water, and the water evaporated.

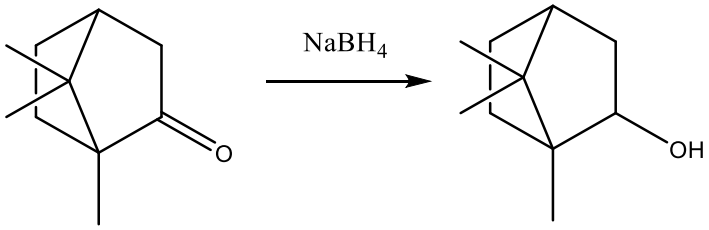
CHEM2050

Synthesis of 9,10-dihydroanthracene-9,10-α,β-succinicanhydride or (15R,19S)-17-Oxapentacyclo[6.6.5.0^{2,7}.0^{9,14}.0^{15,19}]nonadeca-2,4,6,9,11,13-hexaene-16,18-dione



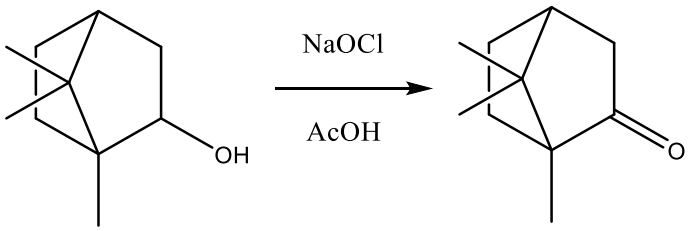
To a 100 mL round bottomed flask were added anthracene (1 g, mmol), maleic anhydride (0.5 g, mmol), and xylene (15 mL). The reaction mixture was then refluxed for 30 minutes, after which the flask was placed in an ice bath for 10 minutes. The reaction mixture was then filtered, and the product dried to yield g (%) of crystals.

Synthesis of isoborneol



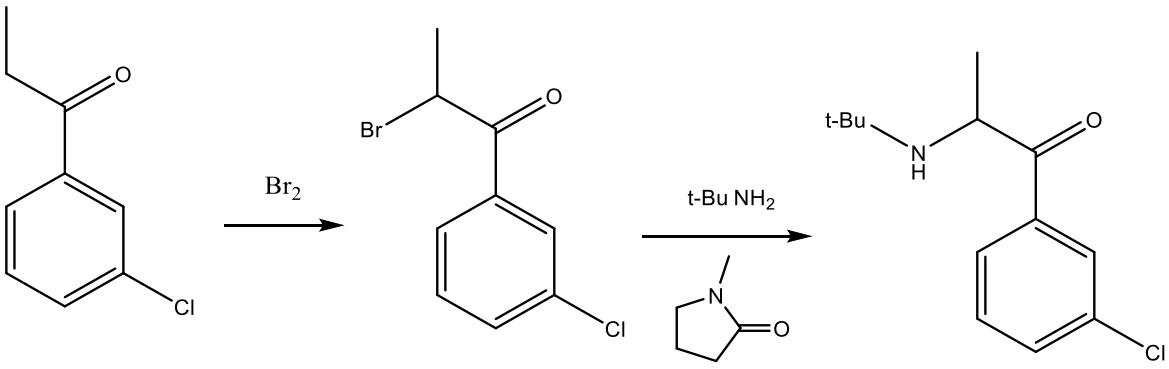
To a 25 mL round bottomed flask was added camphor (1 g, mmol), and methanol (5 mL). Sodium borohydride (480 mg, mmol) was then slowly added, and the mixture refluxed for 15 minutes. After cooling to room temperature, the ice water (12 mL) was added, and the solids filtered off. The solids were dissolved in diethyl ether (7 mL), and dried with anhydrous magnesium sulphate. The solvent was removed, and the product recovered as g (%) melting at °C (lit. °C).

Synthesis of camphor



To a 125 mL Erlenmeyer flask were added isoborneol (5 g, mmol), and glacial acetic acid (15 mL). Sodium hypochlorite solution (50 mL, 5.25%) was then slowly added over a period of 5 minutes. The reaction mixture was then allowed to stand at room temperature for 30 minutes. A positive starch-iodide test was obtained after this time. Saturated sodium bisulphite solution (~100 mL) was then added until the yellow colour of the solution disappeared and the reaction mixture tested negative to starch-iodide. The reaction mixture was then poured into a mixture of brine (100 mL) and ice (100 g), and the precipitate filtered off, then washed with saturated sodium bicarbonate solution and water, then sucked dry under vacuum. The 2,4-DNP derivative was then prepared, and the melting point determined. (lit. 164°C).

Synthesis of (±)-2-(tert-Butylamino)-3'-chloropropiophenone hydrochloride (bupropion)



Synthesis of 2-bromo-3'-chloropropiophenone

To a 100 mL round bottom flask was added m-chloropropriophenone (1 g, mmol), and dichloromethane (5 mL). Bromine solution (6 mL, 1 mol dm^{-3} in dichloromethane) was then added dropwise to the reaction solution with stirring. Gentle warmth was required to initiate the reaction. The reaction flask was then placed in an ice bath, and the bromine solution added dropwise. The solvent was then removed.

Synthesis of bupropion

A mixture of t-butylamine (5 mL, g, mmol) and N-methylpyrrolidinone (5 mL) was then added at room temperature, and the reaction mixture heated to 55-60°C in a water bath for 10 minutes. After cooling to room temperature, water (25 mL) was then added, and the organic layer separated. The aqueous layer was then extracted with diethyl ether (3x 25 mL). The combined organic layers were washed with water (5x 25 mL), and dried with sodium sulphate. The dry ether solution was then chilled in an ice bath, and a mixture of hydrochloric acid (2 mL, 31%) and isopropanol (10 mL) added dropwise to achieve a pH of 3. The mixture was allowed to sit for 10 minutes, then the precipitate was then filtered off, and dried yielding g (80%) of white crystals melting at °C (lit. °C).
DOI: 10.1021/ed077p1479

ChemExplorer Kim

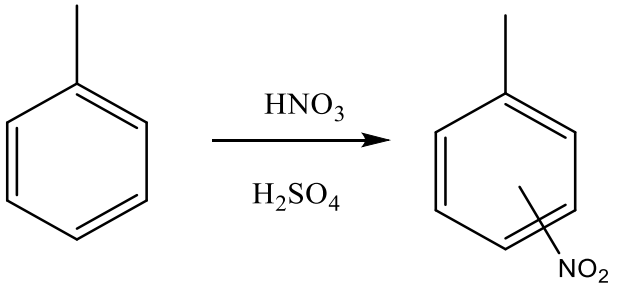
Synthesis of copper(I) oxide

$RCHO + 2\ Cu^{2+} + 5\ OH^- \rightarrow RCOO^- + Cu_2O + 3\ H_2O$

Four solutions were prepared: copper sulphate pentahydrate (50 g) in water (100 mL), citric acid monohydrate (30 g) in water (20 mL), anhydrous sodium carbonate (50 g) in water (100 mL), sucrose (100 g, excess) and citric acid monohydrate (3 g) in water (150 mL). The four solutions were then combined then heated until no further colour change was seen. The solution went from dark blue to green to red. This took about 20 minutes. The precipitate was filtered off and washed well with water, then dried in a vacuum desiccator. Unfortunately, the product had oxidised to copper(II) oxide. The yield was 14.5 g of oxidised product. Based on complete oxidation of the product, this was a 91% yield.

Chem Player

Synthesis of nitrotoluene



To a 250 mL beaker was added nitric acid (75 mL, 105.38 g, 68%, 1.14 mol), and sulphuric acid (90 mL, 166 g, 98%, 18.3 mol dm^{-3}, 1.69 mol). This was then cooled to below 10°C. To a 500 mL three-neck round bottomed flask is added toluene (78 mL, 68 g, 0.74 mol); and the chilled mixed acid added to the addition funnel. The flask was chilled in an ice bath, and the acid mixture added dropwise to the toluene, maintaining the temperature below 15°C over a period of 2 hours. After complete addition, the reaction mixture was stirred for a further hour at room temperature. The upper organic layer was separated off, and washed with water (50 mL), sodium bicarbonate (50 mL), water (2x 100 mL), and dried over calcium chloride. The organic layer was then cooled to -10°C, and the precipitate filtered off. The filtrate was refrozen and filtered 3 further times, until no more solid precipitated.

Synthesis of methyl iodide

$3MeOH + PI_3 \rightarrow 3MeI + P(OH)_3$

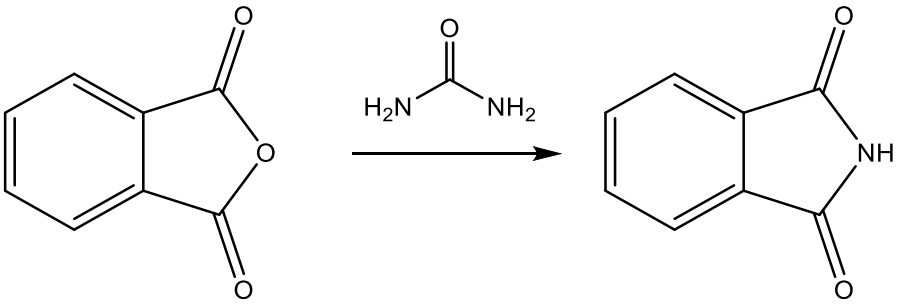
To a 250 mL round bottomed flask was added methanol (60 mL, 1.48 mol), and iodine (75 g, 0.591 mol) then chilled in an ice bath. Red phosphorus (15 g, 0.484 mol) was then slowly added to the reaction mixture, maintaining the temperature below 40°C. After complete addition, the reaction mixture was distilled until no more distillate was collected. The distillate was washed with brine (20 mL), sodium thiosulphate solution (10 mL, 0.1 mol dm^{-3}), water (20 mL) and dried over calcium chloride. The final yield was 86.35 g (81.48%) of clear colourless liquid.

Synthesis of sodium-potassium cyanide

$K_4[Fe(CN)_6] + 2Na \rightarrow 2NaCN + 4KCN + Fe$

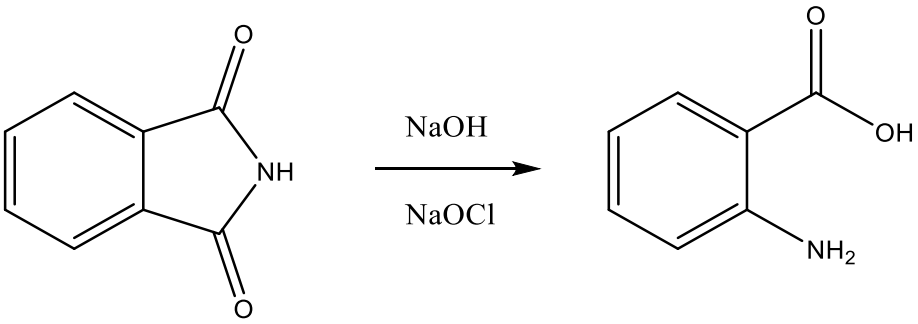
To a crucible was added potassium ferrocyanide (40 g, 109 mmol) and sodium metal (5 g, 217 mmol) in layers, then heated on a gas burner until pasty. The mixture was then stirred until uniform. The reaction mixture was then heated for 15 minutes, then allowed to cool to room temperature. The solid reaction mixture was then dissolved in the minimum of water (60 mL), and the precipitate filtered off. The product was then precipitated by action of ethanol (200 mL), and filtered off. The final yield was 15.9 g (40.7 %) of fine white powder – NaCN.2KCN

Synthesis of phthalimide



To a 500 mL round bottomed flask was added an intimate mixture of phthalic anhydride (35 g, 236 mmol) and urea (11.5 g, 192 mmol). The reaction mixture was heated to melt for 10-15 minutes. The reaction completed when a sudden foaming voluminous mass appeared. The reaction mixture was then allowed to cool, and hot water (150 mL) added and stirred until homogeneous. The precipitate was filtered off, and washed with water (2x 100 mL) and air-dried. The final yield was 31.1 g (90%) of white crystals.

Synthesis of anthranilic acid



To a 600 mL beaker was added sodium hydroxide (18 g, 449 mmol) and water (100 mL). This was then chilled to 10°C, and phthalimide (30 g, 204 mmol) added, then stirred to dissolve. This was rechilled to 10°C, and chilled sodium hypochlorite solution (190 mL, 8.25 %, 211 mmol). The reaction mixture was then stirred for 15 minutes, and heated to 75°C, then allowed to cool to room temperature. Hydrochloric acid (25 mL, 36%, 11.6 mol dm^{-3}) was then added to completely neutralise the solution. Glacial acetic acid (30 mL) was then added to the reaction mixture and stirred vigorously. The precipitate was then filtered off, and washed with water (100 mL). The crude yield was 12.4 g (44%) of light brown powder. Boiling water (>95°C) can be used as a recrystallization solvent.

Synthesis of isopropyl nitrite

$^{i}PrOH + HNO_2 \rightarrow {}^{i}PrONO$

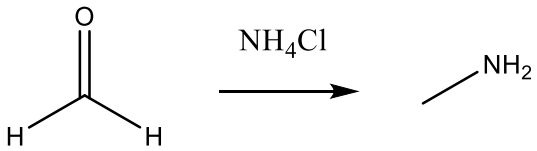
To a 250 mL beaker was added sodium nitrite (72 g, 1.04 mol) and water (120 mL), then stirred to dissolve and chilled to 5°C. To a 600 mL beaker was added isopropanol (90 mL, 1.18 mol) and hydrochloric acid (130 mL, 36%, 11.6 mol dm^{-3}), then chilled to 5°C. The isopropanol-hydrochloric acid solution was added dropwise to the ice-bath chilled sodium nitrite solution with stirring. The solids were filtered off, and the upper organic layer separated and washed with saturated sodium bicarbonate solution (2x 40 mL), then dried with magnesium sulphate. The final yield was 65 g (70%) of yellow liquid.

Synthesis of pyridinium chlorochromate

$py + CrO_3 + HCl \rightarrow pyCrO_3Cl$

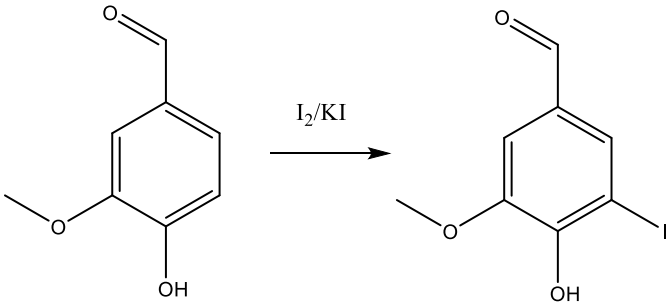
To a 200 mL beaker was added potassium dichromate (20 g, 66 mmol), and dissolved in water (80 mL). To the solution was slowly added sulphuric acid (40 mL, 98%) with stirring. Potassium dichromate (20 g, 66 mmol) was then dissolved, along with sulphuric acid (40 mL, 98%). The reaction mixture was then allowed to cool to room temperature, and the precipitate filtered off. The crude yield was 70 g. This was then added to water (50 mL) and hydrochloric acid (50 mL, 36%, 11.6 mol dm^{-3}). Pyridine (20 mL, 19.6 g, 245 mmol) was then added with stirring, and allowed to cool to room temperature. The precipitate was filtered off, and air dried. The final yield was 35 g (70%) of bright orange crystals.

Synthesis of methylamine hydrochloride



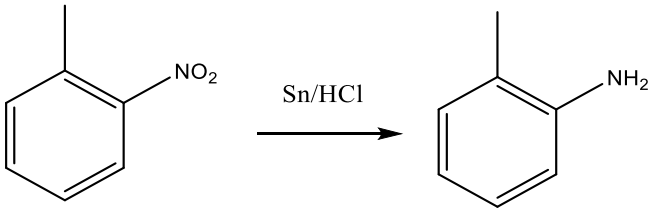
To a 1000 mL three-neck round bottomed flask was added ammonium chloride (230 g, 4.3 mol), formaldehyde solution (410 mL, 460 g, 37%, 5.67 mol). The reaction mixture was then distilled at 104°C for 3 hours at atmospheric pressure, then 2 hours under vacuum. The reaction mixture was then allowed to cool to room temperature, then chilled in the refrigerator, and the precipitate filtered off. The filtrate was then redistilled under a vacuum until white smoke began to collect in the flask ~160°C. The hot reaction mixture was then allowed to cool to room temperature, taking care not to allow it to absorb moisture from the air. Ethanol (200 mL) was then used to wash the crystals, and the precipitate filtered off, then washed with ethanol (3x 50 mL). The filtrate was then chilled to below 10°C, and the precipitate filtered off. This was then washed with chloroform (50 & 25 mL), and dried under vacuum. The filtrate was distilled until only a syrupy liquid remained, and treated in the same way as before. The final yield was 80 g of pure damp white crystals, and 15 g of a second batch of less pure damp white crystals. The total yield was 90%.

Synthesis of 5-iodovanillin



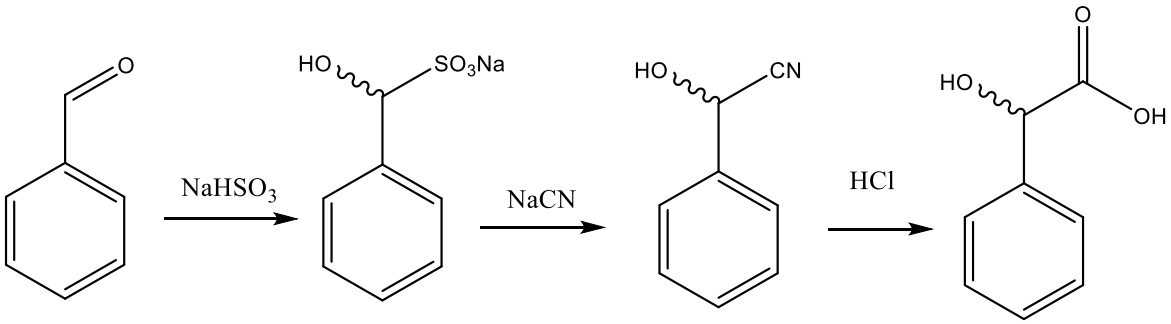
To a 250 mL beaker were added potassium iodide (10 g, 60 mmol) and sodium bicarbonate (5 g) and dissolved in water (200 mL). Vanillin (7.5 g, 49 mmol) and iodine (12.6 g, 99 mmol) were then added to the solution, and stirred for 2 hours. The precipitate was then filtered off, and washed with very dilute sodium thiosulphate solution (50 mL) and water (50 mL). The solids were then dried in a desiccator, and recrystallized from a 20:80 mixture of ethanol:isopropanol (~200 mL). The product was dried in a vacuum desiccator. The final yield was 12.6 g (92%) of off-white crystals.

Synthesis of toluidine



To a 500 mL round bottomed flask set up for reflux was added o-nitrotoluene (26 mL, 30.2 g, 220 mmol), tin metal (49 g, 413 mmol), and hydrochloric acid (110 mL, 36%, 11.6 mol dm^{-3}) in 10 mL portions, allowing the reaction to die down between additions. After complete addition, the reaction mixture was refluxed for 30 minutes. After allowing to cool to room temperature, the reaction mixture was carefully poured into ice-cold sodium hydroxide solution (90 g in 150 mL of water). The reaction mixture was then steam-distilled until 20 minutes after the distillate ran clear. To the distillate was added sodium chloride (35 g), and the upper organic layer separated. The aqueous layer was washed with dichloromethane (2x 20 mL), and the combined organic layers dried with magnesium sulphate. The solvent was removed, yielding 15.94 g (68%) of brown liquid.

Synthesis of (±)mandelic acid



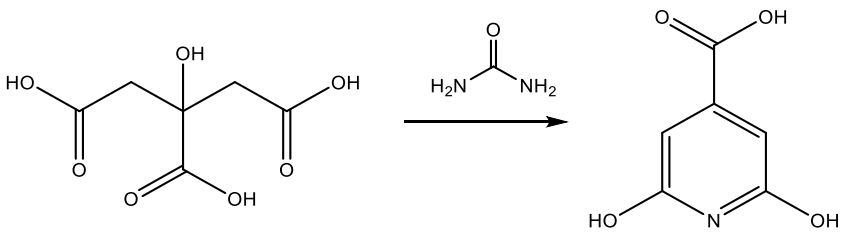
Synthesis of benzaldehyde-sodium bisulphite adduct

To a 250 mL beaker was added sodium bisulphite (30 g, 285 mmol), and water (60 mL), then stirred until all the solids had dissolved. With vigorous stirring, benzaldehyde (15 g, 141 mmol) was added slowly, and stirred for 15 minutes. The precipitate was filtered off, and dried.

Synthesis of mandelic acid

Sodium-potassium cyanide (15 g, 0.251 mol CN^-) was then dissolved in water (40 mL), and the adduct added, and stirred for 2 hours. The upper organic layer was separated off, and the aqueous layer washed with toluene (50 mL). The combined organic layers were washed with saturated sodium bisulphite solution (10 mL), and brine (50 mL). The organic layer was then transferred to a 250 mL round bottomed flask and hydrochloric acid (40 mL, 36 %, 11.6 mol dm^{-3}) added. The toluene was distilled off, and hydrochloric acid (20 mL, 36%, 11.6 mol dm^{-3}) added, then refluxed for a further hour. The solution was then concentrated to 15 mL. Sodium chloride (5 g) was then added to aid precipitation of the product. The chilled reaction mixture was then filtered, and the solids washed with cold chloroform (15 mL). The solid was dissolved in diethyl ether (50 mL), and the precipitate filtered off, and washed with diethyl ether (40 mL). The solvent was removed from the filtrate, and the solids recrystallized from chloroform (15 mL), washed with ice-cold chloroform (2x 15 mL). The final yield was 8.94 g (42%) of off-white powdery crystals.

Synthesis of citrazininc acid



To a 600 mL beaker were added an intimately mixture of citric acid monohydrate (75 g, 360 mmol) and urea (60 g, 1 mol), and fused for 1 hour. The reaction mixture was allowed to cool, and boiling water (150 mL) added, and stirred to dissolve. The pH was adjusted to 9, and the solution filtered. Hydrochloric acid (20 mL, 36%, 11.6 mol dm^{-3}) was then added, and the precipitate filtered off, and washed with a minimum of ice-cold water. The solid was then dried in a desiccator, then washed with methanol (30 mL, 10 mL) and dichloromethane (20 mL). The final yield was 7.73 g (14%) of golden powder.

Synthesis of propionyl chloride

$3EtCOOH + PCl_3 \rightarrow EtCOCl + P(OH)_3$

To a 500 mL round bottomed flask in a water bath was added dry propionic acid (140 mL, 1.89 mol) and phosphorous trichloride (56 mL, 0.63 mol) was added dropwise over 20 minutes. The reaction mixture was then stirred for 1 hour at room temperature, then distilled collecting until 90°C. The final yield was 90 mL (55%) yield. Low yield due to impurities in propionic acid. Redistillation will purify.

Synthesis of propiophenone

$(BzO)_2Ca + (EtCOO)_2Ca \rightarrow PhAc + PhC(=O)Et$

An intimate mixture of calcium benzoate (2 mol) and calcium propionate (1 mol) with a total mass of 110 g was added to a 500 mL round bottomed flask. The powder was dry-distilled with a propane flame, and heated until no more distillate collected, approximately 3 hours. The orange distillate was washed with water (2x 20 mL), and dried with magnesium sulphate. The organic layer was distilled, collecting a colourless fraction (5 mL) at 75-80°C with a petrochemical, fruity, ethyl acetate-like odour. A small (3 mL) colourless second fraction was collected at 120-130°C with a petrochemical, phenolic, spicy, irritating odour. A large (20 mL) yellow fraction was collected at 190-200°C with an intense, honey, sweet, sickly odour. Fraction 3 melted between 5-10°C. Fraction 1 was ethyl acetate, fraction 2 was an aliphatic ketone, fraction 3 was a mixture of acetophenone and propiophenone, along with other impurities.

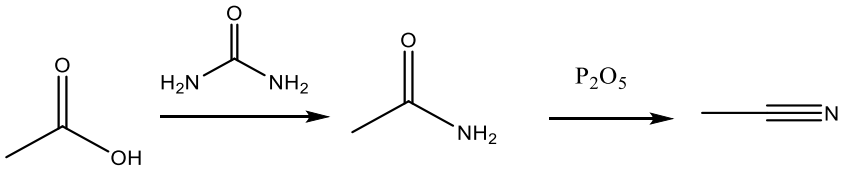
Synthesis of nitroethane

$NaNO_2 + AgNO_3 \rightarrow NaNO_3 + AgNO_2$

$EtI + AgNO_2 \rightarrow EtNO_2 + AgI$

Silver nitrate (42 g, 247 mmol) dissolved in water (100 mL) was added to sodium nitrite (19 g, 275 mmol) dissolved in water (100 mL), and stirred for 15 minutes then the precipitate was filtered off and washed with water (40 mL) and ethanol (2x 40 mL), then dried in a desiccator. The yield was 35 g. This was then added to a 500 mL round bottomed flask, and diethyl ether (60 mL) added. The flask was cooled in an ice-salt bath, and a reflux condenser and addition funnel added. Ethyl iodide (33.7 g, 216 mmol) and diethyl ether (30 mL) added to the addition funnel. The ethyl iodide solution was added dropwise over a period of 45 minutes with good stirring, maintaining the temperature at 8-12°C. After complete addition, the reaction mixture was stirred for a further hour in the ice bath, then 6 hours at room temperature. The precipitate was then filtered off, and washed with diethyl ether (2x 50 mL). The solvent was then removed, and the residue distilled, collecting the fraction at 115-130°C (oil bath temperature). The final yield was 9.2 g (57%) of clear colourless liquid.

Synthesis of acetonitrile



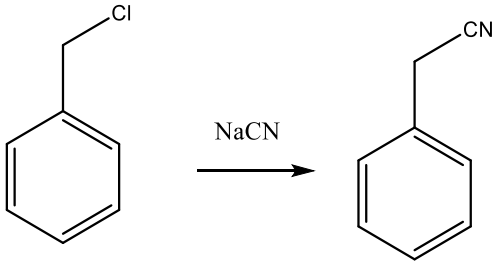
Synthesis of acetamide

To a 500 mL round bottomed flask was added glacial acetic acid (57 mL, 1.00 mol) and urea (61 g, 1.02 mol), then refluxed for 4 hours (3 hours after ammonia first produced), then distilled until no more distillate collected. The residue was poured out of the flask, and allowed to cool. The crude yield was 50 g (85%).

Synthesis of acetonitrile

The crude acetamide was dissolved in hot methanol, and filtered, then the filtrate concentrated to 50 mL, then diethyl ether (50 mL) added to precipitate the acetamide. The precipitate was filtered off, and dried in a desiccator. To a 250 mL flask was added phosphororus pentoxide (35 g, 120 mmol) and dropwise molten crude acetamide (20 g, 339 mmol). The reaction mixture was then distilled until no more distillate was collected, and the distillate washed with saturated sodium carbonate (8 mL), then dried over magnesium sulphate. The final yield was 9.1 g (65% on acetamide).

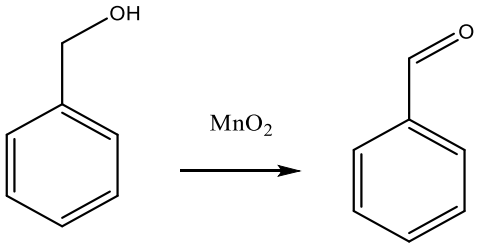
Synthesis of phenylacetonitrile



To a 500 mL round bottomed flask fitted with an addition funnel was added sodium-potassium cyanide solution (30 g in 140 mL water, ~500 mmol CN^-). Benzyl chloride (40 g, 316 mmol) and ethanol (40 mL) were added to the addition funnel, then the flask was heated to 50°C and the benzyl chloride solution added dropwise to the reaction mixture, then refluxed for 3 hours. The reaction mixture was then distilled to remove the ethanol. The organic layer was then separated from the reaction mixture, and washed with sulphuric acid (2x 30 mL, 50%), saturated sodium bicarbonate solution (40 mL), and brine (40 mL). The crude yield was 32 g (86%) of black liquid. Vacuum distillation can be used for purification – vacuum essential to avoid decomposition.

Synthesis of benzaldehyde

Method 1 – Manganese dioxide oxidation



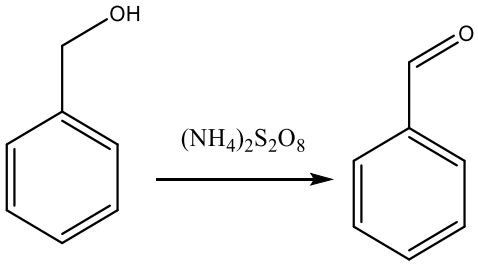
Synthesis of manganese dioxide

To a 600 mL beaker was added potassium permanganate (50 g, 316 mmol), and warm water (100 mL), then stirred until total dissolution. Manganese sulphate monohydrate (70 g, 432 mmol) dissolved in the minimum of water, and added to the potassium permanganate solution, then stirred for 1 hour. The precipitate was then filtered off, and washed with water (400 mL). The solids were then air dried to yield 65 g (99%) of dark brown powder.

Synthesis of benzaldehyde

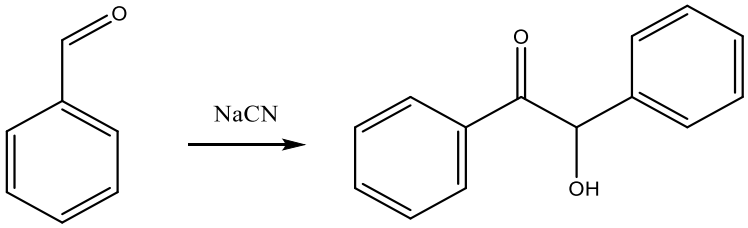
To a 500 mL flask was added benzyl alcohol (30 mL, 289 mmol), and dichloromethane (80 mL). The chemical manganese dioxide prepared earlier (65 g, 748 mmol) was slowly added to the refluxing mixture over a period of 1 hour and allowed to reflux for a further 1 hour. After allowing to cool to room temperature, the precipitate was filtered off and washed with dichloromethane (3x 50 mL). The solvent was removed, and the residue poured into saturated sodium bisulphite solution (150 mL) and stirred strongly for 15 minutes. The precipitate was then filtered off, and washed with dichloromethane (2x 20 mL). The solids were then added to a saturated sodium bicarbonate solution (150 mL), and stirred for 15 minutes. The organic layer was separated off, and dried over magnesium sulphate. The final yield was 12.6 g (43%) of pale yellow liquid with an almond odour.

Method 2 – Ammonium persulphate oxidation



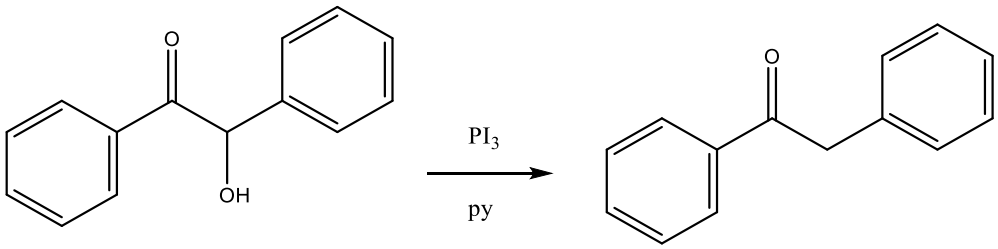
Ammonium persulphate (185 g, 810 mmol) was dissolved in water (150 mL), and added to the addition funnel of a 1000 mL three-neck round bottomed flask fitted with a thermometer and addition funnel. Benzyl alcohol (85 mL, 810 mmol), water (100 mL), and methanol (20 mL) was added to the flask, and stirred vigorously. Strong stirring is important to ensure good mixing of the aqueous and organic layers during the reaction. The reaction mixture was heated to 50°C, and 15-20 mL of ammonium persulphate solution added. The reaction mixture was then carefully heated to 60°C, and the solution added dropwise from the funnel, maintaining the temperature around 65°C; the reaction is exothermic and overoxidation is easy. Do not allow to warm to 70°C. Complete addition took 60-90 minutes, then allowed to cool to room temperature in a water bath over a period of 1 hour. The upper layer was separated, and slowly added to saturated sodium bisulphite solution (250 mL, 1.06 mol) with strong stirring, then stirred for 20 minutes after complete addition. The solids were washed with water (50 mL) and dichloromethane (2x 50 mL), then air dried. The dry solid was then added to a saturated solution of sodium bicarbonate (300 mL) with vigorous stirring, and allowed to stir for 30 minutes. The upper organic layer was separated to yield 74 g (86%) of brown liquid.

Synthesis of benzoin



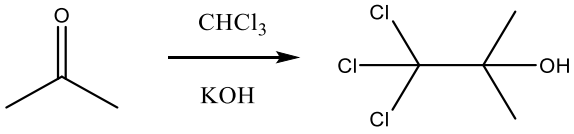
To a 100 mL round bottomed flask was added sodium-potassium cyanide (1.0 g, 16.7 mmol) dissolved in water (3 mL), benzaldehyde (10 g, 94.3 mmol), and ethanol (20 mL), then gently refluxed for 2 hours. The reaction mixture was concentrated until crystals began to form, and allowed to cool to room temperature. The solids were filtered off, and washed with cold water (10 mL), and ice-cold ethanol (10 mL). The final yield was 7.2 g (72%) of white powder.

Synthesis of desoxybenzoin (2-phenylacetophenone)



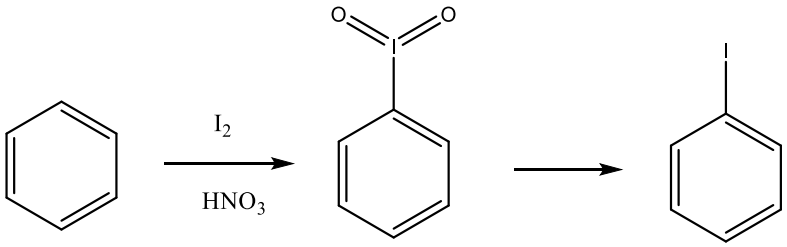
To a 100 mL round bottomed flask was added toluene (30 mL), iodine (7.5 g, 59 mmol), and slowly red phosphorus (0.6 g, 19 mmol). After complete addition of the red phosphorus, the reaction mixture was stirred for 15 minutes, and benzoin (3.5 g, 16 mmol) added in small portion, then stirred for a further 10 minutes. Then pyridine (1.3 g, 16 mmol) dissolved in dichloromethane (10 mL) was added slowly to the reaction mixture, and allowed to stir at room temperature for 3 hours. Sodium thiosulphate pentahydrate (15 g) dissolved in water (30 mL) was added to the reaction flask, and stirred well. The reaction mixture was then filtered, and the organic layer separated. The aqueous layer was extracted with dichloromethane (20 mL), and the combined organic layers dried over magnesium sulphate. The solvent was removed, and recrystallized from methanol (20 mL). The final yield was 0.4 g (12%) of white powder melting at ~50°C.

Synthesis of chlorbutol



To a 250 mL Erlenmeyer flask was added acetone (60 mL, 730 mmol), chloroform (30 mL, 370 mmol) then chilled to 0°C. Potassium hydroxide (2 g, 36 mmol) was then slowly added to the ice-cooled reaction mixture, maintaining the temperature at 10°C. The reaction mixture was then chilled in the freezer for 1 hour, and the precipitate filtered off, then washed with a small amount of acetone. The solvent was removed from the filtrate, and the hot residue poured into ice water (40 mL), and chilled for 1 hour. The precipitate was then filtered off, and washed with ice-cold water (10 mL). The final yield was 17.4 g (25%) of fluffy white crystals.

Synthesis of iodobenzene



To a 250 mL round bottomed flask was added benzene (35 mL, 393 mmol) and iodine (29 g, 229 mmol), then brought to a reflux. Nitric acid (46 g, 68%, 490 mmol) was then added slowly through the top of the condenser over a period of 30 minutes, then refluxed for a further 90 minutes. After cooling, the lower organic layer was separated, and tin metal (3 g), hydrochloric acid (10 mL, 36%, 11.6 mol dm^{-3}) and water (10 mL) added to the organic layer in a 100 mL round bottomed flask. The reaction mixture was allowed to stir at room temperature for 30 minutes, then the lower organic layer

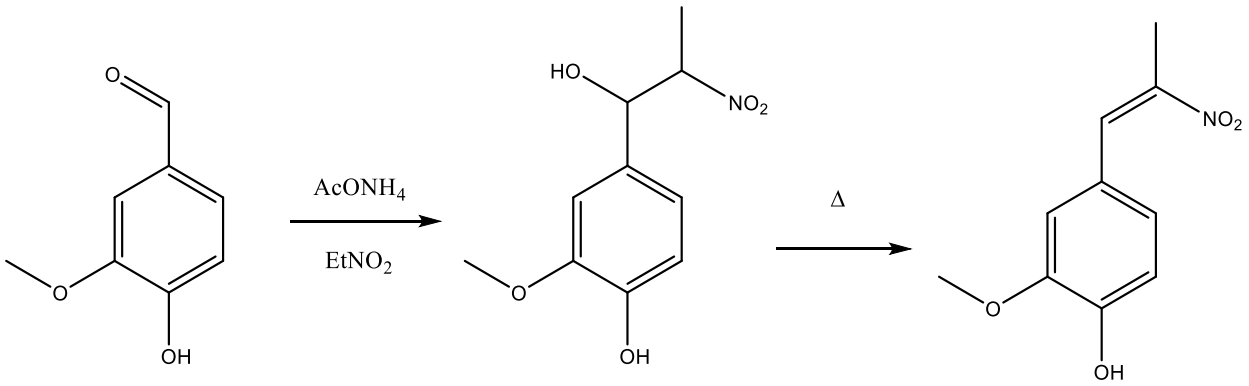
separated and dried over magnesium sulphate. The dried organic layer was distilled, and the benzene removed as a first fraction at 80°C, and the product collected from 180°C. The final yield was 37.1 g (81%) of pale yellow liquid with a density of 1.77 g cm^{-3} (lit. 1.83 g cm^{-3}).

Synthesis of cyanogen iodide

$NaCN + I_2 \rightarrow NaI + ICN$

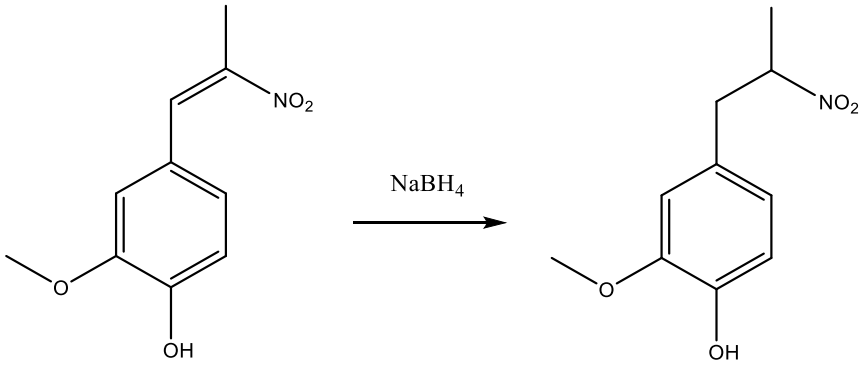
To a 250 mL flask were added sodium-potassium cyanide (10 g, 168 mmol CN^-), iodine (30 g, 236 mmol), and water (30 mL), all cooled to 10°C. The iodine was added slowly to the cooled solution. After complete addition, the reaction mixture was allowed to stir for 10 minutes in the ice-bath. Dichloromethane (40 mL) was then added, and stirred for a further 10 minutes. The upper organic layer was separated, and the aqueous layer extracted with dichloromethane (20 mL). The combined extracts were dried over magnesium sulphate, and transferred to a 250 mL flask, and the solvent removed from the ice-cold solution under vacuum. The concentrated solution was then chilled in the freezer, and the precipitate filtered off. The product was recrystallized from chloroform. The final yield was 3 g (17%) of grey crystals.

Synthesis of 1-(3-methoxy-4-hydroxyphenyl)-2-nitropropene



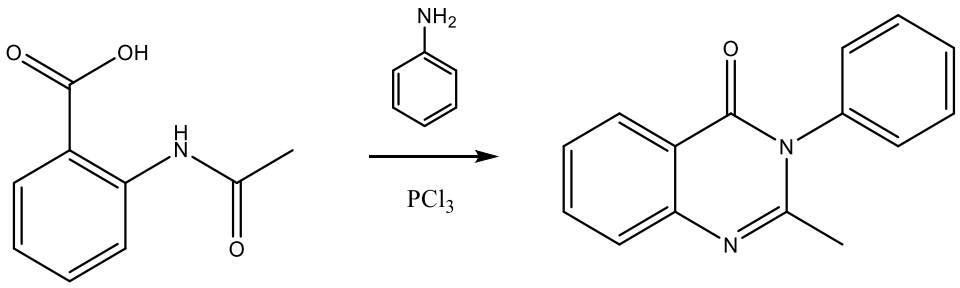
To a 100 mL flask was added vanillin (5 g, 33 mmol), anhydrous ammonium acetate (2 g, 26 mmol), nitroethane (3.5 g, 47 mmol), and glacial acetic acid (10 mL). The covered flask was placed into a 100W microwave oven for 14x 30 seconds with breaks to allow the flask to cool, all over a 30 minute time period. The hot reaction mixture was then added to ice (30 g), and the flask washed with cold water (10 mL). The precipitate was filtered off, and washed with cold water (20 mL). The crude yield was 3.2 g (47%) of bright yellow crystalline powder. The product was recrystallized from a mixture of methanol:water 60:40 (25 mL). The final yield was 2.8 g (41%) of bright yellow plates. The compound is relatively unstable, and must be stored in the freezer.

Synthesis of 1-(3-methoxy-4-hydroxyphenyl)-2-nitropropane



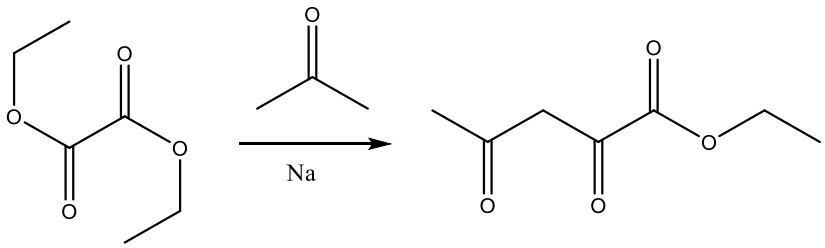
3-methoxy-4-hydroxyphenyl-2-nitropropene (2.2 g, 10.5 mmol) was dissolved in the minimum of a 1:4 ethanol:ethyl acetate mixture. To a 100 mL round bottomed flask was added 20 mL of the same 1:4 solvent mixture, and sodium borohydride (1.5 g, 40 mmol) added. Maintaining the temperature below 20°C, and the 3-methoxy-4-hydroxyphenyl-2-nitropropene solution added slowly through an attached condenser over a period of 30 minutes. After complete addition, the reaction mixture was stirred for 15 minutes. Small amounts of solvent mixture were used to wash the beaker containing the reagent solution and condenser, then cold water (30 mL) was cautiously added to the reaction mixture, and stirred for 10 minutes. After effervescence had ended, the upper organic layer was separated off, and washed with cold water (30 mL). The aqueous layer was extracted with ethyl acetate (10 mL) and the organic layers combined and washed with water (20 mL) and acetic acid (20 mL, 20%), then dried over magnesium sulphate. The solvent was then removed, and the residue collected. The final yield was 1.3 g (59%) of brown oil.

Synthesis of 2-methyl-3*N*-phenylquinazolin-4(3*H*)-one hydrochloride



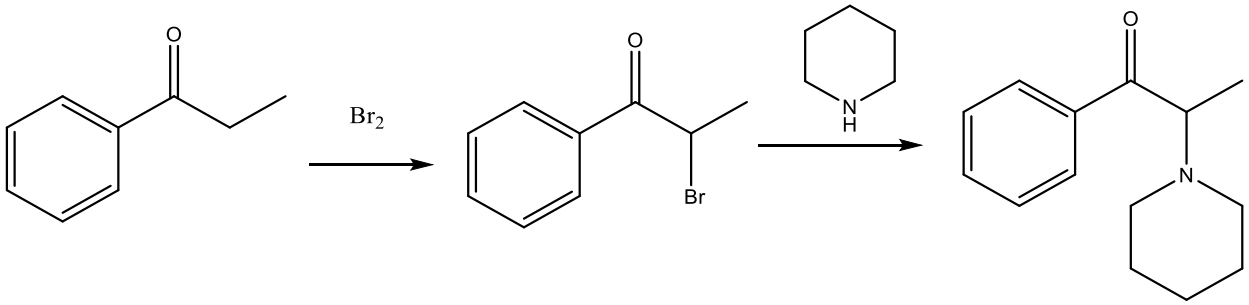
To a 100 mL flask equipped with good stirring was added dry toluene (30 mL), dry N-acetyl-o-anthranilic acid (10 g, 55.8 mmol), dry aniline (5.5 g, 59.1 mmol), and set up for reflux with a calcium chloride drying tube. Phosphorous trichloride (6 mL, 68.6 mmol) dissolved in dry toluene (20 mL) was then added slowly through the condenser to the reaction mixture, maintaining the flask at a cool temperature. Complete addition took about 20 minutes, after which the reaction mixture was refluxed for 90 minutes. The reaction mixture was concentrated to half-volume, and the precipitate filtered off, then washed with toluene (30 mL). The crude product was added a solution of sodium hydroxide (10 g) in water (80 mL), and stirred well. The precipitate was filtered off, and washed with water. The solids were dissolved in the minimum of boiling acetone (10-15 mL) – if a yellow precipitate persists, this is unreacted hydrochloride salt from the sodium hydroxide step. Hydrochloric acid (6 mL, 36%, 11.6 mol dm^{-3}) was then added, and the solvent removed. Toluene (30 mL) was then used to suspend the solids, and the precipitate filtered off. The solids were then suspended in cold acetone (20 mL), and filtered off, then washed with cold acetone (20 mL). The final yield was 7.8 g (56%) of white powder. The product can be recrystallized from hot acetone.

Synthesis of ethyl β-acetopyruvate



To a 250 mL flask was added absolute ethanol (60 mL) and set up for reflux. Sodium metal (3.5 g, 152 mmol) was then added in small portions. After complete reaction, a mixture of dry diethyl oxalate (20 g, 137 mmol) and dry acetone (11 mL, 150 mmol) was then slowly added through the top of the condenser over a period of 30 minutes. The reaction mixture was then stirred for 20 minutes until no longer possible to stir. It was then allowed to rest for 1 hour, and suspended in dry acetone (40 mL) before filtering the fine precipitate. To the solids was then added sulphuric acid (12 mL, 50%), and stirred manually until possible to stir magnetically. At this point, water was added in 10 mL portions until all the solid had dissolved (~50 mL). Dichloromethane (50 mL) was then added, and the lower organic layer separated, and the aqueous layer washed with dichloromethane (25 mL). The combined organic layers were dried over magnesium sulphate, and the solvent removed, and the residue vacuum distilled, collecting the fraction at 155-175°C / 53 mmHg. The final yield was 14 g (65%) of pale yellow liquid.

Synthesis of 1-phenyl-2-(1*N*-piperidinyl)propan-1-one



Synthesis of α-bromopropiophenone

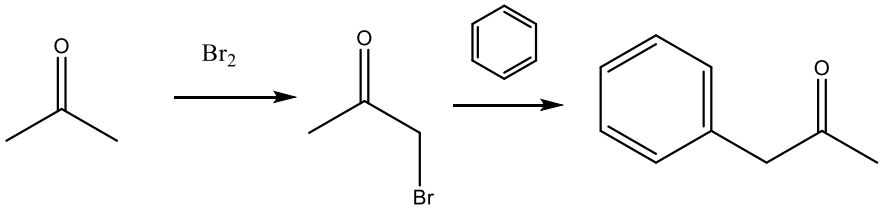
To a 100 mL round bottomed flask was added dry propiophenone (1.5 g, 11.2 mmol), dichloromethane (5 mL). Bromine solution (4g, 45% in dichloromethane, 11.2 mmol) was added dropwise to the reaction mixture, allowing the colour to fade between each addition – 20 minutes total. The reaction mixture was then allowed to stir for 20 minutes, and the solvent removed.

Synthesis of 1-phenyl-2-(1*N*-piperidinyl)propan-1-one

The flask was placed into an ice bath, and piperidine hydrochloride (1.7 g, 14.0 mmol) dissolved in water (3 mL) was added. Sodium hydroxide (1.2 g, 30 mmol) dissolved in water (3 mL) was then added dropwise to the reaction mixture, then heated slowly to 60°C, then allowed to cool to room temperature. Dichloromethane (20 mL) was then added to the reaction mixture, and the lower organic layer separated, and the aqueous layer extracted with dichloromethane (10 mL). The combined organic extracts were then extracted with hydrochloric acid (20 mL, 10%), and the lower organic layer removed. Sodium hydroxide (4 g) dissolved in water (25 mL) was then used to basify the aqueous solution, and extracted with dichloromethane (15 mL). The aqueous layer was extracted

with dichloromethane (15 mL), and the combined organic extracts dried with magnesium sulphate. The solvent was removed, and hydrochloric acid (4 drops, 36%, 11.6 mol dm^{-3}) added to the oily residue. Acetone (2 mL) was then added, and the solution chilled in the freezer to crystallise. The crystals were filtered off, using acetone as a transference medium. The yield was 0.49 g. The filtrate was concentrated, and treated with more acetone. The second batch was 0.36 g. The total final yield was 0.85 g (35%) of white crystalline powder.

Synthesis of phenyl-2-propanone



Synthesis of bromoacetone

Sodium hydroxide (10 g) dissolved in water (250 mL) was used as an emergency quench solution. Acetone (25 mL 340 mmol) was placed into a separatory funnel, and placed over the quench solution in a beaker. Bromine solution (33 mL, 45% in dichloromethane, 165 mmol) was added dropwise to the separating funnel over a period of 40 minutes. Saturated sodium carbonate solution (50 mL) was added to the separating funnel, and the lower organic layer separated off, and dried over magnesium sulphate. The quench solution was then used to 'clean' the aqueous solution in the separatory funnel.

Synthesis of phenyl-2-propanone

To a 250 mL round bottomed flask was added dry benzene (50 mL), anhydrous aluminium chloride (23 g, 172 mmol) and an addition funnel added. The bromoacetone solution was then added to the addition funnel, and added dropwise to the reaction mixture, maintaining the temperature below 50°C over a period of 40 minutes. The reaction mixture was then stirred for 30 minutes at room temperature, then poured into a mixture of hydrochloric acid (5 mL, 36%, 11.6 mol dm^{-3}) and ice (60 g). The mixture was then stirred well, and the upper organic layer separated, then washed with sodium hydroxide (4 g) dissolved in water (60 mL). The organic layer was then steam distilled for 2-3 hours. The upper organic layer was separated off, and the aqueous layer extracted with benzene (10 mL), and the combined organic layers dried over magnesium sulphate, then vacuum distilled collecting the fraction at 130°C / 50 mmHg. The final yield was 3.1 g (14%) of yellow oil with a sweet piny odour.

Synthesis of chocolate truffle mousse cake

Butter was first used to grease a spring-form non-stick tin (16 x 6 cm), which was then assembled. To a medium size pan was added water (3-4 cm), and the water heated to boiling. A bowl was place on top of the pan, and sugar (50 g) added. Eggs (2) were opened, and also added. The mixture was then stirred very thoroughly with a whisk for 1 minute. Once the reaction mixture reached 42°C, the bowl was removed from the heat, and whisked further until it fell in ribbons from the beaters. Natural vanilla extract (¾ tsp) was then added, and whisked to homogeneity. To a bowl was added cake flour (50 g), sodium bicarbonate (¼ tsp) and mixed well. The flour mixture was then sieved into the sugar-egg mixture in small portions, mixing carefully with a whisk between additions. After complete addition, the batter should still fall in ribbons. This was then poured into the cake tin prepared earlier, and heated in an oven at 180°C for 15-20 minutes. A metal skewer should emerge clean from the cooked reaction mixture. It was allowed to cool to room temperature, then placed in

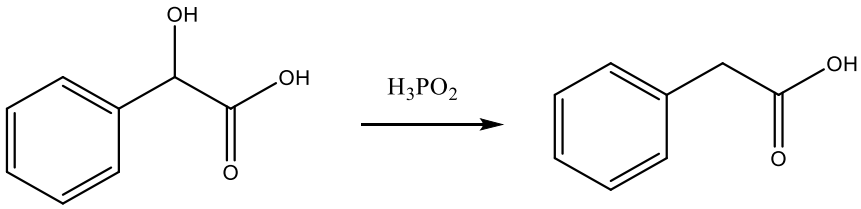
the refrigerator.
Eggs (4) were then opened, and the yolks separated. These were then strongly beaten with a whisk until light yellow. Caster or granulated sugar (60 g) and water (15 mL) were then added to a saucepan, and the sugar melted carefully. Upon reaching 125°C, the heat was turned off, and added slowly to the whisked egg yolks. The reaction mixture was whisked until it had approximately doubled in volume and had attained a smooth fluffy consistency. Fresh whipping cream (120 g, >28% fat) was then whisked until it had attained a solid consistency. This was temporarily stored in a cool place. Araguani cocoa chocolate (100 g, 72% cocoa solids, Valrhona) was then placed in a bowl on a pan of boiling water and heated until molten. At this point, the bowl was removed from the heat. Gelatine (1 g) was dissolved in a little hot water. The egg yolk foam prepared earlier was added to the molten chocolate, and gently stirred to mix, then the gelatine solution added and mixed. Ensuring the mixture is not too hot, the whipped cream was then added, and folded into the mixture. The sponge prepared in the first reaction was then cut in half, and the lower layer placed back into the spring-form tin. Some of the chocolate mousse was then poured on top of the lower layer, and levelled out. The upper layer was placed on top, and the remainder of the mousse used to cover the sponge. The tin was then placed into the refrigerator for 4 hours to allow the gelatine to set. The final product was served in slices accompanied by French meringues, strawberries and cream.

Synthesis of hypophosphorous acid

$NaH_2PO_2 + HCl \rightarrow NaCl + H_3PO_2$

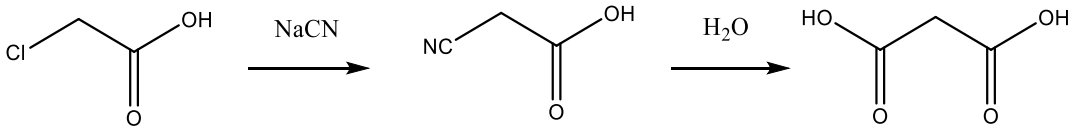
To a 250 mL beaker were added sodium hypophosphite dihydrate (100 g, 807 mmol), and hydrochloric acid (60 mL, 36%, 11.6 mol dm^{-3}, 696 mmol), then stirred and warmed to 80°C, and then chilled to 0°C. The precipitate was filtered off, yielding 40 g of sodium chloride (85%). The filtrate was transferred to a 250 mL flask, and the water vacuum distilled off. The precipitate was filtered off, yielding 4 g of sodium chloride (total: 94%). The final yield was 100 g of syrupy liquid with a density of 1.35 g cm^{-3} (lit. 1.49 g cm^{-3}), so ~70% solution.

Synthesis of phenylacetic acid



To a 250 mL flask was added mandelic acid (10 g, 65.7 mmol) and a slurry formed with the minimum of water (10 mL). Hypophosphorous acid (15 mL, 70%, 160 mmol) was then added to the flask, along with 5 mL of water to achieve total dissolution of the solids. The flask was then chilled in an ice bath, and iodine (20 g, 157 mmol) added slowly. The reaction mixture was then refluxed gently for 3 hours. After cooling to room temperature, the solids were filtered off, and washed with the minimum of ice-cold water. The yield was 5.3 g of flaky plate-like iridescent crystals with a rich sweet honey odour. Dichloromethane (25 mL) was then used to extract the filtrate, and the upper organic layer separated, then the solvent removed. The crystals were filtered off, and washed with the minimum of ice-cold water. This yielded 1.5 g of off-white flaky crystals. The final yield was 6.8 g (76%).

Synthesis of malonic acid



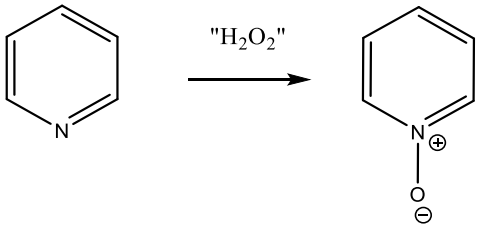
Sodium-potassium cyanide (11 g, 175 mmol CN-) was dissolved in water (25 mL). Chloroacetic acid (15 g, 159 mmol) dissolved in water (30 mL) was neutralised with sodium hydroxide (6.4 g, 160 mmol). The two solutions were warmed to 40°C, and the cyanide solution added to the sodium chloroacetate solution, then stirred at room temperature (on a larger scale an ice bath must be used), then chilled in a cold water bath to keep the temperature below 60°C. The reaction mixture was allowed to exotherm for 30 minutes, then heated to 80°C for 30 minutes. Sodium hydroxide (7 g, 175 mmol) was then dissolved in water (20 mL) and added to the reaction mixture. The reaction mixture was then refluxed for 3 hours, then allowed to cool to 0°C. Hydrochloric acid (25 mL, 36%, 11.6 mol dm-3) was then added to acidify the solution. The water was then removed, and the solids extracted with diethyl ether (50 mL) for 30 minutes with stirring. The solids were filtered off, and the extraction was repeated twice more, and the solvent removed from the filtrate. The final yield was g (%) of white crystals.

Synthesis of carbamide peroxide (urea-hydrogen peroxide adduct)

$CO(NH_2)_2 + H_2O_2 \rightarrow CO(NH_2)_2 \bullet H_2O_2$

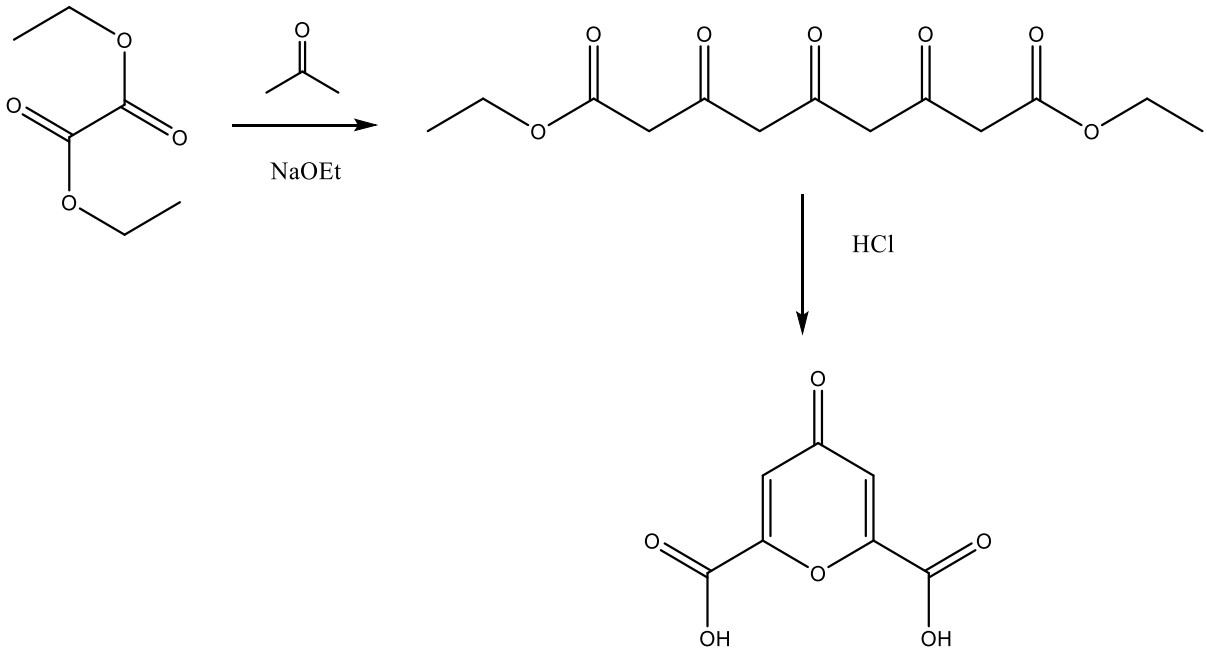
To a 250 mL beaker were added urea (66 g, 1.10 mol), and hydrogen peroxide (150 g, 25%, 1.10 mol). The reaction mixture was then warmed to achieve total dissolution of the urea, then allowed to cool to room temperature, then chilled in an ice-bath. The crystalline precipitate was filtered off, and dried on the pump. The yield was 46 g of long white shard-like crystals. The filtrate was chilled further, and a second batch obtained of 34 g of identical crystals. The final yield was 80 g (77%) of long white shard-like crystals.

Synthesis of pyridine N-oxide



To a 250 mL round bottomed flask was added pyridine (20 g, 255 mmol), and warmed in an oil bath to 90°C. Carbamide peroxide (24 g, 255 mmol) was then added slowly with stirring, maintaining the temperature between 90-100°C. The reaction mixture was then stirred in the oil bath for 1 hour, then allowed to cool to 0°C, and the crystals (urea) filtered off. These were washed with dichloromethane (20 mL). The solvent was removed from the filtrate, and the residue allowed to cool to room temperature. The crude yield was 8.5 g (35%) of brown waxy hygroscopic solid. The solid were washed with acetone yielding a tan powder.

Synthesis of chelidonic acid monohydrate



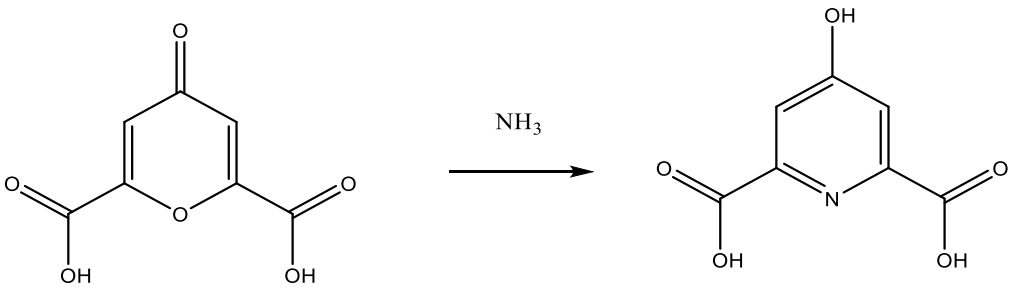
Synthesis of diethyl 3,5,7-trioxononanedioate

To an oven-dried 250 mL round bottomed flask was added sodium ethoxide (14 g, 206 mmol), and dissolved in the minimum of absolute ethanol (40 mL). Dry diethyl oxalate (26 g, 178 mmol) was then added to the reaction flask, and dry acetone (5.2 g, 89 mmol) dissolved in absolute ethanol (20 mL) added dropwise over a period of 40 minutes. Half-way through the addition, the reaction mixture was slowly warmed to 60°C over 20 minutes, then warmed to 75°C for 1 hour. The reaction mixture was then allowed to cool to 0°C. Very cold (<0°C) hydrochloric acid (20 mL, 36%, 11.6 mol dm^{-3}) was then added dropwise to the cold reaction mixture. The precipitate was then filtered off, and washed with ice-water (20 mL). The filtrate stained extremely well, and may have contained more product.

Synthesis of chelidonic acid

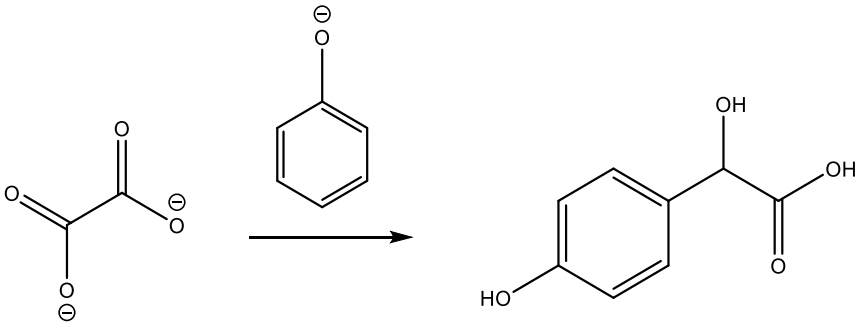
The solids were added to the 250 mL round bottomed flask, along with hydrochloric acid (40 mL, 36%, 11.6 mol dm^{-3}). This was then refluxed for 2 hours, and an additional 10 mL of hydrochloric acid added, then refluxed for a further hour. The reaction mixture was then boiled without a condenser for a further hour. The chilled reaction mixture was then filtered, and the precipitate washed with ice-water (10 mL), then dried in the over at 120°C. The final yield was 8.4 g (47%) of light brown powder.

Synthesis of chelidamic acid monohydrate



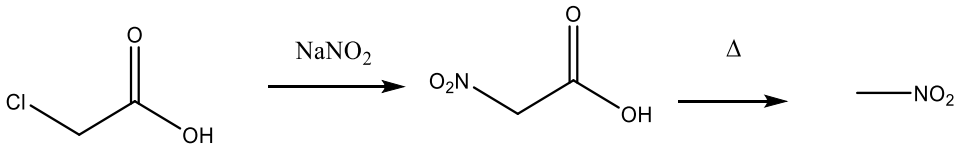
To a 250 mL round bottomed flask was added, ammonium bicarbonate (14 g, 177 mmol) and water (50 mL). Chelidonic acid monohydrate (8.0 g, 39.6 mmol) was then slowly added, then water (10 mL) was added. The reaction mixture was then heated to 90°C for 4 hours, with intermittent addition of an arbitrary amount of ammonium bicarbonate to maintain a high concentration of ammonia. The solvent (water) was then removed keeping the temperature below 95°C, then allowed to cool to room temperature. Hydrochloric acid (7 mL, 36%, 11.6 ml dm^{-3}) was then added dropwise to the solution, and the precipitate filtered off, and washed with a small amount of cold water. The final yield was 4.5 g (57%) of brown powder.

Synthesis of hydroxymandelic acid?



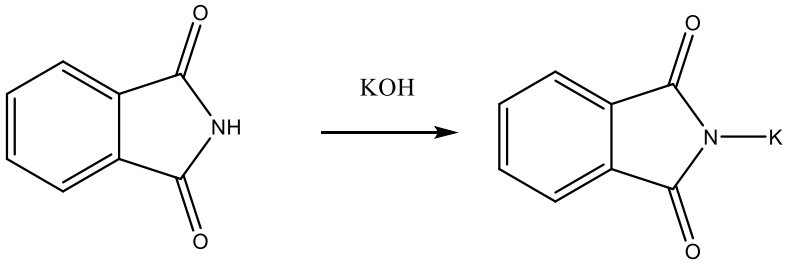
Oxalic acid dihydrate (30 g, 238 mmol) was dissolved in water (100 mL), and magnesium shavings (6 g, 247 mmol) added slowly. An ice bath was used to control the temperature. After complete addition, the reaction mixture was stirred for 1 hour to ensure complete reaction. Potassium hydroxide (13.4 g) dissolved in water (20 mL) was then added to the reaction mixture, and the precipitate filtered off. Phenol (24 g, 262 mmol) was then dissolved in potassium hydroxide (15 g) dissolved in water (70 mL), then added to the filtrate. The reaction mixture was then allowed to stand at room temperature for 48 hours. The reaction mixture was filtered to remove any solids, and hydrochloric acid (30 mL, 36%, 11.6 mol dm^{-3}) was then added until the pH 3. The upper phenol layer was removed, and the reaction mixture chilled in the refrigerator for 1 week. The precipitate was filtered off, to yield a 3.2 g of acidic crystalline white powder insoluble in water, dichloromethane, and diethyl ether, but soluble in sodium hydroxide solution. If hydroxymandelic acid, 6.5% yield.

Synthesis of nitromethane



Sodium hydroxide (45 g, 1.125 mol) was dissolved in water (120 mL) and chilled in the refrigerator. Chloroacetic acid (100 g, 1.06 mol) was dissolved in water (60 mL) and ice (120 g), then the sodium hydroxide solution was added slowly, maintaining the temperature below 22°C. This solution was then added to a 1000 mL round bottomed flask, and sodium nitrite (74 g, 1.07 mol) dissolved in water (70 mL) was added after setting up for distillation. The reaction mixture was then heated strongly without stirring until the temperature reached around 80°C. At this point the exotherm became self-sustaining, and heating was removed. The self-sustaining reaction lasted about 40 minutes, after which gentle heat was applied, maintaining the temperature at around 100°C. Distillation was continued until no more oil was collected. To the distillate was added sodium chloride (20 g), and the lower organic layer separated, and dried with calcium chloride. The final yield was 22 g (34 %) of very pale yellow liquid.

Synthesis of potassium phthalimide



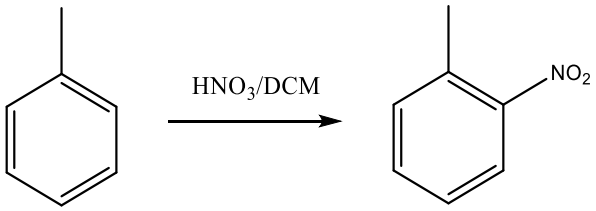
To a 250 mL beaker was added ethanol (100 mL) and potassium hydroxide (8 g, 143 mmol), then stirred to dissolve, and heated to 70°C. Phthalimide (17.5 g, 119 mmol) was then added, and the reaction mixture stirred at 70°C for 1 hour. The reaction mixture was allowed to cool to room temperature, then the precipitate was filtered off and washed with absolute ethanol (15 mL) and dried at the pump. The final yield was 23 g (99%) of fine white powder.

Synthesis of ethyl iodide

$EtOH + AlI_3 \rightarrow EtI + Al(OH)_3$

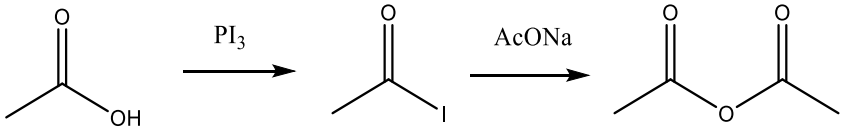
To a 250 mL round bottomed flask was added iodine (30 g, 236 mmol), ethanol (40 mL), and aluminium foil (5 g, 185 mmol), and the flask set up for reflux. The reaction mixture was allowed to stir until the reaction was complete – 2.5 hours. When it began to cool, the apparatus was set up for distillation, and the reaction mixture distilled to dryness. The distillate was then washed with brine (2x 50 mL), and brine with a little sodium bisulphite (50 mL), then the organic layer dried over calcium chloride. The crude yield was 24 g (65%) of yellow liquid.

Synthesis of o-nitrotoluene



To a 250 mL Erlenmeyer flask was added sulphuric acid (25 mL, 98%, 18.3 mol dm^{-3}, 466 mmol), dry, finely-powdered potassium nitrate (40 g, 396 mmol) and stirred for 30 minutes at room temperature. Dichloromethane (90 mL) was then used to extract the nitric acid from solution and stirred for 1 hour, after which the liquid was decanted. The solution fumed incredibly strongly, on a par with dry ice and water. The nitric acid solution was then chilled in an ice bath to 5°C and toluene (25 mL, 205 mmol) was slowly added, then allowed to stir in the ice bath for 1 hour, then stirred at room temperature for 1 hour. Water (80 mL) was then added, and stirred well, then the lower organic layer separated and washed with saturated sodium bicarbonate solution (50 mL) and water (50 mL). The solvent was removed from the organic layer, yielding 9.5 g (29%) of yellow liquid with a strong sweet almond and floor polish odour. The liquid did not freeze at -12°C.
Tetrahedron Letters, Vol 42(7), 1387 – 1389 (2001)

Synthesis of acetic anhydride



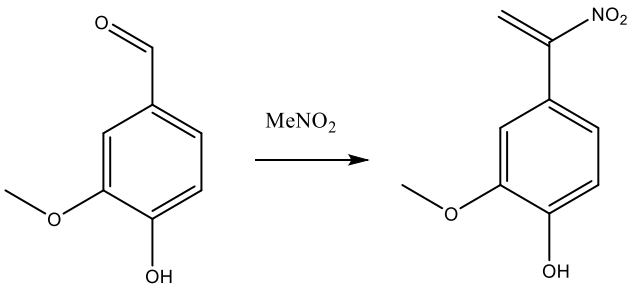
Synthesis of acetyl iodide

To a 100 mL round bottomed flask was added iodine (20 g, 158 mmol), glacial acetic acid (15 mL, 262 mmol, but stoichiometric amount recommended), and slowly red phosphorus (2.5 g, 81 mmol), then the reaction mixture was stirred for 15 minutes. The reaction mixture was then distilled until no more distillate collected. The distillate reacted rapidly with water with evolution of heat, and generated the sharp smell of acetic anhydride on contact with anhydrous sodium acetate. It also did not freeze at -10°C.

Synthesis of acetic anhydride

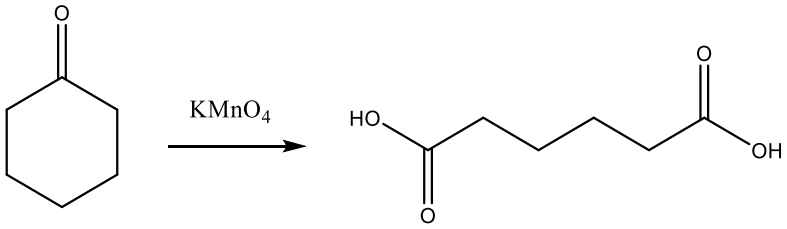
Anhydrous sodium acetate (15 g, 183 mmol) was then slowly added to the chilled distillate. The reaction mixture was then distilled collecting the distillate at 130-140°C until no more distillate collected. The yield was 22 g of acetic acid and acetic anhydride.

Synthesis of 1-(4-hydroxy-3-methoxyphenyl)-2-nitrostyrene



To a 100 mL round bottomed flask was added vanillin (10 g, 65.7 mmol), nitromethane (4.4 g, 72.1 mmol), absolute ethanol (15 mL) and stirred to dissolve. Methylamine hydrochloride (1 g) dissolved in absolute ethanol (10 mL) was added to potassium hydroxide (0.5 g) dissolved in ethanol (5 mL), and 1 mL of the methylamine solution added to the 100 mL round bottomed flask. The reaction mixture was then gently refluxed for 2 hours, and the solvent boiled off for a further 30 minute. After cooling to room temperature, cold water was added dropwise until crystals began to form, at which point the reaction mixture was chilled in the refrigerator for a couple of hours. The solids were then filtered, and washed with cold methanol (30 mL). The crude yield was 7.9 g (61%) of yellow flakes. The crude product was then recrystallized from methanol to yield 6.5 g (51%) of fluffy bright yellow crystals.

Synthesis of adipic acid



To a 500 mL round bottomed flask was added warm (40°C) water (150 mL), potassium permanganate (32 g, 203 mmol), and sodium hydroxide (0.5 g) dissolved in water (5 mL). Cyclohexanone (10 g, 102 mmol) was added slowly, with the reaction mixture cooled in an ice bath, keeping the temperature below 65°C. After complete addition, the reaction mixture was allowed to stir for 1 hour at room temperature, then filtered. Hydrochloric acid (10 mL, 36%, 11.6 mol dm^{-3}) was then added, and the reaction mixture chilled in the freezer, and the precipitate filtered off. The yield was 3.5 g (24%) of off-white crystals.

Synthesis of sodium dithionite

$$2NaHSO_3 + Zn \rightarrow Na_2S_2O_4 + ZnO + H_2O$$

To a 250 mL beaker was added sodium bisulphite (50 g, 480 mmol), water (50 mL), zinc powder (19 g, 290 mmol), and the beaker cooled in an ice bath. The reaction mixture must be above 10°C before starting. After the reaction had finished, water (10 mL) was added, and the precipitate filtered off, then washed with warm water (2x 10 mL). The filtrate is fit for purpose as a reducing agent, but the solid can be precipitated through use of ethanol. A portion of the filtrate (15 mL) was added to absolute ethanol (75 mL), then chilled to 0°C and the precipitate filtered off. The final yield was 8 g (63%) of white powder. This is the dihydrate. The solid is however highly unstable, heating up

considerably in a matter of minutes on being left out in the open air. However, it is stable in low pH solutions.

Synthesis of manganese(III) acetate

$MnSO_4 + Na_2CO_3 \rightarrow MnCO_3 + Na_2SO_4$

$MnCO_3 + 2AcOH \rightarrow Mn(OAc)_2 + CO_2 + H_2O$

$2Mn(OAc)_2 + KMnO_4 \rightarrow Mn(OAc)_3 + AcOK + H_2O$

Manganous sulphate (2.3 g, 136 mmol) dissolved in water (80 mL) was added to anhydrous sodium carbonate (16 g, 151 mmol) dissolved in water (250 mL) and stirred for 10 minutes, then the precipitate filtered off, then washed with water (50 mL) and dried in the oven. To a 500 mL round bottom flask was added glacial acetic acid (100 mL) and the manganous carbonate, then refluxed for 4 hours, then after cooling slightly, glacial acetic acid (70 mL) and potassium permanganate (5 g, 32 mmol) added, then stirred at 100°C for 15 minutes before allowing to cool to 0°C. The precipitate was then filtered off, and the solids dried over calcium oxide. The filtrate was chilled for 1 week in the refrigerator, then filtered again and also dried over calcium oxide. The final yield was 40 g of brown cinnamon-coloured powder with a faint acetic acid odour.

Synthesis of ammonium methylsulphate

$HSO_3NH_2 + MeOH \rightarrow NH_4SO_3Me$

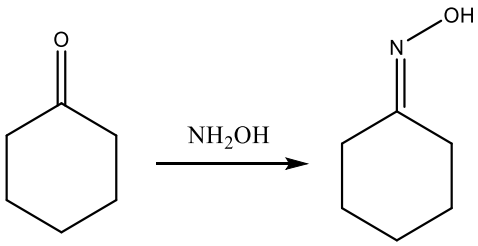
To a 500 mL flask was added sulphamic acid (125 g, 1.29 mol) and dry methanol (225 mL, 5.55 mol), then refluxed with a drying tube at the top of the condenser for 5.5 hours. The solvent was then removed, and the residue allowed to cool to room temperature. The solid was remelted, and poured out to cool as a plate. The crude yield was 157 g (<94%) of white solid, ~90% pure.

Synthesis of methylamine

$NH_4SO_3Me \rightarrow MeNH_2 + SO_2 + H_2O$

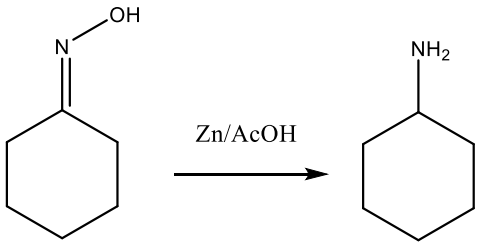
To a 1000 mL beaker was added ammonium methylsulphate (100 g, 1.2 mol), and heated to 275°C, then held there for 10 minutes before cooling to room temperature. The product was highly hygroscopic. The solid was first dissolved in water (80 mL), then added to a large (1000 mL) conical flask. Sodium hydroxide solution (300 mL, 7 mol dm^{-3}) was then added dropwise to the hot solution, and the gases produced dissolved in water (40 mL). After complete addition, the reaction mixture was boiled for 30 minutes until no more soluble gas was produced. Hydrochloric acid (20 mL, 36%, 11.6 mol dm^{-3}) was then added to the methylamine/ammonia solution, and the neutralised solution boiled to remove the water. The dry liquid was then allowed to cool to room temperature covered. To the solids, absolute ethanol (40 mL) was used to dissolve, and the precipitate filtered off, and washed with hot absolute ethanol (25 mL). The filtrate was chilled in the freezer for a few hours, then the crystals filtered off. The final yield was ~25 g (~30%) of flaky white crystals.

Synthesis of cyclohexanone oxime



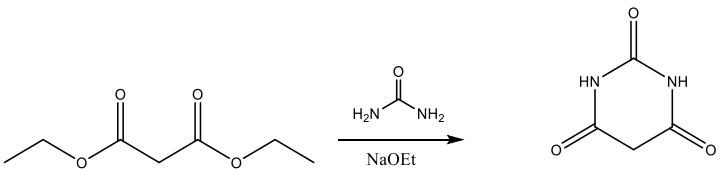
Sodium hydroxide solution (4.5 g, 113 mmol in 20 mL water) was added to hydroxylamine hydrochloride (7.8 g, 112 mmol) dissolved in water (10 mL). Cyclohexanone (10 g, 102 mmol) was then added to the hydroxylamine freebase solution dropwise with stirring, and was allowed to stir for a further 30 minutes. The precipitate was then filtered off and washed with water (20 mL) to yield 12 g (99%) of slightly damp white crystalline powder.

Synthesis of cyclohexylamine



At room temperature with stirring, zinc dust (21 mg, 321 mmol) was added to a solution of cyclohexanone oxime (12 g, 106 mmol) in glacial acetic acid (60 mL, 1.05 mol). Stirring was continued for another 2.5 hours in a water bath. The reaction mixture was then neutralised with sodium hydroxide (47 g, 1.18 mol) dissolved in water (300 mL), then steam distilled until perfectly clear distillate was condensing. The distillate was then extracted with dichloromethane (2x 15 mL), and the combined organic layers dried over magnesium sulphate. The solvent was removed to yield 11g (105%) of clear colourless liquid with an amine and cyclohexanone odours – hydrolysis occurs. JOC 57, 6324 (1992)

Synthesis of barbituric acid



To a dry 100 mL round bottomed flask fitted with a dry condenser and a drying tube was added anhydrous ethanol (20 mL) and sodium (1.1 g, 50 mmol). After complete reaction, dry diethyl malonate (4.0 g, 25 mmol), and dry urea (2.2 g, 37 mmol) dissolved in anhydrous ethanol (15 mL). The reaction mixture was then refluxed for 2 hours, then warm (60°C) water was added through the condenser. Hydrochloric acid (8 mL, 36%, 11.6 mol dm^{-3}) was then added, yielding a clear acidic solution. The reaction mixture was then chilled to 0°C for 6 hours, and the precipitate filtered off, and washed with ice-water and air dried. The crude yield was 2.3 g (72%) of fluffy cream crystals.

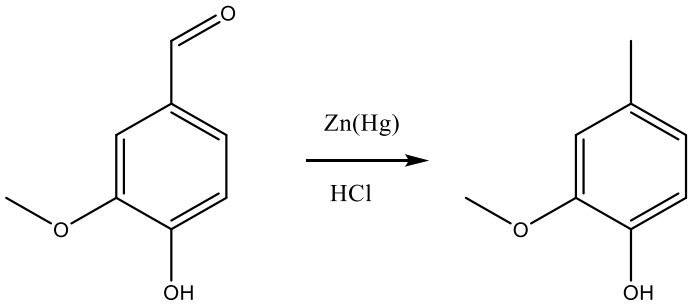
Synthesis of hydrazine sulphate

$NaOCl + CO(NH_2)_2 \longrightarrow N_2H_4 + CO_2 + NaCl$

$N_2H_4 + H_2SO_4 \longrightarrow N_2H_5{\cdot}HSO_4$

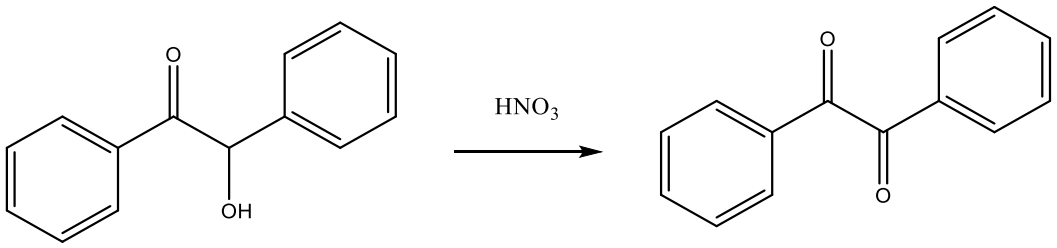
To a 1000 mL beaker was added chilled bleach (500 mL, 5.25%, 7.06 mol dm^{-3}, 353 mmol) and sodium hydroxide (16 g, 400 mmol), then chilled back to 7°C, then another 16 g of sodium hydroxide was added before being rechilled. Gelatine (0.75 g) was dissolved in warm distilled water (40 mL). Urea (22 g, 366 mmol) was then dissolved in the minimum of warm water (40 mL), and combined with the gelatine solution. To the vigorously stirred cold bleach, the urea solution was rapidly added, and the reaction mixture heated to 85°C for 5 minutes, then allowed to cool to 0°C. Chilled (0°C) sulphuric acid (100 mL, 50%, 9.1 mol dm^{-3}) was then added slowly to neutralise the reaction mixture, and form the salt. The reaction mixture was then chilled to just above 10°C, then the precipitate filtered off. The final yield was 22.6 g (49%) of white crystals. To the filtrate was added anhydrous copper sulphate (20 g) dissolved in water (80 mL), and the precipitate filtered off. The yield was 6.9 g (7%) of additional hydrazine sulphate as the copper sulphate double salt.

Synthesis of 2-methoxy-4-methylphenol



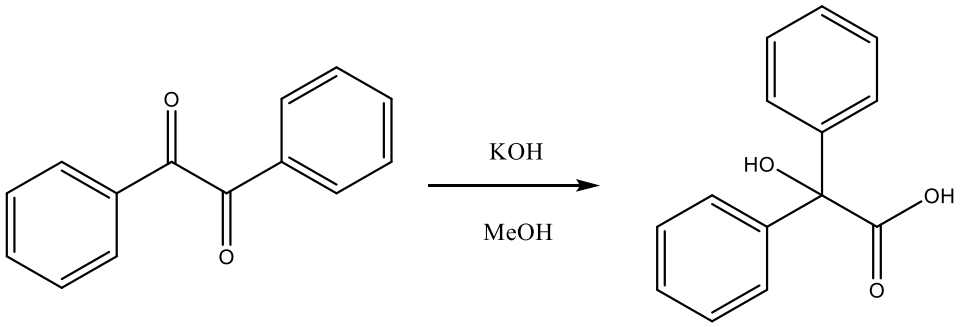
To a 50 mL beaker was added mercury (0.45 g, 2.2 mmol), and aqua regia added (5 mL) added until the mercury dissolved. The solution was then diluted with water (50 mL) and neutralised with saturated sodium carbonate solution (25 mL). Zinc powder (50 g, 765 mmol) was then added to the mercury solution, and stirred for 1 hour. Vanillin (15 g, 99 mmol) was dissolved in ethanol (45 mL), and hydrochloric acid (100 mL, 36%, 11.6 mol dm^{-3}) added. The zinc/mercury mixture was filtered, and washed with a little cold water. To a 1000 mL round bottomed flask fitted with an addition funnel was added the zinc amalgam, and water (20 mL) added, and the vanillin solution added to the addition funnel. The vanillin solution was added dropwise to the stirred reaction mixture over a period of 1 hour. Hydrochloric acid (20 mL, 36%, 11.6 mol dm^{-3}) was then added dropwise to the reaction mixture, and the reaction mixture stirred at 70°C during addition, and then for 45 minutes after complete addition. The reaction mixture was allowed to cool to 5°C, and the precipitate filtered off. The solids were washed with dichloromethane (25 mL), and the lower organic layer separated from the filtrate, then the aqueous layer extracted with dichloromethane (25 mL). The combined organic layers were dried over magnesium sulphate, and the solvent removed. The crude yield was 9.6 g (70%) of orange-brown liquid with a woody, slightly smoky, balsamic, sweet candy-like phenolic odour.

Synthesis of benzil



To a 50 mL Erlenmeyer flask was added nitric acid (15 mL, 68%, 15.8 mol dm^{-3}, 119 mmol), benzoin (5 g, 23.6 mmol), and the reaction mixture heated to 70°C for 15 minutes, then allowed to cool slightly and water (40 mL) added. The reaction mixture was then chilled, and the precipitate filtered off. The crude yield was 5 g. The crude product was recrystallized from ethanol, yielding 4.8 g (97%) of cream powder. Through testing with sodium hydroxide solution, the product was found to be contaminated with benzoin (purple colouration).
$CuSO_4$ and py: orgsyn.org/demo.aspx?prep=CV1P0087

Synthesis of benzilic acid



To a 100 mL round bottomed flask fitted with a reflux condenser was added methanol (20 mL), benzil (4.8 g, 22.8 mmol), then stirred and heated to dissolve. Potassium hydroxide (1.9 g, 34.3 mmol) dissolved in water (5 mL) and added dropwise through the top of the condenser, then refluxed for 30 minutes. After cooling to <0°C, the precipitate was filtered off, and the solvent removed from the filtrate, then the sludgy residue filtered and washed with ice-cold methanol (10 mL). The combined batches were dissolved in the minimum of boiling water (~100 mL), and hydrochloric acid (10 mL, 36%, 11.6 mol dm^{-3}) added. The reaction mixture was allowed to cool to 0°C, and the precipitate filtered off. The final yield was 1.3 g (25%) of off-white flakes.

Preparation of steak

Shallots (100 g) were prepared by separating the cloves and trimming the top and bottom, then removing the dry outer layers, and the first white layer. The shallots were then finely sliced. An arbitrary quantity of fresh green peppercorns were then stripped from the stalk.
To a heavy bottomed saucepan was added unsalted butter (25 g), the sliced shallots and the mixture heated on a low heat. The mixture was stirred, taking care to keep the heat low enough to avoid Maillard reaction products, but still cook the shallots. After a few minutes of stirring and heating, the green peppercorns were added, and cooked for a further few minutes. At this point, brandy (15 mL, whiskey can be substituted) was added and the mixture flambéed. The mixture was allowed to cool slightly, and whipping cream (60 mL) added then heated gently, not allowing the mixture to boil. A

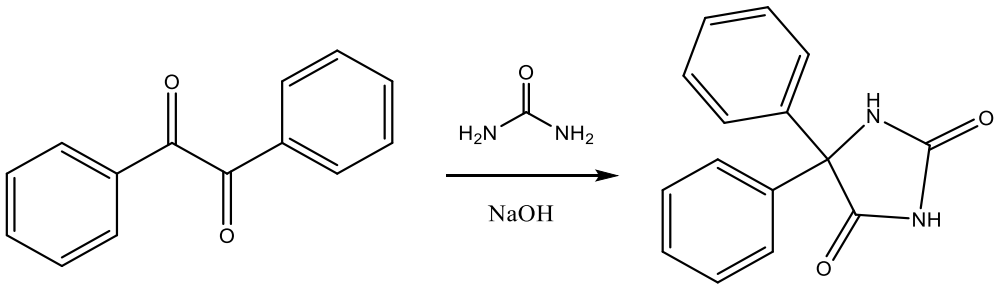
small amount of powdered white pepper and powdered nutmeg was then added, and the powders stirred into the sauce. The resulting sauce was then put to one side, and kept warm.
A heavy-based cast iron frying pan was wetted with olive oil. A rib-eye steak fillet (250 g) was sprinkled with flaky sea-salt and freshly powdered black pepper, then drizzled with a little olive oil. The frying pan was heated until the oil began to smoke, and the steak added, and allowed to sit for 4 minutes on each side. The steak was then transferred to a warm plate, and allowed to sit in a warm oven for 10 minutes. The sauce prepared earlier was then reheated, and the juices from the cooked steak added to the sauce.
The steak was then placed on a plate, with a portion of sauce in a small dish and half a dozen spears of asparagus added. Recommended liquid accompaniments are Burgundy, Bordeaux, Sangiovese.

Synthesis of sodium azide

$N_2H_4 \bullet H2O + {}^{i}PrONO + KOH \rightarrow KN_3 + {}^{i}PrOH + 3H_2O$

To a 250 mL beaker were added dry hydrazine sulphate (30 g, 231 mmol) and absolute ethanol (50 mL), and cooled in an ice bath. Sodium hydroxide (9.2 g, 230 mmol) was then added and stirred well until no more exotherm was detected, ~20 minutes. An additional portion of sodium hydroxide (9.2 g, 230 mmol) was then added, and stirred well for 10 minutes. The supernatant was then decanted, and the solids washed with absolute ethanol (20 mL).
To a 500 mL round bottomed flask fitted with a reflux condenser and dropping funnel was added the hydrazine solution prepared earlier, ethanol (20 mL), and sodium hydroxide (9.2 g, 230 mmol), then stirred to dissolve. Dry isopropyl nitrite (28 mL, 277 mmol) was then added to the addition funnel, and the isopropyl nitrite added dropwise over a period of 30 minutes to the stirred reaction mixture. The reaction mixture was then refluxed for 20 minutes, then allowed to cool to 5°C, and the precipitate filtered off and washed with cold ethanol (20 mL). The final yield was 12.8 g (85%) of fluffy white powder.

Synthesis of phenytoin

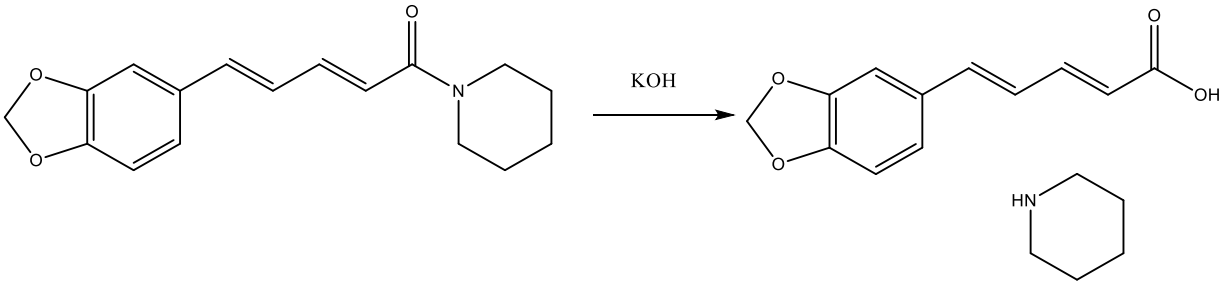


To a 250 mL round bottomed flask was added benzil (5 g, 23.8 mmol), ethanol (40 mL), urea (2.9 g, 48.3 mmol), and stirred and heated to dissolve. Sodium hydroxide (3.8 g, 95.1 mmol) dissolved in water (10 mL) was then added, and the reaction mixture refluxed for 90 minutes. After cooling to room temperature, water (10 mL) added and the reaction mixture filtered. To the filtrate was dropwise added hydrochloric acid (10 mL, 36%, 11.6 mol dm^{-3}) to neutralise the solution, then chilled in the refrigerator. The precipitate was then filtered off, and washed with cold water (10 mL). The crude yield was 4.3 g (72%) of fluffy pale yellow powder. The product can be recrystallized from ethanol.

Preparation of Type A Urushibara nickel

Basic nickel carbonate tetrahydrate (5 g, 13.3 mmol) was added to a 50 mL beaker, and hydrochloric acid (8 mL, 36%, 11.6 mol dm^{-3}) added carefully until all the solids had dissolved. Zinc dust (10 g, 153 mmol) was used to cover the bottom of a large (250 mL) beaker, and the nickel chloride solution added rapidly. The more violent the reaction, the better the catalyst generated. The precipitate was then filtered off, and washed with deionsised water (50 mL) then sucked dry.
Type A – nitro reduction
To the solids was added glacial acetic acid (15 mL) dissolved in water (80 mL), then allowed to sit and react for 30 minutes until the faintest hint of green appeared. The precipitate was then filtered off, and washed thoroughly with deionised water (50 mL) and absolute ethanol (2x 10 mL). The final yield was 4.1 g of black light fluffy powder.
Type B – oxime, ketone, imine reduction

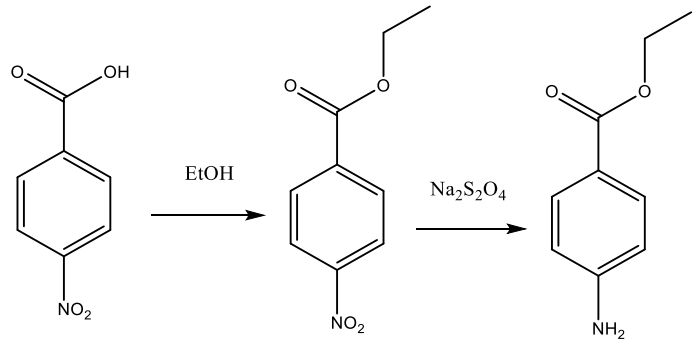
Synthesis of piperidine and piperinic acid



To a 100 mL Erlenmeyer flask was added piperine (7 g, 24.5 mmol), potassium hydroxide (5 g, 89.1 mmol), and ethanol (40 mL), then the reaction mixture was refluxed for 3 hours. The reaction mixture was then distilled to dryness.[1] Water (50 mL) was added to the reaction flask, and stirred for 10 minutes, then filtered and washed with a little hot water. Hydrochloric acid (15 mL, 36%, 11.6%) was then added dropwise, and the precipitate filtered off and washed with cold water (30 mL), then dried in the oven. The crude yield of piperinic acid was 4.6 g (86%) of beige powder with a slightly sweet, pungent bubble-gum odour.
Hydrogen chloride gas was then passed into the distillate until strongly acidic, then the solvent was removed. The crude yield of piperidine hydrochloride was 1.3 g (44%) of off-white crystalline powder.

(1) Addition of 30 mL of ethanol at the end of the first distillation run, mixing thoroughly and distilling to dryness again improves the yield of piperidine (~60-70% yield).

Synthesis of benzocaine



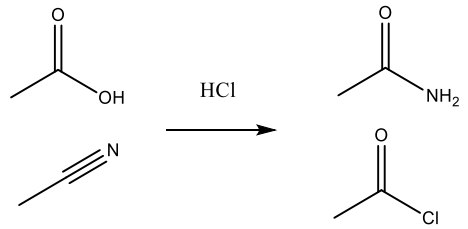
Synthesis of ethyl p-nitrobenzoate

To a 250 mL round bottomed flask was added p-nitrobenzoic acid (5 g, 29.9 mmol), absolute ethanol (40 mL), sulphuric acid (5 mL, 98%, 18.3 mol dm^{-3}), then refluxed for 3 hours, after which the solution was concentrated slightly. The cooled reaction mixture was then poured into cold water (75 mL), and the precipitate filtered off. The crude product was then sucked dry, and dried over calcium chloride. The crude yield was 5.9 g (101%) of light brown powder.

Synthesis of ethyl p-aminobenzoate

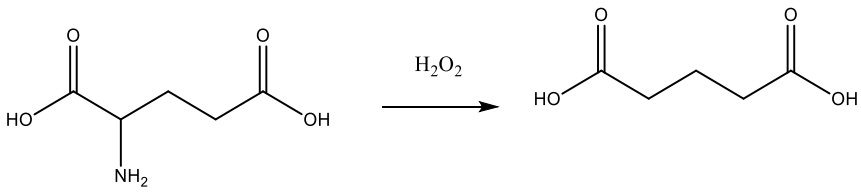
Sodium bisulphite (50 g) was dissolved in water (50 mL) and cooled in a water bath. Zinc powder (19 g) was then added and stirred until the reaction was complete. The precipitate was filtered off, and to the filtrate (sodium dithionite solution) was added sodium carbonate (8 g) and dissolved. The solution was added to a 250 mL round bottomed flask, and ethyl p-nitrobenzoic acid (4 g, 20.5 mmol) dissolved in ethanol (20 mL) added slowly, then heated just below boiling for 2.5 hours. Dichloromethane (40 mL) was then added to the cooled solution, and the mixture filtered. To the filtrate was added sodium hydroxide (3 g) dissolved in water (20 mL), and the lower organic layer separated. The aqueous layer was extracted with dichloromethane (20 mL), and hydrochloric acid (15 mL, 36%, 11.6 mol dm^{-3}) dissolved in water (100 mL) added. The upper aqueous layer was separated, and sodium hydroxide solution added until no more precipitate formed. The reaction mixture was chilled, and the precipitate filtered off. The yield was 1.1 g (32%) of white powder.

Synthesis of acetyl chloride



To a 500 mL round bottomed flask was added dry glacial acetic acid (50 mL, 873 mmol) and dry acetonitrile (46 mL, 880 mmol), then chilled in an ice-bath to below 5°C. Hydrogen chloride gas (3 mol) was then passed into the reaction flask until 20 minutes after no more gas dissolved, maintaining the temperature around 3°C. Total reaction time was 1.5 hours. The reaction flask was then chilled in the freezer for 24 hours, and then distilled, collecting the fraction at 50-60°C. The yield was 20.5 g (30%) of clear colourless liquid that fumed strongly in humid air. The remaining liquid in the boiling flask was chilled for 24 hours in the refrigerator, and the crystals filtered off. The yield of acetamide hydrochloride was ~22g of white crystals with the odour of acetic acid.

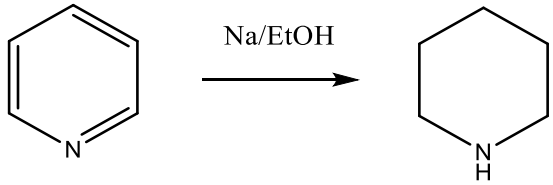
Synthesis of succinic acid



Monosodium glutamate (40 g, 237 mmol) was dissolved in water (70 mL), and hydrochloric acid (18 mL, 36%, 11.6 mol dm^{-3}, 230 mmol) added, then the reaction mixture was chilled and the precipitate filtered off and washed with cold water, then sucked dry. The yield of glutamic acid was 36 g (>100%) of fluffy white crystals.
Glutamic acid (25 g, 167 mmol) was added to a 250 mL beaker, and water (60 mL) added. Ammonium bicarbonate (13.4 g, 169 mmol) was then added slowly with stirring. Hydrogen peroxide (58 g, 30%, 512 mmol) was then added slowly to the warmed (65°C) solution. The reaction mixture was cooled in a water bath when the temperature exceeded 75°C. Total addition took 30 minutes, after which the reaction mixture was heated to 80°C for 1.5 hours. Sulphuric acid (10 mL, 50%) was then added to the cooled reaction mixture, then chilled in the refrigerator. The precipitate was filtered off to yield 5.5 g (27%) of damp white powder.

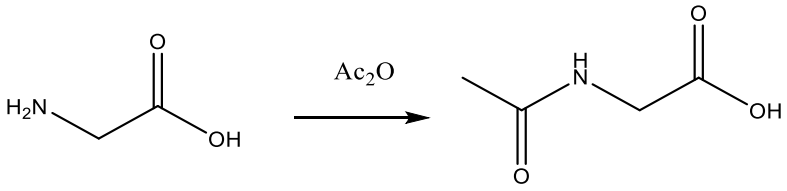
Note on the oxidation of glutamic acid and aspartic acids by means of hydrogen peroxide, H. D. Dakin (J. Biol. Chem.)

Synthesis of piperidine



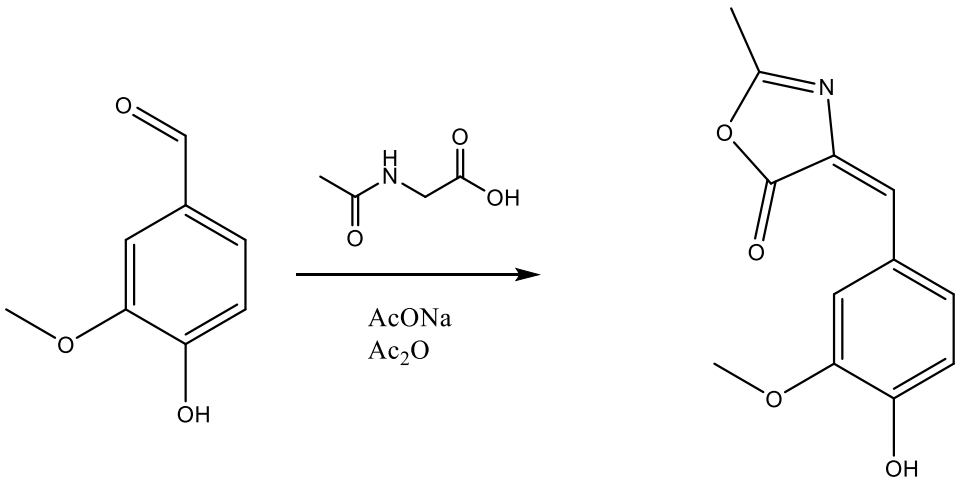
To a 1000 mL round bottomed flask fitted with a condenser was added dry pyridine (15 g, 190 mmol), anhydrous ethanol (300 mL), and sodium metal (56 g, 2.43 mol) slowly added. It was found that the addition of methanol (30 mL) increased the rate of reaction towards the end of the addition. The ethanol was then distilled off, and water (250 mL) gradually added through the distillation. After the ethanol had been removed, a steam distillation was carried out to separate the piperidine, collecting the distillate until it was monophasic. Hydrochloric acid (25 mL, 20%) was then added to neutralised the distillate. The solvents were then removed, resulting in a crude yield (19 g) of piperidine hydrochloride. This was then dissolved in hot absolute ethanol, and the supernatant decanted, and distilled. The final yield was 15.4 g (67%) of slightly off-white powder. Recrystallization can be achieved through use of a methanol/diethyl ether solvent/antisolvent system.

Synthesis of acetylglycine



To a 60 mL beaker was added glycine (5 g, 66.6 mmol) dissolved in water (20 mL), then acetic anhydride (9.5 mL, 100 mmol) added and the reaction mixture chilled in an ice-bath, then stirred vigorously for 20 minutes. After chilling for 2 hours, the precipitate was filtered off, and washed with the minimum of ice-water, and sucked dry. The final yield was 5.6 g (72%) of fine white powder.

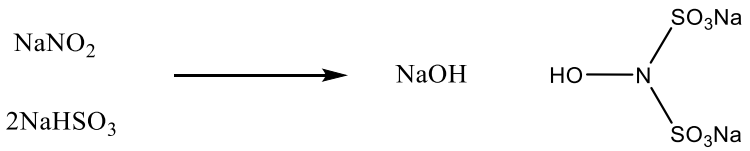
Synthesis of 4-(4-hydroxy-3-methoxybenzylidene)-2-methyl-1,3-oxazol-5(4*H*)-one



To a 100 mL round bottomed flask were added acetylglycine (5.6 g, 47.8 mmol), vanillin (11 g, 71.3 mmol), anhydrous sodium acetate (10 g, 122 mmol), and acetic anhydride (10 mL, 106 mmol). The reaction mixture was heated slowly and gently to melt it (it was found that if heating was too quick, an orange-red solution formed). The reaction mixture was then heated and stirred at 100°C for 45 minutes, then allowed to cool to <0°C. The solidified mixture was then dissolved with hot chloroform (40 mL), and the precipitate filtered off, and washed with chloroform (10 mL). The filtrate was then chilled in the freezer for 24 hours, and the precipitated crystals filtered off. The crystals were then washed thoroughly with petroleum ether (2x 20 mL).[1] The final yield was 6.2 g (55%) of intensely yellow crystals.

(1) It was found that the evaporation of solutions in dichloromethane or chloroform led to a sticky oil; likewise, some solvents like water or ether caused the crystals to revert to an oil.
(2) Sodium hydroxide solution was found to be good at removing yellow stains; the product hydrolyses into an apple-red unsaturated tyrosine derivative.

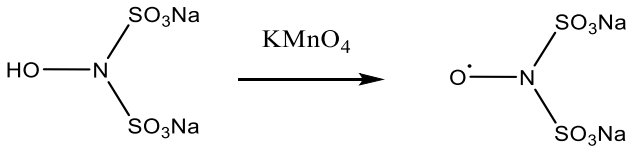
Synthesis of potassium hydroxylamine disulphonate



Sodium bisulphite (90 g, 865 mmol) was suspended in the minimum volume of cold water (120 mL). To a 600 mL beaker was added sodium nitrite (30 g, 435 mmol), and water (24 mL), then chilled to -10°C in an ice-salt bath. The slurry of sodium bisulphite was added dropwise, maintaining the temperature below -5°C over a period of 30 minutes, then allowed to stir at -2°C for 20 minutes. Potassium hydroxide (45 g) dissolved in ice (45 g)[1] was then chilled to <0°C, and added to the chilled reaction mixture, then left overnight to chill in the refrigerator. The crystals were then filtered off, and dried at the pump. The yield was 23 g (~50-60%) of damp white crystals.

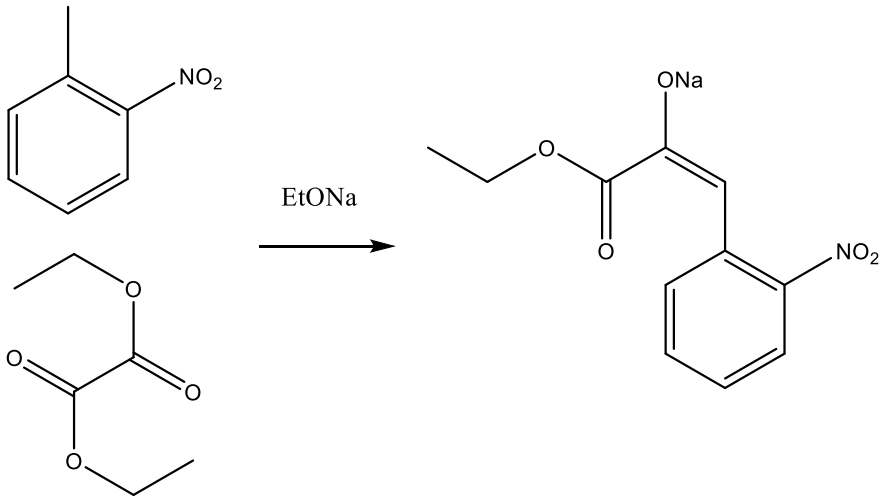
(1) Methyl ethyl ketone can be used to extract the oxime as a separate layer, but acetone cannot be used – the oxime is highly water-soluble

Synthesis of Fremy's salt?



Potassium hydroxylamine disulphonate (5 g, 21.1 mmol) was dissolved in water (5 mL), and added to potassium permanganate (1 g, 6.3 mmol) dissolved in water (10 mL), then allowed to stir for 5 minutes. The precipitate was then filtered off, and the filtrate chilled in the refrigerator for 24 hours. The precipitate was then filtered off, and washed with acetone. The yield was 2.2 g of light brown powder. The product was highly unstable and decomposed overnight to a white powder.

Synthesis of sodium O-ethyl *o*-nitrophenylpyruvate



To a dry 250 mL round bottomed flask fitted with a dry condenser and drying tube was added dry ethanol (40 mL) and dry methanol (10 mL), and sodium metal (4.5 g, 196 mmol), then allowed to react. Dry diethyl oxalate (19.2 g, 131 mmol) was then added and cooled to below 30°C, then o-nitrotoluene (18 g, 131 mmol) dissolved in dry diethyl ether (30 mL) was added with vigorous

stirring. The reaction mixture was then refluxed for 1 hour, then allowed to cool to room temperature, after which diethyl ether (30 mL) was added via the condenser, and the mixture stirred to suspend the solids. The reaction mixture was then chilled overnight in the refrigerator, and the precipitate filtered on a Büchner funnel. The damp paste was then washed with diethyl ether[1] (150 mL) to yield 26.5 g (78%) of dark brown powder.

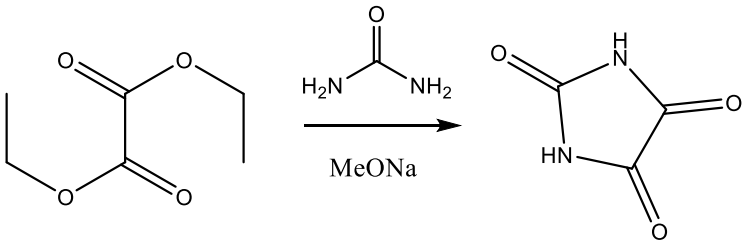
(1) The product is insoluble in diethyl ether and petroleum ether, but soluble in alcohols and dichloromethane

Synthesis of guanidine

$NH_4SCN \rightarrow HNC(NH_2)_2 \bullet H_2CO_3$

To a two-neck 500 mL round bottomed flask fitted with a drying tube and a thermometer was added ammonium thiocyanate (100 g, 1.31 mol), then heated to 190°C for 6 hours. The cold solids were then digested with boiling water (100 mL), then potassium carbonate (15 g) added and stirred dissolve. The reaction mixture was then filtered, and the filtrate evaporated to dryness. The solids were then dissolved in absolute ethanol (150 mL), and the precipitate filtered off, and washed with warm ethanol. The crude yield was 3.9 g (3%) of white powdered guanidinium hydrogencarbonate.

Synthesis of parabanic acid



To a 250 mL round bottomed flask fitted with a condenser was added methanol (70 mL) and sodium metal (5 g, 217 mmol), then allowed to react. Urea (6.5 g, 108 mmol) was then dissolved in the reaction mixture, and then dimethyl oxalate (12.8, 108 mmol) dissolved in methanol (40 mL) added, and the reaction mixture stirred at room temperature for 1 hour. Hydrochloric acid (20 mL, 36%, 11.6 mol dm^{-3}) was then added carefully, and the precipitate filtered off. The solvent was stripped from the filtrate, and the residue recrystallized from water. The final yield was 9.0 g (73%) of white powder.

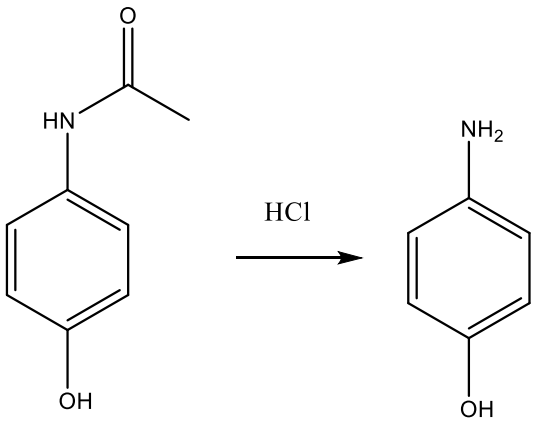
Synthesis of truffles

Marou chocolate (100 g, 72% cocoa solids, Mekong) was broken up into pieces, and put aside. Cold whipping cream (100 g, 35% fat) and flaky sea salt (0.2 g) were then added to a stainless steel bowl, and the mixture whipped until solid, then chilled in the refrigerator. The chocolate was then gently melted over a steam-bath,[1] then allowed to cool slightly until just above body temperature. The molten chocolate was then carefully but thoroughly incorporated into the cream with a fork[2], then allowed to chill for 20 minutes, until just hard enough to remain intact upon delicate manipulation, but not crumbly or sticky to cut. A teaspoonful of mixture was then placed on a section of plastic wrap, and folded up and squeezed to form a ball. The truffle was then lightly rolled in Dutch processed sieved cocoa powder. This process was repeated to finish the mixture.

(1) If water comes in contact with the chocolate, it could cause it to crystallise

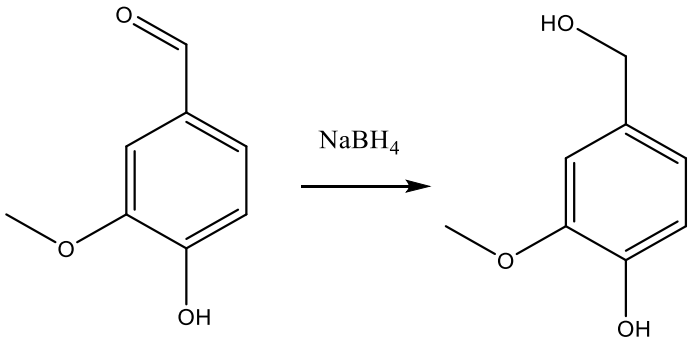
(2) Flavourings (Grand Marnier, Cognac, vanilla, etc.) can be incorporated into the mixture, but there is a risk of crystallisation of the chocolate. Ideally these would be incorporated when the cream was whipped. There is no remedy for crystallisation.

Synthesis of p-aminophenol



To a 100 mL round bottomed flask was added p-(acetylamino)phenol (5 g, 31.1 mmol), and sodium hydroxide (4 g, 100 mmol) dissolved in water (30 mL), then stirred at heated to 80°C for 1 hour. The hot reaction mixture was then filtered, and washed with water (50 mL). The filtrate was neutralised with hydrochloric acid (<10 mL, 36%, 11.6 mol dm^{-3}), then chilled to 0°C and the precipitate filtered off and sucked dry. The crude product was recrystallized from water to yield 2.8 g (78%) of brown powder.

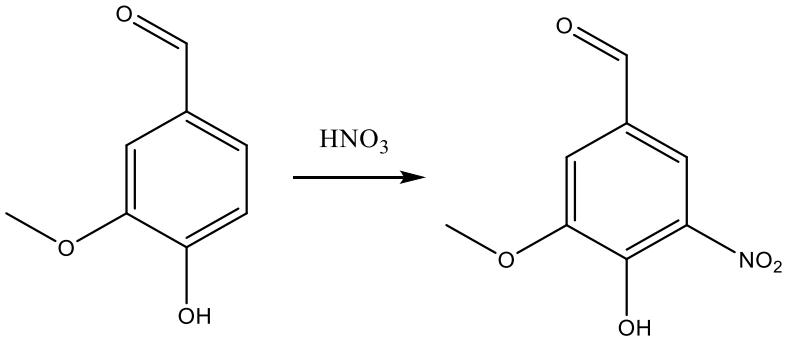
Synthesis of vanillyl alcohol



To a 600 mL beaker was added vanillin (25 g, 164 mmol), water (200 mL), and sodium hydroxide (7.3 g, 183 mmol), then stirred to dissolve, then chilled to 5°C. Sodium borohydride (7.0 g, 185 mmol) was then slowly added to the chilled solution over a period of 20 minutes, maintaining the temperature below 10°C.[1] The reaction mixture was then allowed to stir at room temperature for 30 minutes, then chilled to 5°C. Hydrochloric acid (35 mL, 36%, 11.6 mol dm^{-3}) was added to ice (60 g), and allowed to chill to 5°C. The hydrochloric acid was then added dropwise to the reaction mixture, maintaining the temperature below 10°C, then chilled in the refrigerator for 4 hours. The precipitate was then filtered off, and washed with ice-water, then dried at the pump. The final yield was 23.9 g (94%) of white powder. The product can be recrystallized from ethyl acetate.

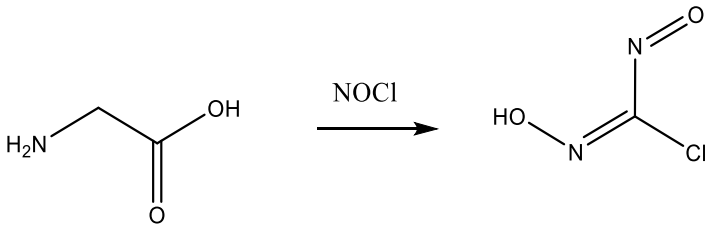
(1) Allowing the mixture to get too warm leads to a “gloopy oil” which is likely the Clemmensen reduction product – a cresol

Synthesis of 5-nitrovanillin



To a 250 mL beaker was added vanillin (25 g, 164 mmol) and dissolved in dichloromethane (110 mL), then chilled in an ice bath. Nitric acid (16 mL, 68%, 15.8 mol dm^{-3}, 316 mmol) was added dropwise to the reaction mixture, then allowed to stir in the ice bath for 30 minutes, then allowed to warm to room temperature over 20 minutes. Water (45 mL) was then added, and chilled in the refrigerator for 2 hours, and the precipitate filtered off and washed with cold water, then sucked dry. The crude yield was 36 g. Ethanol and acetone proved to be poor recrystallization solvents, but chloroform may be possible. A poor-quality melting point determination for the product recrystallized from ethanol – 162-170°C (lit. 175°C)

Synthesis of essence of Smurf



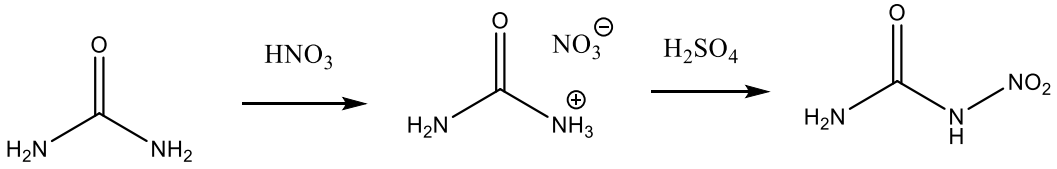
To a 600 mL beaker was added glycine (15 g, 200 mmol), and hydrochloric acid (100 mL, 36%, 11.6 mol dm^{-3}), and ice (60 g) and the reaction mixture stirred and cooled in an ice and salt bath. Maintaining the temperature below 7°C, sodium nitrite (22 g, 320 mmol) was slowly added over a period of 1 hour, then allowed to stir for 10 minutes in the ice bath, then allowed to warm to room temperature. The reaction mixture was then filtered, and the filtrate extracted with diethyl ether (17 mL). The solvent was removed to yield a blue-green liquid. The product is denser than water, and immediately dissolves. It reacts with dilute sodium hydroxide rapidly to yield an intense yellow-brown solution. It burns readily with no residue, and decomposes slowly at room temperature to nitrogen dioxide (in air).

Note: The blue substance is a chemical escape artist, and 'creeps' out of the container. Do NOT try to handle the container, even using gloves (latex, PVC) as it also goes straight through these. If a small amount is spilt on the skin, scrubbing with soap has no effect, and after 3 minutes the stain begins to itch and sting. After 5-6 minutes the skin turned white and peeled off. The vapour is also highly irritating.

Synthesis of tiramisu

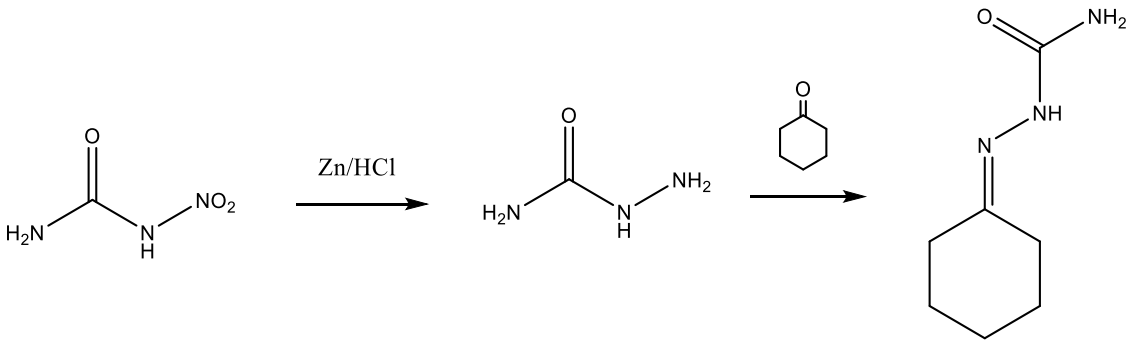
To freshly brewed strong coffee (50 mL) were added cognac (15 mL), whipping cream (50 mL), caster sugar (1 tbsp), and stirred well then chilled in the refrigerator. To a bowl was added egg whites (3), and whisked to a foam, then sugar (60 g) and cream of tartar (1 pinch) were added, and whisked until stiff peaks were formed. Sugar (60 g) was then added, and the mixture whisked once more to a solid foam. Egg yolks (3) were then added, and whisked to incorporate. Cake flour was then sieved into the meringue, and folded to mix. Milk (50 mL, ~4% fat) and butter (30 g) were then melted together, and added slowly to the mixture while carefully incorporating. The reaction mixture was then added to a buttered spring-form pan, and heated in the oven at 175°C for 45 minutes. The cooked reaction mixture was then turned out upside down onto a plate and allowed to cool to room temperature. Whipping cream (250 mL), mascarpone cheese (250 g), eggs (3, separated), caster sugar (50 g). To a bowl was added egg yolks (3) and sugar (50 g), and the mixture whisked for 10 minutes to a thick pale foam. Mascarpone cheese (250 g) was then added to the reaction mixture, and carefully stirred until homogeneous. To a chilled bowl was added whipping cream (250 mL), and whipped at a medium speed until a stiff foam is formed. The mascarpone-egg mixture was then gently incorporated into the whipped cream. Egg whites (3) were then whipped to medium-stiff peaks, and gently folded into the reaction mixture. The cake was then cut into thin slices, and layered in a glass dish; cake, followed by coffee mixture, followed by cream mixture, and repeated until the dish was full. Cocoa powder was then sieved across the final product until uniform.

Synthesis of nitrourea



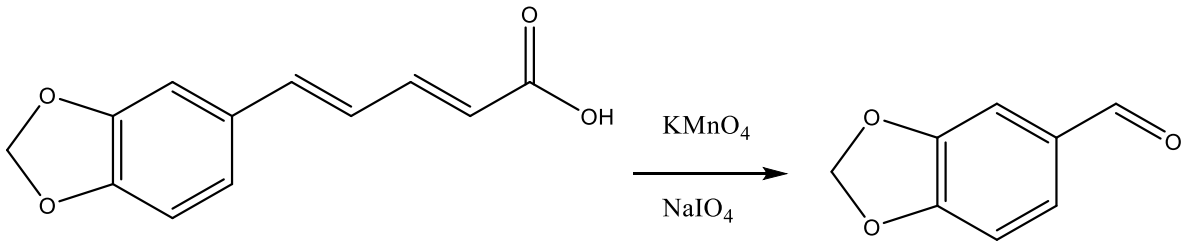
To a 200 mL beaker was added urea (25 g, 416 mmol), water (10 mL) and slowly nitric acid (32 mL, 68%, 15.8 mol dm^{-3}, 490 mmol), then stirred for 15 minutes, and chilled in the refrigerator for 1 hour. The precipitate was dried at the pump, to yield 55g of damp white powder. To a 500 mL beaker was added sulphuric acid (150 mL, 98%, 18.3 mol dm^{-3}), and chilled to -10°C, then placed into an ice-salt bath. To the chilled reaction mixture was slowly added the crude urea nitrate (55 g), maintaining the temperature below 0°C over a period of 40 minutes, then stirred for a further 30 minutes in the ice bath. Ice (200 g) was then slowly added, and the precipitate filtered off, and washed well with ice-water. The final yield was 31 g (71%) of fine white powder.

Synthesis of semicarbazide



To a 200 mL beaker was added nitrourea (10 g, 95.1 mmol) and hydrochloric acid (75 mL, 36%, 11.6 mol dm^{-3}), then chilled to below 0°C. A 500 mL beaker was added to an ice-salt bath, and zinc powder (25 g, 38.2 mmol) and crushed ice added to cover bottom of the beaker. The starting temperature was -5°C. The chilled hydrochloric acid solution was then added dropwise to the zinc maintaining the temperature below 0°C over a period of 2 hours. Sodium chloride (35 g) was then added to the chilled reaction mixture and stirred to dissolve as much as possible, then filtered. Cyclohexanone (9 g, 9.2 mmol) was then added to the filtrate, then chilled in the refrigerator for 24 hours, and the precipitate filtered off. 2.1 g of cyclohexanone semicarbazone zinc chloride adduct was isolated. Ammonia solution (10 mL, 20%) was then added to the solids, and stirred well, then the precipitate filtered off. The yield was 0.9 g (6%) of crude cyclohexanone semicarbazone as a white crystalline powder melting at 151-154°C (underestimated, lit. 165°C).

Synthesis of piperonal



To a 1000 mL beaker was added piperinic acid (3.7 g, 17.0 mmol), water (100 mL) and sodium hydroxide (0.69 g, 17.0 mmol), then stirred to dissolve for 10 minutes. Potassium periodate (3.9 g, 17 mmol) and potassium permanganate (5.4 g, 34 mmol) were dissolved in water (500 mL). A quench solution was prepared by dissolving sodium hydroxide (2.1 g, 51 mmol) and sodium bisulphite (5.3 g, 104.1 mmol) in water (75 mL). With vigorous stirring, the oxidising solution was added to the sodium piperinate solution, and allowed to react for about 2 minutes, when a brown precipitate became visible. At this point, the quench solution was added, and the precipitate filtered off, and washed with a little water, and dichloromethane (2x 30 mL). The lower organic layer was then separated, and the aqueous layer extracted with dichloromethane (15 mL). The combined organic layers were dried over magnesium sulphate, and the solvent removed. The crude yield was 0.5 g (20%) of yellow-brown crystals melting at 34-37°C (lit. 37°C).

Synthesis of dichloroacetic acid

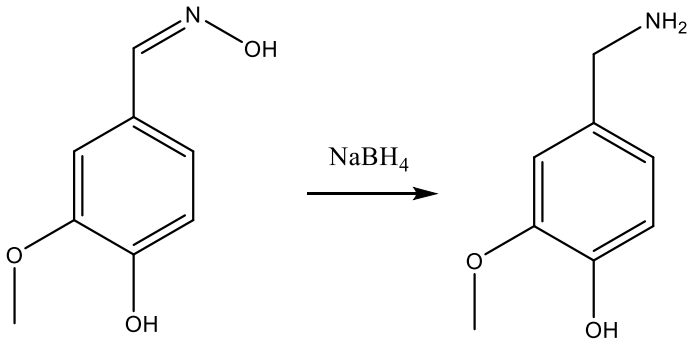
$Cl_3CCOOH + Zn \rightarrow Cl_2HCCOO^- + 2ZnCl^+$

To a 250 mL beaker was added water (50 mL) and trichloroacetic acid (25 g, 153 mmol), then stirred to dissolve, and the beaker cooled in an ice bath. Zinc powder (11g, 168 mmol) was then added slowly to the reaction mixture with stirring, keeping the temperature under control over a period of 20 minutes, then allowed to stir at room temperature for 20 minutes. Sulphuric acid (10 mL, 50%, 9 mol dm^{-3}) was then added to the chilled reaction mixture. Diethyl ether (40 mL) was then used to extract the solution, and dried with magnesium sulphate, and the solvent removed, along with any residual water. The crude yield was 13.5 g of very pale yellow oily liquid boiling at 170°C. The product appears to be a mixture of the three chloroacetic acids, due to inability to freeze, and slightly reduced activity with zinc powder.

Synthesis of seduction

A large salmon fillet was prepared by trimming the tail, sides, and skin, then deboning. The prepared fillet was then chilled in the refrigerator. To a small heavy-bottomed saucepan was added finely chopped peeled shallots (40 g), white wine (3 tbsp), white wine vinegar (2 tbsp), bay leaves (2), and fresh thyme (2, fresh sprigs), then heated gently with stirring until no liquid remains, then removed from the heat. Water (20 mL) was added, and the mixture warmed. Chilled butter (125 g, unsalted) was then cut into pieces and added with stirring, allowing each piece to melt before adding the next, maintaining the temperature just hot enough to melt the butter. After addition was complete, and the butter completely melted, the reaction mixture was strained to yield a beurre-blanc sauce. Sodium chloride and powdered peppercorns were added to improve flavour, then set in a warm place to stay liquid.
The salmon fillet was then sprinkled on both sides with flaky sea salt and olive oil, then seared on a smoking, oiled, heavy cast-iron skillet. Once crispy on the outside, the fillet was allowed to rest on a warm plate. Residual heat cooked the inside of the fillet. A handful of spinach was heated in a hot pan with a little butter for a few minutes, then the excess water and butter drained. The fish was then placed on the bed of spinach, and the juices from the salmon poured over the fillet. The warmed beurre-blanc sauce was then pooled around the fish and spinach, and served with a Première Cru from Northern Burgundy.

Synthesis of vanillyl amine



To a 200 mL beaker was added absolute ethanol (20 mL), vanillin oxime (2 g, 12.0 mmol), nickel chloride (2.3 g, 15.8 mmol), and stirred to dissolve. Sodium borohydride (1.8 g, 47.8 mmol) was then added slowly to the stirred reaction mixture over a period of 30 minutes, then stirred for 1 hour. Ethanol (20 mL) was then added, and stirred for a further 30 minutes. Hydrochloric acid (10 mL,

25%) was then added dropwise, and the precipitate filtered off, and the solids washed with ethanol (20 mL). A saturated solution of potassium hydroxide in ethanol (50 mL) was then made up, added to the reaction mixture, and the precipitate filtered off, and washed with ethanol. The solvent was then removed, and the solids dissolved in water (20 mL), and hydrochloric acid (~5 mL, 20%) used to neutralise the solution. - workup incomplete.

ChemPracs

Synthesis of sodium hypochlorite solution

$Ca(ClO)_2 + Na_2CO_3 \rightarrow 2\ NaClO + CaCO_3$

To a 250 mL round bottomed flask was added sodium carbonate (10 g), distilled water (48 mL) and chilled in an ice bath. Calcium hypochlorite (14 g) was then added to the reaction flask and allowed to stir for 20 minutes ensuring the temperature did not rise above 10°C. The precipitate was then filtered off, and the filtrate chilled in the freezer overnight, then refiltered. The final yield was 25 mL of concentrated solution.

ChemX

Synthesis of diethyl ether

$2\ EtOH \rightarrow Et_2O + H_2O$

To a 500 mL three necked round bottomed flask cooled in an ice bath was added absolute ethanol (90 mL) and allowed to cool to 5°C. Sulphuric acid (80 mL, 98%) was then added dropwise from an addition funnel over a period of 15 minutes. Sand (20 g) was then added, and the apparatus set up for fractional distillation. The reaction mixture was heated to 140-145°C, and ethanol (230 mL) added at the same rate as distillate was collected. The distillate was washed twice with saturated sodium bicarbonate (50 mL) and once with brine (50 mL) and dried over anhydrous calcium chloride for 1 hour. The ether was then distilled at 50°C to dryness without removing the calcium chloride. The final yield was ~250 mL (~85%). Sodium or potassium hydroxide can be used to minimise peroxide formation.

Synthesis of ethyl bromide

$EtOH + HBr \rightarrow EtBr + H_2O$

To a 500 mL round bottomed flask cooled in an ice bath was added ethanol (118 g, 2 mol), slowly hydrobromic acid (340 mL, 3 mol), and slowly sulphuric acid (100 g, 98%, 1 mol). The reaction mixture was then fractionally distilled, collecting the fraction at 38°C. The distillate was washed twice with cold sulphuric acid (20 mL, 98%), water (20 mL), saturated sodium bicarbonate solution (20 mL), water (20 mL), then dried over anhydrous calcium chloride. The final yield was 157.5 g (72.3%).

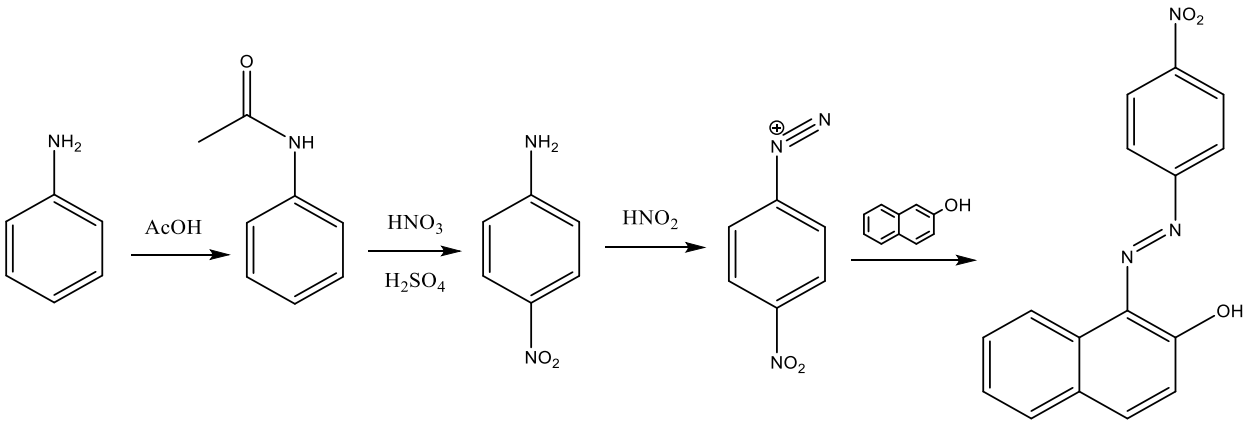
Synthesis of hydrobromic acid

$KBr + H_2SO_4 \rightarrow HBr + KHSO_4$

To a beaker was added potassium bromide (120 g, 1 mol), distilled water (200 mL) and stirred to dissolve, then chilled in an ice bath. Sulphuric acid (90 mL, 96%) was then added dropwise, and the precipitate filtered off. The filtrate was distilled, collecting the fraction at 123-128°C. The final yield was 120 mL of azeotropic hydrobromic acid (48% by weight). The product could be redistilled over red phosphorus to remove traces of bromine.

chemx01

Synthesis of para red



Synthesis of acetanilide

To a 250 mL round bottomed flask were added aniline (41 mL), and acetic acid (63 mL), then the reaction mixture was refluxed for 2.5 hours. After this time, the reaction mixture was poured into ice-water (500 mL), and the precipitate filtered off. The solids were recrystallized from water to yield 47.8 g (82%) of white crystals.

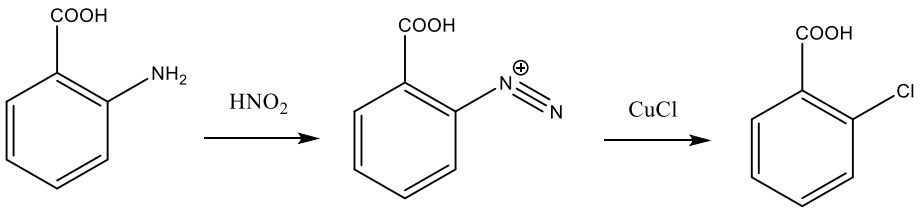
Synthesis of p-nitroaniline

To a 500 mL beaker were added acetanilide (100 g), and sulphuric acid (222 mL, 18.4 mol dm^{-3}), then the reaction mixture cooled in an ice bath. To the chilled solution was added dropwise a mixture of sulphuric acid (89 mL, 18.4 mol dm^{-3}) and nitric acid (89 mL, 70%, mol dm^{-3}), maintaining the temperature below 35°C with strong stirring. After the addition, the reaction mixture was stirred for 5 minutes at room temperature. The reaction mixture was then added to water (1040 mL) in a round bottomed flask, and brought to a reflux. The mixture was then allowed to cool to room temperature, and added to ice-water (1000 mL) and sodium hydroxide solution added (6 mol dm^{-3}) until the pH was 9-10. The precipitate was filtered off to yield 76 g (74.5%) of light brown powder.

Synthesis of para red

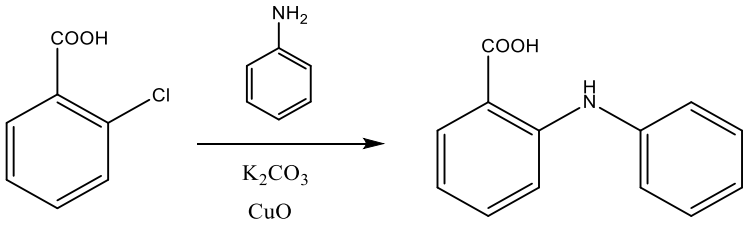
To a 1000 mL round bottomed flask was added p-nitroaniline (50 g) and sulphuric acid (550 mL, 1.75 mol dm^{-3}), and chilled in an ice bath for 30 minutes. A solution of 2-naphthol (50 g) in sodium hydroxide solution (500 mL, 2.5 mol dm^{-3}) was then prepared, and chilled to 10°C. A chilled (5°C) sodium nitrite solution (25 g in 100 mL water) was added to the p-nitroaniline solution dropwise, maintaining the temperature below 10°C. After complete addition had taken place, the 2-naphthol solution was rapidly added with strong stirring. The solution was then stirred in the ice bath for 10 minutes, and filtered. The final yield was 77.4 g (73%) of red powder.

Synthesis of o-chlorobenzoic acid



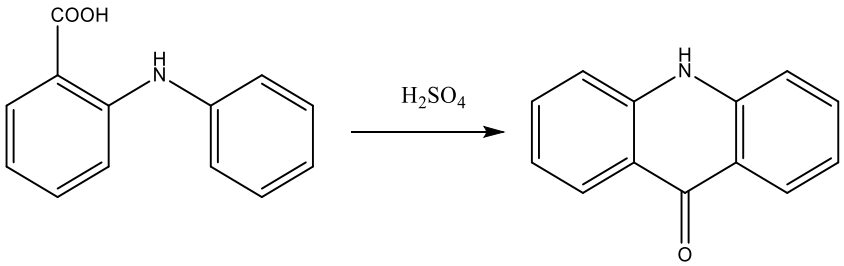
To a 250 mL beaker was added anthranilic acid (7 g, 51 mmol) and hydrochloric acid (112 mL, 1.5 mol dm^{-3}), and chilled to 5°C in an ice bath. To this solution, chilled (5°C) sodium nitrite solution (3.5 g/13 mL water, 50.7 mmol) was added dropwise maintaining the temperature below 10°C. This was added to a solution of cuprous chloride (5.3 g) in hydrochloric acid (22 mL, 31%), and allowed to stir at room temperature for 2.5 hours. The precipitate was then filtered off, and dissolved in boiling water (300 mL) and ethanol (20 mL), and a little activated charcoal added. The charcoal was filtered off, and the solution chilled to precipitate the product. The crystals were filtered off, yielding 5.3 g (66%) of white crystals melting at 138-140°C.

Synthesis of N-phenylanthranilic acid



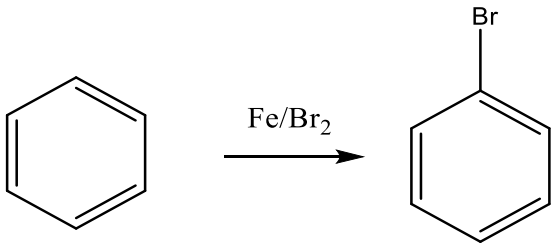
To a 250 mL round bottomed flask was added aniline (15.5 g, 166 mmol), o-chlorobenzoic acid (4.1 g, 26 mmol), potassium carbonate (4.1 g, 30 mmol), and cupric oxide (0.1 g). This was then refluxed for 2 hours, and steam distilled until 500 mL of distillate had been collected. To the reaction flask was added activated charcoal (2 g), and refluxed for 15 minutes. The carbon was filtered off, and the filtrate added to hydrochloric acid (9.6 mL, 18.6 %), then chilled to 5°C in an ice bath. The precipitate was filtered off to yield 4.2 g (76%) of white powder.

Synthesis of acridone



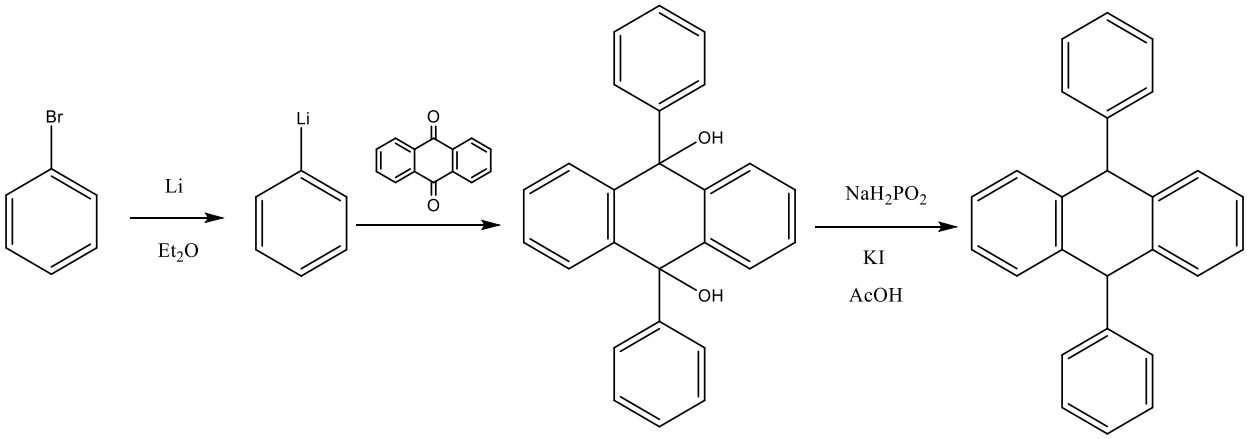
To a 25 mL Erlenmeyer flask was added N-phenylanthranilic acid (2.76 g, 13 mmol), and sulphuric acid (6.5 mL, 18 mol dm^{-3}), and heated to 100°C for 4 hours. The hot reaction mixture was then carefully poured into boiling water (65 mL), and boiled for 5 minutes. The precipitate was filtered off, and boiled in a solution of sodium carbonate (1.95 g, 18 mmol) in water (26 mL) for 5 minutes. The precipitate was filtered off, and dried under vacuum to yield 1.61 g (64%) of yellow powder.

Synthesis of bromobenzene



To a 500 mL three-necked round bottomed flask fitted with an addition funnel and a reflux condenser was added benzene (114 mL, 100 g, 1.28 mol), iron filings (4 g, mol%), and bromine (80 mL, 250 g, 1.56 mol) added to the addition funnel. To the outlet of the condenser is attached a PVC hose, and the end immersed in water (400 mL). The reaction flask was warmed to 30°C, and the bromine added dropwise with stirring such that reflux occurs. After complete addition, the reaction mixture was stirred at 30°C for 20 minutes, then heated to 70°C for 45 minutes. The reaction mixture was then washed with water (2x 100 mL), sodium hydroxide solution (100 mL, 10%) until the aqueous layer basic to phenolphthalein, then washed with water (100 mL). The organic layer was dried over calcium chloride, and distilled, collecting the fraction at 150-170°C, then the distillate redistilled, collecting the fraction at 154-157°C. The final yield was 100 g (50%) of clear colourless liquid.

Synthesis of 9,10-diphenylanthracene



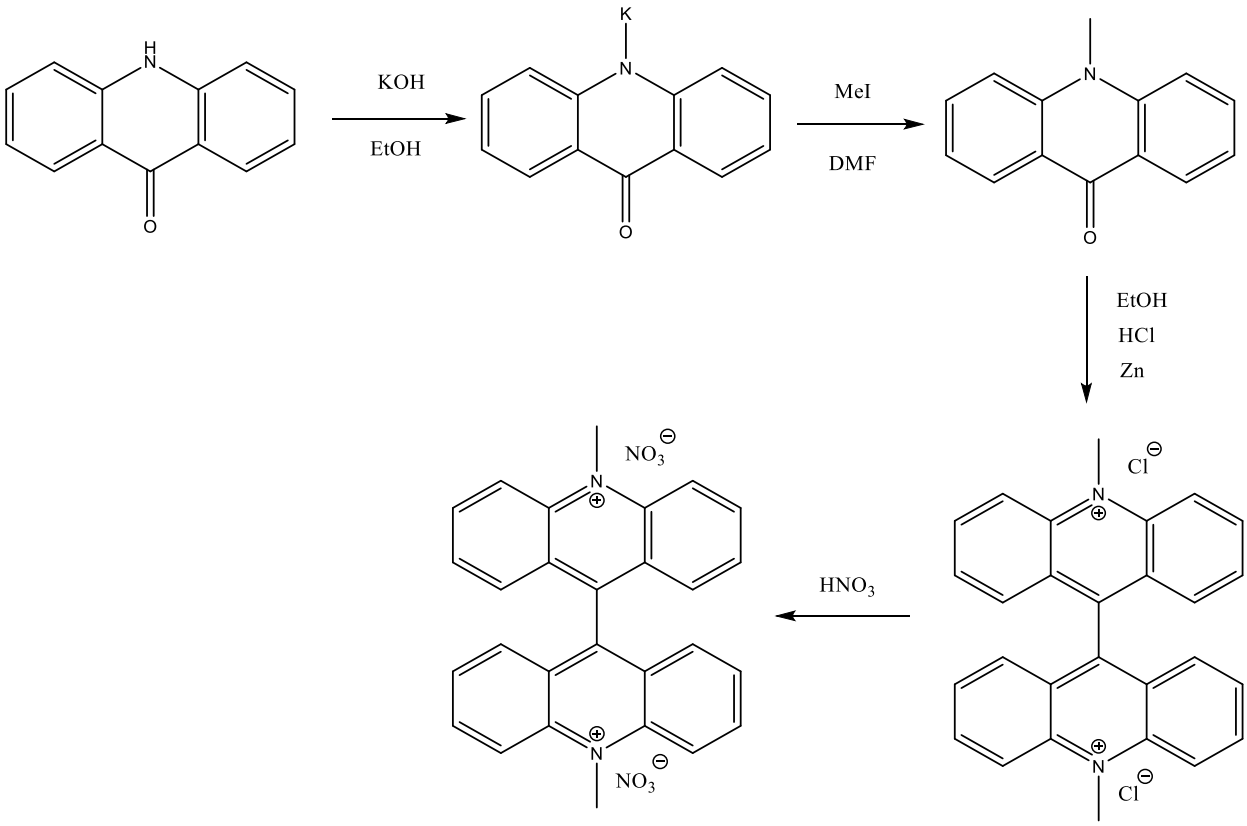
Synthesis of 9,10-diphenyl-9,10-dihydroanthracen-9,10-diol

To a 250 mL round bottomed flask fitted with a reflux condenser were added clean lithium flakes (0.8 g), sodium-dried diethyl ether (50 mL), and the vessel purged with nitrogen. To an addition funnel was added dry bromobenzene (4 mL, 6 g, 38 mmol) and dry diethyl ether (50 mL). The bromobenzene solution was slowly added to the reaction flask with stirring. The reaction mixture was then refluxed for 1 hour. Anthraquinone (5 g, 24 mmol) was then added to the cooled reaction mixture under nitrogen. The reaction mixture was then refluxed for 15 minutes, and then chilled in an ice bath. The reaction mixture was then added with stirring to water (100 mL) and stirred until no more lithium could be seen. The precipitate was filtered off, and a crude yield of the dihydroxy compound was 10 g.

Synthesis of 9,10-diphenylanthracene

Potassium iodide (15 g, 90 mmol), sodium hypophosphite monohydrate (15 g, 140 mmol), and glacial acetic acid (100 mL) were mixed with the 9,10-diphenyl-9,10-dihydroanthracen-9,10-diol, and refluxed for 15 minutes. The reaction mixture was then chilled in an ice bath, and the precipitate filtered off. The solids were recrystallized from toluene to yield 4.06 g (52%) of off-white powder melting at 243-245°C.

Synthesis of lucigenin



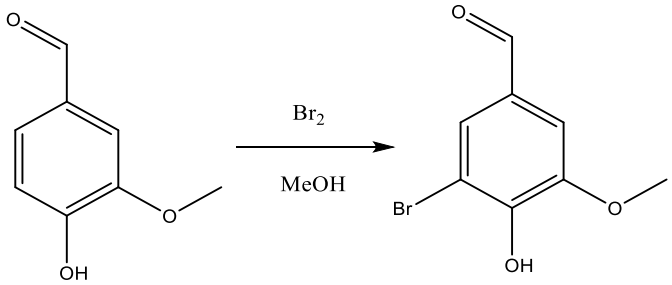
Synthesis of N-methylacridone

To a 25 mL round bottomed flask was added potassium hydroxide (0.3 g, 5 mmol), acridone (0.96 g, 5 mmol), and ethanol (12 mL), and was heated until all solids had dissolved, at which point the solvent was removed. The residue was dissolved in N,N-dimethylformamide (12 mL), and methyl iodide (0.36 mL, 0.82 g, 6 mmol) was added with stirring, then the reaction mixture was heated on a steam bath for 15 minutes. The reaction mixture was allowed to cool, and was added to water (100 mL). The precipitate was filtered off to yield 0.92 g (89%) of off-white powder (N-methylacridone).

Synthesis of lucigenin

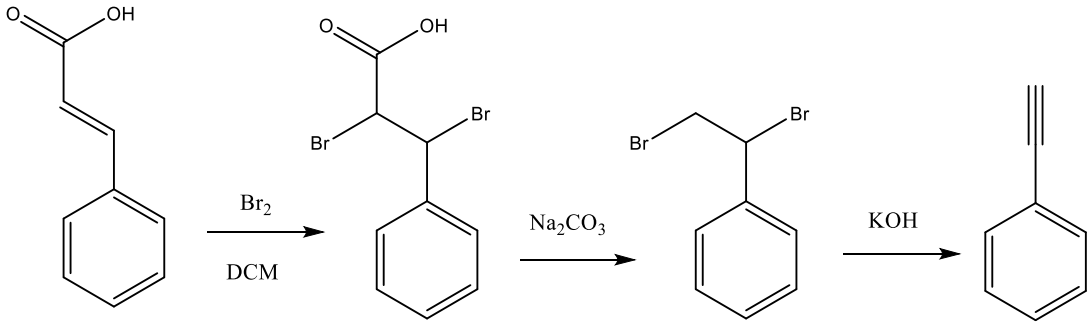
To a 250 mL round bottomed flask was added N-methylacridone (0.92 g, mmol), ethanol (46 mL), and hydrochloric acid (12 ml, 31%) and brought to reflux. Zinc powder (2.94 g, 45 mmol) was then slowly added with stirring, and the mixture allowed to reflux for 30 minutes. After cooling to room temperature, the reaction mixture was added to water (100 mL) and the precipitate filtered off. The solids were dissolved in nitric acid (55 mL, 1 mol dm^{-3}) and heated on a steam bath for 15 minutes. The liquid was hot filtered to remove any impurities, and the filtrate cooled in an ice bath. The precipitate was filtered off to yield 0.65 g (73%) of orange crystals.

Synthesis of 5-bromovanillin



To a 250 mL Erlenmeyer flask were added vanillin (8.19 g, 54 mmol), and methanol (54 mL) and stirred to dissolve. The mixture was then cooled in an ice bath to 0°C, and bromine (9.47 g, 59 mmol) was added dropwise maintaining the temperature below 10°C. After full addition, the reaction mixture was allowed to stir at room temperature for 1 hour. It was then recooled to 0°C, and cold water (30 mL) added slowly over a period of 30 minutes. The precipitate was filtered off, and washed with cold water (4x 30 mL) and ice-cold methanol/water solution (30 mL, 70%). The final yield was 11.86 g (96%) of yellowish powder.

Synthesis of phenylacetylene



Synthesis of dibromocinnamic acid

To a 1000 mL flask was added cinnamic acid (74 g, 0.5 mol), and dichloromethane (300 mL). The reaction mixture was heated to dissolve the cinnamic acid, then cooled in an ice bath. Bromine solution (76 mL, 10 mol dm^{-3} in dichloromethane) was then added rapidly in 3 portions, then the reaction mixture was allowed to stir for 30 minutes in the ice bath, after which the precipitate was filtered off.

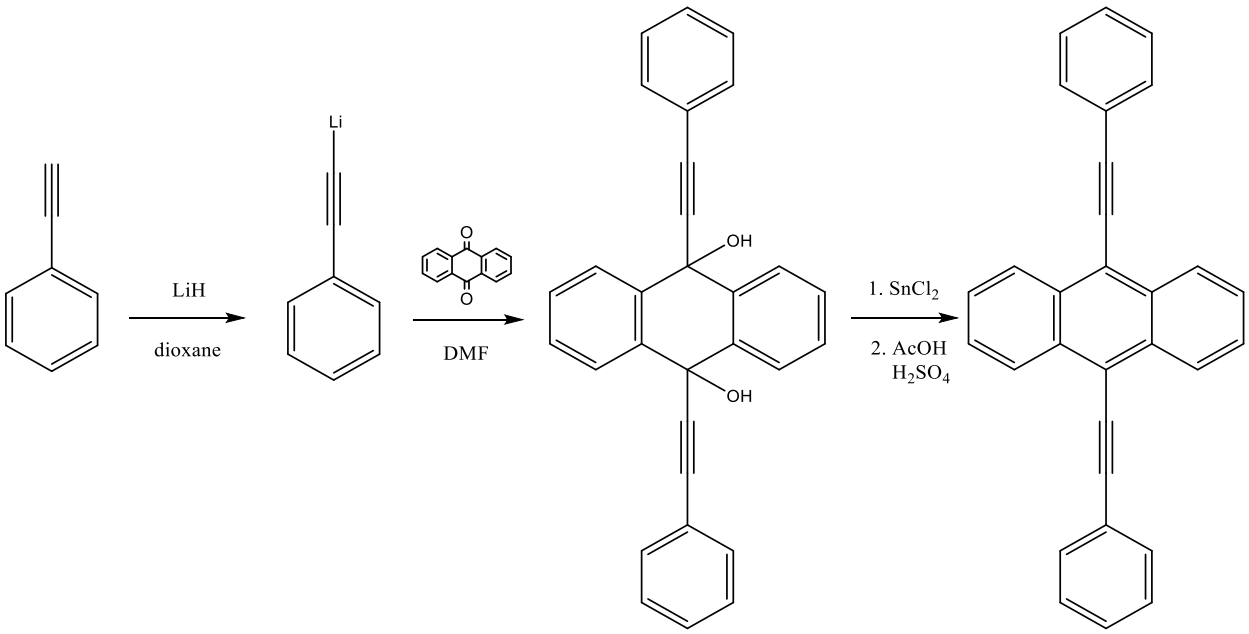
Synthesis of dibromostyrene

The dried precipitate was added to a 2000 mL round bottomed flask containing sodium carbonate solution (750 mL, 10%), and refluxed for 2 hours. The reaction mixture was allowed to cool to room temperature, and the lower organic layer separated off. The aqueous layer was extracted with diethyl ether (2x 75 mL). The combined organic layers were dried over calcium chloride, and the solvent removed. The yield of bromostyrene was 54.53 g (60%) of pale yellow liquid.

Synthesis of phenylacetylene

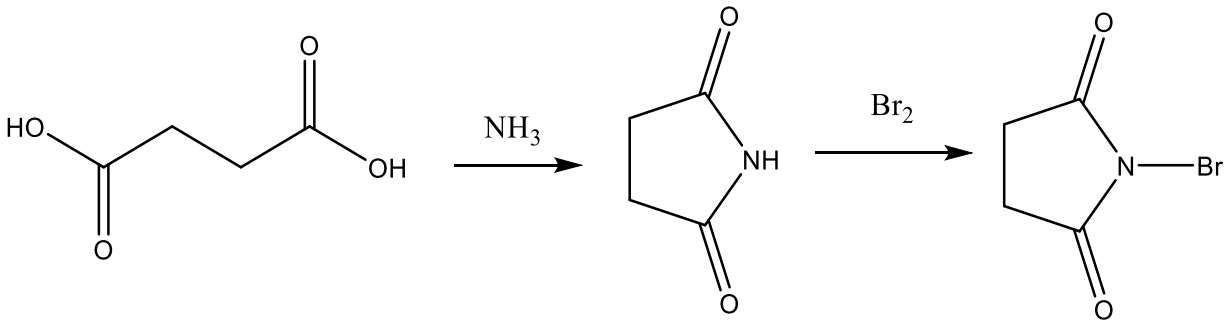
To a 250 mL round bottomed flask was added potassium hydroxide (100 g, 1.5 mol) moistened with water (2 mL). The apparatus was set up for distillation, and the bromostyrene added dropwise to the molten potassium hydroxide. The temperature was held at 220°C with an oil bath. After complete addition, the temperature was raised to 230°C to distil any remaining product. The lower aqueous layer was drained from the distillate, and the organic layer dried with potassium hydroxide. The organic layer was distilled, collecting the fraction at 142-144°C. The final yield was 15.53 g (30%) of clear colourless liquid.

Synthesis of 9,10-bis(phenylethynyl)anthracene



To a 500 mL round bottomed flask were added phenylacetylene (15.53 g, 152 mmol), anhydrous dioxane (100 mL), and lithium hydride (1.21 g, 152 mmol), with a calcium chloride drying tube to protect the contents of the flask from moisture. The reaction mixture was then refluxed for 2 hours, then cooled to 50°C and anthraquinone (14.4 g, 60 mmol), and N,N-dimethylformamide (100 mL) added. The reaction mixture is stirred for 4 hours at 50°C, then cooled to room temperature. A suspension of stannous chloride dihydrate (29.55 g, 116 mmol) in N,N-dimethylformamide (60 mL) added and the solution stirred for 15 hours at 25°C. The solution must be protected from light during this time. After this time, glacial acetic acid (8.4 mL) and sulphuric acid (59 mL, 8 mol dm^{-3}) added. The solution is cooled in an ice bath to precipitate crystals of product. The precipitate was filtered off and washed with methanol (130 mL). The final yield was 2.4 g (9%) of bright orange crystals.
δ_H (ppm) – 8.70, 1H, sex; 8.325, 0.59H, sex; 7.79, 1.62H, sex; 7.64, 1.26H, sex; 7.42, 2.12H, m

Synthesis of N-bromosuccinimide



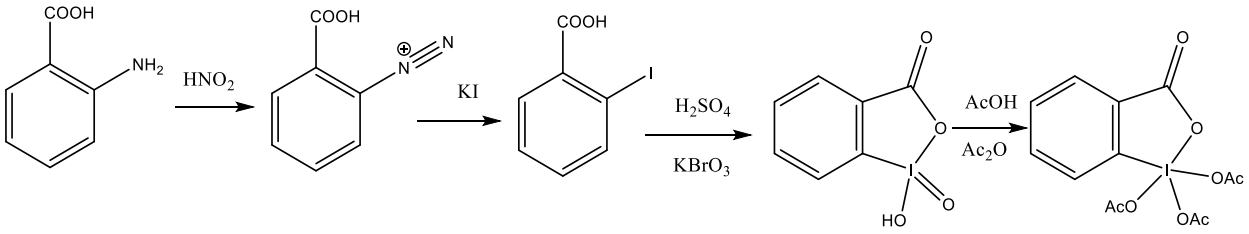
Synthesis of succinimide

To a 500 mL round bottomed flask was added succinic acid (93 g, 0.79 mol), then ammonia solution (110 mL, 26%, 1.8 mol) was added slowly and with cooling. The reaction mixture was then distilled to remove water, and distilled at 280°C until decomposition becomes evident – evolution of yellow fumes. The distillate was allowed to cool, then recrystallized from ethanol (1 mL / 1 g), and washed with cold ethanol (20 mL). The yield of succinimide was 47 g (60%) of white powder.

Synthesis of N-bromosuccinimide

To a 400 mL was added sodium hydroxide (19 g, 0.48 mol) dissolved in water (118 mL), and chilled to 10°C, then crushed ice (90 g) added, and the reaction mixture placed in an ice bath. Succinimide (47 g, 0.47 mol) was then dissolved in the solution, and bromine (25 mL, 78 g, 0.99 mol) added with vigorous stirring, and stirred for 5 minutes. The precipitate was filtered off, and washed with a small amount of ice-water. The final yield was 66 g (78%) of white crystalline powder.

Synthesis of Dess-Martin periodinane



Synthesis of 2-iodobenzoic acid

To a 250 mL beaker was added sodium nitrite (25 g, 0.36 mmol), and water (85 mL) and stirred to dissolve, then chilled in a refrigerator. To a 2000 mL beaker was added sulphuric acid (50 mL, 18.4 mol dm^{-3}), water (350 mL), and o-anthranilic acid (50 g, 0.36 mol). To the chilled solution (0°C) was dropwise added the sodium nitrite solution, maintaining the temperature below 7°C. Potassium iodide (92.9 g, 0.56 mol) was dissolved in sulphuric acid (180 mL, 2 mol dm^{-3}), and the solution slowly added to the diazonium salt solution. After complete addition, the reaction mixture was stirred at room temperature for 5 minutes, then heated to 80-90°C for 15 minutes. The reaction mixture was then allowed to cool to room temperature, then cooled further in an ice-bath. The precipitate was filtered off, and washed with a little cold water, sodium thiosulphate (2x ,0.1 mol dm^{-3}), then water again.

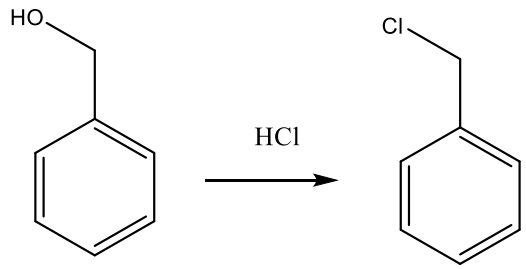
Synthesis of iodoxybenzoic acid

The dried solids were suspended in the minimum of water for stirring, and saturated sodium bicarbonate solution added until the pH is just basic. Activated charcoal was added, and the solution boiled for 5 minutes. The charcoal was filtered off, and sulphuric acid was added to acidify the solution, and the precipitate filtered off to yield 69.15 g (76%) of beige powder – o-iodobenzoic acid. In a 1000 mL three-neck round bottomed flask were added sulphuric acid (410 mL, 2 mol dm^{-3}), and potassium bromate (38.5 g, 0.23 mol), and the solution heated to 60°C and stirred to dissolve the solids. Then 2-iodobenzoic acid (38.5 g, 0.155 mol) was added over 40 minutes in 5 g portions. After complete addition had taken place, the solution was heated to 65°C for 2.5 hours. The reaction mixture was then cooled in an ice bath to precipitate as much solid as possible. While cooling, nitrogen was bubbled into the mixture to expel the bromine present. The precipitate was filtered off, and washed with cold water (2x 125 mL), methanol (2x 40 mL), and water (2x 125 mL). The yield of moist beige powder was 46.6 – iodoxybenzoic acid.

Synthesis of Dess-Martin periodinane

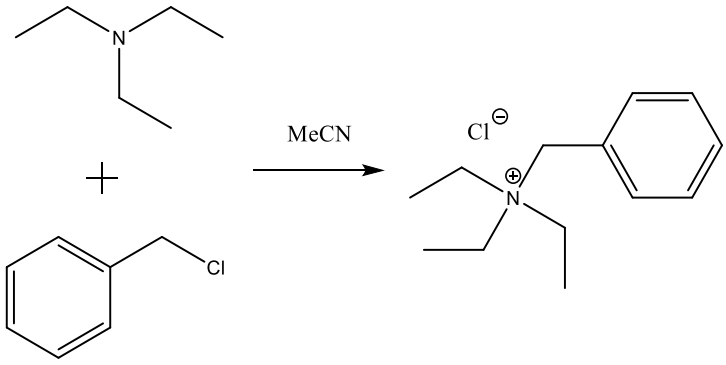
The moist powder was added to a 500 mL three-neck round bottom flask with acetic acid (72 mL) and acetic anhydride (145 mL) under nitrogen. The reaction mixture was slowly heated to 85°C over a period of 30 minutes. It was then allowed to cool to room temperature over a period of 24 hours without stirring, then cooled in ice. The precipitate was then filtered under nitrogen, and washed with dry diethyl ether (3x 40 mL). The final yield was 32.96 g (50%) of yellowish powder.

Synthesis of benzyl chloride



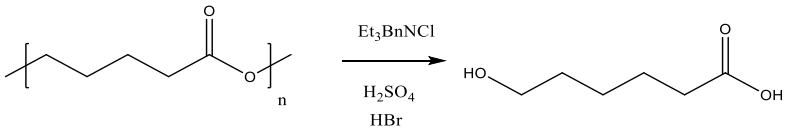
To a 2000 mL round bottomed flask fitted with a thermometer was added hydrochloric acid (1200 mL, 31%, 9.4 mol dm^{-3}, 11.6 mol), benzyl alcohol (156 mL, 1.5 mol), and the solution heated slowly to 70°C, then refluxed for 10 minutes. After cooling to room temperature, the upper organic layer was separated, and washed with water (2x 100 mL), then dried over potassium carbonate. The crude product was then distilled, collecting the fraction at 176-180°C. The final yield was 141.25 g (74%) of clear colourless liquid.

Synthesis of benzyltriethylammonium chloride



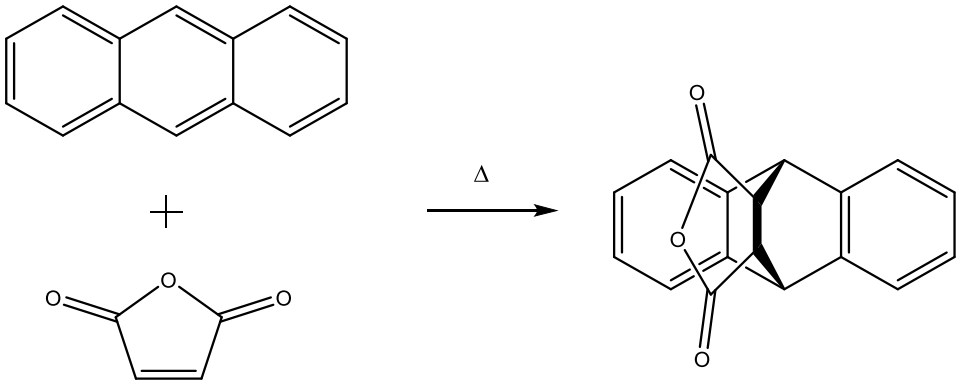
To a 100 mL round bottomed flask was added acetonitrile (10 mL), triethylamine (31 mL, 0.22 mol), and benzyl chloride (25 mL, 0.217 mol). The reaction mixture was then refluxed for 2 hours, then allowed to cool to room temperature, and diethyl ether (20 mL) added. The crystals were filtered off, and washed with diethyl ether, then dried under vacuum and stored in a tightly stoppered bottle. The yield was 32.46 g (66%) of fine white very hygroscopic powder.

Synthesis of 6-bromohexanoic acid



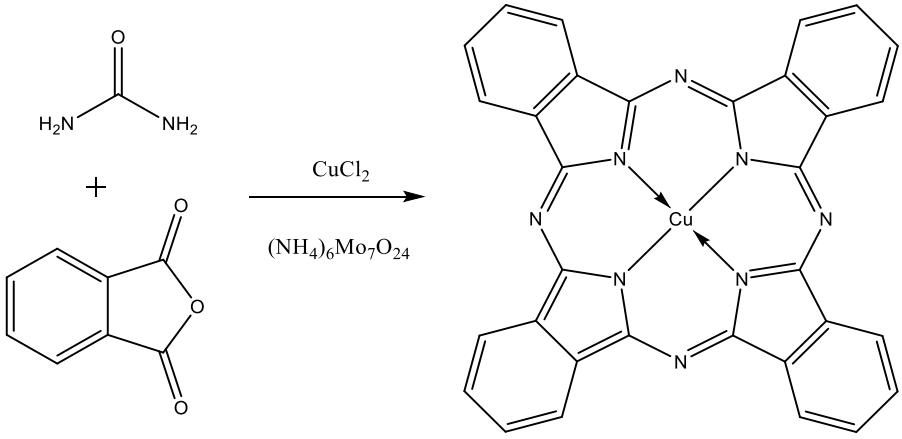
To a 500 mL flask was added sulphuric acid (7 mL, 0.13 mol, 18.4 mol dm^{-3}, 14 mol%), hydrobromic acid (234 ml, 1.84 mol, 48%), benzyltriethylammonium chloride (10.5 g, 46 mmol, 5 mol%), and polycaprolactone (105 g, 0.92 mol). The reaction mixture was then refluxed for 6 hours. After cooling to room temperature, the lower layer was separated and washed with water (100 mL), sodium sulphite solution (100 mL, 1 mol dm^{-3}), and water (100 mL). Emulsion formation was likely, to remedy this dichloromethane or diethyl ether was added (100 mL), or if that failed, filtration through Celite. If dichloromethane was added, the organic layer was dried over magnesium sulphate and vacuum distilled until the temperature exceeded 150°C / 10 mmHg. The final yield was 92.3 g (51%) of fuming white crystals.

Synthesis of 9,10-dihydroanthracene-9,10-α,β-succinic anhydride



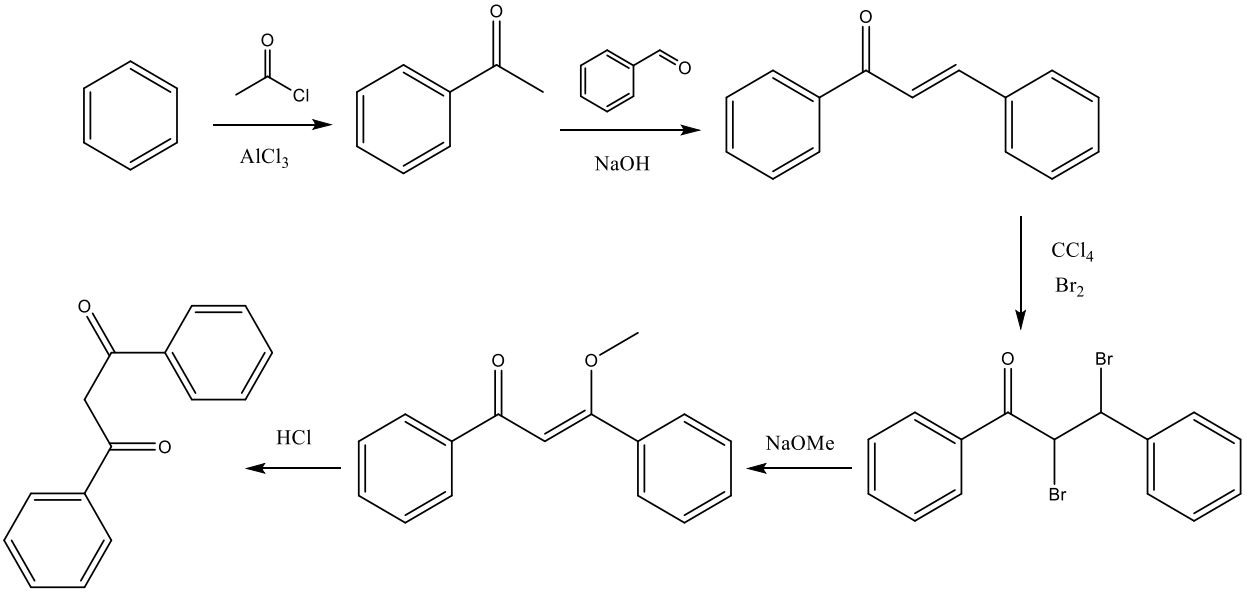
To a 250 mL round bottomed flask was added maleic anhydride (4.22 g, 43 mmol), xylene (100 mL), and anthracene (7.64 g, 43 mmol), then the reaction mixture was refluxed for 30 minutes. After cooling to 6°C, the crystals were filtered off, and recrystallized from xylene (ca 90 mL) to yield 7.97 g (67%) of yellow-white crystals.

Synthesis of copper phthalocyanine



To a 250 round bottomed flask were added phthalic anhydride (20 g, 0.14 mol) and urea (27 g, 0.45 mol) that had been intimately ground together. Cupric chloride dihydrate (5.8 g, mmol) and ammonium molybdate (0.1 g, 0.5 mmol) were then also ground together and added to the flask. Kerosene (60 mL) was then added, and the mixture heated to 200°C for 2 hours. After cooling to room temperature, the product was washed with warm petroleum ether (100 mL) and filtered on a Buchner funnel. This was repeated twice. In the same manner, the product was washed with boiling water (100 mL) and warm acetone (100 mL). The final yield was 14.31 g (73%) of deep purple powder.

Synthesis of dibenzoylmethane



Synthesis of acetophenone

To a 500 mL round bottomed flask was added anhydrous aluminium chloride (80 g, 0.6 mol), and anhydrous benzene (200 mL, 176 g, 2.25 mol) and set up to reflux with a dropping funnel containing acetyl chloride (58 mL, 64 g, 0.82 mol). The acetyl chloride was slowly added over a period of 50 minutes with cooling in a cold water bath. After addition was complete, the mixture was heated on a water bath to 60°C for 1 hour. The mixture was then cooled to room temperature, and poured into a mixture of water (250 mL) and ice (500 g). The upper organic layer was separated and washed with sodium hydroxide solution (2x 100 mL, 5%) and water (2x 100 mL), then dried over magnesium sulphate. The solvent was removed, and the crude acetophenone distilled at 190-200°C. The distillate was then redistilled, and the fraction boiling at 198-202°C collected. The final yield was 20.08 g (20%) of clear colourless liquid.

Synthesis of chalcone

To a 500 mL beaker was added sodium hydroxide solution (50 mL, 8.2 mol dm^{-3}), ethanol (32 mL), and acetophenone (13.56 g), 0.11 mol) and chilled to 5°C. Then benzaldehyde (11.5 ml, 0.11 mol) was rapidly added, and the use of ice around the beaker was used to maintain the temperature at 25°C. The mixture was stirred at this temperature for 3 hours, then chilled in the freezer for 15 hours. The product was filtered off, and washed with ice-cold ethanol (5 mL). The yield of benzalacetophenone (chalcone) was 22.32 g (97%) of a yellowish powder.

Synthesis of dibromochalcone

To a 250 mL was added chalcone (22.32 g, 0.107 mol) and carbon tetrachloride (65 mL). The solution was cooled to 5°C in an ice bath, and bromine (5.6 ml, 0.109 mol) added slowly maintaining the temperature below 20°C. The mixture was then stirred for 15 minutes at room temperature, before being cooled in the ice bath to precipitate as much product as possible. The precipitate was filtered off, and washed with hot ethanol (2x 28 mL). The yield of dibromochalcone was 30.6 g (78%) of nearly white fine powder.

Synthesis of dibenzoylmethane

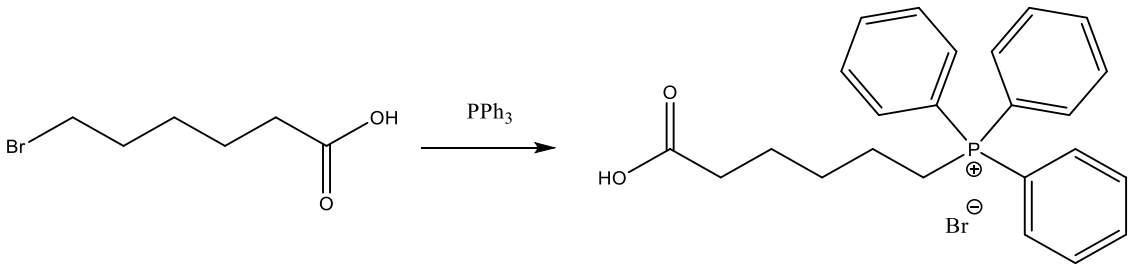
To a 250 mL three-neck round bottomed flask fitted with a reflux condenser, nitrogen inlet and a dropping funnel was added sodium metal (3.9 g, 0.17 mol) and dry methanol (38 mL). The flask was kept cool with ice, and heat used to complete dissolution. To a 250 mL three-neck round bottomed flask fitted with a reflux condenser, nitrogen inlet and a dropping funnel was added chalcone dibromide (30.6 g, 83 mmol) and dry methanol (27 mL). The sodium methoxide solution was added to the dropping funnel, and added rapidly to the reaction flask after flushing the apparatus with nitrogen. The reaction mixture was then refluxed for 1 hour, after which hydrochloric acid (2.1 mL, 31 %) was added, and boiled for 5 minutes. Then water (26 mL) was added, and the reaction mixture cooled in an ice bath. The precipitate was filtered off, and washed with methanol (8 mL, 50% in water), then cold water (40 mL). The crude product was recrystallized from methanol (24 mL) at 55°C to yield 9.52 g (51%) of pure product and 0.59 g (3%) of less pure product.

Synthesis of isopropyl nitrite

$^{i}PrOH + HNO_2 \rightarrow {}^{i}PrONO + H_2O$

Isopropanol (60 mL, 49 g, mol), hydrochloric acid (95 mL, 31%) were cooled to below 0°C in the freezer. A solution of sodium nitrite (45 g, 0.65 mol) was then dissolved in water (80 mL) and also chilled in an ice-salt bath. The mixed isopropanol and hydrochloric acid was then added slowly to the sodium nitrite solution with vigorous stirring over a period of 20 minutes. After complete addition, the reaction mixture was stirred for a further 5 minutes in the ice bath. The upper yellow layer was then separated off, and washed with cold saturated sodium bicarbonate solution (2x 100 mL), then dried over anhydrous potassium carbonate and stored in the freezer. The final yield was 46.5 g (80%) of yellow liquid.

Synthesis of (6-carboxyhexyl)triphenylphosphonium bromide



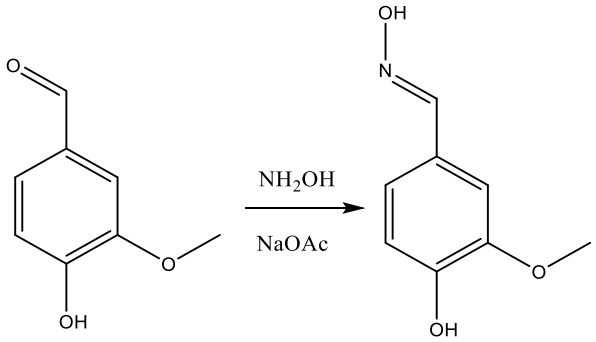
To a 200 mL flask was added triphenylphosphine (20.97 g, 79.9 mmol), and 6-bromohexanoic acid (15.6 g, 79.9 mmol), then the reaction mixture was heated to 145°C for 4 hours. The cooled glassy reaction mixture was dissolved in the minimum of hot chloroform, and ethyl acetate was added to precipitate the product, then chilled in the freezer. The crystals were filtered off, and washed with cold ethyl acetate. The final yield was 23.8 g (65%) of white powder.

Synthesis of potassium azide

$N_2H_4 \bullet H2O + {}^{i}PrONO + KOH \rightarrow KN_3 + {}^{i}PrOH + 3H_2O$

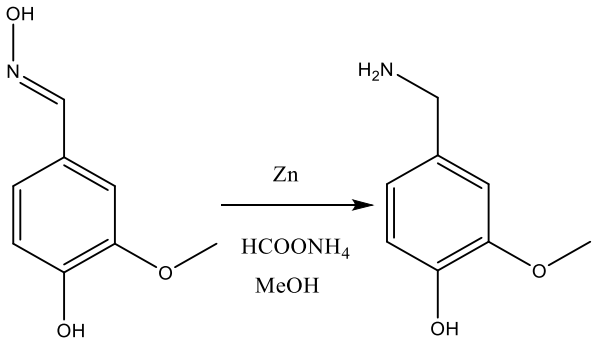
To a 500 mL round bottomed flask fitted with an addition funnel and an efficient reflux condenser was added absolute ethanol (230 mL), hydrazine hydrate (18.5 mL, 19 g, 0.38 mol), potassium hydroxide (26.6 g, 0.4 mol), and the solids dissolved with stirring. Isopropyl nitrite (39 g, 0.43 mol) was then added dropwise with strong stirring over a period of 45 minutes. The reaction mixture was then refluxed for 15 minutes, then stirred in an ice bath for 3 hours, and the precipitate filtered off and washed with cold absolute ethanol and diethyl ether. The final yield was 19.1 g (63%) of white crystalline powder.

Synthesis of vanillyl oxime



To a 100 mL flask were added vanillin (3.8 g, 25 mmol), hydroxylamine hydrochloride (1.95 g, 27.4 mmol), sodium acetate (4.1 g, 50 mmol), and water (35 mL). The reaction mixture was refluxed for 15 minutes, then cooled in an ice bath, and the crystals filtered off. The solids were washed with cold water (3x). The final yield after drying was 3.27 g (78%) of white powder.

Synthesis of vanillyl amine



To a 100 mL round bottomed flask were added vanillin oxime (3.27 g, 19.6 mmol), methanol (40 mL), ammonium formate (3.7 g, 58.6 mmol), and zinc powder (2.6 g, 39.8 mmol). The reaction mixture was then stirred under reflux for 15 minutes. After reflux, the solution was filtered to removed excess zinc, and the residue washed with methanol (10 mL). The solvent was then removed, and the solids washed with water (50 mL). The suspension was filtered, and the product washed with cold ethanol. The final yield was 1.06 g (35 %) of white powder, melting at °C (lit. 179-181°C).[4] Low yield due to use of warm ethanol in washing step.

College Chemistry

Synthesis of copper dichloroisocyanurate

To a beaker was added copper sulphate pentahydrate (10g), hot water (250 mL), and to a separate beaker sodium dichloroisocyanurate (10g) and dissolved in cold water. The solutions were combined, and allowed to stir for 10 minutes. The precipitate was then filtered off, washed well with water and allowed to dry. The formula for the product is not known.

US Patent US3055889

Synthesis of Chevreul's salt

$3CuSO_4 + 8NaHSO_3 \rightarrow Cu_3(SO_3)_2 + 4Na_2SO_4 + 5SO_2 + 4H_2O$

Copper sulphate pentahydrate (7.25 g) was dissolved in water (75 mL) and added to sodium metabisulphite (6.14 g) dissolved in water (32 mL) and boiled for 5 minutes. The precipitate was then filtered off and washed with water (10 mL).

Synthesis of Prussian blue

$3\ K_4Fe(CN)_6 + 4\ FeCl_3 \rightarrow Fe_7(CN)_{18} + 12\ KCl$

A solution of potassium ferricyanide (11.1 g) in water (50 mL) and combined with a saturated solution of ferrous sulphate heptahydrate (200 mL). The precipitate was filtered off, washed well with water and allowed to dry. The final yield was 5.5 g.

Synthesis of tetramminecopper(II) sulphate

$[Cu(H_2O)_6]SO_4 + 2\ NH_4OH \rightarrow [Cu(OH)_2(H_2O)_4] + (NH_4)_2SO_4$

$[Cu(OH)_2(H_2O)_4] + 4\ NH_3 \rightarrow [Cu(NH_3)_4(H_2O)_2](OH)_2 + H_2O$

To a solution of copper sulphate was added ammonia solution until the precipitate so formed redissolved.

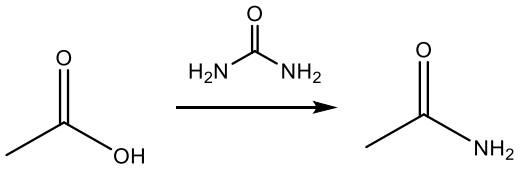
Doug's lab

Synthesis of isopropyl nitrite

$^{i}PrOH + HNO_2 \rightarrow {}^{i}PrONO$

To a 400 mL beaker was added, isopropanol (86 g, 70%), and sodium nitrite (79 g) then the solution was chilled to 0°C, and chilled hydrochloric acid (110 g, 31%) added dropwise. After reaction mixture was then stirred for a further 5 minutes, and water (40 mL) added. The upper organic layer separated off, and dried with brine (100 mL). The product was then stored in an amber glass bottle.

Synthesis of acetamide



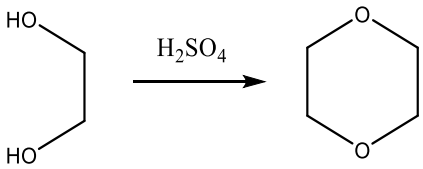
To a 500 mL two-neck round bottomed flask was added a thermometer, urea (100 g), and glacial acetic acid (100 g), then refluxed with an air condenser for 4 hours. After cooling to room temperature, the reaction mixture was short-path distilled, collecting the fraction at 219-222°C. The final yield was 104 g (75% of Vogel's) of white crystals.

Synthesis of sulphur monochloride

$S_8 + 4Cl_2 \rightarrow 4S_2Cl_2$

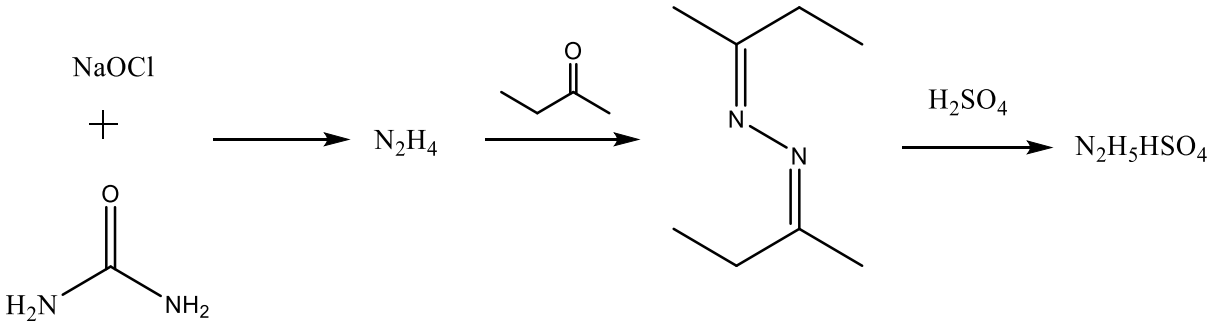
To a 500 mL round bottomed flask was added sulphur (96 g, 3 mol) and dry chlorine passed in to the molten sulphur until all the sulphur had been consumed and the product distilled over as the reaction progressed; approximately 3 hours. Sulphur (15 g) was then added to the distillate, and the reaction mixture refluxed until the liquid turned yellow, approximately 20 minutes. The reaction mixture was then distilled, and 194 g (%) of orange distillate was collected.

Synthesis of 1,4-dioxane



To a 500 mL round bottomed flask was added ethylene glycol (~200 mL, g) and sulphuric acid (25 mL, 93%). The reaction mixture was then distilled, and product collected until distillate stopped coming over. The dioxane was salted out with potassium carbonate, and the upper organic layer separated. This was dried with potassium carbonate and potassium hydroxide for several days. The upper organic layer was separated, and distilled, collecting the fraction at 100-103°C. The final yield was g (%) of clear colourless liquid.

Synthesis of hydrazine sulphate



To a 1000 mL beaker was added water (189 mL) and urea (126 g), and stirred and heated to dissolve. To a 150 mL beaker was added water (8 mL) and sodium hydroxide (8 g), and stirred to dissolve. To a 4000 mL Erlenmeyer flask was added sodium hypochlorite solution (1356 mL, 1562 g, 10%), and all solutions were chilled to -10°C. The sodium hydroxide solution was added to 2000 mL beaker along with the urea solution. The final addition was from the beaker to the flask. The freezing hypochlorite solution was added in 100 mL portions to the urea solution, maintaining the temperature below -4°C. To a 100 mL beaker was added sodium hydroxide (150 mL) and water (150 mL), then stirred to dissolve. Gelatine (1g) was also dissolved in the minimum of water. The gelatine solution was added to the reaction mixture in the 4000 mL flask, and the sodium hydroxide solution was also added rapidly. The reaction mixture was then allowed to cool to room temperature, and methyl ethyl ketone (400 mL) added then stirred at room temperature for 2 hours, and the upper organic layer separated. The organic layer was added to water (200 g) and sulphuric acid (200 g, 93%), then distilled to separate the methyl ethyl ketone from the hydrolysis equilibrium. When water began distilling over (seen by the higher density of the distillate), the reaction mixture was allowed to cool to room temperature, then chilled in the freezer, and the crystals filtered off, and washed with a small amount of ice-cold water. The solids were dried over a water bath, and to yield 73.6 g (24.9%) of fine white crystals.

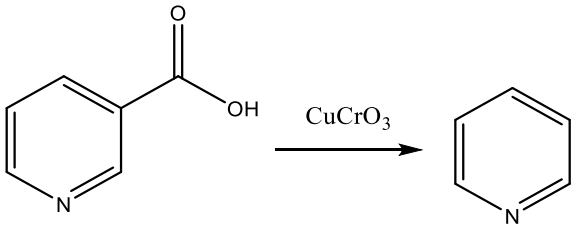
Synthesis of copper chromite

$CuSO_4 + (NH_4)_2CrO_4 \longrightarrow CuCrO_4$

$CuCrO_4 \longrightarrow CuCrO_3$ etc.

To a 600 mL beaker was added ammonium dichromate (12.6 g, 0.1 mol) dissolved in water (33mL) and ammonia solution (42 mL, 10% in water). Copper sulphate pentahydrate (25 g, 0.1 mol) dissolved in water (60 mL) was then added and allowed to stir for 5 minutes. The precipitate was then filtered off, washed with water and dried on a water bath. The dry powder was then transferred to a crucible, and heated to 350-400°C for 10 minutes with a propane flame to thermolyze it to copper chromite. The black powder was then washed with acetic acid (2x 150 mL, 10%). The final yield was 12.4 g of coarse grey-green powder.

Synthesis of pyridine



To niacin (300 g) was added copper chromite catalyst (4.6 g) and stirred thoroughly. The mixture was then added to a 1000 mL round bottomed flask, and distilled at 100°C until no more distillate was coming over. The distillate was then redistilled, and the final yield was 167.1 g (86.6%) of clear colourless liquid with the characteristic odour of pyridine.

Synthesis of pyridinium chlorochromate

$$py + CrO_3 + HCl \longrightarrow pyCrO_3Cl$$

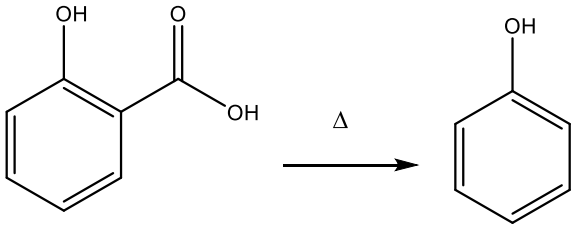
To a 400 mL beaker was added hydrochloric acid (180 mL, 6 mol dm^{-3}) and chromium trioxide (50 g), and stirred to dissolve, then cooled to 0°C. Over a period of 10 minutes, pyridine (40 mL) was added carefully with rapid stirring. The precipitate was then filtered off, and dried on a hot water bath. The final yield was 99.2 g (%) of bright orange crystals.

Synthesis of hexachloroethane

$$C_2Cl_4 + Cl_2 \longrightarrow C_2Cl_6$$

To a two-neck 250 mL round bottomed flask was added tetrachloroethylene (83 g) and ferric chloride (~1 g). Dry chlorine (0.5 mol) was passed through the refluxing reaction mixture, and after complete addition (3.5 hours), the reaction mixture was allowed to cool to room temperature. The solvent was then removed, and the product sublimed onto a cold finger to purify/separate.

Synthesis of phenol



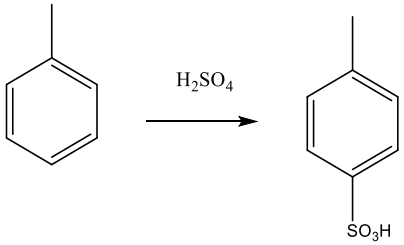
To a 500 mL round bottom flask was added salicylic acid (200 g) then distilled at 165°C with a short path distillation apparatus until no more distillate came over. The phenol was then redistilled, collecting the product from 99°C (water-phenol 9:91 azeotrope) as a first fraction, and dry phenol at 180-190°C. The final yield was 53 g of white crystalline product with a phenolic odour.

Synthesis of silver tetraiodomerurate(II)

2 AgNO3 + Hg(NO3)2 + 4 KI = Ag2HgI4 + 4KNO3

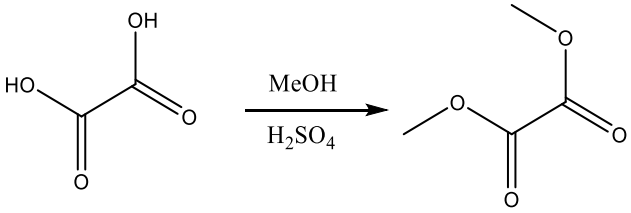
To a beaker were added silver turnings (0.634 g), mercury (0.59 g), and nitric acid (10 mL, 68%). During this reaction, potassium iodide (0.59 g) was dissolved in hot water (100 mL). The two solutions were then combined, and the precipitate filtered off.

Synthesis of p-toluenesulphonic acid



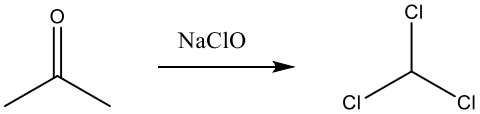
To a 500 mL round bottomed flask set up for reflux with a Dean-Stark trap was added toluene (130 mL), sulphuric acid (13 mL) and refluxed for 4 hours when water had stopped collecting in the Dean-Stark trap. The reaction mixture was then allowed too cool after the addition of water (9 mL). The precipitate was then filtered off and dried over a water bath.

Synthesis of dimethyl oxalate



To a 1000 mL beaker was added oxalic acid dihydrate (126 g), methanol (90 mL), sulphuric acid (40 mL, 98%), and heated gently and stirred for 10 minutes. The reaction mixture was then chilled to 0°C, and the precipitate filtered off. The crystals were then dried over a steam bath, then dried in a vacuum desiccator.

Synthesis of chloroform



To bleach (13.5 L) cooled to -16°C was slowly added acetone (390 mL) in two portions, allowed to sit for 20 minutes then cooling to -10°C between each addition. The reaction mixture was then allowed separate, and the chloroform recovered. The product was distilled, collecting the fraction at 58-63°C. The distillate was dried over calcium chloride and stored with 1% of ethanol in an amber glass bottle. The final yield was 300 mL (66%).

Synthesis of chromyl chloride

$K_2Cr_2O_7 + 2H_2SO_4 \rightarrow 2KHSO_4 + 2CrO_3 + H_2O$

$NaCl + H_2SO_4 \rightarrow NaHSO_4 + HCl$

$CrO_3 + 2HCl \rightarrow CrO_2Cl_2 + H_2O$

To a 500 mL round bottomed flask was added intimately ground dry potassium dichromate (55.8 g, 0.2 mol) and dry sodium chloride (46.7 g, 0.8 mol), cold sulphuric acid (143 g, 93%, 0.4 mol) and set up for distillation, then allowed to sit for 10 minutes. The reaction mixture was then distilled at 120°C. When the vapours turned yellow, the reaction was considered complete. The glassware was cleaned with sodium sulphite. The final yield was 44 g (23 mL).

The preparation was a modification of [A laboratory Manual of Organic Chemistry: a compendium of laboratory methods for the use of chemists, physicians, and pharmacists] by Dr. Lassar-Cohn, Alexander Smith. Macmillian and co. ltd., 1895 Page 254.

Preparation of bromine

$KBr + H_2SO_4 \rightarrow KHSO_4 + HBr$

$2\ HBr + H_2O_2 \rightarrow H_2O + Br_2$

To a 500 mL round bottomed flask was added water (100 mL) and sulphuric acid (74 mL, 94%) then allowed to cool, and hydrogen peroxide (140 mL, 29%) to an addition funnel. Potassium bromide (150 g), The hydrogen peroxide was added dropwise to the reaction mixture, and the product distilled off into an ice-cooled collection flask. The distillate was washed with ice-cold sulphuric acid (50 mL, 94%), and stored in a lab freezer.

Synthesis of nitric acid

$KNO_3 + H_2SO_4 \rightarrow HNO_3 + KHSO_4$

To a 1000 mL round bottomed flask was added water (200 mL), potassium nitrate (404 g), and sulphuric acid (292 mL, 93%). The reaction mixture was then distilled until the reaction mixture began to foam up. The distillate was then redistilled, collecting the fraction at 120°C. The final yield was ~200 mL of azeotropic nitric acid. The forerun of the distillation summed 300 mL of ~25% nitric acid.

Synthesis of chromium trioxide

$Na_2Cr_2O_7 + 2\ H_2SO_4 \rightarrow 2\ CrO_3 + 2\ NaHSO_4 + H_2O$

To a 1000 mL beaker was added sodium dichromate dihydrate (100 g) and dissolved in water (250 mL). To the solution was added sulphuric acid (400 mL, 93%), then allowed to cool to 0°C. The precipitate was then filtered off, and washed with nitric acid. The final yield of crude chromium trioxide was 71 g.

EH Productions Videos

Synthesis of tetramminecopper(II) sulphate

$CuSO_4 + 4\ NH_3 \rightarrow [Cu(NH_3)_4]SO_4$

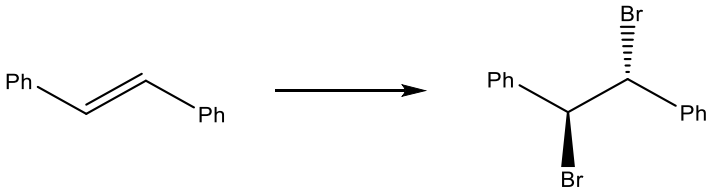
Ammonia was passed into a test tube charged with a small portion of anhydrous copper sulphate until no further colour change was seen.

eku_chem_lab

Extraction of β-carotene from spinach

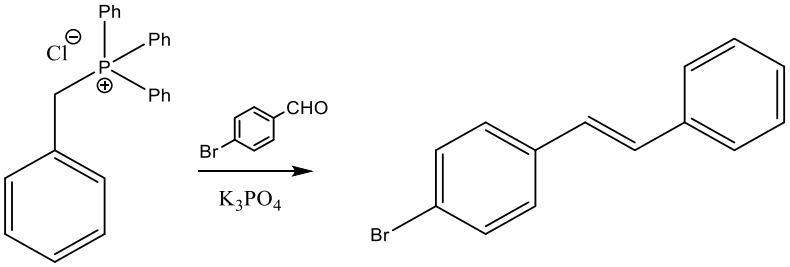
Spinach (0.5 g) were ground with anhydrous magnesium sulphate (0.5 g) and sand (1.0 g) and the mixture extracted with acetone (3 mL). A silica gel microcolumn was set up in a pipette and hexanes (10 mL) used to flush the column. The acetone solution was removed from the extract and evaporated to dryness. The extract was then redissolved in hexanes (1 mL), and added to the column. Hexanes (5 mL) were then used to elute the β-carotene to the middle of the column, at which point the eluent was switched to 9:1 hexanes:ethyl acetate. The yellow band was separated, and the fraction analysed by TLC (solvent: 7:3 hexane:acetone).

Synthesis of meso-stilbene dibromide



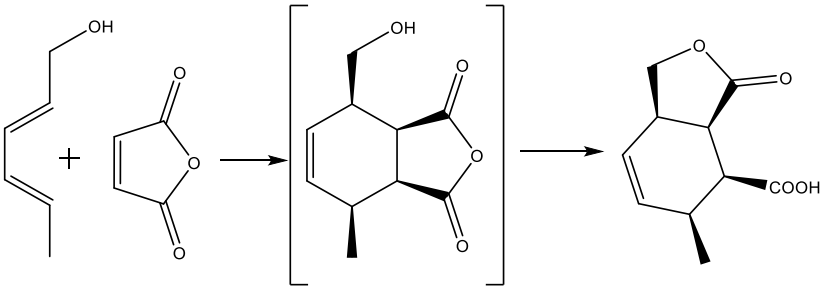
To a 25 mL Erlenmeyer flask was added absolute ethanol (10 mL), trans-stilbene (250 mg) and stirred to dissolve. Pyridinium tribromide (500 mg) was then added and stirred and warmed for 10 minutes. The reaction mixture was then cooled to 0°C, and the precipitate filtered off and washed with ice-cold methanol.

Synthesis of 4-bromostilbene



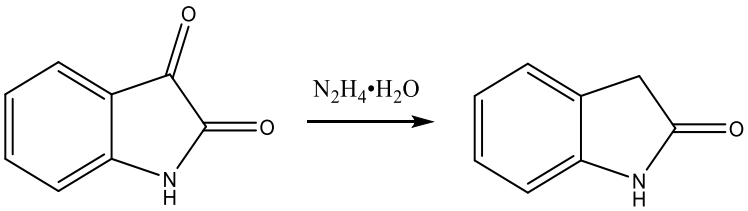
4-Bromobenzaldehyde (100 mg), benzyltriphenylphosphonium chloride (200 mg), and anhydrous potassium phosphate tribasic (425 mg) were intimately ground together for 15 minutes and suspended in distilled water, then filtered. The product was then recrystallised from absolute ethanol.

Synthesis of 1,3,3a,4,5,7a-hexahydro-5-methyl-3-oxo-4-isobenzofurancarboxylic acid



To a test tube was added sorbic alcohol (110 mg), maleic anhydride (112 mg), toluene (3 mL) and boiled for 10 minutes in a sand bath. The reaction mixture was allowed to cool slowly to 0°C, and the crystals filtered off.

Synthesis of oxindole



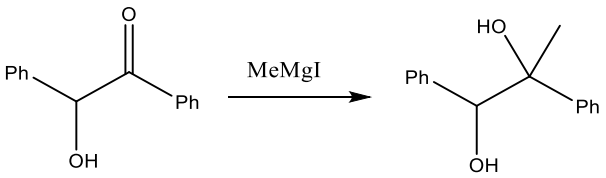
Method 1 – Traditional heating

To a 50 mL beaker was added isatin (250 mg), hydrazine hydrate (300 mg, 55% in water), ethylene glycol (1 mL), and microwaved at 700W for 30 seconds. The yellow product was suspended in ice-cold ethanol and filtered off.

Method 2- Microwave heating

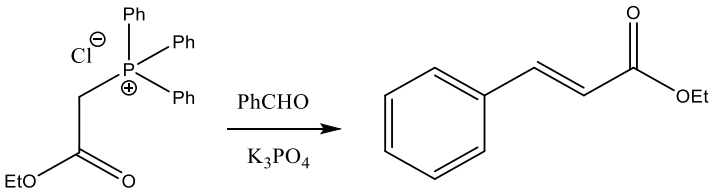
To a 50 mL beaker was added isatin hydrazone (200 mg), potassium hydroxide (1.1 eq), ethylene glycol (2 mL) and mixed before microwaving at 700W for 10 seconds. The solution was then acidified with hydrochloric acid (6 M) to pH 2 and extracted three times with diethyl ether (5 mL), dried over anhydrous sodium sulphate. The solvent was removed, and the product recrystallised from water.

Synthesis of 1,2-diphenyl-1,2-propanediol



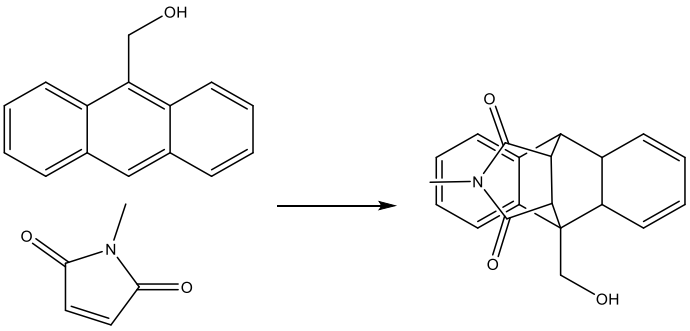
To a 25 mL Erlenmeyer flask was added benzoin (250 mg), dry dichloromethane (5 mL) and stirred to dissolve. Methyl magnesium iodide (1.2 mL, 3 M in Et_2O) was then added dropwise to the solution cooled in an ice-water bath and stirred for 15 minutes. Hydrochloric acid (2 mL, 10%) was then added, followed by diethyl ether (5 mL) and the upper organic layer separated. The aqueous layer was extracted with a further portion of diethyl ether (5 mL). The combined organic layers were dried over anhydrous sodium sulphate, and the solvent removed. The product was recrystallised from toluene, and then from hexanes.

Synthesis of ethyl trans-cinnamate



To a 5 mL conical flask was added benzaldehyde (48 µL, 50 mg), carbethoxymethylenetriphenylphosphine (200 mg) and stirred for 10 minutes at room temperature. Hexanes (3 mL) was then added and stirred for another 10 minutes. The solids were filtered off, washed with hexanes, and the solvent removed from the filtrate.

Synthesis of 9,10-Dihydro-9-hydroxymethyl-9,10-[4,5](N-methyl-1,3-dioxopyrrolidino)anthracene



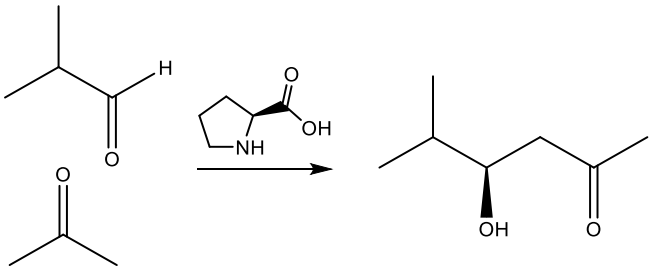
Method 1- Traditional heating

To a 100 mL round bottomed flask was added 9-anthracenemethanol (100 mg), N-methylmaleimide (180 mg, 3 eq), water (50 mL) and heated to reflux for 2 hours. The reaction mixture was allowed to cool to 0°C, and the precipitate filtered off and dried under vacuum.

Method 2 – Microwave heating

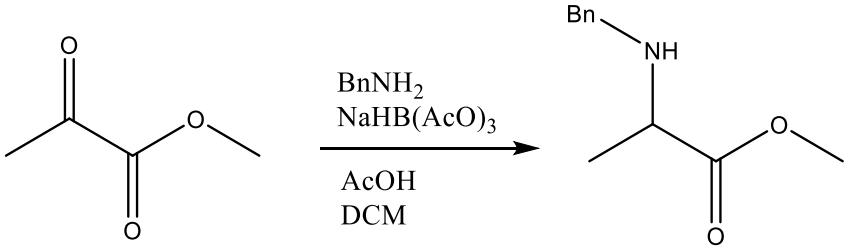
To a test tube was added 9-anthracenemethanol (10 mg), N-methylmaleimide (18 mg, 3 eq), water (7 mL) and microwaved at 200W for 3 minutes. The precipitate was filtered off, and dried under vacuum. The final yield was 8 mg (50%).

Synthesis of (4R)-4-hydroxy-5-methyl-2-hexanone



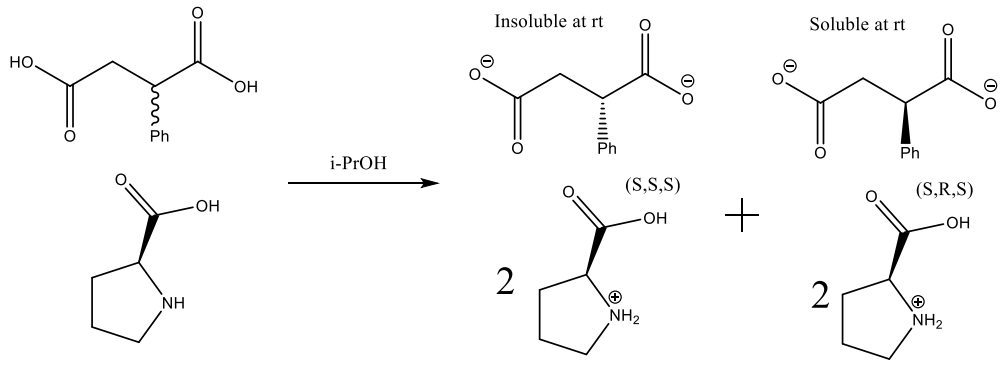
To a 25 mL Erlenmeyer flask was added isobutyraldehyde (1 mL), acetone (14 mL), and L-proline (230 mg) and stirred vigorously at room temperature for 2 hours, then allowed to sit for 1 week in the dark. Brine (50 mL) was then added, and the mixture extracted twice with diethyl ether (15 mL). The ether extract was dried over anhydrous sodium sulphate, and the solvent removed. The final product had an enantiomeric excess of 77%.

Synthesis of methyl 2-(N-benzyl)aminopropanoic acid



To a test tube was added methyl pyruvate (102 mg, 1 mmol), benzylamine (127 mg, 1.1 mmol), dichloromethane (3 mL), sodium triacetoxyborohydride (251 mg, 1.1 mmol), glacial acetic acid (70 mg) and stirred for 15 minutes at room temperature. Saturated sodium bicarbonate solution (3 mL) and the reaction mixture stirred thoroughly until effervescence had ceased. The lower aqueous layer was separated, and the aqueous layer extracted twice with dichloromethane (3 mL), and the organics dried with anhydrous sodium sulphate, and the solvent removed. The final yield was 158 mg.

Resolution of phenylsuccinic acid



To a 100 mL Erlenemeyer flask was added racemic phenylsuccinic acid (1.0 g), L-proline (600 mg), isopropanol (30 mL) and refluxed for 30 minutes. The reaction mixture was then allowed to cool to 0°C. The precipitate was filtered off and washed well with ice-cold acetone. The yield of the (S,S,S) diastereomeric salt was 715 mg (65%). The product was added to hydrochloric acid (6 mL, 6 M), stirred well and cooled to 0°C, and the precipitate filtered off and washed with ice-water. The yield of (S)-phenylsuccinic acid was 165 mg (33%) with an enantiomeric excess of 78%. The product was then recrystallised from water, yielding 50 mg with 97.6 %ee.

elementguy27

Synthesis of chromium(IV) triamine diperoxide

$K_2CrO_4 + 3NH_3 + 3H_2O_2 \rightarrow Cr_3(NH_3)(O_2)_2 + 2H_2O + 2KOH + O_2$

Ammonium dichromate (0.3 g) dissolved in the minimum of ammonia solution (~5 mL, 30%), and chilled to 0°C. Hydrogen peroxide (3 mL, 30%) was then added dropwise to the solution, and allowed to chill for 30 minutes. The precipitate was then filtered off, and washed with ethanol. The yield was of rust-red crystalline powder.

Extractions&Ire

Synthesis of sodium nickel(IV) periodate

$NiSO_4 + NaIO_4 + 2\ Na_2S_2O_8 + 2\ H_2O \rightarrow NaNiIO_6 + 2\ Na_2SO_4 + O_2 + 2\ H_2SO_4$

Nickel(II) sulphate heptahydrate (2.0 g, 7.1 mmol) was dissolved in distilled water (200 mL) and treated with sodium periodate (5.0 g, 17 mmol) and dilute sulfuric acid (0.5 mL, 1 M), and the mixture heated to ca. 80°C, more water being added if necessary to completely dissolve the periodate. Sodium persulphate (8.0 g, 34 mmol) was added in small portions to the hot solution, which slowly darkened in colour. A fine black solid slowly separated, and after ca. 4 h the solution was cooled and allowed to stand overnight. The precipitate was filtered off, rinsed with hot dilute sodium persulphate solution, and with a small quantity of hot water, and dried in vacuo. The final yield was 1.1 g (17%) of purple-black powder. Typical yields are rather variable, but typically 40-50% on Ni.

Currie DB, Levason W, Oldroyd RD, Weller MT. Synthesis, spectroscopic and structural studies of alkali metal–nickel periodates MNilO6 (M= Na, K, Rb, Cs or NH4). Journal of the Chemical Society, Dalton Transactions. 1994 Jan 1(9):1483-7.

Synthesis of sodium orthoperiodate

$NaI + 5\ H_2O + 4\ Cl_2 + NaOH \rightarrow Na_2H_3IO_6 + 8\ HCl$

To a 500 mL beaker was added sodium hydroxide (50 g) and dissolved in water (220 mL). Sodium iodide (10 g) was then added and the mixture stirred and heated to boiling until all the solids had dissolved. Chlorine was then passed in until no more precipitate formed, then filtered. The precipitate was washed with ice-water and dried at the pump. The product was recrystallised from water. The final yield was 16 g (86%).

Synthesis of nitric acid

$KNO_3 + NaHSO_4 \rightarrow HNO_3 + NaKSO_4$

To a 500 mL round bottomed flask set up for distillation was added potassium nitrate (50 g) and sodium hydrogen sulphate (85 g), and the reaction mixture distilled until no more distillate collected. The final yield was 25 g.

Synthesis of sodium sulphide

$Fe + 2\ Al + 4\ S \rightarrow FeS + Al_2S_3$

$FeS + Al_2S_3 + 8\ HCl \rightarrow FeCl_2 + AlCl_3 + 4\ H_2S$

$H_2S + 2\ NaOH \rightarrow Na_2S + 2\ H_2O$

Iron filings (5 g), aluminium powder (10 g), and sulphur (20 g) were mixed thoroughly and ignited. The cooled reaction product was transferred to a flask, and hydrochloric acid added via a thistle funnel to generate hydrogen sulphide. The gas so produced was passed into sodium hydroxide solution (40 g in 100 mL). The water was boiled off, leaving the product as a soft white powder. The final yield was 25 g (64%).

Frankium

Synthesis of bromine

$2\ KMnO_4 + 10\ NaBr + 14\ H_2SO_4 = 2\ KHSO_4 + 10\ NaHSO_4 + 8\ H_2O + 5\ Br_2 + 2\ MnSO_4$

To a 250 mL round bottomed flask set up for distillation was added sodium bromide (100 g), potassium permanganate (20 g), and distilled water (150 mL), and sulphuric acid (60 mL, 98%) to an addition funnel. The sulphuric acid was added slowly to the reaction mixture, and the reaction mixture heated gently towards the end of the reaction. The distillate was then dried over sulphuric acid (5 mL). The final yield was 12 mL.

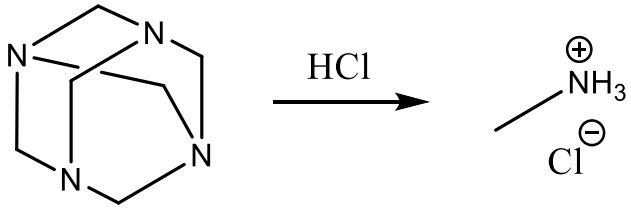
Full Modern Alchemist

Synthesis of benzyl chloride

$BnOH + HCl \rightarrow BnCl + H_2O$

To a 500 mL round bottomed flask was added benzyl alcohol (75 mL), hydrochloric acid (300 mL) and refluxed for 30 minutes. The upper aqueous layer was separated, and washed with saturated sodium carbonate (2x 50 mL) and brine (50 mL), then dried over calcium chloride. The drying agent was filtered off, and the product distilled collecting the fraction at 170-175°C. The final yield was 74 mL (90+%).

Synthesis of methylamine



To a 500 mL three-necked round bottomed flask set up for distillation was added hexamine (70 g) dissolved in water (100 mL), hydrochloric acid (200 mL, 36%) and distilled to dryness. The solids were digested with ethanol, and the solvent distilled from the filtrate. The solids were suspended in acetone, and the solids filtered off. (Care must be taken not to leave the acetone and product in contact for a long time to avoid condensation products from forming.) The final yield was 36%.

Synthesis of sodium ethyl sulphate

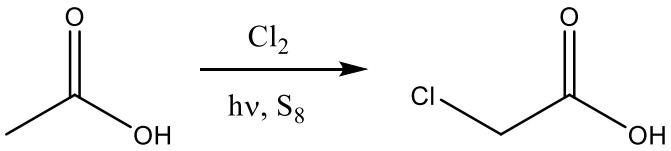
$EtOH + H_2SO_4 \rightarrow EtHSO_4$

$2\ EtHSO_4 + Na_2CO_3 \rightarrow EtNaSO_4 + H_2O + CO_2$

To a 250 mL round bottomed flask was added ethanol (160 mL), and slowly sulphuric acid (80 mL, 98%) and the reaction mixture was refluxed for 2-3 hours. The reaction mixture was allowed to cool, and calcium carbonate added until bubbling ceased. The precipitate was filtered off, and sodium carbonate added to the filtrate until no more precipitate formed. The precipitate was filtered off again, and the solution evaporated to dryness. The final yield was ~70%.

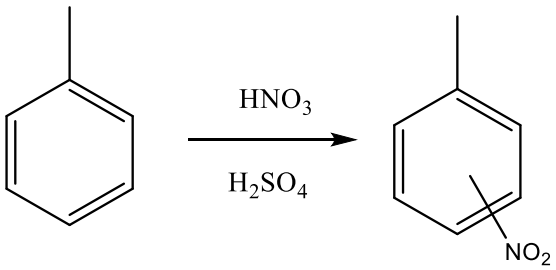
Hazel Chem

Synthesis of chloroacetic acid



To a 250 mL two necked round bottomed flask fitted with a reflux condenser were added glacial acetic acid (101 g) and sulphur (10 g). A chlorine generator was set up using TCCA (100 g) and hydrochloric acid (116 mL, 32%), and the chlorine dried over sulphuric acid. The reaction mixture was heated to 90-95°C with a water bath and irradiated with a halogen lamp. Chlorine was passed into the reaction mixture until the generator was exhausted, and the water (30 mL) was added to the reaction mixture and stirred for 30 minutes. The sulphur was filtered off, and the filtrate cooled to 0°C. The precipitate was filtered off, and the liquor evaporated to crystallise further product out.

Synthesis of nitrotoluenes



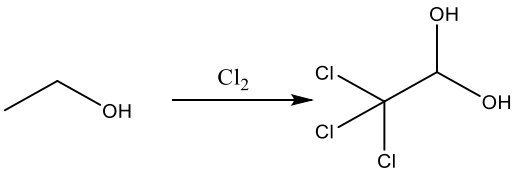
To a 250 mL round bottomed flask were added sulphuric acid (95 g, 98%) and nitric acid (75 mL, 65%), and the mixed acid cooled to 0°C in an ice bath. This was then added dropwise to toluene (80 mL) in a 250 mL round bottomed flask, with total addition taking 30-60 minutes. The upper aqueous layer was separated and washed with water (100 mL), sodium carbonate (2x 100 mL, 10%), and dried over calcium chloride. The mixture of isomers were then separated by freezing the para isomer out at -8°C. The para isomer was then dried in a desiccator over sodium hydroxide. The ortho isomer was then re-dried over calcium chloride.

Synthesis of oxalic acid

$C_6H_{12}O_6 + 18\ HNO_3 \rightarrow 18\ NO_2 + 12\ H_2O + 3\ (COOH)_2$

To a 500 mL beaker was added glucose (50 g), then nitric acid (260 mL, 65%) was added carefully. With stirring, the reaction was gently heated until the reaction initiates. It is then allowed to proceed with voluminous evolution of nitrogen oxides. The reaction mixture was allowed to react for 1 hour with vigorous stirring. The reaction mixture was then reduced in volume until crystals formed, then cooled to 0°C. The product was filtered off, and washed with ice-cold ethanol.

Synthesis of chloral hydrate



A chlorine generator was set up with TCCA (317 g, 1.36 mol) and hydrochloric acid (400 mL, 32%, 3 mol). To a 100 mL two-necked round bottomed flask was added ethanol (46.07 g, 1 mol), and chlorine passed in until the generator was exhausted. The reaction flask was slowly heated to boiling over the course of the reaction. The reaction mixture was then distilled to dryness. The final yield was 2.8 g, melting at 55°C.

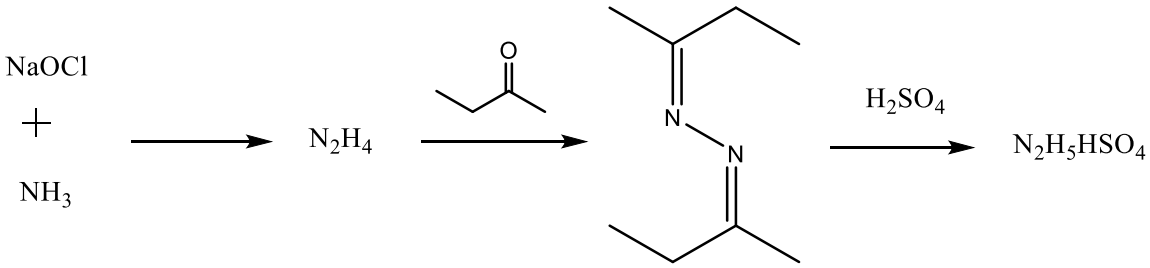
Synthesis of potassium cyanide

$KOH + HCN \rightarrow KCN + H_2O$

To a 500 mL two-necked round bottom flask fitted with an addition funnel was added potassium ferrocyanide (50 g), and phosphoric acid (46 mL, 85%) was added to the addition funnel. Potassium hydroxide (29 g) was dissolved in water (50 mL), and the gas formed from the reaction of the potassium ferrocyanide and phosphoric acid was passed in through a wash bottle. After complete reaction, the liquid in the wash bottle was diluted with three times its volume of ethanol, and the precipitate filtered off and washed with ice-cold ethanol. The final yield was 14.55 g (37%) of fine white powder.

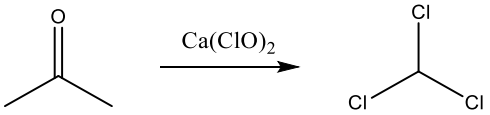
hkparker

Synthesis of hydrazine sulphate



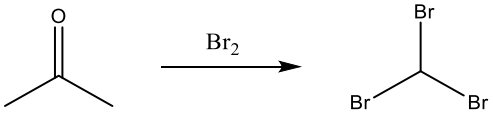
To a 1000 mL beaker was added ammonia solution (250 mL, 35%), methyl ethyl ketone (100 mL), and sodium hypochlorite (180 g, 10%, 0.25 mol) was added slowly with strong stirring. After complete addition, the solution was allowed to separate and the upper organic layer separated. Water (100 mL) and sulphuric acid (20 mL, 98%) were then added to the organic layer, and then heated to boil the methyl ethyl ketone off, then allowed to cool to 0°C. The precipitate was then filtered off, and washed with ice water. The final yield was ~2 g.

Synthesis of chloroform



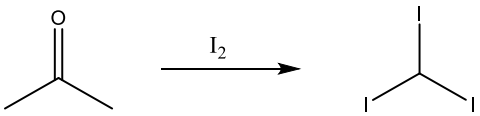
To a three necked round bottomed flask cooled in an ice bath fitted with a pressure equalising addition funnel and a reflux condenser was added water (200 mL), calcium hypochlorite (~130 g) and acetone (200 mL) was added dropwise with strong stirring until the temperature ceased to rise. The reaction mixture was then distilled, and the lower organic layer separated and washed with brine.

Synthesis of bromoform



To a 250 mL beaker was added sodium bromide (7.4 g) dissolved in water (100 mL), acetone (4 mL), and bleach (150 mL). The reaction mixture was allowed to separate, and the lower organic layer separated.

Synthesis of iodoform



Method 1 – Iodine generated in situ

To a 250 mL beaker was added potassium iodide (12 g) dissolved in water (100 mL), acetone (4 mL), and bleach (150 mL). The reaction mixture was then filtered, and the precipitate washed with ice-water.

Method 2 – From elemental iodine

To a saturated solution of iodine in acetone was added 20x its volume of water, then sodium hydroxide solution (10%) was added dropwise until the brown colour of iodine had disappeared. The precipitate was filtered off and washed with ice-water.

Preparation of silicon

$$3\ SiO_2 + 6\ Al + 3\ S \rightarrow 3\ Si + 2\ Al_2O_3 + Al_2S_3$$

Sulphur powder[1] (72 g), silicon dioxide powder (54 g), aluminium powder (60 g) were mixed intimately and then ignited with flash powder. After allowing to cool, the reaction mixture was digested with water, then hydrochloric acid (2 M) and the precipitate filtered off.

(1) The purpose of using sulphur was to use the heat of reaction of aluminium and sulphur to drive the reduction of silicon dioxide with aluminium, which is endothermic.

Synthesis of iodine chlorides

$$I_2 + Cl_2 \rightarrow 2\ ICl$$

A chlorine generator was set up using potassium permanganate and hydrochloric acid (31%) and passed into a test tube containing iodine. Both iodine monochloride and iodine trichloride were formed.

LuminolSynthesis

Extraction of furfural from corncobs

To a 100 mL round bottom flask was added ground corncob (10 g), sodium chloride (10 g), hydrochloric acid (50 mL), and the reaction mixture distilled for 30 minutes at 135°C. Approximately 10 mL of distillate was collected, and extracted with dichloromethane (25 mL), and the organic layer dried over anhydrous magnesium sulphate. The solvent was removed, leaving the residue. The final yield was ~1 mL.

mabakken

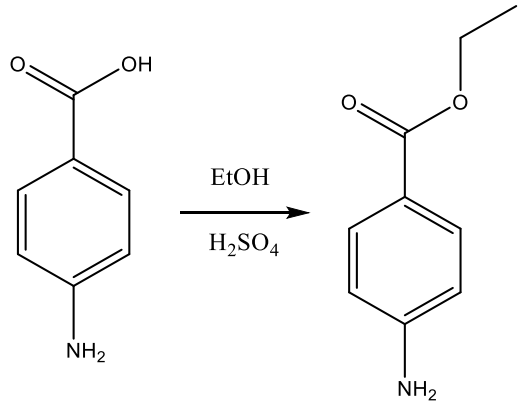
Synthesis of sulphur trioxide

$Na_2S_2O_8 \rightarrow Na_2SO_4 + SO_3$

To a 250 mL round bottomed flask is added sodium persulphate (g, mol), and sulphuric acid (10 mL, g, 18.4 mol dm^{-3}). The reaction mixture is then distilled, and the sulphur trioxide collected.

Matter Manipulation

Synthesis of benzocaine



To a 500 mL two-necked round bottomed flask was added p-aminobenzoic acid (10.20 g), absolute ethanol (100 mL), and sulphuric acid (10 mL, 96%) and refluxed for 80 minutes using molecular sieves in a pressure-equalising funnel as drying agent to remove water as it formed. The reaction mixture was added to water (200 mL), and sodium bicarbonate (30 g) dissolved in water (100 mL) was added to achieve pH 8. The precipitate was then filtered off, and washed with water. The final yield was 10.56 g (86%).

Synthesis of auric chloride

$$2\ Au + HNO_3 + 4\ HCl \rightarrow HAuCl_4 + 3\ H_2O + 3\ NO_2$$

To a 50 mL beaker was added gold (1.0 g) and aqua regia (10 mL), then allowed to react. After complete reaction, the solution was evaporated to a syrup and then hydrochloric acid (5 mL) added, then reevaporated. This process was repeated until nitrogen oxides were no longer evolved, and the solution evaporated to dryness at less than 120°C. Due to the hygroscopicity of the product, a yield measurement could not be taken.

Synthesis of fuming nitric acid

$$2\ NaNO_3 + H_2SO_4 \rightarrow 2\ HNO_3 + Na_2SO_4$$

To a 500 mL round bottomed flask set up for distillation was added sodium nitrate (170 g, 2 mol), and sulphuric acid (100 g, 98%, 1 mol). The reaction mixture was distilled until the fumes became a lighter colour, signifying an end to the reaction.

mrhomescientist

Synthesis of Chevreul's salt

$3CuSO_4 + 8NaHSO_3 \rightarrow Cu_3(SO_3)_2 + 4Na_2SO_4 + 5SO_2 + 4H_2O$

Copper sulphate (2.4 g) was dissolved in water (25 mL) and added to sodium metabisulphite (2.0 g) dissolved in water (10 mL), and boiled for 5 minutes. The precipitate was then filtered off, and washed with water (10 mL).

Synthesis of hexamminenickel(II) chloride

$[Ni(H_2O)_6]Cl_2 + 3NH_3 \rightarrow [Ni(NH_3)_6]Cl_2$

To a cooled saturated solution of nickel chloride (3 mL) was added cooled ammonia solution (25%) until the precipitate dissolved. Acetone was then added to precipitate the product as a lavender precipitate. This was then filtered off, and dried. The yield was 0.6 g.

Preparation of boron

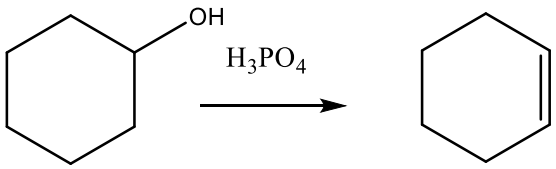
$2\ H_3BO_3 \rightarrow B_2O_3 + 3\ H_2O$

$B_2O_3 + 3\ Mg \rightarrow 2\ B + 3\ MgO$

To a large crucible was added boric acid (15 g) and was heated strongly until steam ceased to be evolved. The glassy material was allowed to cool and was chipped out of the crucible. The resulting hard material was crushed with difficulty to a powder, and mixed with magnesium powder (9 g) and ignited with a potassium permanganate/glycerine mixture. The reaction mixture was allowed to cool to room temperature, and the result digested with dilute hydrochloric acid, and the precipitate filtered off.

myst32YT

Synthesis of cyclohexene



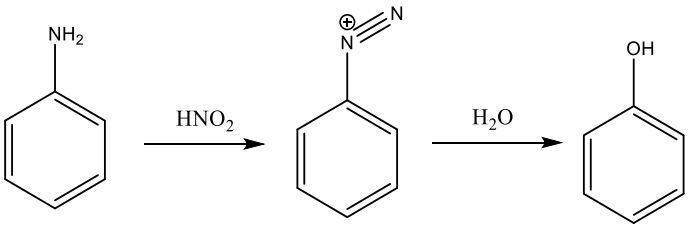
To a 100 mL pear-shaped flask were added cyclohexanol (70 mL, g, mol), and phosphoric acid (5 mL, g, 85%, mol dm^{-3}). The reaction mixture was then distilled, collecting the fraction at around 80°C.

Synthesis of sodium peroxide

$Na + O_2 \rightarrow NaO_2$

To a 100 mL crucible was added sodium metal (13 g) and heated strongly until ignition occurs, at which point the heat source was removed. Oxygen gas was then passed into the crucible until the reaction ceased. The reaction mixture was then allowed to cool to room temperature and stored. The product was contaminated with sodium oxide.

Synthesis of phenol



To a 250 mL beaker was added water (150 mL), slowly sulphuric acid (27.5 mL, 98%), and slowly aniline (24 mL). This solution was then diluted with water (200 mL) and chilled to 0°C. Sodium nitrite (20 g) was dissolved in water (15 mL) and added dropwise to the aniline sulphate solution, maintaining the temperature below 5°C. After complete addition, the reaction mixture was allowed to warm slowly to 50-55°C, and held at that temperature for 30 minutes, then allowed to cool slightly. The reaction mixture was then steam distilled until 500 mL of distillate had collected.[1] The distillate was then extracted four times with diethyl ether (16 mL), and the combined organic layers stirred with potassium carbonate (5 g) for 1 hour. The solvent was then removed, and the residue distilled twice collecting the fraction at 178-184°C. The final yield was 7 g. Typical yields are closer to 10-14 g.

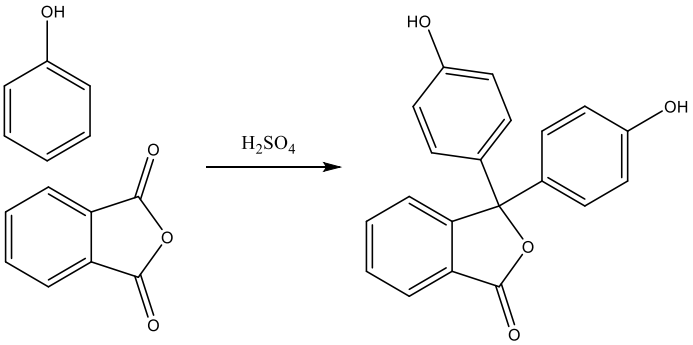
(1) Clean up is made easy with concentrated sodium hydroxide solution

Preparation of bromine

$2\ KBr + Cl_2 \rightarrow 2\ KCl + Br_2$

To a 500 mL beaker was added potassium bromide (250 g) and made into a solution with water to a total volume of 400 mL. The solution was then added to a 1000 mL two necked round bottomed flask set for distillation. A chlorine generator was then set up with calcium hypochlorite (300 g) and hydrochloric acid (400 mL, 37%) and the chlorine so formed passed to the bottom of the potassium bromide solution. The main reaction flask (containing potassium bromide) was gently heated, and the chlorine passed in until no more bromine was distilled. The bromine was then dried over sulphuric acid (60 mL, 98%) and ampouled for storage. The final yield was 142 g (85%).

Synthesis of phenolphthalein



To a test tube was added phenol (100 mg), phthalic anhydride (100 mg), and sulphuric acid (3 drops, 98%) and mixed thoroughly. The reaction mixture was heated carefully until just molten, then allowed to cool. Water (5 mL) was then added, and dilute sodium hydroxide solution added until the solids had all dissolved. Sulphuric acid was then added dropwise to the solution until the colour had dissipated. This generated a solution of phenolphthalein.

Synthesis of carbon disulphide

$C + 2\ S \rightarrow CS_2$

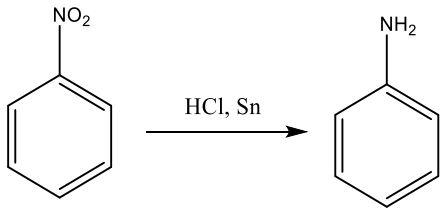
A 12 inch ceramic tube was filled half full with packed sulphur powder, and the rest of the way with carbon. A glass tube and rubber stopper used to collect the vapours. The tube was then placed in a kiln and heated white-hot with an oxy-propane torch until no more distillate collected in a collection vessel half-full of water placed in an ice bath. After 40 minutes of heating, the lower layer of product was separated and distilled, collecting the fraction at 38-42°C. The final yield was 4 mL.

Synthesis of benzene

$PhCOOH + CaO \rightarrow PhH + CaCO_3$

To a 250 mL flat-bottomed flask was added benzoic acid (10 g) and calcium oxide (10 g) that had been mixed intimately. The reaction mixture was then distilled until no more distillate collected, which took approximately 30 minutes. The crude yield was 4 g (~50%).

Synthesis of aniline



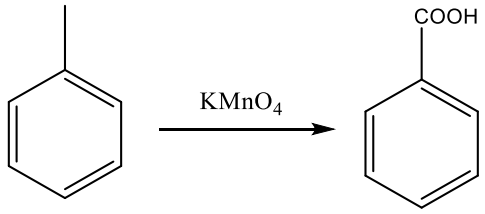
To a 1 L three necked round bottom powder fitted with an overhead mixer and a pressure equalising addition funnel was added iron powder (120 g, 2 mol), distilled water (140 mL), and nitrobenzene (82 mL) to the addition funnel. Hydrochloric acid (15 mL, 31%) was then added, and heated to 80°C and nitrobenzene added dropwise, maintaining a temperature of 80-90°C. This took about 1 hour. The reaction mixture was the refluxed for 30 minutes, then potassium carbonate (20 g) was added with good mixing. The reaction mixture was then steam distilled until the distillate collected was no longer turbid. To the distillate was added sodium chloride (100 g) and stirred until dissolved. The upper organic layer was then separated and distilled to remove water. The apparatus was then dissembled, washed and dried with a heat gun, and the residue distilled, collecting the fraction at 180°C. The final yield was 56 mL, and stored in an amber bottle.

Synthesis of diethyl ether

$2\ EtOH \rightarrow Et_2O + H_2O$

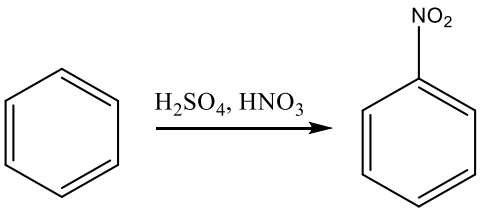
To a 1000 mL two necked round bottomed flask cooled in an ice bath was added sulphuric acid (160 mL, 98%) and absolute ethanol (200 mL) dropwise. The flask was then set up for simple distillation, and sand (5 g) added to the flask. To an addition funnel was added ethanol (500 mL), and the reaction mixture heated to 145-149°C (below 150°C to avoid elimination product). When distillate began to collect, ethanol was added at a similar rate to distillate collection. The distillation was continued for 15 minutes after complete addition. The distillate was then washed with sodium hydroxide solution (200 mL, 10%), then brine added to force separation of the ether. The upper organic layer was then separated and distilled, collecting the fraction at 34-36°C. The distillate was dried over calcium chloride (30 g). The final yield was ~500 mL.

Synthesis of benzoic acid



Potassium permanganate (30 g) was dissolved in water (350 mL) and added to toluene (70 mL) in a 1000 mL round bottomed flask and refluxed for 2.5 hours. The precipitate was then hot-filtered off, and the upper organic layer removed from the filtrate. The aqueous layer was then concentrated to 100 mL and allowed to cool to room temperature. Hydrochloric acid (37%) was then added dropwise until no more precipitate formed. The precipitate was filtered off and washed well with cold water.

Synthesis of nitrobenzene



To a 250 mL beaker cooled in an ice bath was added sulphuric acid (80 mL, 98%) and nitric acid (75 mL, 90%) added slowly, ensuring the temperature remained low. Benzene (16 mL) was then added dropwise, maintaining the temperature below 50°C over a period of 1 hour. After complete addition, the reaction mixture was allowed to sit at room temperature for 1 hour. The upper organic layer was separated and washed thrice with water (40 mL) then distilled, collecting the fraction at 207-211°C. The distillate was dried over calcium chloride. The final yield was 15 mL (83%).

Synthesis of 2-bromopropane

$^{i}PrOH + HBr \rightarrow {}^{i}PrBr + H_2O$

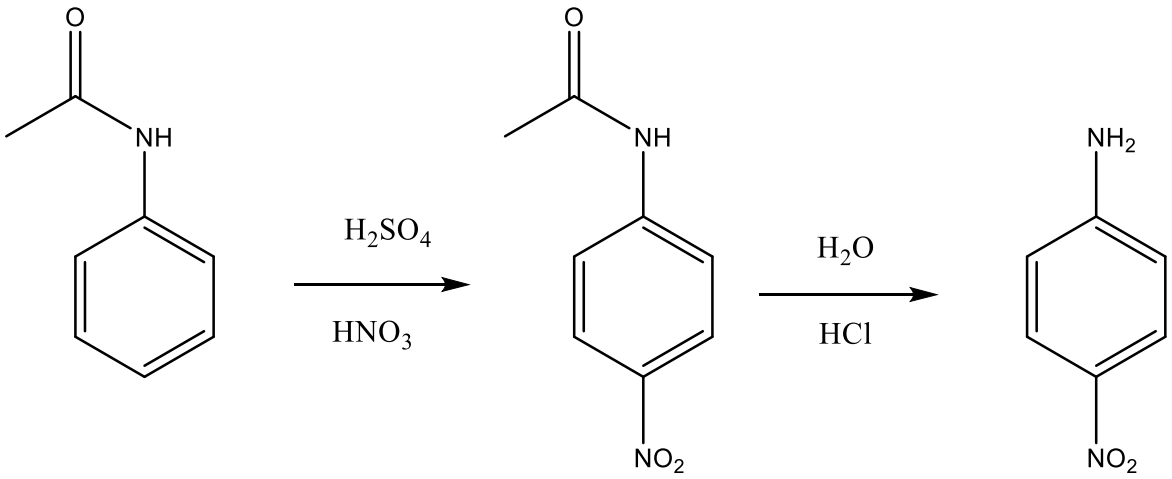
To a 250 mL round bottom flask was added hydrobromic acid (180 mL), isopropanol (50 mL) and the reaction mixture distilled until the temperature rose above 70°C. To the distillate was added brine (20 mL), and the upper organic layer separated and dried over anhydrous magnesium sulphate.

Preparation of Dragendorff's reagent

To a beaker was added bismuth subnitrate (0.5 g) and dissolved in water, and hydrochloric acid (10 mL, 37%) added. Potassium iodide (4 g) was dissolved in water and added to the bismuth subnitrate solution to form the Dragendorff's reagent.

NileRed

Synthesis of p-Nitroaniline



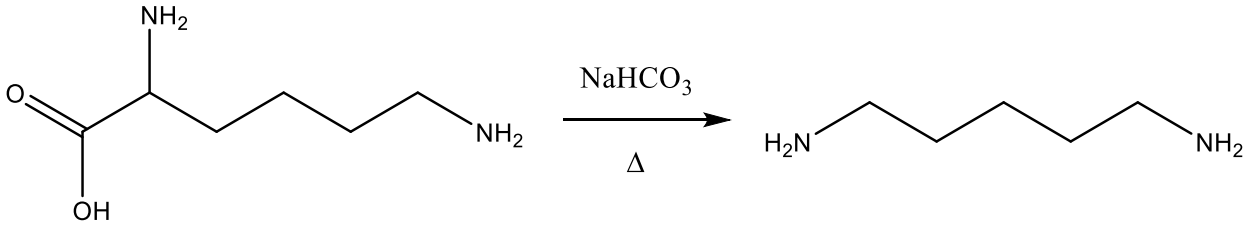
Synthesis of p-nitroacetanilide

To a 250 mL round bottomed flask were added acetanilide (34 g, mmol), and glacial acetic acid (40 mL). This was then heated under stirring to dissolve all the acetanilide. The solution was allowed to cool to below 40°C, then ice-cold sulphuric acid (80 mL, g, 18.4 mol dm^{-3}) was slowly added. The flask was then added to an ice-bath, and allowed to cool below 5°C. An ice-cold nitrating mixture of sulphuric acid (30 mL, g, 18.4 mol dm^{-3}) and nitric acid (20 mL, g, mol dm^{-3}) was then prepared. The nitrating mixture was then added dropwise to the solution of acetanilide in glacial acetic acid maintaining the temperature below 20°C. After addition, the reaction mixture was allowed to sit at room temperature for 40 minutes, then poured into ice-cold water (200 mL). The reaction flask was rinsed with water (50 mL). After stirring to ensure homogeneity of the diluted reaction mixture, the precipitate was filtered off, and washed with cold water.

Synthesis of p-nitroaniline

The product was then added to hydrochloric acid (200 mL, 6 mol dm^{-3}), and refluxed for 20 minutes. The reaction mixture was then added to water (150 mL), and allowed to cool to room temperature, at which point it was added to ice-cold ammonia solution (750 mL, 2.5 mol dm^{-3}). The precipitate was then filtered, and washed with cold water (100 mL). The product was then recrystallized from ethanol to yield g (%) of flaky yellow crystals melting at 137°C (lit. 145°C).

Synthesis of cadaverine



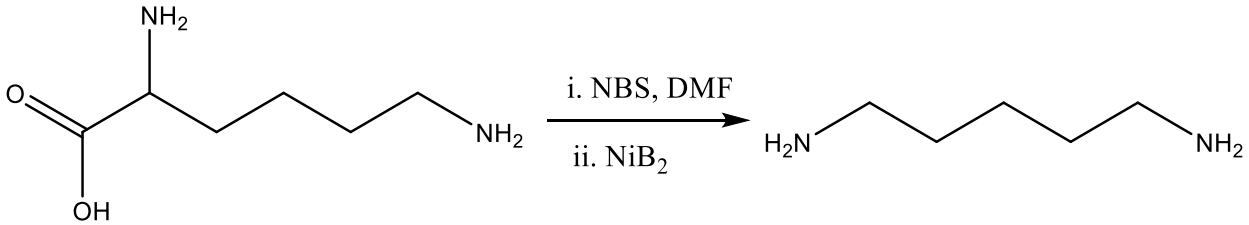
Method 1a – Simple dicarboxylation

To a 10 mL round bottom flask were added lysine hydrochloride (0.5 g, mmol), sodium bicarbonate (0.75 g, mmol), and mixed. The mixture was then heated strongly until the reaction was judged complete. After cooling, ethanol (5 mL) was added and stirred to dissolve the solids. The solvent was then removed.

Method 1b

A mixture of lysine hydrochloride (1.3 g) and sodium bicarbonate (1 g) were added to a test-tube flushed with nitrogen. The mixture was then heated strongly, and the vapours condensed in water.

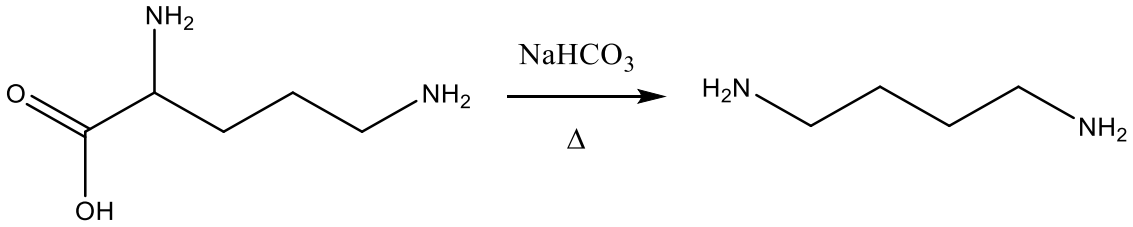
Method 2 – Via nickel boride



To a 250 mL Erlenmeyer flask were added disodium phosphate dodecahydrate (3.2 g, mmol), citric acid (0.86 g, mmol), and water (90 mL). To the buffer solution so prepared were added L-lysine hydrochloride (3.53 g, mmol), and dropwise a solution of N-bromosuccinimide (10.3 g, mmol) in dimethylformamide (20 mL). The reaction mixture was then stirred for 30 minutes, and then nickel chloride hexahydrate (22.9 g, mmol) was added, followed by sodium borohydride (5.84 g, mmol) slowly, then stirred for 20 minutes. The nickel boride was then filtered off (Celite recommended), and sodium hydroxide solution (6 mol dm^{-3}) added to increase the pH to 11-12. The solution was then extracted with diethyl ether (3x 30 mL), and the combined organic layers dried with brine (50 mL), and anhydrous magnesium sulphate. The solvent was removed, and the gel washed with acetone to yield 0.5 g (25%) of yellow-orange goo.

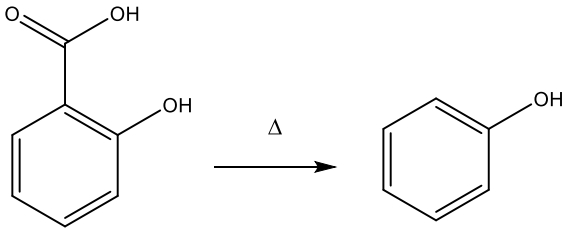
Laval G, Golding BT. One-pot sequence for the decarboxylation of α-amino acids. Synlett. 2003 Mar;2003(04):0542-6.

Synthesis of putrescine



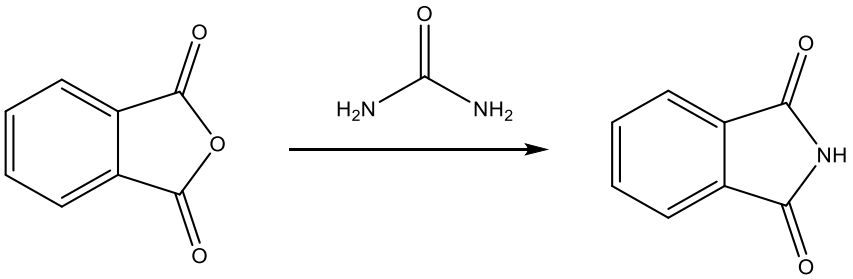
A mixture of ornithine hydrochloride (1.3 g) and sodium bicarbonate (0.9 g) were added to a test-tube flushed with nitrogen. The mixture was then heated strongly, and the vapours condensed.

Synthesis of phenol



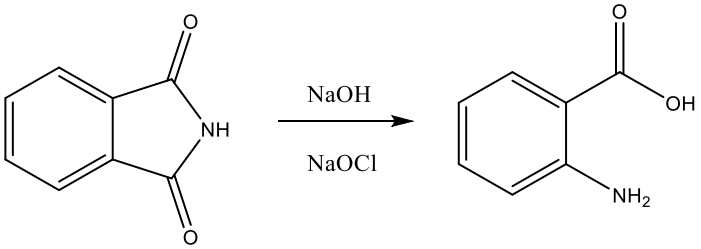
A 100 mL round bottomed flask was charged with salicylic acid (10 g), and boiling chips added. A short-path distillation apparatus was then set up, and the salicylic acid was distilled, collecting the fraction boiling between 145-165°C. The distillate was then redistilled twice under reduced pressure, and the fraction boiling at 85-90°C / 40 mmHg. The final yield was 3.0 g (44%) of white crystals.

Synthesis of phthalimide



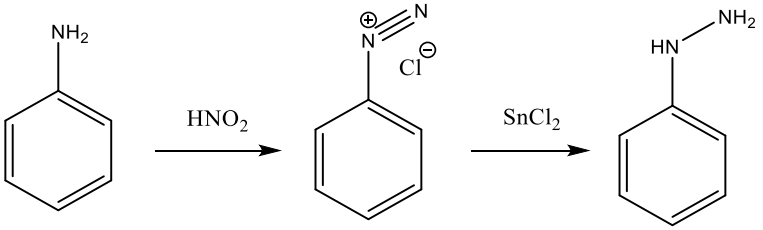
To a 100 mL round bottomed flask were added urea (2 g, mmol) and phthalic anhydride (10 g, mmol), then heated on an oil-bath to 135℃. The reaction is considered complete when the reaction mixture froths up. Upon cooling to room temperature, distilled water (10 mL) were added to the flask to dissolve any unreacted urea. The insoluble phthalimide was then recrystallized from methanol (300 mL) to yield 7.4 g (76%) of white needles.

Synthesis of o-anthranilic acid



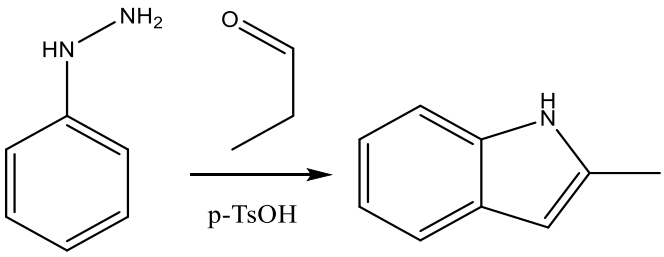
To a 100 mL round bottomed flask were added sodium hydroxide (3 g, mmol) and distilled water (12 mL), then cooled in an ice-bath to below 5°C. Maintaining the temperature below 5°C, sodium hypochlorite solution (13 mL, 16.7 mmol, 8.6 %) was added, followed by phthalimide (2.6 g, 17.7 mmol). The reaction mixture was allowed to warm to room temperature with stirring. Then sodium hydroxide solution (2.25 g in 8 mL water) was added, then heated to 80°C for 30 minutes. The reaction mixture was then cooled to 0°C, and hydrochloric acid (6 mol dm^{-3}) added until a pH of 3.5 was obtained. The precipitate was then filtered off, and the product dried at the pump. The final product was 1 g of light brown powder. A recrystallization from water could be performed to achieve white crystals.

Synthesis of phenylhydrazine hydrochloride



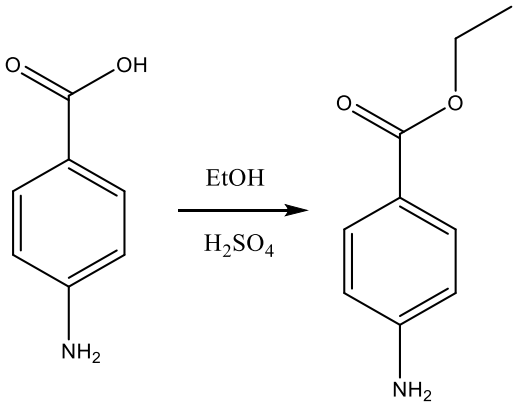
To a 250 mL round bottomed flask fitted with a stir bar were added water (18 mL), hydrochloric acid (32 mL, 31.45%), dropwise aniline (4.6 mL, g, mmol). The reaction mixture was then added to an ice bath, and cooled to below 5°C. Sodium nitrite (3.63 g, mmol) dissolved in water (8 mL) was then added dropwise to the reaction mixture, maintaining the temperature below 5°C. The solution was then stirred at 0°C for 1 hour. Stannous chloride solution (50 mL, 2 mol dm^{-3}) chilled to 0°C was then added dropwise to the reaction mixture. The reaction mixture was then allowed to sit at 0°C for 1 hour, then the precipitate filtered off. The solids were washed with hydrochloric acid, and dried in a vacuum desiccator. The final yield was 10 g (133%) of brown powder.

Synthesis of skatole



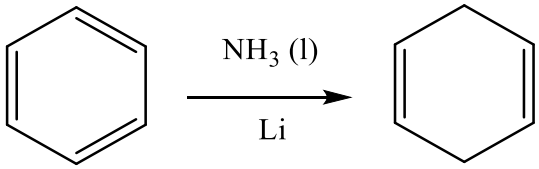
To a 25 mL round bottomed flask were added phenylhydrazine (0.5 g, 4.6 mmol), anhydrous ethanol (10 ml), and dropwise propionaldehyde (0.291 g, 5 mmol). The reaction flask was then allowed to stir at room temperature for 10 minutes, after which p-toluenesulphonic acid (0.761 g, 4 mmol) was added, and refluxed for 2-3 hours. The solvent was then removed, and water (50 mL) used to transfer the solids to a separatory funnel, along with dichloromethane (75 mL), and the organic layer removed. The aqueous layer was washed with dichloromethane (2 x 75 mL). The combined organic extracts were washed with saturated sodium bicarbonate solution (100 mL), and brine (100 ml), then dried with anhydrous magnesium sulphate. The solvent was removed, and the dark brown oil obtained (0.58 g) was redissolved in ethanol. Water was then added to precipitate the skatole, and the tan precipitate filtered off.

Synthesis of benzocaine



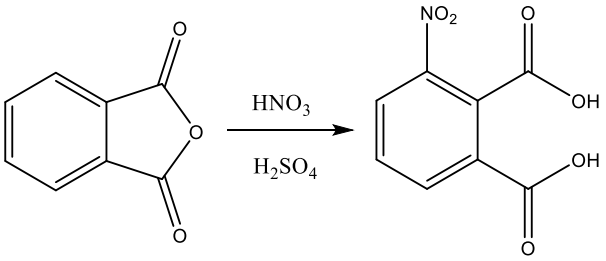
To a 500 mL round bottom flask were added p-aminobenzoic acid (20 g, mmol), anhydrous ethanol (200 mL). Sulphuric acid (25 mL, 18.4 mol dm^{-3}) was added slowly, and then the reaction mixture was refluxed for 90 minutes. Saturated sodium bicarbonate solution (50 mL) was then added slowly to achieve a pH of 8. The precipitate of benzocaine was then filtered off, and washed with water. The final yield was of 15 g (67%) of fine white powder.

Synthesis of 1,4-cyclohexadiene



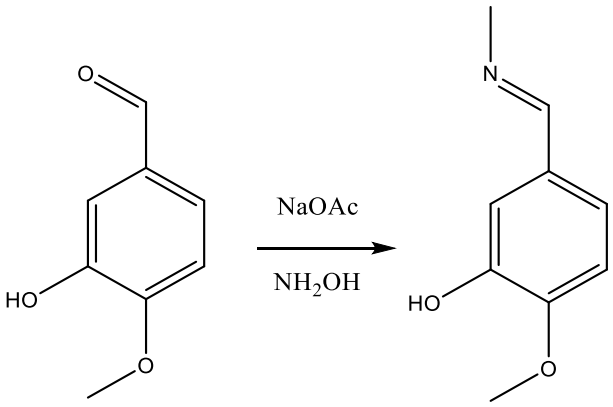
To a 25 mL round bottom flask cooled in a dry-ice acetone bath was added liquid ammonia (17 mL), an ethanolic solution of benzene (1 ml in 1.6 ml ethanol). To the stirred solution was added lithium (0.21 g) in small portions of 0.04 g. Once a persistent blue colour was achieved, the mixture was stirred for 1 hour at -78°C, then the dry-ice acetone bath was removed to allow the ammonia to evaporate. To the crude mixture was added water (20 mL), and stirred for 15 minutes. The mixture was then extracted with diethyl ether (3 x 5 mL), and the combined organic extracts washed with brine (25 mL), then dried with anhydrous magnesium sulphate. The solvent was removed, and the product collected as a yellow oil. The final yield was 0.35 g (39%), with a boiling point of 84°C (lit. 82°C).

Synthesis of 3-nitrophthalic acid



To 250 mL Erlenmeyer flask were added phthalic anhydride (30g, mmol), nitric acid (28.4 mL, g, mmol, 15.7 mol dm^{-3}), and slowly sulphuric acid (28.4 mL, g, 18.4 mol dm^{-3}). The reaction mixture was then heated using a water bath to 100°C for 2 hours. The reaction mixture was then allowed to cool to room temperature, and poured into distilled water (75 mL). The flask was rinsed with water (25 mL). The reaction mixture was then chilled in a freezer to 0°C, then the precipitate was filtered off, and washed with water. The product was then recrystallized from water to yield 12.81 g (30%) of soft white powder.

Synthesis of vanillyl oxime



Into a 250 mL round bottomed flask were placed vanillin (9.8 g, 64.4 mmol), anhydrous sodium acetate (10.55 g, 128 mmol), hydroxylamine hydrochloride (5.0 g, 72.0 mmol), and distilled water (90 mL). The reaction mixture was the refluxed with stirring for 20 minutes. The reaction mixture was then allowed to cool to room temperature, then chilled in an ice bath. The white crystals are then filtered off, and washed with ice-cold water, then allowed to dry. The final yield was 8.85 g (82%) of white flaky crystals melting at °C (lit. 118-122°C)[2]

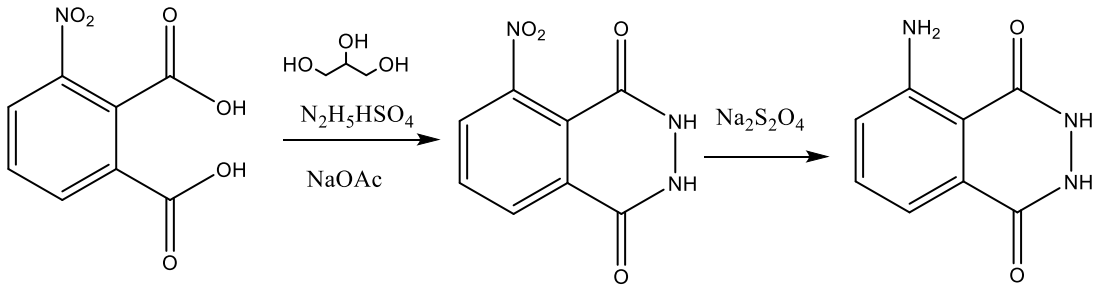
Synthesis of hydrazine sulphate

$NaOCl + CO(NH_2)_2 \longrightarrow N_2H_4 + CO_2 + NaCl$

$N_2H_4 + H_2SO_4 \longrightarrow N_2H_5{\cdot}HSO_4$

To a 1000 mL beaker were added sodium hypochlorite solution (500 mL, mmol, 5%), and was chilled in an ice bath to 8°C. Sodium hydroxide (32 g, mmol) was then added slowly, keeping the temperature below 20°C. To a small beaker was added hot distilled water (15 mL) and gelatin (0.75 g), and stirred to dissolve. Urea (22 g, mmol) was then dissolved in water (30 mL), and the gelatin solution was added to the urea solution. The urea solution was then rapidly added to the very vigorously stirred solution of sodium hypochlorite, after removal from the ice bath. After the foam had died down, the solution was heated to 85°C for 5 minutes to expel carbon dioxide, with a cover to minimize hydrazine loss. The solution was then cooled to 0°C in an ice-water bath, and ice-cold sulphuric acid (60 mL, 9.2 mol dm^{-3}) was added slowly, followed by a rapid additional volume of 40 mL. The solution was then cooled to 15°C to precipitate as much hydrazine sulphate as possible, then filtered. Washing with water was not conducted due to considerable potential losses. The final yield was 20.25 g (48%) of a white powder.

Synthesis of luminol



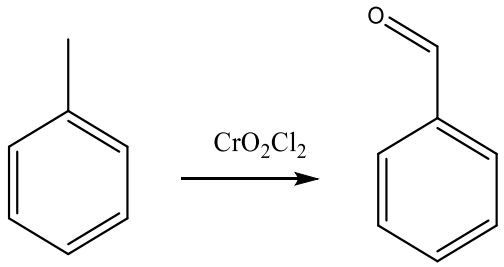
Synthesis of 4-nitrophthalhydrazide

To a 150 mL beaker were added 3-nitrophthalic acid (5 g, mmol), hydrazine sulphate (3.5 g, mmol), sodium acetate trihydrate (5 g, mmol), distilled water (20 mL), then stirred to dissolve. The reaction mixture was then brought to a boil with strong stirring, and glycerol (25 mL) was then added. The beaker was then heated until the temperature reached 200°C, at which point it was held at 200-225°C for 5 minutes. It was then allowed to cool, and water (80 mL) was added to dissolve water-soluble byproducts. The precipitate was then filtered off, and washed with water.

Synthesis of 4-aminophthalhydrazide

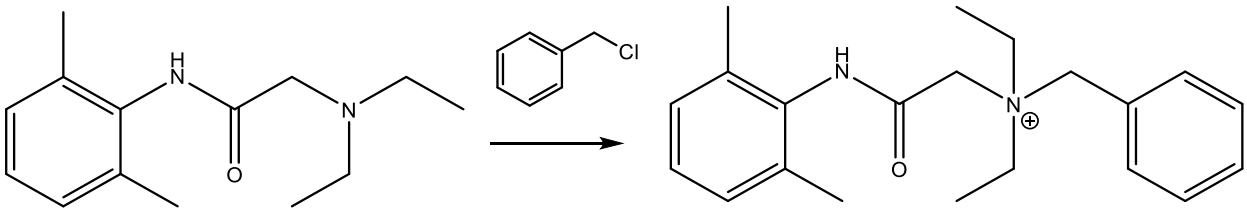
The solids were then added to sodium hydroxide solution (50 mL, 2.5 mol dm^{-3}). Sodium dithionate was then added (15 g, mmol), then heated to boiling for 5 minutes. Glacial acetic acid (25 mL, mmol) was then added to neutralise the solution. The precipitate was the filtered off, and washed with a little water, then ethanol. The final yield was 0.5 g (%) of light brown powder.

Synthesis of benzaldehyde



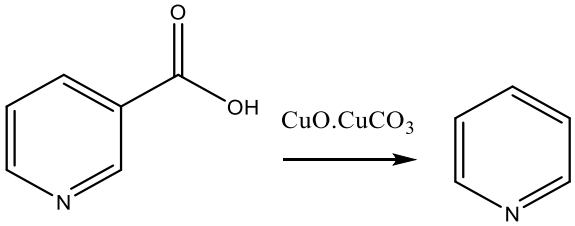
To a 1000 mL three-necked round bottomed flask fitted with an addition funnel, a condenser fitted with a calcium chloride drying tube and a thermometer was added toluene (9.2 g, mmol) and dichloromethane (96 mL) and to the addition funnel, dichloromethane (50 mL) and chromyl chloride (34 g, 110 mmol). The flask was cooled in an ice bath, and the chromyl chloride solution added dropwise with strong stirring over a period of 1 hour. After this time, dichloromethane (25 mL) and chromyl chloride (17 g, mmol) was added to the addition funnel, and added dropwise to the reaction mixture over a period of 1 hour. After complete addition, the reaction mixture was allowed to stir in the ice bath for 1 hour, then at room temperature for 2 hours. The reaction mixture was then poured into saturated sodium sulphite solution (500 mL) and ice (200 g), and the flask rinsed with saturated sodium sulphite solution. The lower organic layer was separated, and the aqueous layer washed with dichloromethane (3x 50 mL). The combined organic layers were washed with saturated sodium bicarbonate solution (50 mL), water (5x 200 mL), and brine (200 mL), then dried over molecular sieves (3A). The solvent was removed, and the residue passed through a small plug of silica gel with the aid of dichloromethane, and the solvent removed again. The final yield was 5.4 g (51%) of yellow liquid.

Synthesis of denatonium benzoate



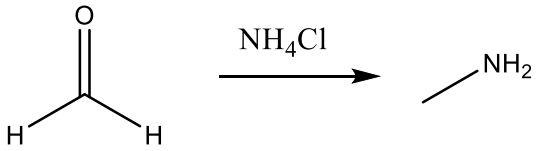
To a 25 mL round bottomed flask were added lidocaine (2.5 g, mmol) and distilled water (6 mL), and heated to 80°C with an oil bath. Then, benzyl chloride (mL, 1.76 g, mmol) was added, and held at 80°C for 24 hours. After allowing to cool to room temperature, toluene (2x 5 mL) was used to extract the aqueous layer. The toluene extracts were washed with water, and the aqueous layers combined, and sodium hydroxide solution (1 mL, 12.5 mol dm^{-3}) added. Water (25 mL) was then added to the reaction mixture, and the solids filtered off. Benzoic acid (1 g, mmol) dissolved in acetone was then added to an acetone suspension of the solids filtered of earlier. The solution was concentrated, and the precipitate filtered off. The final yield was 0.6 g of white powder.

Synthesis of pyridine



To a 250 mL round bottomed flask was added an intimate mixture of niacin (50 g, mmol) and basic copper carbonate (12 g), then the reaction mixture was dry distilled at 250°C until no more distillate collected. To the distillate was added sodium hydroxide (3 g), and stirred until all the solids had reacted, the filtered. The filtrate was then distilled, collecting the fraction at 110-115°C. The final yield of pyridine was 15 mL (46%) of clear colourless liquid.

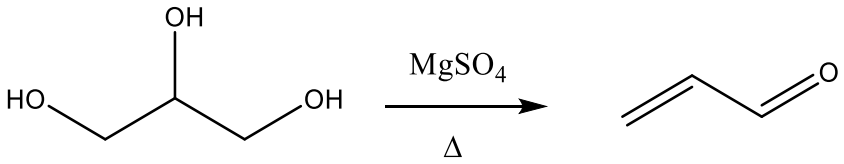
Synthesis of methylamine



To a 1000 mL round bottomed flask was added ammonium chloride (250 g, mol) and formaldehyde solution (500 g, 37% aqueous), then heated at 104°C for 3 hours in a distillation apparatus. A vacuum was then applied to the apparatus, and heated at 104°C until ammonium chloride begins to crystallise. The reaction mixture was then allowed to cool to 0°C, and the precipitate filtered off, and washed with the minimum of water. The procedure was repeated until the temperature of the boiling flask reaches 160°C, then allowed to cool slightly, and poured into a beaker. The flask was then washed with methanol (200 mL), and used to dissolve the solids with heating, then the precipitate filtered off, then washed with warm methanol (2x 50 mL). The filtrate was allowed to cool to 0°C for 24 hours, and the crystals filtered off, and washed with cold methanol and dichloromethane (50 mL).

The filtrate was then concentrated, and the crystals filtered off to obtain a second batch. Both batches were then dried under vacuum, to give a final yield of 50 g (44%) of white crystals.

Synthesis of acrolein



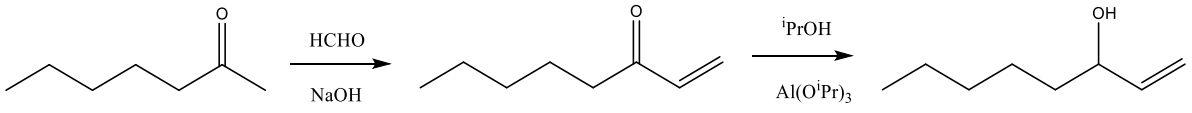
To a 1000 mL flask flask fitted with an addition funnel set up for distillation directly into a two-neck round bottom was added glycerol (67 g, mol) and anhydrous magnesium sulphate (54 g, mol). Glycerol (100 g, mol) was then added to the addition funnel, and sodium chloride (50 g, mol) to the collection flask. This was also set up for distillation, and the whole apparatus covered with aluminium foil. The central flask was heated to 110°C, and the reaction flask was then heated, and glycerol added from the addition funnel at the same rate as distillate collected in the final collection flask. After distillate stopped collecting, the distillation was stopped, and any water present was removed from the distillate, then sodium bicarbonate (5 g) added to remove any acidic species present. The distillate was filtered, and hydroquinone (0.1 g, mol%) added, then redistilled, collecting the fraction at 50-55°C. To the distillate was added hydroquinone (1.0 g, mol%), and comprised 25 g (25%) of clear colourless liquid.

Synthesis of aluminium *iso*-propoxide

$Al + 3^{i}PrOH \rightarrow Al(O^{i}Pr)_3$

To a 250 mL three-neck round bottomed flask equipped with an addition funnel and a condenser was added aluminium foil (27 g, 1 mol), iodine (0.2 g), and anhydrous iso-propanol (260 mL, mol). A solution of mercuric chloride (1.65 g) and anhydrous isopropanol (40 mL, mol) was made up, and added to the addition funnel. This was then drained into the reaction flask. The reaction mixture was the refluxed until all the aluminium had been consumed. The isopropanol was then removed, and a vacuum distillation carried out to isolate the aluminium iso-propoxide at 135°C / 10 mmHg. The final yield was 189 g (93%) of a clear colourless liquid, which eventually solidified (supercooled) to a white solid.

Synthesis of 1-octen-3-ol



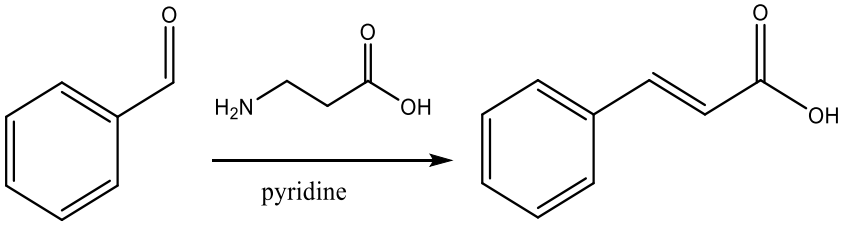
Synthesis of pentyl vinyl ketone

To an addition funnel attached to a round bottomed flask was added water (10 mL), formaldehyde (20 mL, 37%), and sodium hydroxide solution (10 mL, 2.5 mol dm^{-3}). To the round bottomed flask was added 2-heptanone (30 mL, 24 g, mmol). The mixture in the addition funnel was added dropwise to the round bottomed flask, and allowed to stir at room temperature for 5 hours. The reaction mixture was then neutralised with hydrochloric acid, and the lower aqueous layer separated, and extracted with toluene (3 x 15 mL). The combined organic extracts were dried with brine (25 mL), and anhydrous magnesium sulphate. The solvent was removed, and the 1-octen-3-one was collected by vacuum distillation at 60-85°C / 15 mmHg.

Synthesis of 1-octen-3-ol

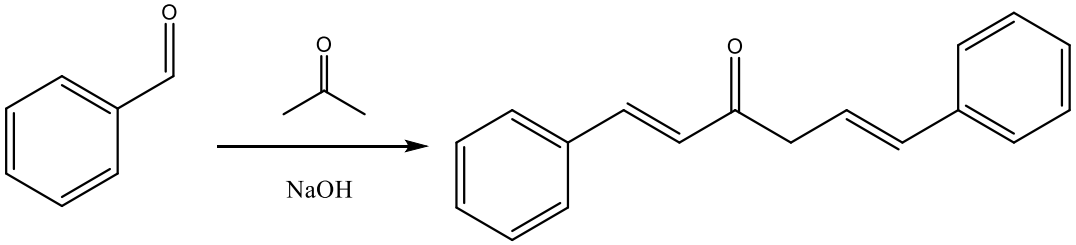
The distillate was then dissolved in anhydrous isopropanol (150 mL), and aluminium iso-propoxide (3.1 g) dissolve in isopropanol (15 mL) was added. A fractional distillation was then carried out to distil acetone out of the flask. The reaction was judged complete when the distillate no longer tested positive for acetone. To the reaction flask was added sulphuric acid (50 mL, 3.7 mol dm^{-3}), and the organic layer separated and washed with distilled water (20 mL) and dried with anhydrous magnesium sulphate. The organic layer was then distilled, and the product collected in the third fraction. The final crude yield was 0.5 g (2%) of clear colourless liquid.

Synthesis of trans-cinnamic acid



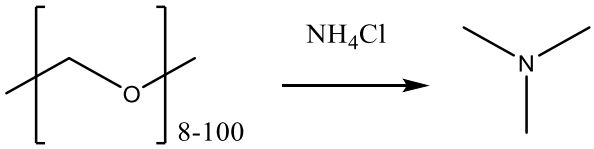
To a 250 mL round bottomed flask were added (25 g, mmol), β-alanine (2 g, mmol), benzaldehyde (10 mL, mmol) and pyridine (50 mL). The reaction mixture was refluxed for 1.5 hours. After cooling to room temperature, the reaction mixture is poured into ice-water (200 mL). Then hydrochloric acid (150 mL, 6 mol dm^{-3}) is slowly added, and the precipitate filtered off, and washed with water. The crude white solid was then recrystallized from water to yield 12.6 g (87%) of flaky white crystals melting at 125°C (lit. 133°C).

Synthesis of dibenzalacetone



To a 1000 mL beaker were added benzaldehyde (32 mL, mmol), ethanol (250 mL), sodium hydroxide solution (150 mL, 3 mol dm^{-3}), and acetone (10 mL). The reaction mixture was stirred at room temperature for 30 minutes, then water (200 mL) was added, and allowed to stir for 30 minutes. The precipitate was filtered off, and recrystallized from ethanol to yield 22.3 g (70%) of yellow crystals melting at 109-111°C (lit. 112-114°C).

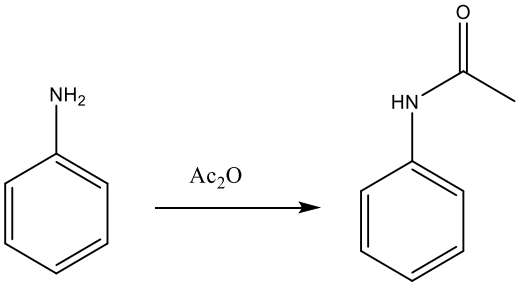
Synthesis of trimethylamine hydrochloride



To a 1000 mL two neck round bottomed flask fitted with a reflux condenser was added paraformaldehyde (185.7 g, mol), and ammonium chloride (71.5 g, mol). The reaction mixture was then heated to 105°C, and held at that temperature for 1.5 hours. The reaction was sufficiently exothermic for external heat to be unnecessary. The reaction mixture was then heated to 160°C for 30 minutes. The flask was then set up for distillation, and an addition funnel with sodium hydroxide solution (240 mL, 16.7 mol dm^{-3}) added. A hydrochloric acid bubbler was used to trap the trimethylamine as the hydrochloride. The sodium hydroxide was added dropwise, and the gas collected as hydrochloride in the bubbler. The solution in the bubbler was then evaporated until crystals began to form, then allowed to cool to room temperature, then chilled in an ice bath overnight. The crystals were then filtered off. The filtrate was evaporated further to yield a second batch of product. The product was dried in a vacuum desiccator over concentrated sulphuric acid to yield 113g (89%) of very deliquescent white crystals.

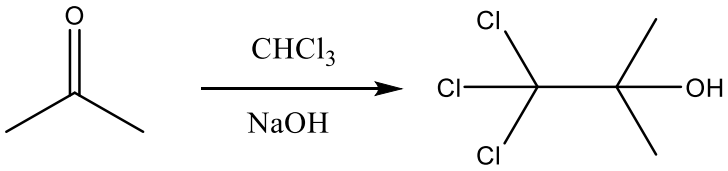
http://www.orgsyn.org/demo.aspx?prep=CV1P0531

Synthesis of acetanilide



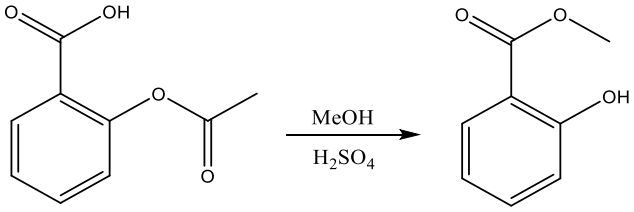
To a 1000 mL beaker was added water (600 mL) and hydrochloric acid (21 mL, 31.45%). Aniline (20 g) was then added slowly and stirred until homogeneous. Acetic anhydride (25 mL) was then added and stirred until homogenous. Sodium acetate trihydrate (35.2 g) dissolved in water (125 mL) was then added then chilled to 0°C, and the precipitate filtered off and washed with water. The product was then recrystallised from water (600 mL). The final yield was 21.4 g (74%).

Synthesis of chlorobutanol



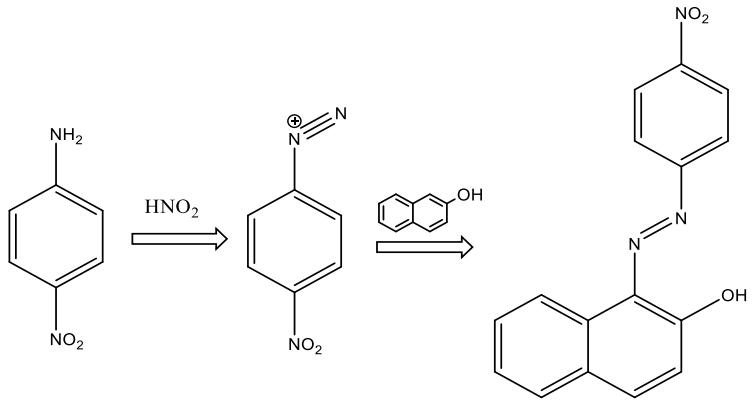
To a 100 mL Erlenmeyer flask chilled in an ice bath was added acetone (80 mL), and chilled to 0°C. Chloroform (10 mL) was then added and allowed to cool back to 0°C. Potassium hydroxide (2 g) was then added slowly and stirred for an hour. The precipitate was then filtered off, and the filtrate evaporated at 90°C. The hot melt was then quenched in ice-water (75 mL), and the precipitate filtered off. It is not recommended to recrystallise the product due to its tendency to form an oil in preference to crystals. If desired however, it can be crystallised from isopropanol/water. The final product was dried in a vacuum desiccator. The final yield was 8 g (36%), with losses due to recrystallisation and volatilisation under vacuum.

Synthesis of methyl salicylate



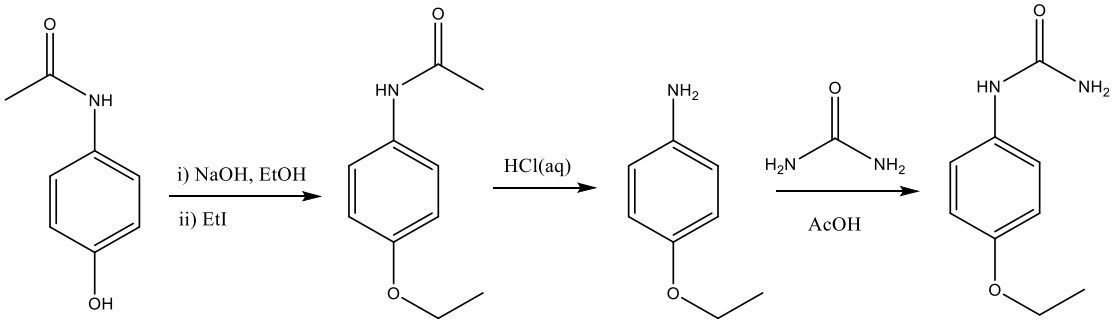
To a 500 mL round bottomed flask was added methanol (190 mL), and acetylsalicylic acid (25 g) and stirred to dissolve. Sulphuric acid (25 mL, 98%) was then added slowly and the reaction mixture refluxed for 1 hour. The volume was reduced by half and poured into ice-water (100 mL). The lower organic layer was separated, and washed with saturated sodium bicarbonate solution (2x 50 mL), and dried over anhydrous magnesium sulphate. The final yield was 4.85 g (24%).

Synthesis of para red



To 100 mL beaker was added water (50 mL), sulphuric acid (5 mL, 98%), and p-nitroaniline (5 g) and stirred and cooled to 5°C. Sodium nitrite solution (2.7 g in 10 mL water) was then added slowly. 2-naphthol (0.5 g) was then dissolved in hot water (100 mL) with the aid of sodium hydroxide, then chilled to 5°C. The two solutions were then combined, and dilute sulphuric acid added until pH 1-2. The precipitate filtered off and washed with ice water, then allowed to dry.

Synthesis of dulcin



Synthesis of phenacetin

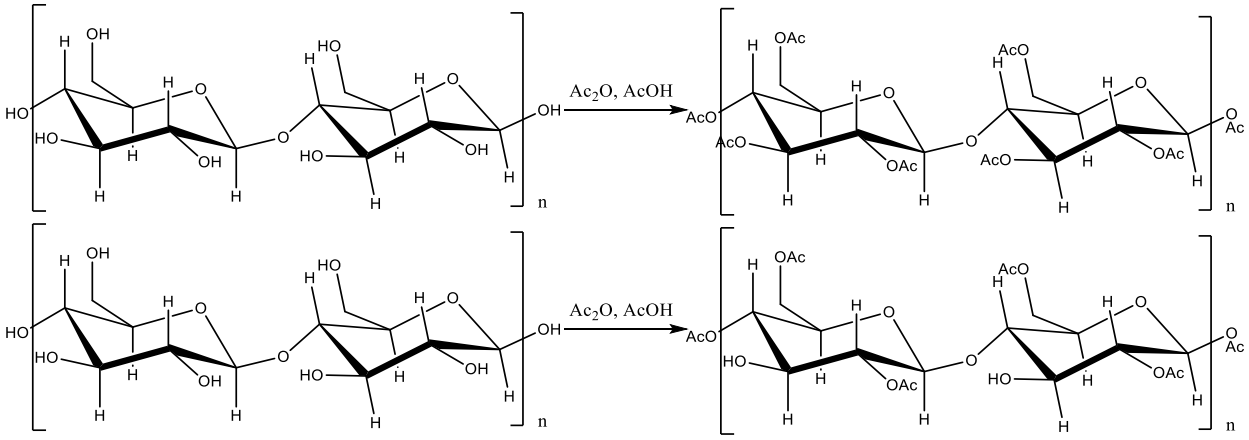
To a 500 mL round bottomed flask was added N-acetyl-4-aminophenol (12.5 g) and sodium hydroxide (7.4 g) dissolved in ethanol (190 mL), then refluxed for 20 minutes. Ethyl iodide (25 mL) was then added and refluxed for a further 20 minutes. The reaction mixture was then poured into water (400 mL) and the precipitate filtered off. The product was then recrystallised from ethanol/water. The final yield of the intermediate phenacetin was 11 g.

Synthesis of dulcin

To a 100 mL round bottomed flask was added phenacetin (5 g), hydrochloric acid (25 mL, 6 M) and the reaction mixture refluxed for 15 minutes. After cooling, sodium bicarbonate was added until pH 6-6.5. Urea (6.7 g) and acetic acid (20 drops) were then added and refluxed for 1 hour. The reaction mixture was allowed to cool to -10°C, and the precipitate filtered off. The product was then recrystallised twice from water (250 mL). The final yield was 2.2 g (44%) melting at 169-144°C (lit. 174°C).

J. Chem. Educ., 2000, *77* (3), p 357
DOI: 10.1021/ed077p357

Synthesis of cellulose acetate



To a 250 mL Erlenmeyer flask was added filter paper (10 g), glacial acetic acid (50 mL), sulphuric acid (0.5 g, 98%), then mixed thoroughly and allowed to sit for 1 hour. Acetic anhydride (50 mL) and glacial acetic acid (20 mL) were then added and heated in a hot water bath to 50°C for 30 minutes.

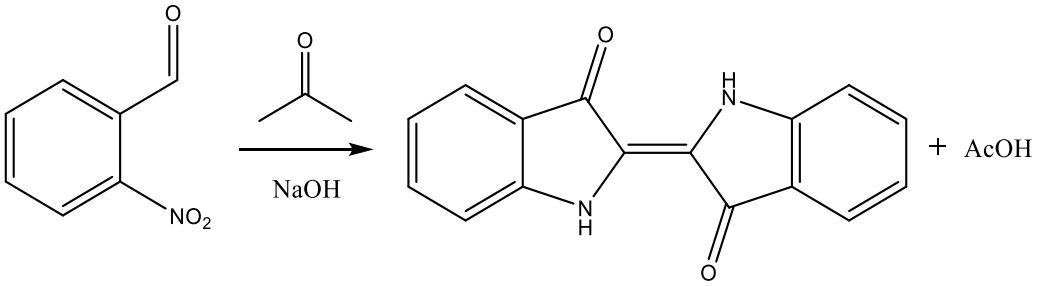
Synthesis of cellulose triacetate

Half of the resulting solution was then heated to 60°C in a hot water bath, and acetic acid (25 mL, 80%) and allowed to stir for 15 minutes. Water (25 mL) was then added with strong stirring, followed by a further 200 mL. The precipitate was filtered off and washed with water until the pH was neutral. The final yield of cellulose triacetate after drying 6.37 g.

Synthesis of cellulose diacetate

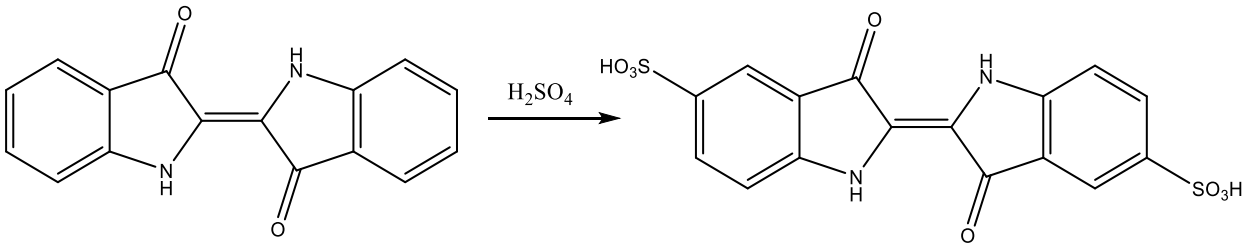
The other half of the solution was then heated to 60°C, and acetic acid (50 mL, 70%) and sulphuric acid (0.14 g, 98%) was added. Water (25 mL) was then added with strong stirring, followed by a further 200 mL. The precipitate was filtered off and washed with water until the pH was neutral. The final yield after drying was 5 g.

Synthesis of indigo



To a 1000 mL beaker was added 2-nitrobenzaldehyde (25 g) dissolved in a mixture of acetone (250 mL) and water (250 mL), followed slowly by sodium hydroxide (5 g) dissolved in water (125 mL). The precipitate was filtered off and washed with water, ethanol, and diethyl ether. The final yield was 11 g (51%).

Synthesis of indigo carmine



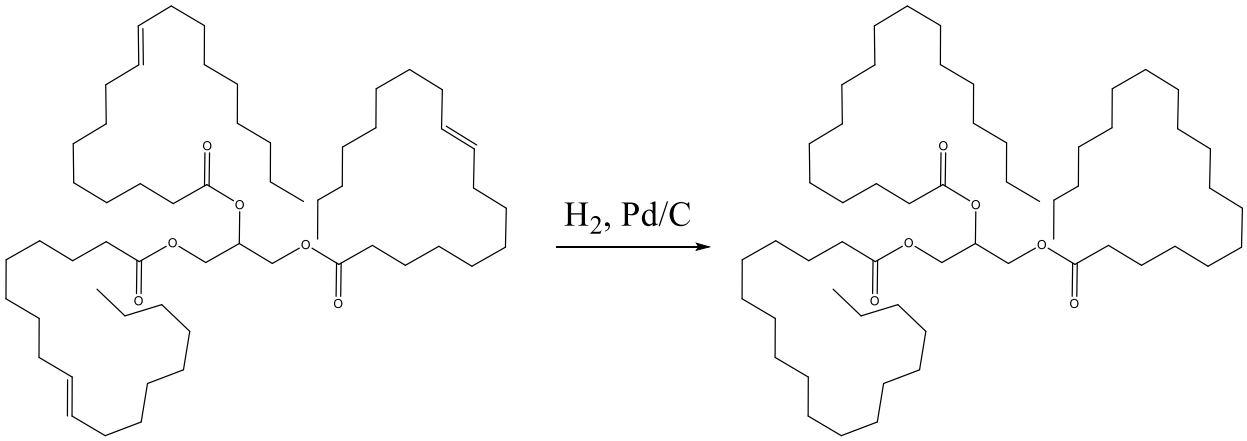
To a 50 mL beaker was added indigo (2.5 g) and sulphuric acid (30 mL, 98%) and stirred at 90°C for 30 minutes. The hot solution was then poured into cold water (150 mL), and any solids precipitated off. To the filtrate was added sodium chloride (30 g) and allowed to sit for 12 hours. The precipitate was gravity filtered off and washed with water. The crude yield was 2.8 g (63%). The product was the recrystallised from water/isopropanol as antisolvent (1:1, but 1:4 recommended). The final yield was 0.9 g (21%).

Synthesis of fuming nitric acid

$KNO_3 + H_2SO_4 \rightarrow HNO_3 + KHSO_4$

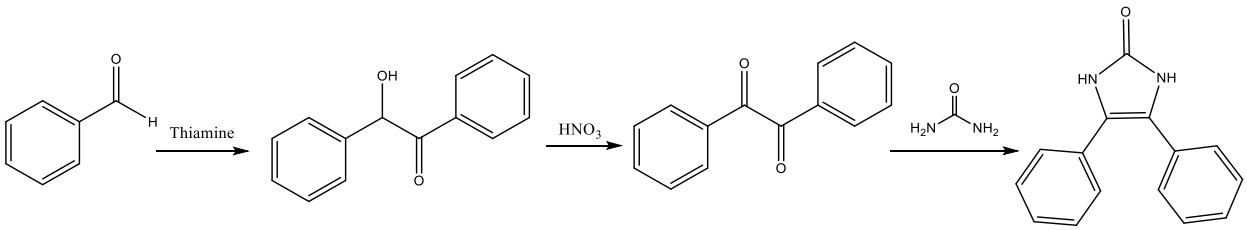
To a 500 mL round bottomed flask was added dry potassium nitrate (110 g) and sulphuric acid (60 mL, 98%), and the reaction mixture distilled. The final yield was 28.5 mL, 43.1 g with a density of 1.51 g/mL or 99%.

Synthesis of margarine



A hydrogen generator was set up by using aluminium foil and concentrated sodium hydroxide (~50%) solution. The gas was dried over calcium chloride and collected in a balloon. To 500 mL round bottomed flask was added olive oil (15 g), methanol (200 mL), and palladium on carbon (350 mg). The reaction flask was evacuated and replaced with hydrogen three times. The hydrogen atmosphere was maintained until it appeared that hydrogen was only leaving the balloon through diffusion out of the equipment. The reaction mixture was then heated to dissolve the fats that had precipitated, and the solution filtered through celite and washed with hot methanol. The solution was then chilled to 0°C for 24 hours, and the precipitate filtered off. The product was melted and heated to 90°C to remove all traces of solvent. The final yield was 13 g (86% assuming solely glyceryl trioleate originally).
The product was combined with an equal volume of olive oil, water, and a little lecithin to form the final margarine.

Synthesis of phenytoin



Synthesis of benzoin

A solution of sodium hydroxide (0.84 g) in water (7 mL) and a solution of thiamine hydrochloride (3.5 g) in water (8 mL) and ethanol (35 mL, 95%) were combined with strong stirring after being cooled to 0°C. This was allowed to stir for 5 minutes, then benzaldehyde (20 mL) was added. It was ensured the pH was 8-9, then warmed to 60°C for 90 minutes then allowed to cool to 0°C. The precipitate was filtered off and recrystallised from ethanol. The final yield of benzoin was 4.95 g (24%).

Synthesis of benzil

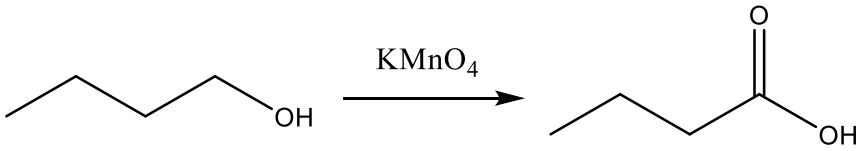
To a 50 mL beaker was added nitric acid (17 mL, 68%) and benzoin (4.95 g), then stirred and heated to 90°C for 1 hour. Ice-water (50 mL) was then added and cooled to 0°C and the precipitate filtered off. The product was recrystallised from ethanol and water, yielding 4.2 g (86%) of benzil.

Synthesis of dulcin

To a 100 mL round bottomed flask was added benzil (2.0 g), urea (0.96 g), ethanol (50 mL, 95%), potassium hydroxide solution (3.2 g) in water (6 mL) and refluxed for 2 hours. The reaction mixture was then poured into ice-water (50 mL) and stirred briefly before being filtered. Dilute hydrochloric acid was then added until pH 1-2 was reached, then cooled to 0°C. The precipitate was filtered off and washed with ice-water. The product was recrystallised from ethanol (40 mL) yielding 1.18 g (49%).

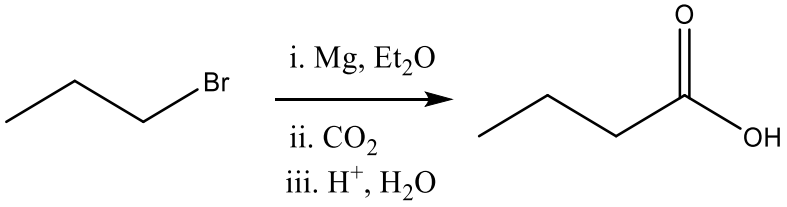
Synthesis of butyric acid

Method 1 – Oxidation



To a 1000 mL flask was added sodium carbonate (1.5 g) dissolved in water (150 mL) and n-butanol (6.2 mL). Potassium permanganate (14 g) dissolved in water (275 mL) was then added to an addition funnel, and dripped into the reaction mixture with strong stirring over a period of 3 hours. After complete addition, the reaction mixture was cooled 0°C overnight. Hydrogen peroxide (90 mL, 3%) was then added slowly with stirring, and the reaction mixture filtered and washed twice with water (100 mL). The filtrate was concentrated to 20 mL and sulphuric acid (2M) used to acidify the solution to pH 0, which was then extracted five times with diethyl ether (5 mL). The combined organic layers were dried over anhydrous magnesium sulphate, and the solvent removed. The final yield was 3.75 mL (60%).

Method 2 – Grignard Reaction



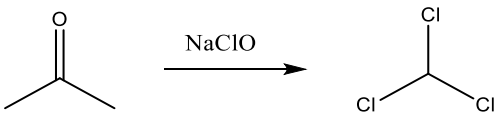
To a dry 100 mL three-necked round bottomed flask fitted with a condenser was added magnesium metal (1.5 g), and dry diethyl ether (15 mL). Dry bromopropane (5.0 mL) was then added slowly until the reaction started, then the remainder was added dropwise. Ether was added as necessary to maintain a constant volume. The reaction mixture was allowed to cool to room temperature, and a carbon dioxide generator set up using dry ice. Carbon dioxide was then passed into the reaction mixture, topping up the ether as necessary. The reaction mixture was then poured into hydrochloric acid (60 mL, 3 M). When effervescence had ceased, the reaction mixture was filtered to remove excess magnesium and washed with ether (50 mL). The lower aqueous layer was removed, and organic layer washed with water (30 mL), and sodium hydroxide (2x 20 mL, 3 M), and water (15 mL) retaining the aqueous layer each time for the last three washings. The aqueous layers were then reacidified with hydrochloric acid (50 mL, 3 M), and extracted three times with diethyl ether (30 mL). The combined organic layers were then washed twice with brine (50 mL), and dried over anhydrous sodium sulphate. The solvent was then removed.

Synthesis of diethyl ether

$2\ EtOH \rightarrow Et_2O + H_2O$

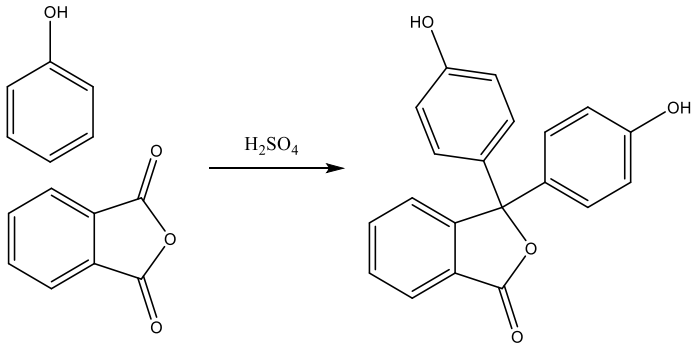
To a 500 mL three necked round bottomed flask cooled in an ice bath was added absolute ethanol (90 mL) and allowed to cool to 5°C. Sulphuric acid (80 mL, 98%) was then added dropwise from an addition funnel over a period of 15 minutes. Sand (20 g) was then added, and the apparatus set up for fractional distillation. The reaction mixture was heated to 140-145°C, and ethanol (230 mL) added at the same rate as distillate was collected. The distillate was washed twice with saturated sodium bicarbonate (50 mL) and once with brine (50 mL) and dried over anhydrous calcium chloride for 1 hour. The ether was then distilled at 50°C to dryness without removing the calcium chloride. The final yield was 208 mL (~70%). Sodium or potassium hydroxide can be used to minimise peroxide formation.

Synthesis of chloroform



The concentration of bleach was determined by reacting a small portion of the bleach (5 mL) with hydrogen peroxide (3%) and collecting the gas produced. To a jug of bleach (3.6 L, 1.28 M, 8.6%) cooled to -2°C and acetone (100 mL) was added. This represents an 11% excess of bleach, however 3-5% is preferable. The jug was shaken and allowed to react in a cool place and separate. The upper aqueous layer was decanted, and the lower organic layer separated. The product was washed once with brine, then dried over calcium chloride and distilled collecting the fraction at 60°C. The final yield was 58 mL (53%).

Synthesis of phenolphthalein



To a 25 mL round bottomed flask was added phenol (2.0 g), phthalic anhydride (1.5 g), sulphuric acid (1 mL, 98%) and then heated to 150°C for 2 hours. Upon cooling to room temperature, water (10 mL) and dichloromethane (10 mL) was added and stirred rapidly. The lower organic layer was separated, and the aqueous layer washed with dichloromethane (10 mL). The combined organic layers were washed with sodium hydroxide (5 mL, 2 M) and water (10 mL) added. The upper organic layer was separated and added to hydrochloric acid (200 mL, 2 M) and the precipitate filtered off. The final yield was 1 g.

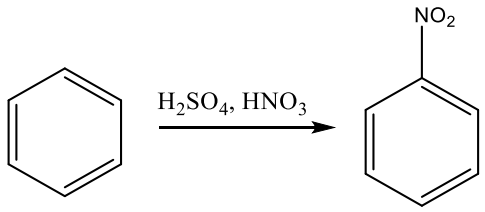
Preparation of bromine

$TCCA + 3\ HCl \rightarrow 3\ Cl_2 + ICA$

$Cl_2 + 2\ NaBr \rightarrow Br_2 + 2\ NaCl$

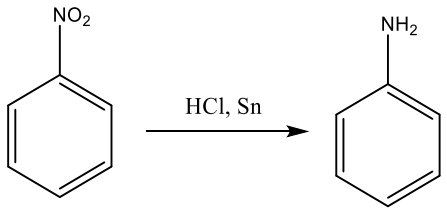
To a 1000 mL two necked round bottomed flask set up for distillation was added sodium bromide solution (400 mL, 35%) and TCCA powder (72 g), and hydrochloric acid (60 mL, 16%) to an addition funnel. Concentrated sulphuric acid was used instead of grease. With strong stirring, the hydrochloric acid was added dropwise, then the reaction mixture distilled until no more distillate collected. The lower bromine layer was separated, and washed with sulphuric acid (20 mL, 98%). The final yield was 109 g (75%). The yield could be improved by using more hydrochloric acid.

Synthesis of nitrobenzene



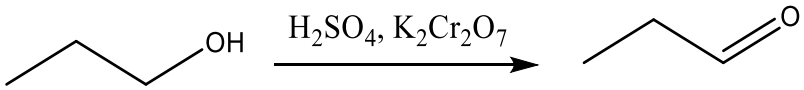
To a 100 mL round bottomed flask cooled in an ice bath was added nitric acid (35 mL, 70%), and with stirring cold sulphuric acid (40 mL, 98%). Benzene (30 mL) was then added dropwise, maintaining the temperature below 55°C. After complete addition, the solution was heated to 60°C for 1 hour, then allowed to cool to room temperature. The upper organic layer was washed twice with water (50 mL), then distilled collecting the fraction at 208°C. The distillate was dried over calcium chloride. The final yield was 20 mL (57%). Typical yields are closer to 80-90%.

Synthesis of aniline



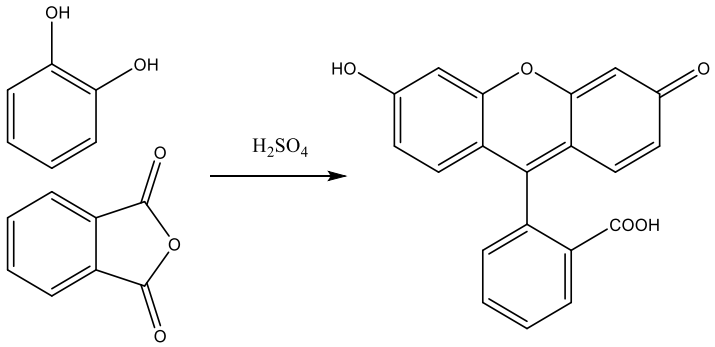
To a 250 mL round bottomed flask set up for reflux was added nitrobenzene (15 mL), granulated tin (34 g), and hydrochloric acid (75 mL, 31.45%) was added in 15 mL portions to prevent the reaction getting out of control. After complete addition, the reaction mixture was heated to 60°C for 1 hour. A solution of sodium hydroxide (60 g) in water (100 mL) was prepared. The end point of the reaction was determined by adding an aliquot of reaction mixture to water and seeing no turbidity. Once this point had been reached, the sodium hydroxide solution was added slowly with good stirring. The reaction mixture was then distilled until 100 mL of clear distillate had been collected after cloudy distillate had ceased to be collected. A total of 200 mL of distillate was collected. Sodium chloride (40 g) was then added to the distillate and stirred to dissolve. The product was extracted from the solution with dichloromethane (3x 25 mL), and the combined organic layers dried over potassium hydroxide. The solution was then distilled, collecting the fraction at 180-184°C. The final yield was 10 mL (78%).

Synthesis of propionaldehyde



To a 500 mL three necked round bottomed flask fitted with an addition funnel and a reflux condenser warmed with water at 60°C itself connected to a cold water condenser was added n-propanol (25 mL), and sulphuric acid (24 mL, 98%) and potassium dichromate (33 g) dissolved in water (200 mL) to the addition funnel. The n-propanol was brought to reflux, and then the potassium dichromate solution added dropwise. After complete addition, the distillation was continued for a further 15 minutes. The distillate was dried over 3Å molecular sieves, then fractionally distilled collecting the fraction at 48-53°C. The final yield was 11 mL (46%), which is consistent with literature (45-50%).

Synthesis of fluorescein



To a 100 mL round bottomed flask was added phthalic anhydride (2 g), resorcinol (3 g), mixed well and sulphuric acid (5 drops, 98%) added. The reaction mixture was then heated to 190-200°C for 30 minutes, then allowed to cool to room temperature. Acetone (15 mL) was then added, and stirred well to dissolve the product, and transferred to a beaker. This was repeated until all the solids had dissolved. This took about 50 mL of acetone. The solvent was then removed, and ether (40 mL) and water (4 mL) added to dissolve the product. The solvent was transferred to separatory funnel, and the process repeated thrice more. Water was used to aid dissolution when stubborn. The lower aqueous layer was separated, and washed with water (50 mL), brine (50 mL), and dried over 3Å molecular sieves and the solvent was removed. The final yield was 3 g.

Synthesis of chromyl chloride

$K_2Cr_2O_7 + 2H_2SO_4 \rightarrow 2KHSO_4 + 2CrO_3 + H_2O$

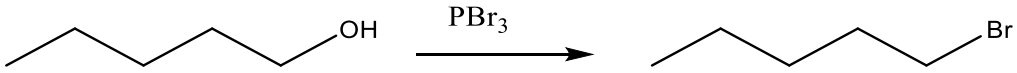
$NaCl + H_2SO_4 \rightarrow NaHSO_4 + HCl$

$CrO_3 + 2HCl \rightarrow CrO_2Cl_2 + H_2O$

To a 500 mL three necked round bottomed flask was added intimately ground dry potassium dichromate (80 g) and dry sodium chloride (50 g), and set up for distillation with an addition funnel. The joints were greased with sulphuric acid. To the addition funnel was added sulphuric acid (150 mL, 98%), and added dropwise to the reaction mixture. The reaction mixture was then distilled at 120°C. When the vapours turned yellow, the reaction was considered complete. The glassware was cleaned with sodium sulphite. The final yield was 44 g (23 mL).

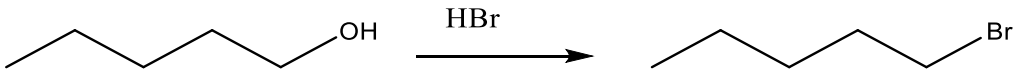
Synthesis of 1-bromopentane

Method 1 – with phosphorous tribromide



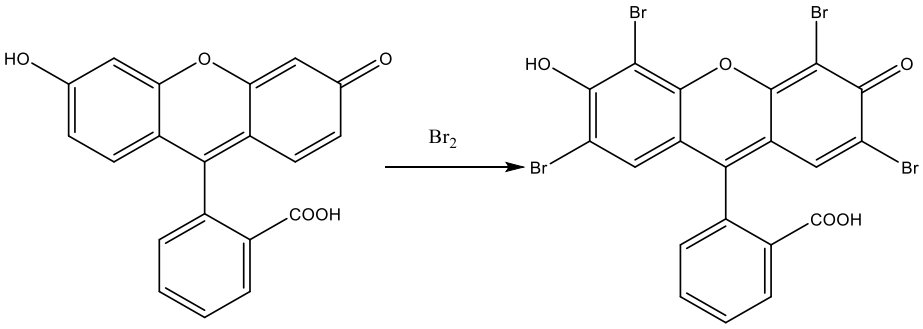
To a 100 mL round bottomed flask fitted with an addition funnel was added cold dry 1-pentanol (31 mL, -10°C) and phosphorous tribromide (9.9 mL) to the addition funnel, which was then added dropwise to the reaction mixture, maintaining the temperature below 0°C. After complete addition, the reaction mixture was allowed to warm to room temperature, then poured into water (50 mL). Brine was then added to increase the density of the aqueous layer. The lower organic layer was then separated and washed with saturated sodium bicarbonate solution (25 mL), water (25 mL), and brine (25 mL), then dried over anhydrous magnesium sulphate. The product was then distilled, collecting the fraction at 128-130°C (lit. 130°C) and dried over molecular sieves. The final yield was 14 mL (39%). Typical yields are typically 40-70%.

Method 2 – with hydrogen bromide



To a 500 mL round bottomed flask was added distilled water (75 mL) and sodium bromide (78 g) then allowed to cool to 5°C, then sulphuric acid (60 mL, 98%) was added slowly. 1-Pentanol (65 mL) was then added dropwise and the reaction mixture refluxed for 2 hours, then distilled until the still head temperature rose above 135°C. Water (100 mL) was added to the distillate and the lower organic layer separated and washed with sulphuric acid (25 mL, 98%), water (100 mL), saturated sodium bicarbonate (100 mL) then dried over anhydrous calcium chloride. The product was then fractionally distilled collecting the fraction at 125-130°C. The final yield was 60 g (66%). Typical yields are 80-90%.

Synthesis of Eosin Y (2',4',5',7'-tetrabromofluorescein)



To a 25 mL round bottomed flask was added fluorescein (2.5 g), and ethanol (15 mL), and chilled in an ice bath. Bromine (5.4 g) was then added and the reaction mixture stirred for 5 minutes, then allowed to warm to room temperature for 20 minutes. The precipitate was filtered off and washed with ethanol, then dried in an oven at 110°C. The final yield was ~3 g. The low yield was likely due to impurities in the fluorescein.

Synthesis of methyl iodide

$3\ MeOH + PI_3 \rightarrow 3\ MeI + P(OH)_3$

To a 500 mL round bottomed flask was added iodine (83.3 g) and methanol (120 g), then cooled in an ice bath. Red phosphorus (42.3 g) was then added in small portions, maintaining the temperature below 30°C. After complete addition, the reaction mixture was distilled, collecting the fraction boiling below 45°C. The distillate was washed with brine (60 mL) with a single crystal of sodium thiosulphate dissolved, then dried over calcium chloride. The final yield was 70 g, (71%). The product was stored over freshly polished copper pieces in an amber bottle.
The excess red phosphorus was filtered from the residue from the distillation, and washed with dilute sodium thiosulphate solution, water, and methanol. The recovery was 38 g.

Synthesis of ammonium metavanadate

$V_2O_5 + Na_2CO_3 \rightarrow 2\ NaVO_3 + CO_2$

$NaVO_3 + NH_4Cl \rightarrow NH_4VO_3 + NaCl$

To a 1000 mL beaker was added water (400 mL), sodium carbonate (25 g) and heated to 90°C. Vanadium pentoxide (40.8g) was then added slowly with strong stirring, then brought to a boil. Water (300 mL) was then added to dissolve the majority of the solids. Some impurities were then filtered off, and ammonium chloride (50 g) dissolved in water (75 g) is then added and the reaction mixture was allowed to cool to 0°C. The precipitate was then filtered off, and washed with ice water. The final yield was 42 g (80%).

Synthesis of lithium peroxide

$LiOH + H_2O_2 \rightarrow LiOOH + 2\ H_2O$

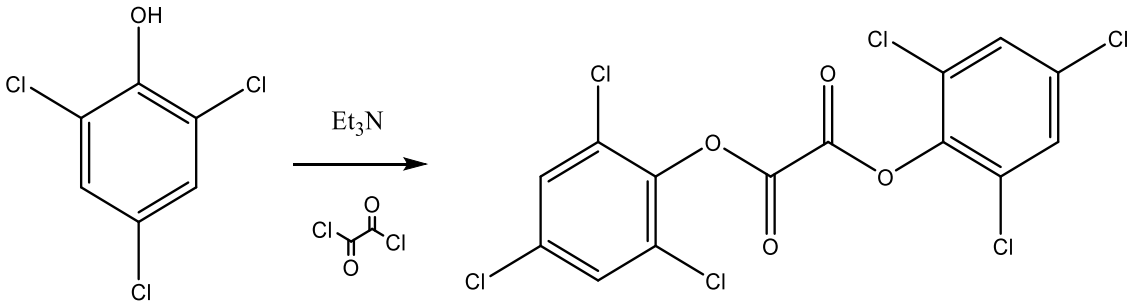
$2\ LiOOH \rightarrow Li_2O_2 + H_2O_2$

To a 1000 mL beaker was added lithium hydroxide (50 g), hydrogen peroxide (125 mL, 35%) and the temperature maintained below 60°C. Water (100 mL) was added to aid stirring. The reaction mixture was then allowed to stir for 15 minutes, and heated to 100°C then immediately filtered. The solids were then transferred to a 250 mL round bottomed flask and heated to 120°C under vacuum for 1 hour. The final yield was 25 g (52%) of off-white powder.

US patent US3446588

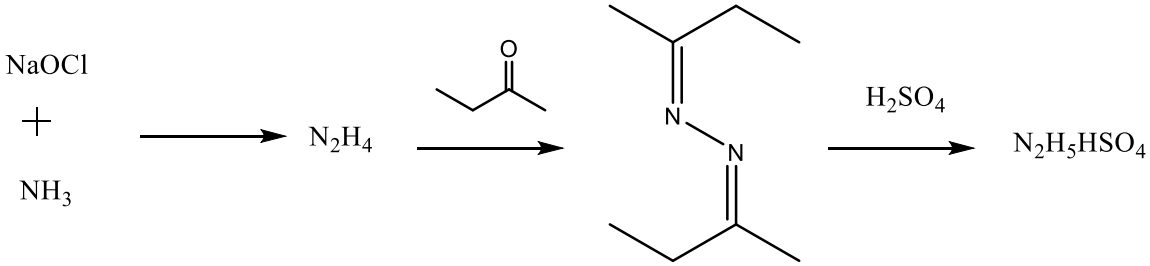
NurdRage

Synthesis of bis(2,4,6-trichlorophenyl)oxalate (TCPO)



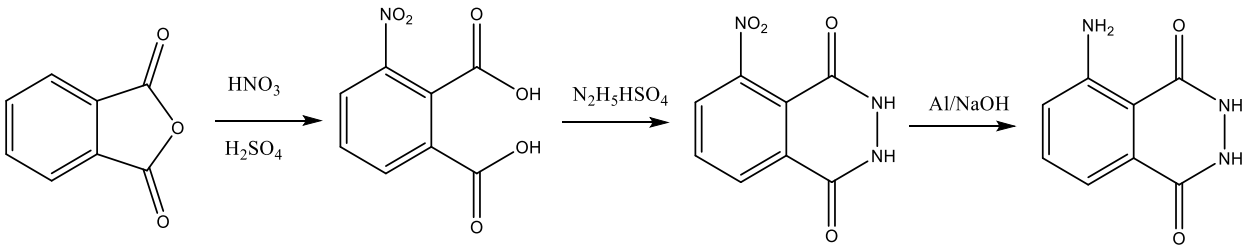
To a 10 mL Erlenmeyer flask was added 2,4,6-trichlorophenol (700 mg, 3.55 mmol) and dry toluene (10 mL). The flask was sealed with a suba-seal, and triethylamine (0.261 mL, 359 mg, 3.55 mmol) was injected into the flask, and cooled in an ice-water bath. Then oxalyl chloride (0.152 mL, 225 mg, 1.775 mmol) was added dropwise to the solution, and allowed to warm to room temperature. The precipitate was filtered off and washed with methanol (25 mL).

Synthesis of hydrazine sulphate



To a 1000 mL beaker were added ammonia solution (250 mL, mol, 30%), methyl ethyl ketone (100 mL, mol), and gelatin (250 mg), then chilled in an ice bath. To this chilled mixture, sodium hypochlorite solution (186g, 0.25 mol, 10%) was dropwise added with strong stirring, then the reaction mixture was allowed to separate into two layers. The upper organic layer was separated off, and added to sulphuric acid (120 mL, 3.7 mol dm^{-3}). The stirred reaction mixture was heated to 85°C for 1 hour, and then allowed to cool to room temperature, then chilled in an ice bath. The precipitate was then filtered off, and dried. The filtrate was reheated, and the process repeated until no more precipitate was formed. The final was g (60%) of white powder.

Synthesis of luminol



Synthesis of 4-nitrophthalic acid

To a 200 mL beaker were added sulphuric acid (45 mL, 18.4 mol dm^{-3}), phthalic anhydride (13 g, mmol), and sodium nitrate (19 g, mmol). The reaction mixture was then heated to 110°C for 1 hour. After cooling to room temperature, the reaction mixture was added to water (150 mL), and stirred vigorously, then allowed to stand overnight. The precipitate was filtered off, and dried to yield g (%) of white powder.

Synthesis of 4-aminophthalhydrazide

To a small beaker were added 3-nitrophthalic acid (1 g, mmol), hydrazine sulphate (660 mg, mmol), sodium acetate trihydrate (1.4 g, mmol), and water (1 mL). The water was evaporated, and polyethylene glycol (5 mL) added, then heated to 200-230°C for 10 minutes. After cooling to room temperature, the reaction mixture was dissolved in 100 mL of water, and sodium hydroxide (13 g, mmol), sodium metabisulphite (10 g, mmol), and water (50 mL) were added and stirred to dissolve. The reaction mixture was transferred to a 500 mL round bottomed flask, and a reflux condenser added. Aluminium foil (20 g, mmol) was added in 5 g portions, and allowed to react until effervescence had ceased. The solids were filtered off, and washed with water (50 mL). To the filtrate was added sodium bisulphate solution (25 g, mmol in 200 mL water), and acetone (100 mL), and stirred well. The solids were filtered off, and the filtrate evaporated to dryness. The residue was dissolved in the minimum of water (100 mL), and filtered.

Synthesis of triphenylphosphinebis(pyridine)thiocyanatocopper(I)

$$CuSCN + 2py + Ph_3P \longrightarrow [Cu(Ph_3P)(py)_2(SCN)]$$

To a small flask were added copper thiocyanate (121 mg, mmol), triphenylphosphine (262 mg, mmol), and pyridine (5 mL). The solution was heated to dissolve the solids, then allowed to cool to room temperature, and the solvent to evaporate until crystals formed. The supernatant was removed, and the crystals washed with toluene (3x 3 mL), then allowed to dry.
http://dx.doi.org/10.1021/ed2001494

Synthesis of europium tetrakis(dibenzoylmethide)triethylammonium nitrate

$$Eu(NO_3)_3 + Et_3N + Bz_2CH_2 \longrightarrow [Eu(Et_3N)(Bz_2CH_2)_4](NO_3)_3$$

To a 150 mL Erlenmeyer flask was added dry ethanol (100 mL), dibenzoylmethane (2.93 g, mmol), europium nitrate pentahydrate (1.4 g, mmol), and triethylamine (1.9 mL, mmol). The mixture is heated until everything dissolves and then allowed to cool slowly to obtain crystals that are filtered off and washed with ethanol.

Preparation of potassium (alcohol-catalysed magnesium-reduction process)

$2\ KOH + 2\ Mg \rightarrow 2\ K + H_2 + 2\ MgO$

To a 50 mL Erlenmeyer flask was added magnesium turnings[1] (2.4g, 0.1 mol), potassium hydroxide (5 g, 89 mmol), tetrahydronaphthalene[2] (20 mL), and the flask fitted with a reflux condenser and the top covered with aluminium foil to reduce diffusion of oxygen into the reaction mixture. The reaction mixture was heated to 200°C, then 2-methyl-2-butanol[3] (0.4 mL) was injected into the reaction mixture. Every 10 minutes,[4] a further 0.1 mL of tertiary alcohol was injected for a total of 0.6 mL. The reflux was continued until the reaction was judged complete. If the spheres of potassium formed turned dark, an additional injection of 0.1 mL of t-amyl alcohol was used to clean the product. Injections of alcohol catalyst can be used to ensure coalescence of spheres. After cooling to room temperature, the potassium metal was separated manually and cleaned by melting under paraffin oil and adding a few drops of isopropanol to clean the surface.

Additional notes:
Clean up can be achieved by carefully adding isopropanol to the waste(s). Stirring is recommended to increase rate of reaction but do so manually after cooling the reaction mixture. Magnesium can be cleaned by refluxing under the high-boiling solvent with a small amount (0.1 mL) of t-amyl alcohol.

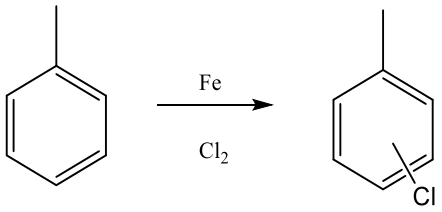
(1) Powder is better, and the reaction is faster
(2) Paraffin oil is better, since tar slowly forms. Avoid aromatics in the solvent which must boil over 200°C
(3) Any tertiary alcohol can be used, but not secondary or primary
(4) Staged addition was used to ensure reproducibility. This is not necessarily required

Synthesis of trimethyl orthoformate

$CHCl_3 + 3MeONa \longrightarrow HC(OMe)_3 + 3NaCl$

To a 500 mL Erlenmeyer flask was added methanol (200 mL, mol), sodium hydroxide (40 g, 1 mol), and stirred to dissolve. Molecular sieves (100 g, 3A) were then added, and allowed to sit for a few days until the liquid volume ceased to fall over a 24 hour period. The liquid was decanted into a 500 mL oven-dried Erlenmeyer flask, and brought to a reflux. The heating was removed, and dry chloroform (30 mL) was added slowly through the oven-dried condenser as so to maintain reflux. After complete addition, the reaction mixture was refluxed for 1 hour, then allowed to cool to $_{room}$ temperature. The precipitate was filtered off, and fractionally distilled to obtain the product at 101°C to yield 7 g (20%) of clear colourless liquid.

Synthesis of o- and p-chlorotoluene



Method 1 – Using chlorine gas

To a 1000 mL Erlenmeyer flask was added steel wool (2 g, mmol), and toluene (500 mL, mol). Dry chlorine (4 mol) was then passed through the reflux condenser connected to the flask, and into the reaction mixture. Water (500 mL) was then added, and the lower aqueous layer removed. Saturated sodium bicarbonate solution (500 mL) was then added, and the lower aqueous layer removed. The reaction mixture was then fractionally distilled, and the fraction at 160°C collected as chlorotoluene.

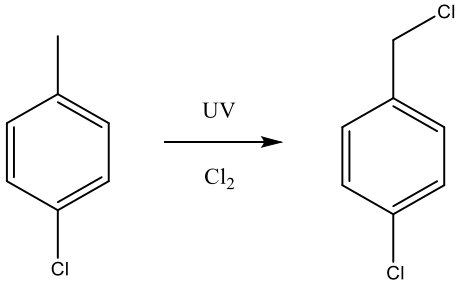
Method 2 – Using TCCA

To a 1000 mL Erlenmeyer flask fitted with a Claisen adapter and a condenser was added anhydrous ferrous chloride (125 g, mol), and dry toluene (500 mL, mol). Trichloroisocyanuric acid (160 g, mol) was then added in 10 g portions over a period of 4 hours with stirring. When reflux occurred, the reaction mixture was allowed to cool before continuing addition. The reaction mixture was then allowed to stir overnight. Water (500 mL) was then added, and the reaction mixture distilled to remove the organic material. Filtration proved ineffective due to fine particle size of the precipitate.

Separation of isomers

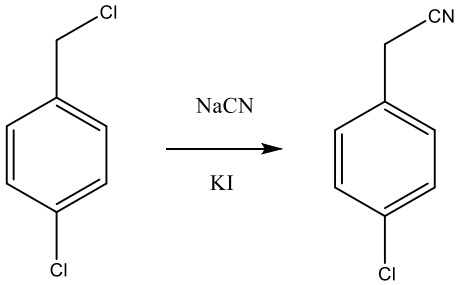
To a 1000 mL Erlenmeyer flask were added chlorotoluene (450 g, mol), and sulphuric acid (65mL, 18.4 mol dm^{-3}). The reaction flask was then set up for distillation with a Dean-Stark to return the denser layer of distillate. Distillation was carried out until no more water distilled over. To the reaction flask, an equal volume of water (500 mL) was added, and then set up for distillation with a Dean-Stark to return the less dense layer of distillate. The distillate was then tested for purity of p-chlorotoluene, while the liquid in the reaction flask was processed to obtain pure o-chlorotoluene. (A rough melting-point test determines the extent of separation – the distillate should be enriched in p-chlorotoluene. If a sample remains liquid in the freezer, it is quite impure. If it solidifies in the freezer, but melts in ice-water, it has reasonable purity. If it remains solid in ice-water, it is quite pure.) The process was repeated until the extent of separation was acceptable. The liquid from the reaction flask, containing o-chlorotoluene, was distilled, maintaining the temperature at 175-180°C to minimise decomposition to the phenol. The final yield was 70 ml of p-chlorotoluene.

Synthesis of p-chlorobenzyl chloride



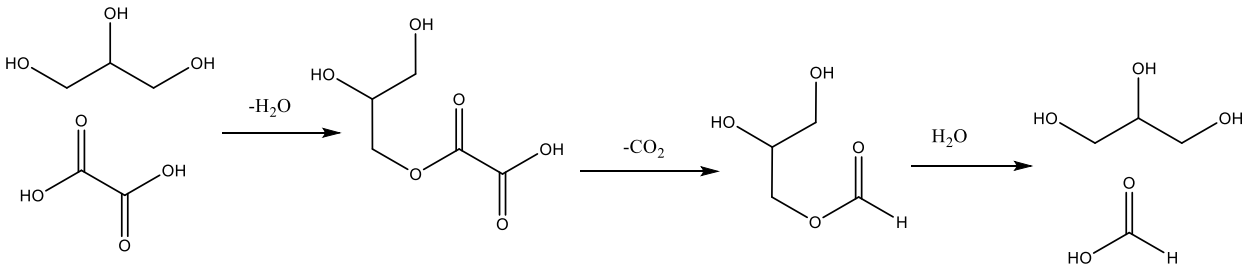
To a 250 mL Erlenmeyer flask was added p-chlorotoluene (mL, g, 1 mol), and set up for reflux. An ultraviolet light source was fixed below the condenser, and dry chlorine gas (1 mol) passed into the apparatus. After the chlorine was exhausted, the reaction mixture was fractionally distilled, collecting the fraction at 200-230°C. The final yield was 50 g (65%) of cloudy colourless liquid.

Synthesis of p-chlorophenylacetonitrile



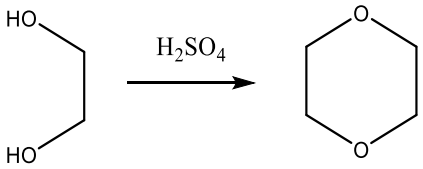
To a 250 mL Erlenmeyer flask was added p-chlorobenzyl chloride (50 g, mmol), potassium iodide (7.2 g, mmol), sodium cyanide (19.3 g, mmol), and dry acetone (250 mL). The reaction mixture was then refluxed for 24 hours. The solids were then filtered off, and washed with acetone. The solvent was then removed, and water (150 mL), and sodium metabisulphite (15 g) were added, then stirred for 1 hour. The organic layer was separated off, and washed with hydrochloric acid (30 mL), and water (30 mL), then dried with brine (30 mL) and anhydrous magnesium sulphate. The organic layer was then chilled in an ice bath to freeze the product, then the crystals filtered off. These were then remelted in a hot water bath, and then the liquid allowed to slowly cool at 15°C to form large crystals. The crystals were separated, and dried, yielding 6 g (12%) of exceptionally pure product.

Synthesis of formic acid



To a 250 mL Erlenmeyer flask was add glycerol (100 mL), oxalic acid dihydrate (100 g), and distilled at 105-120°C. When carbon dioxide evolution had ceased, a further portion of oxalic acid dihydrate (100 g) was then added. This process was repeated until the glycerol had decomposed too far to continue the reaction. At this point, water (50 mL) was added, and distillation continued to complete the hydrolysis. The distillate was then redistilled, discarding any material boiling below 100°C (consisting mainly of acrolein and allyl alcohol). The distillate between 100-104°C was considered water, and final distillate collected at 104-109°C. This crude distillate of azeotropic formic acid was then considered the final yield.

Synthesis of dioxane



To a 500 mL Erlenmeyer flask was added ethylene glycol antifreeze (300 mL), sulphuric acid[1] (30 mL, 98%) and the mixture distilled. Distillate was collected at 2-3 drops per second and was considered complete when foaming became unmanageable.[2] Sulphuric acid (10 mL, 98%) was then added to the distillate, and fractionally distilled collecting the fraction at 85-94°C as the dioxane-water azeotrope. Sodium hydroxide[3] (40 g) was then added and stirred until full reaction had occurred. If the result was monophasic, a further portion of sodium hydroxide was added. This was allowed to stir overnight. The liquid was then decanted and dried over sodium or sodium-magnesium oxide aggregate. The product was then distilled, and stored over sodium or sodium hydroxide.

(1) Sodium bisulphate does work but requires much higher temperatures and takes much longer.
(2) Residual tar can be removed with sodium hydroxide.
(3) Potassium hydroxide is a better substitute.

Preparation of sodium via sodium-magnesium oxide aggregate

$2NaOH + 2Mg \rightarrow 2Na + 2MgO + H_2O$

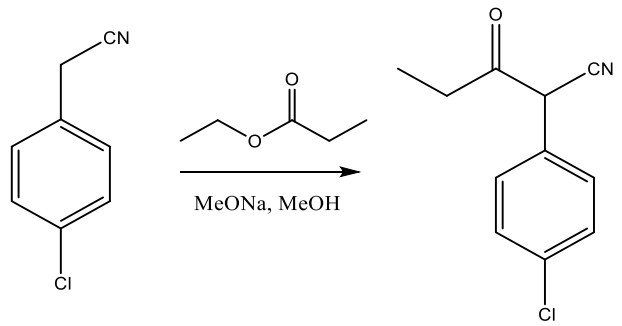
First 40g of sodium hydroxide and 30g of magnesium are place in a steel container with a fuse or sparkler and lit. A heavy lid is quickly placed over the mixture and allowed to burn. The mixture then allowed to cool, and the resulting aggregate of sodium metal and magnesium oxide is chiselled out. The aggregate is then ground in a blender until the consistency of coarse sand. It is then poured into a flask and covered with 2x-4x the volume in dioxane. A stir bar is added, and a distillation apparatus is outfitted on the mixture. It is then distilled with vigorous stirring. When the sodium is clearly visible on the surface of the liquid the stirring is reduced. When the height of the liquid is less than the diameter of the sodium globules the stirring is stopped completely. Distillation continues until dryness. Once dry the heating is stopped and the sodium allowed to solidify. It can then be dislodged from the slag and stored under mineral oil. The sodium may additionally be cleaned by heating until molten under mineral to which a few drops of alcohol has been added. Light agitation may be applied to dislodge any residual slag.

Synthesis of potassium chlorate

$KCl + 3\ H_2O \rightarrow KClO_3 + 3\ Cl_2$

For the anode, carbon, platinum, or mixed metal oxide works best. For the cathode, almost any metal can be used but titanium is preferred. The electrodes are simply inserted into a solution of potassium chloride salt and a current is passed through. The anode is the positive connection while the cathode is the negative connection. The current should be adjusted to match the surface of the electrode. For carbon that's around 40ma/cm^2, for platinum 300ma/cm^2, for mixed metal oxide 200ma/cm^2. You can use less current for lower heating and wear, but the production will take longer. The electrolysis must be kept in a well-ventilated area since it produces hydrogen gas as well as small amounts of chlorine gas. As the reaction progresses the potassium chlorate will precipitate our and the potassium chloride will be depleted. This has the overall effect of lowering the solution concentration. Every so often the salts must be topped up. Saturated potassium chloride solution has a density of 1.16g/mL. I recommend adding more potassium chloride salt when the density drifts below 1.1g/mL. To process it, remove the electrodes and decant and filter the solution from the potassium chlorate crystals. The filtrate still has some potassium hypochlorite in it so if you want to squeeze out every last gram of yield then you can boil the solution to disproportionate it. Upon cooling a small amount of potassium chlorate will crystallize out. You can add this to the original crop of crystals. The filtrate may be used in future potassium chlorate electrolytic runs. The potassium chlorate itself is relatively pure and consists of large crystals. If you wish to purify it and/or produce smaller crystals, mix an equivalent of 100mL of water per 60g of potassium chlorate and boil the mixture until it dissolves. Let it cool and fine crystals of potassium chlorate will precipitate out.

Synthesis of 2-(p-chlorophenyl)-3-oxopentanenitrile



To a 500 mL Erlenmeyer flask fitted with a reflux condenser was added dry methanol (300 mL), and sodium (15 g) added slowly until all had reacted. The solvent was then distilled off. p-Chlorophenylacetonitrile (30 g) was dissolved in ethyl propionate (30 mL), and added to the cooled residue from the distillation. Dioxane was then added to achieve a total volume of 300 mL. The reaction mixture was then refluxed for 2-3 hours. The reaction was then quenched with water (1000 mL) and stirred for 10 minutes. Hydrochloric acid (100 mL, 30%, 10 M) was then added, and stirred for another 10 minutes. The lower organic layer was then separated and washed with water (2x 100 mL). The crude yield was 41 g (99%).

A large scale for this type of reaction is very helpful to avoid destruction through hydrolysis from small amounts of water inevitably present.

Synthesis of bromoethane

$EtOH + HBr \rightarrow EtBr + H_2O$

To a 1000 mL round bottomed flask in an ice bath and set up for distillation was added hydrobromic acid (350 g, 48%), sulphuric acid (55 mL, 98%), and ethanol (120 mL), then to an addition funnel was added sulphuric acid (90 mL, 98%). The reaction flask was heated gently with a water bath, and the sulphuric acid added slowly as the reaction progressed. The water bath was heated to 40°C and slowly ramped to 70°C over the course of the distillation. The distillate was then washed with water (300 mL), sodium bicarbonate solution (350 mL, 5%), and water (350 mL). The product was then dried over anhydrous magnesium sulphate, and fractionally distilled, collecting the fraction at 38°C. The final yield was 116 g (50%).

Synthesis of 1-bromohexane

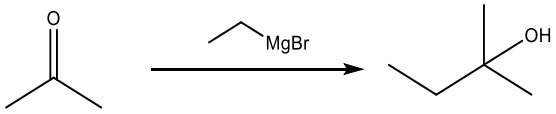
$n\text{-}C_6H_{13}OH + NaBr + H_2SO_4 \rightarrow n\text{-}C_6H_{13}Br + H_2O + NaHSO_4$

To a 500 mL Erlenmeyer flask was added sodium bromide (150 g), water (150 mL), 1-hexanol (102 g). A pressure-equalizing addition funnel was then set up with sulphuric acid (100 mL, 98%), and added slowly. The reaction mixture was then refluxed for 6 hours. The upper organic layer was then separated, and distilled collecting the fraction at 150-160°C. The distillate was washed with water (300 mL), sodium bicarbonate solution (350 mL, 5%), and water (350 mL). The crude was then distilled, removing any components boiling over 150°C. The final yield was 155 g (93%).

Synthesis of 2-methyl-2-octanol

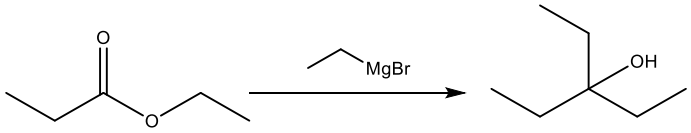
To a 500 mL Erlenmeyer flask set up for reflux and an addition funnel was added dry magnesium shavings (12 g), dry diethyl ether (200 mL). The magnesium was then activated with a small amount of bromohexane. Bromohexane (mL) in diethyl ether (mL) was then added dropwise. After complete addition, acetone (40 mL) in diethyl ether (100 mL) was then added dropwise via the addition funnel. The reaction mixture was then quenched with ice-cold water (400 mL), and then hydrochloric acid (30%) was added slowly until the cloudiness had dissipated. The upper organic layer was separated and distilled. The ether was removed, and the residue fractionally distilled to 90°C. Then a Dean-Stark trap was fitted, and the fractional distillation continued, removing all the water. The residue was then distilled, collecting the fraction at 177-182°C. The final yield was 23 g (44%).

Synthesis of t-amyl alcohol



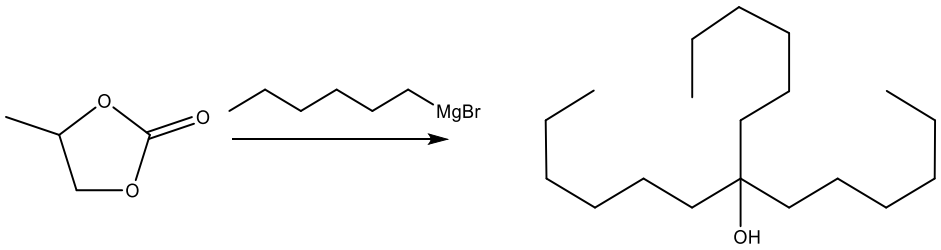
To a 500 mL Erlenmeyer flask set up for reflux and an addition funnel was added dry magnesium shavings (20 g), dry diethyl ether (150 mL). The magnesium was then activated with a small amount of bromoethane. Bromoethane (80 g) in diethyl ether (180 g) was then added dropwise. After complete addition, acetone (50 g) in diethyl ether (100 mL) was then added dropwise via the addition funnel. The reaction mixture was then quenched with ice-cold water (400 mL), and then hydrochloric acid (30%) was added slowly until the cloudiness had dissipated. The upper organic layer was separated and distilled. The ether was removed, and the residue distilled to 80°C. Then a Dean-Stark trap was fitted, and the distillation continued, removing all the water. The residue was then fractionally distilled, collecting the fraction at 95-102°C. The final yield was 23 g (35%).

Synthesis of 3-ethyl-3-pentanol



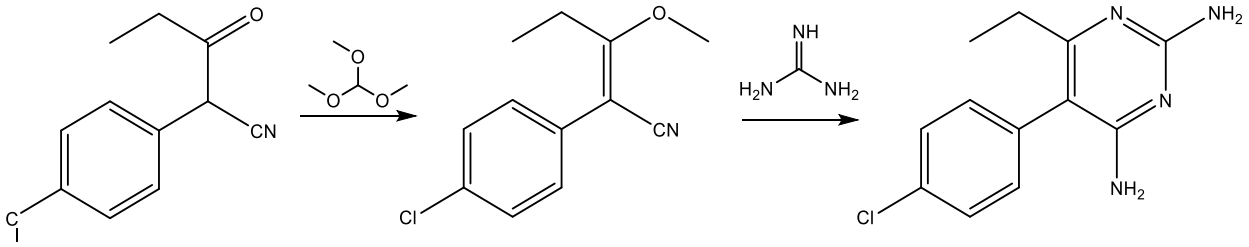
To a 500 mL Erlenmeyer flask set up for reflux and an addition funnel was added dry magnesium shavings (20 g), dry diethyl ether (150 mL). The magnesium was then activated with a small amount of bromoethane. Bromoethane (80 g) in diethyl ether (125 mL) was then added dropwise. After complete addition, ethyl propionate (40 g) in diethyl ether (100 mL) was then added dropwise via the addition funnel. The reaction mixture was then quenched with ice-cold water (400 mL), and then hydrochloric acid (30%) was added slowly until the cloudiness had dissipated. The upper organic layer was separated and distilled. The ether was removed, and the residue distilled to 85°C. Then a Dean-Stark trap was fitted, and the distillation continued, removing all the water. The residue was then fractionally distilled, collecting the fraction at 140-145°C. The final yield was 14 g (32 %).

Synthesis of 7-hexyl-7-tridecanol



To a 500 mL Erlenmeyer flask set up for reflux and an addition funnel was added dry magnesium shavings (15 g), dry diethyl ether (150 mL). The magnesium was then activated with a small amount of bromohexane. Bromohexane (75 g) in diethyl ether (50 mL) was then added dropwise. After complete addition, propylene carbonate (15 g) in diethyl ether (50 mL) was then added dropwise via the addition funnel. The reaction mixture was then quenched with ice-cold water (300 mL), and then hydrochloric acid (30%) was added slowly until the cloudiness had dissipated. The upper organic layer was separated and distilled. The ether was removed and washed with water (150 mL), and the residue distilled to 160°C. Water (150 mL) was then added, and a heavy-return Dean-Stark apparatus set up, and steam distilled for a week. The final yield was 28 g (43%).

Synthesis of pyrimethamine



Synthesis of is (2Z)-2-(4-chlorophenyl)-3-methoxypent-2-enenitrile

To a 250 mL Erlenmeyer flask was added 2-(p-chlorophenyl)-3-oxopentanenitrile (41 g), toluene (50 mL) and set up for drying via a Dean-Stark apparatus. The mixture was refluxed until the reaction mixture was dry. Trimethyl orthoformate (30 mL), dry methanol (75 mL), sulphuric acid (0.5 mL, 98%) were then added, and refluxed for 3-4 hours.

Synthesis of pyrimethamine

A solution of sodium methoxide in methanol was then prepared by reacting methanol (200 mL) and sodium (15 g) in a 500 mL Erlenmeyer flask fitted with a reflux condenser. Guanidinium chloride (20 g) was then added and stirred for 5 minutes. The product in solution from earlier was then added and refluxed for 36 hours. 100 mL of solvent was then distilled off, and an equal volume of water added, then distillation continued until water began to distil. Sodium hydroxide (23 g) was then added, and the reaction mixture cooled in an ice bath. Diethyl ether (150 mL) was then added, and water (200 mL) added to solubilize undissolved salts. The precipitate was then filtered off, and washed with water twice, then ether and allowed to air dry. The crude yield was 5.5 g. The product was then recrystallized from ethanol (90 mL). The final yield was 1.8 g.

Synthesis of manganese(II) sulphate (from battery MnO_2)

To a 600 mL beaker was added water (500 mL) and crude manganese dioxide (100 g) and stirred thoroughly. The solids were filtered off and washed well with water.

Method 1 – reduction with oxalic acid

$$MnO_2 + (COOH)_2 + H_2SO_4 \rightarrow MnSO_4 + CO_2 + 2\ H_2O$$

To a 600 mL beaker was added oxalic acid (30 g), water (300 mL), and sulphuric acid (13mL, 98%) and stirred until all the solids had dissolved. Then the manganese dioxide was continually added until the solution stopped bubbling (~100 g). The precipitate was filtered off, and the solution concentrated to 50 mL.

If iron contamination is present:

$$4\ Mn(OH)_2 + 4\ FeSO_4 + O_2 + 2\ H_2O \rightarrow 4\ MnSO_4 + 4\ Fe(OH)_3$$

One third of the solution was separated, and sodium hydroxide solution was added until no more precipitation occurred. The precipitate was filtered off and washed well with water, then added back to the stock solution. Assuming precipitate is present after complete addition of the precipitate, the solution was then allowed to react with oxygen in the air overnight. The precipitate was then filtered off, and evaporated to dryness. The product was then recrystallised from water.

Method 2[1] – reduction with sulphur dioxide

$$MnO_2 + SO_2 \rightarrow MnSO_4$$

To a 500 mL round bottomed flask was added a slurry of manganese dioxide (100 g in water). Sulphur dioxide was passed into the slurry for 2 hours. The reaction mixture was filtered, and the filtrate evaporated to dryness, then recrystallized from water.

(1) This method is far more forgiving as no iron contamination occurs

Synthesis of manganese dioxide

$$MnSO_4 + KHSO_5 + H_2O \rightarrow MnO_2 + H_2SO_4 + KHSO_4$$

Manganese sulphate (10 g) was dissolved in water (30 mL) and added to potassium peroxymonosulphate (25 g) dissolved in water (100 mL) and the precipitate filtered off when bubbling had ceased and washed well with water. The product was then dried in an oven at 130°C.

Synthesis of cadmium selenide quantum dots

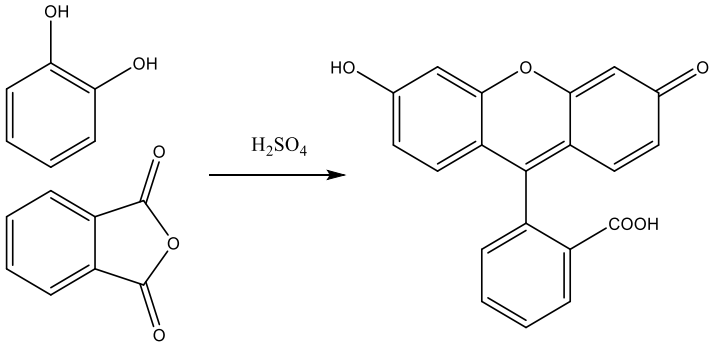
$Se + P(C_8H_{17})_3 \rightarrow SeP(C_8H_{17})_3$

$CdO + 2\ C_{17}H_{33}COOH \rightarrow Cd(C_{17}H_{33}COO)_2$

$SeP(C_8H_{17})_3 + Cd(C_{17}H_{33}COO)_2 \rightarrow CdSe + byproducts$

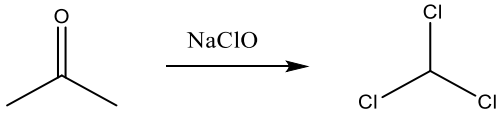
To a vial was added selenium powder (30 mg), 1-octadecene (5 mL), and trioctylphosphine (0.4 mL) then heated and stirred to dissolve. The vial was then sealed and allowed cool.
To a 25 mL Erlenmeyer flask was added cadmium oxide (13 mg), oleic acid (0.6 mL), 1-octadecene (1 mL) and heated to 225°C. As soon as this temperature was reached, to the flask was added a portion of the solution prepared earlier (1 mL), shaken well, and 0.5 mL aliquots of the solution were successively withdrawn upon a change in colour. The resulting solutions contain cadmium selenite quantum dots.

Synthesis of fluorescein



To a vial was added phthalic anhydride (166 mg), resorcinol (220 mg), and sulphuric acid (3 drops, 98%) then heated to 200°C for 5 minutes, then allowed to cool to room temperature. Water (10 mL) and sodium hydroxide (300 mg) was then added to generate a solution of fluorescein.

Synthesis of chloroform



To a 1500 mL beaker was added cold sodium hypochlorite (1000 mL, 7%, -10°C) and chilled in an ice bath. Methyl ethyl ketone (22 g) was added slowly with strong stirring and allowed to stir until the temperature had returned to room temperature. The solution was then allowed to sit and separate. The lower organic layer was then separated, giving a crude yield of 17 g (45%).

Synthesis of propionic acid

To the waste solution (4500 mL) from the synthesis of chloroform from bleach and butanone was added hydrochloric acid (600 mL, 31%) and allowed to sit for 24 hours. The solution was then fractionally distilled collecting everything boiling at 95-103°C, discarding the first 5% of distillate. Sodium carbonate was added to the distillate until effervescence ceased, then the water removed. Sulphuric acid (1 mL/g of solids, 98%) was then added and the reaction mixture distilled to dryness. The distillate was dried over anhydrous sodium sulphate, and redistilled collecting the fraction at 141°C. The final yield was 42 g (42%).

Synthesis of sodium cyanide

$3\ NaOH + H_3N_3C_3O_3 \rightarrow Na_3C_3N_3O_3 + 3\ H_2O$

$Na_3C_3N_3O_3 \rightarrow 3\ NaCNO$

$2\ NaCNO + C \rightarrow CO_2 + NaCN$

To a steel or nickel crucible was added sodium hydroxide (100 g), cyanuric acid[1] (43 g), carbon (12 g), and heated to 600°C over a period of 1 hour, then heated at 600°C for a further hour, then allowed to cool to room temperature. The reaction mixture was then dissolved in methanol (500 mL) and sodium bicarbonate (100 g) added with stirring, then stirred for 30 minutes. The precipitate was filtered off,[2] and the solvent removed. The crude yield was 19 g (36%).
Yield could be improved by more intimately mixing the reagents, and possibly by using potassium hydroxide instead. KCNO decomposes at lower temperatures than NaCNO.

(1) Urea can also be used
(2) Test for cyanide with Fe^{II}/Fe^{III}

Synthesis of trimethyl borate

$Na_2B_4O_7 + 12\ CH_3OH + 2\ H_2SO_4 \rightarrow 4\ B(OCH_3)_3 + 2\ NaHSO_4 + 7\ H_2O$

To a 1000 mL Erlenmeyer flask fitted with a reflux condenser was added borax (200 g), methanol (650 mL), and through the condenser, sulphuric acid (100 g, 98%). The reaction mixture was then distilled collecting the fraction at 54°C. The distillate is trimethyl borate methanol azeotrope (75% trimethyl borate).

Nux,s Channel

Synthesis of sodium ethyl sulphate

$EtOH + NaHSO_4 \rightarrow EtNaSO_4 + H_2O$

To a 100 mL round bottomed flask set up for reflux was added ethanol (90 g), sodium hydrogensulphate (90 g), and the reaction mixture refluxed for 45 minutes, then allowed to cool to room temperature. The precipitate was filtered off, and to the filtrate was added sodium carbonate until a neutral pH was reached, and the solution filtered again. The solution was evaporated to dryness to yield 21.74 g.

Synthesis of nitroethane

$EtNaSO_4 + NaNO_2 \rightarrow EtNO_2 + Na_2SO_4$

To a retort-crucible set up for distillation was added sodium ethyl sulphate (12 g), sodium nitrite (7 g), water (10 mL), and sodium carbonate (0.9 g). The reaction mixture was then heated to 150°C, and the distillate collected over water. The crucible was heated until no more distillate came over. The distillate-water mixture was then extracted with dichloromethane (2x 25 mL), dried over calcium chloride, and the dichloromethane distilled off. The final yield was 1.9 mL of a dark yellow liquid.

Synthesis of red phosphorus

$nP_4 \rightarrow P_n$

To a sealable crucible was added potassium iodide (0.05 g), white phosphorus (1.14 g), then sealed and heated to 280°C for 6 hours. After cooling, the reaction mixture was tipped into sodium hydroxide solution (600 mL, 0.2 %), and the product filtered off. The final yield was 1.08 g of red powder.

Synthesis of sodium hypophosphite

$12\ NaOH + 5\ P_4 + 12\ H_2O \rightarrow 8\ PH_3 + 12\ NaHPO_2$

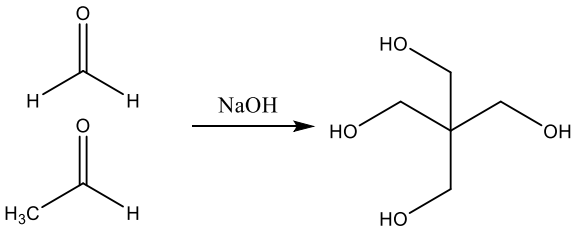
To a 50 mL round bottomed flask set up for reflux was added sodium hydroxide (1.6 g), calcium hydroxide (0.860 g), water (10 mL), and white phosphorus (1.0 g). The reaction mixture was refluxed until all the white phosphorus had reacted, with vapours being destroyed by feeding into a flame. The reaction mixture was then filtered, and the filtrate evaporated to dryness to yield 2.77 g of product.

Preparation of white phosphorus

$4\ AlPO_4 + 5\ C \rightarrow P_4 + 2\ Al_2O_3 + 5\ CO_2$

To a stainless steel crucible was added powdered charcoal (1 briquette), monoaluminium phosphate (20 g), and calcium hydroxide (1 tsp). The reaction mixture was then heated in a furnace to red heat, and the vapours passed into water. The final yield was 2.20 g of yellow waxy solid.

Synthesis of pentaerythritol



To a 250 mL Erlenmeyer flask chilled in an ice bath was added formaldehyde solution (40 g, 20%). Sodium hydroxide solution (7.1 g, 50%) cooled to below 5°C was then added to the cooled formaldehyde solution with stirring, maintaining the temperature below 15°C. Acetaldehyde (2.8 g) was then added over the course of an hour, maintaining the temperature below 20°C. The solution was then warmed to 25°C and stirred for 4 hours, then warmed to 60°C and stirred for 2 hours. Thymolphthalein indicator was then used to measure pH, and formic acid added until the blue colour had disappeared, then 2 or 3 extra drops of formic acid were added to bring the final pH to 7. The solution was then evaporated to crystallisation, and the product filtered off. The final yield was 4.46 g of white solid.

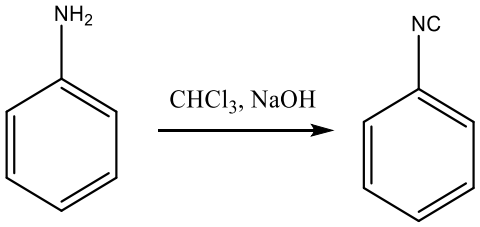
Rhodanide

Synthesis of diiodoacetylene

$2KI + NaOCl + HC{\equiv}CH \rightarrow IC{\equiv}CI + NaCl + 2\ HI + H_2O$

Potassium iodide was dissolved in water (200 mL), and sodium hypochlorite solution (5%) added to achieve a red colour. Acetylene was then passed into the solution generated from calcium carbide and water. When the solution turned yellow-orange, and a precipitate had formed, a further portion of sodium hypochlorite solution (20 mL) was added. Acetylene addition was continued. The precipitate was filtered off and washed with water. The product is very flammable, and soluble in acetone. Store under water.

Synthesis of phenyl isocyanide



To a 500 mL Erlenmeyer flask was added ethanol (75 mL) chloroform (15 mL) dissolved in ethanol (60 mL, 90%), and aniline (15 mL) dissolved in ethanol (60 mL, 90%). Sodium hydroxide (5 g?) dissolved in ethanol (25 mL) and refluxed for 2 hours? The solvent was removed, and the product distilled off.

Extraction of parietin from Xanthoria parietina lichen

To a 250 mL beaker was added ethanol (100 mL), lichen (11 g) and boiled for 30 minutes, then hot filtered and allowed to cool to room temperature. The precipitate was filtered off, washed with ice-cold ethanol and allowed to dry.

Synthesis of chloropentamminecobalt(II) chloride

$CoCl_2 + 4\ NH_4Cl + 16\ NH_3 + 2\ H_2O_2 + 6\ H_2O \rightarrow [Co(NH_3)_5Cl]Cl_2 + O_2$

To a 500 mL beaker was added ammonia solution (40 mL, 10%), ammonium chloride (6 g), cobalt chloride dihydrate (12 g), and stirred to dissolve. Hydrogen peroxide (10 mL, 35%) was then added dropwise, then allowed to stir for 30 seconds. The hydrochloric acid (30 mL, 31%) was then added slowly then heated to 85°C with stirring for 20 minutes and then allowed to cool slowly 0°C. The precipitate was filtered off, and allowed to dry.

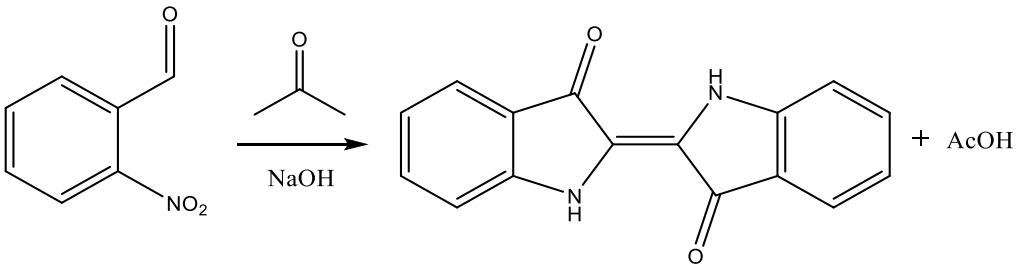
Random Experiments

Synthesis of potassium dithioferrate(III)

$6Fe + 4K_2CO_3 + 13S \rightarrow 6KFeS_2 + K_2SO_4 + 4CO_2$

Iron filings (2 g), sulphur powder (12 g), potassium carbonate (10 g), sodium carbonate (2 g) were intimately mixed with a pestle and mortar. The mixture is then compressed into a porcelain crucible and heated until vigorous gas evolution has ceased, then heated red hot for 2 hours, then allowed to cool to room temperature. The solid was then digested with water, and the crystalline material filtered off. The product was dark purple fine needles, whose size depended on time and temperature of heating.

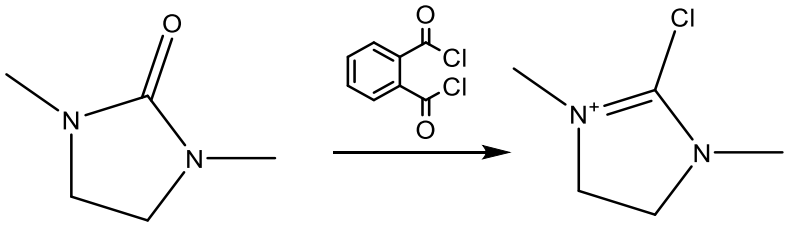
Synthesis of indigo



To a 150 mL beaker was added o-nitrobenzaldehyde (1 g), acetone (10 mL), water (5 mL), and stirred to dissolve. Sodium hydroxide solution (4 mL, 1 mol dm^{-3}) was then added, and the reaction mixture stirred for 10 minutes. The precipitate was then filtered off, and washed with ethanol, to yield 0.5 g (58%) of blue-black powder.

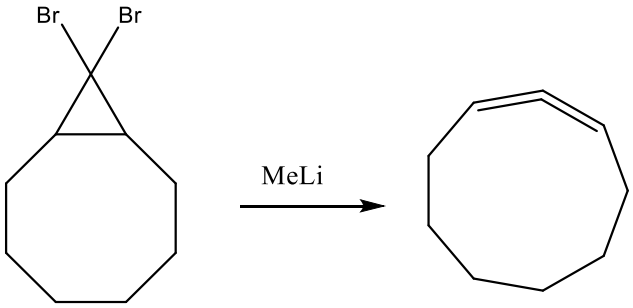
Smokin' Hot Science

Synthesis of dimethylimidoyl chloride (DMC)



A Schlenk tube is flame dried under vacuum and backfilled with nitrogen. 1,3-Dimethyl-2-imidazolidinone (3.35 mL, 3.50 g, 30.7 mmol) and o-phthaloyl chloride (6.54 g, 32.4 mmol) were then added, and the reaction mixture heated to 140°C in an oil bath for 5 hours. After allowing to cool to room temperature, dry dioxane (25 mL) was added and all solids brought into solution or suspension. The solids were then filtered off under nitrogen, washed with dry dioxane (2x 100 mL), dry ether (5 mL) and dried under vacuum. The final yield was 2.72 g (53 %) melting at 94.6°C (lit. 95-100°C).

Synthesis of cyclononallene



To a dry 100 ml round bottom flask flushed with nitrogen was added dry diethyl ether (35 mL), 9,9-dibromobicyclo[6.1.0]nonane (7.15 g, 24.5 mmol) and cooled in a dry ice-acetone bath. Methyl lithium solution (19.05 mL, 1.6 M, 30.48 mmol) was then added dropwise at a rate of 0.3 mL min^{-1}. The reaction mixture was then allowed to warm to room temperature overnight and water (0.2 mL) was added. Anhydrous magnesium sulphate was then added, and the mixture filtered through silica gel, and rinsed with pentane. The solvent was removed, and the product recovered. The final yield was 2.746 g (88%).

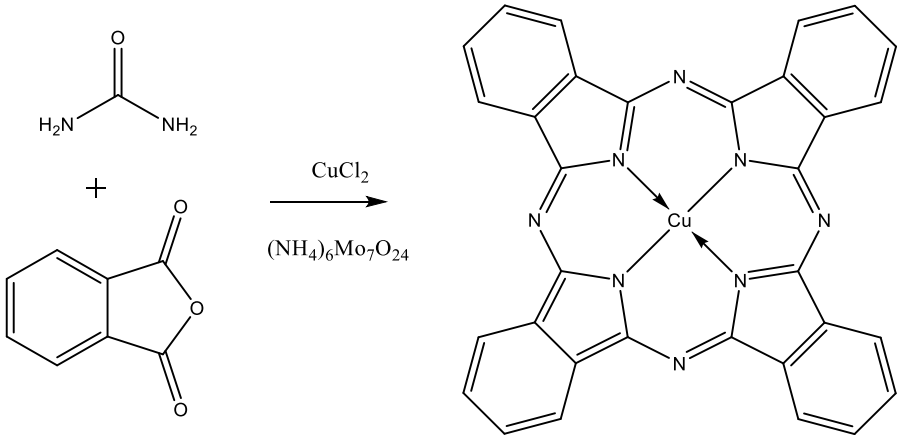
Texium

Synthesis of phosphorus tribromide

$2\ P + 3\ Br_2 \rightarrow 2\ PBr_3$

To a 50 mL round bottomed flask was added bromine (10 mL), and chloroform (10 mL), then set up for reflux. Red phosphorus (5.0 g) was then added slowly through the reflux condenser, then the chloroform was distilled off. The apparatus was then cleaned and reassembled. The phosphorus tribromide was then distilled off at 170°C. The final yield was 30.4 g (87%).

Synthesis of copper phthalocyanine



Phthalimide (4.0 g, 27 mmol), urea (1.62 g, 27 mmol), cupric chloride dihydrate (1.16 g, 7 mmol), ammonium molybdate[1] (30 mg, 20 µmol) are ground intimately and heated in a ceramic crucible on a sand bath to >200°C for 1 hour. After cooling, the glassy solid was scraped out and washed three times with distilled water, twice with dilute sodium hydroxide solution, twice with acetic acid (15%), once more with distilled water, and continued washing with acetone until filtrate is colourless. The final yield was 0.86 g (22%).

(1) Boric acid can be substituted instead

The Canadian Chemist

Synthesis of lithium peroxide

$2\ Li + H_2O \rightarrow 2\ LiOH + H_2$

$2\ LiOH + H_2O_2 \rightarrow Li_2O_2 + 2\ H_2O$

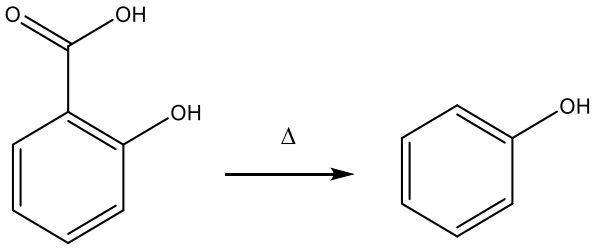
To a beaker was added lithium (4 g) and water (100 mL) and allowed to react. The water was removed, and hydrogen peroxide (45 mL, 35%) then brought to a boil and hot filtered. The precipitate was allowed to dry, and the product dried under vacuum.

Synthesis of fuming nitric acid

$NaNO_3 + H_2SO_4 \rightarrow HNO_3 + NaHSO_4$

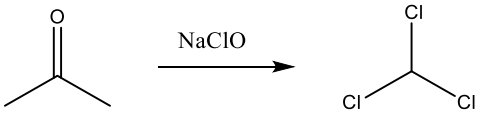
To a 500 mL round bottomed flask set up for distillation was added sodium nitrate (200 g, 2.3 mol), and sulphuric acid (230 g, 98%, 2.3 mol). The reaction mixture was distilled until the fumes became a lighter colour, signifying an end to the reaction. The final product had a density of 1.5 g cm^{-3}.

Synthesis of phenol



A 100 mL round bottomed flask was charged with salicylic acid (30 g), and boiling chips added. A short-path distillation apparatus was then set up, and the salicylic acid was distilled. The distillate was then redistilled under reduced pressure, and the fraction boiling at 85-90°C / 40 mmHg. The final yield was 10 g (49%) of white crystals.

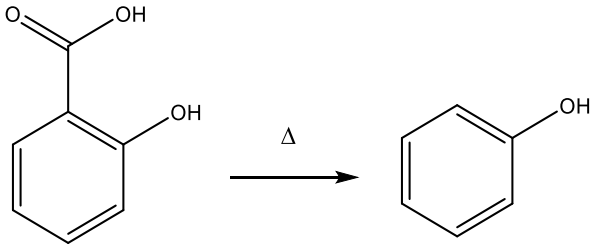
Synthesis of chloroform



To a jug of bleach (4.5 L, 8%) cooled to -2°C and acetone (180 mL) was added. The jug was shaken and allowed to react in a cool place and separate. The upper aqueous layer was decanted, and the lower organic layer separated. The product was washed once with brine, then dried over calcium chloride and distilled collecting the fraction at 60°C.

TheChemiKid

Synthesis of phenol



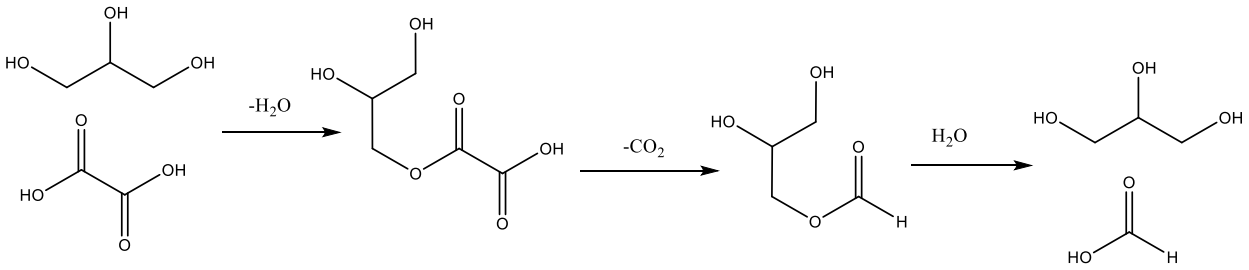
To a 100 mL round bottomed flask fitted with an air condenser was added salicylic acid (13.812 g, 0.1 mol) and heated strongly until no more gas was evolved. The product was allowed to cool to 50°C. The crude yield was 8.38 g (89%).

Synthesis of t-butyl bromide

$^{t}BuOH + HBr \rightarrow {}^{t}BuBr + H_2O$

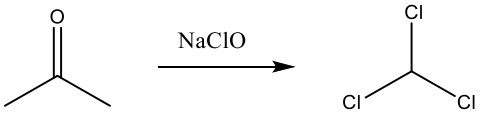
To a 50 mL round bottomed flask was added t-butyl alcohol (7.2 mL), sodium bromide (10 g), water (7.5 mL), and sulphuric acid (7.5 mL, 98%) dropwise. The reaction mixture was then distilled until the vapour temperature reached 90°C. The crude yield was 8.2 g (80%).

Synthesis of formic acid



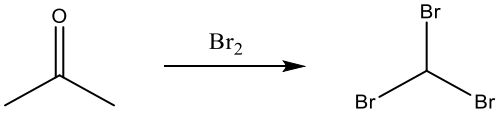
To a 50 mL round bottomed flask was added dry glycerine (30 mL) and oxalic acid dihydrate (24 g), then the reaction mixture distilled until the reaction mixture turned dark brown.

Synthesis of chloroform



To bleach (3.4 L, 8%) cooled to below 0°C was added ice-cold acetone (150 mL) and mixed well. The reaction mixture was then allowed to separate, and the lower organic layer separated and dried over anhydrous magnesium sulphate. The final yield was 32 g (70%).

Synthesis of bromoform



To a beaker was added sodium bromide (10 g) dissolved in water (125 mL) and bleach (205 mL) added. Acetone (4 mL) dissolved in water (10 mL) was then added to the reaction mixture. The mixture was allowed to separate, and the lower organic layer separated.

The Chemistry Shack

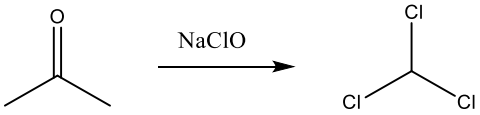
Synthesis of t-butyl chloride

$^{t}BuOH + HCl \rightarrow {}^{t}BuCl + H_2O$

In a 500 mL separatory funnel are placed t-butanol (37 g, 48 mL, 1 mol) and hydrochloric acid (124 mL, 36%, 1.5 mol). After shaking, the layers are allowed to separate (15-20 minutes) and the upper layer is drawn off and washed first with sodium bicarbonate solution (5%), water until neutral to moist litmus paper. The product is dried over calcium chloride and then distilled, collecting the fraction boiling at 49.5–52°C. The final yield is 40 g (90%).

Organic Syntheses, Coll. Vol. 1, p.144 (1941); Vol. 8, p.50 (1928).

Synthesis of chloroform



To bleach (7.28 L, 8.25%) cooled to below 0°C was added acetone (175 g, 110 mL) in two portions, allowing to cool between additions. The reaction mixture was then allowed to separate, and the lower organic layer separated and dried over an equal volume of brine, then over anhydrous calcium chloride. The product was then distilled, collecting the fraction at 60-63°C (lit. 62.1°C). The final yield was 182.8 g (45%).

Synthesis of silicon tetrachloride

$Si + 2\ Cl_2 \rightarrow SiCl_4$

An excess of chlorine generated from TCCA and hydrochloric acid and dried over sulphuric acid (98%) was passed over dry silicon powder (2.7 g) heated strongly in a fused quartz tube and the vapours condensed with a cold condenser.

Preparation of silicon

$SiO_2 + 2\ Mg \rightarrow Si + 2\ MgO$

Silicon dioxide (3.1 g) was mixed with magnesium powder (5 g) and ignited with a magnesium ribbon. After allowing to cool, the reaction mixture was digested with water, then hydrochloric acid (10%), and the precipitate filtered off and washed with water. The final yield was 3.82 g (103%).

Synthesis of copper dichloroisocyanurate

To a beaker was added copper sulphate pentahydrate (10g), hot water (250 mL), and to a separate beaker sodium dichloroisocyanurate (10g) and dissolved in cold water. The solutions were combined and allowed to stir for 10 minutes. The precipitate was then filtered off, washed well with water and allowed to dry. The formula of the product is not known.
US Patent US3055889

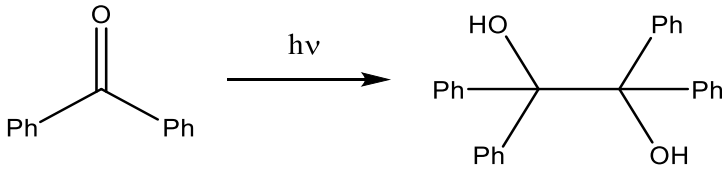
Thoisoi2

Preparation of matches

A large number of toothpicks were soaked in ammonium dihydrogen phosphate solution (2% in water), then in molten paraffin wax. The matchhead mixture was then made by mixing sand (39%), sulphur (4.7%), gelatin (7%), potassium dichromate (1%), sodium alginate (1%), and water added sufficient to form a slurry. Potassium chlorate (47%) was then added, and the slurry mixed thoroughly. The tips of the dried matchsticks were then dipped in the slurry and allowed to dry. The striking surface was then prepared by mixing red phosphorus with PVA glue to form a paste and painted onto the side of the matchbox. Masking tape was used to keep the edges of the surface clean. When everything was dry, the matches were tested, with great success.

Tom's Lab

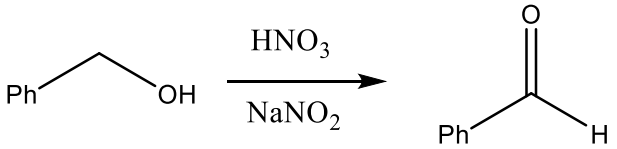
Synthesis of benzopinacol



To a 125 mL Erlenemeyer flask was added benzophenone (10.9 g), dry isopropanol (80 mL), glacial acetic acid (0.5 mL), and sat in sunlight for 5 days (irradiated for 15-20 hours total).[1] The precipitate was filtered off and washed with isopropanol. The final yield was 1.99 g (18.2%)[2] melting at 173-177°C.

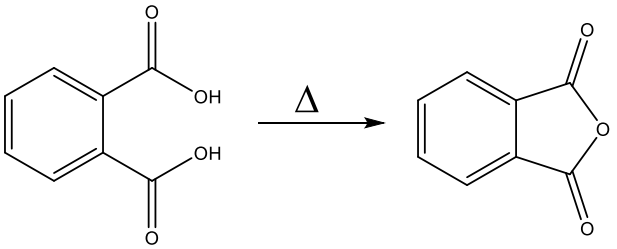
(1) Longer irradiation would lead to more product.
(2) Yields can be up to 90%

Synthesis of benzaldehyde



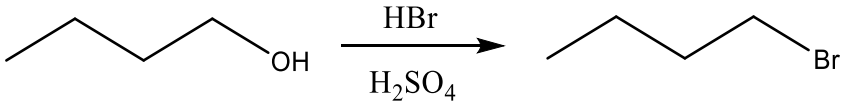
To a 250 mL round bottomed flask was added benzyl alcohol (31.1 mL), nitric acid (13.1 mL, 70%), sodium nitrite (1 g), water (100 mL) and refluxed for 4 hours, then allowed to cool to room temperature. The lower organic layer was washed with water, saturated sodium bicarbonate solution, and brine. The crude was 23.43 g (72.2 %)

Synthesis of phthalic anhydride



To a 400 mL beaker was added phthalic acid (60 g), and heated until water was no longer emitted. An evaporating dish was placed in the mouth of the beaker and used to minimise loss of product through sublimation. Once the solids had completely melted, the liquid was poured out onto a sheet of aluminium foil and allowed to cool.

Synthesis of 1-bromobutane

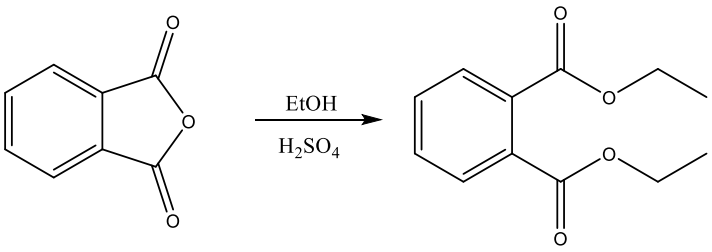


To a 50 mL round bottomed flask was added n-butanol (11 mL), sulphuric acid (7.4 mL), hydrobromic acid (18.5 mL) and refluxed for 3 hours, then distilled until no more oily or cloudy distillate was collected. The lower organic layer was washed with equal volumes of water, hydrochloric acid, water, saturated sodium bicarbonate solution, and water. The organic layer was then dried over anhydrous calcium chloride, and distilled collecting the fraction at 105-108°C. The final yield was 7.96 g (48.3%). Low yield was likely due to a spillage.

Preparation of choline chloride/urea eutectic

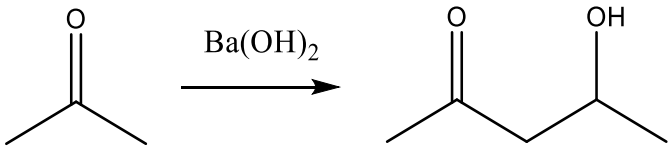
To a small vial was added anhydrous chloline chloride (2.23 g), urea (1.92 g) and the mixture gently heated to 60°C to melt the solids. Upon cooling to room temperature, the liquid did not freeze.

Synthesis of diethyl phthalate



To a 250 mL round bottomed flask was added phthalic anhydride (16.25 g), ethanol (150 mL), sulphuric acid (6 mL, 98%) and set up for reflux over a Soxhlet extractor filled with molecular sieves (15 g, 3Å) and refluxed for 2 hours. The excess ethanol was then distilled off, and the residue washed with an equal volume of water, saturated sodium carbonate solution, and water, then dried over anhydrous magnesium sulphate. The final yield was 15.77 g (65%). The low yield was due to mechanical loss.

Synthesis of diacetone alcohol



A Soxhlet extractor fitted with a thimble full of barium hydroxide was set up, and acetone (130 mL) was refluxed through until the contents of the flask had a boiling point above 100°C. This took 80-100 hours. Excess acetone was then distilled off, and the residue vacuum distilled, collecting the fraction at 85-95°C at 30 Torr. The final yield was 21 g. The low yield was due to the reaction time being cut short to 35 hours.

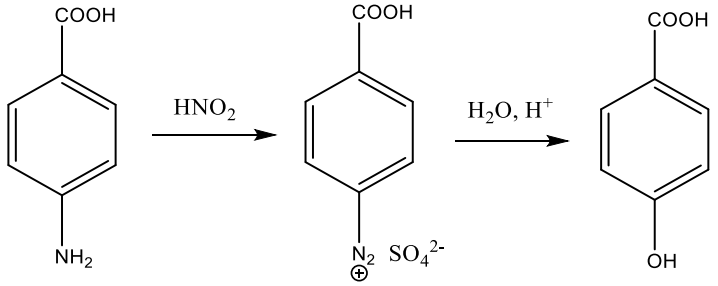
Synthesis of magnesium nitride

$NH_4Cl + NaNO_2 \rightarrow N_2 + 2\ H_2O + NaCl$

$3\ Mg + N_2 \rightarrow Mg_3N_2$

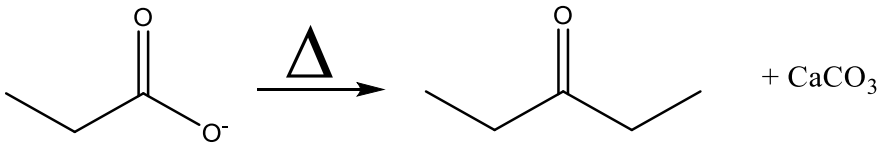
To a 250 mL Erlenemeyer flask was added ammonium chloride (2.76 g), sodium nitrite (3.56 g), and water (100 mL). The gas was then lead through a calcium chloride drying tube to a fused quartz tube into which had been placed magnesium powder (0.5 g). The nitrogen generator was then heated, and the apparatus purged for 10 minutes. The magnesium was then heated strongly for 10 minutes then allowed to cool under a flow of nitrogen. The product was recovered from the tube and tested with water and damp pH paper. Conversion was far from complete.

Synthesis of 4-hydroxybenzoic acid



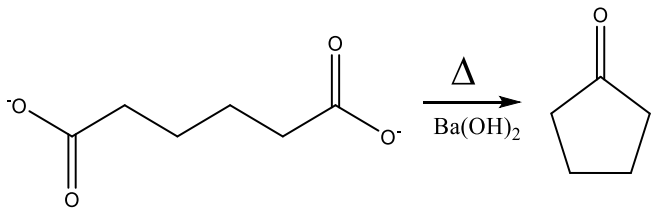
To a 50 mL beaker was added 4-aminobenzoic acid (3.45 g), water (7.5 mL), and slowly sulphuric acid (5.5 mL, 98%). One third of the volume of the suspension was added to a 50 ml three necked flask and cooled to 0-5°C. Sodium nitrite (0.6 g) dissolved in water (2 mL) was then added very slowly dropwise to the reaction mixture over a period of 25 minutes, then allowed to stir for a further 20 minutes. The reaction mixture was then added dropwise to sulphuric acid (15 mL, 50%) maintaining a temperature of 60-70°C over a period of 10 minutes. When the bubbling had ceased, the reaction mixture was allowed to cool to room temperature, and the precipitate filtered off. The final yield was 0.68 g (59%) melting at 205°C (lit. 215°C).

Synthesis of 3-pentanone



To a 50 mL test tube was added calcium propanoate (18 g) and fitted for distillation. The test tube was then heated strongly until no more distillate collected. The upper organic layer was separated and dried over calcium chloride. The yield was (13%) boiling at 101°C (lit. 102°C) with a density of 0.78 g mL^{-1} (lit. 0.81 g mL^{-1}).

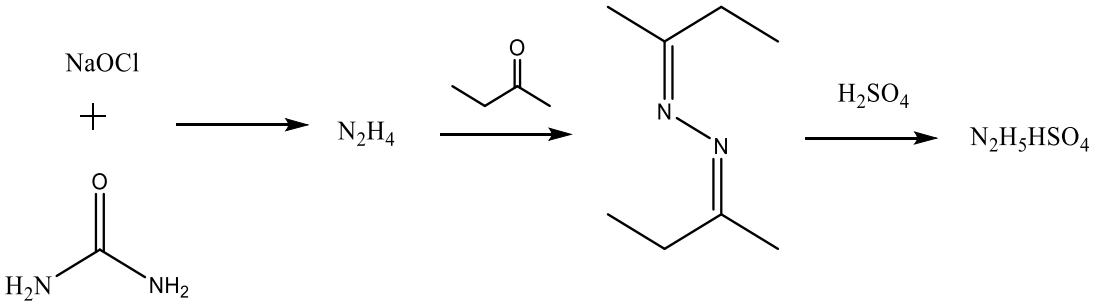
Synthesis of cyclopentanone



To a 500 mL flask set up for distillation was added adipic acid (100 g) and barium hydroxide octahydrate (5 g) mixed very thoroughly. The solids were heated to 290-295°C and distilled until no more distillate collected. The lower aqueous layer was removed, and the product dried over anhydrous potassium carbonate. The product was fractionally distilled, collecting the fraction at 93.5°C (water azeotrope) and 128-130°C. The final yield was 33 g (60%). The low yield was due to stopping heating too early.

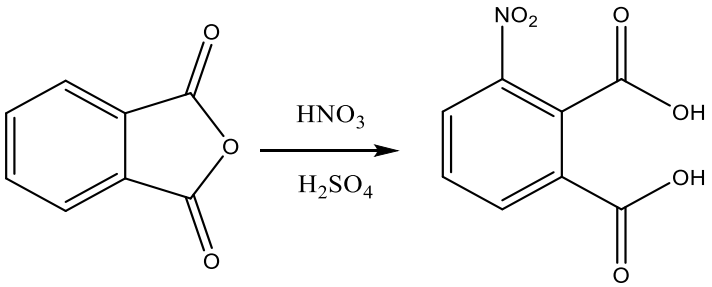
UC235

Synthesis of hydrazine sulphate



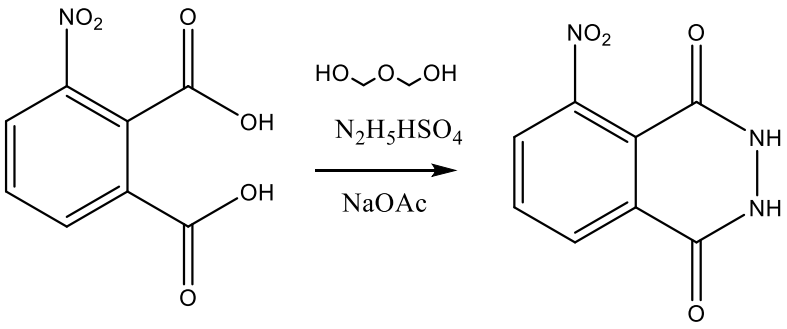
To a 1000 mL Erlenmeyer flask was added sodium hypochlorite solution (314 g, mol, 12.5%) and chilled in an ice bath to 0°C. To the chilled solution was slowly added sodium hydroxide (53 g, mol) with stirring. To a 1000 mL round bottomed flask was added urea (39 g, mol), gelatine (0.6 g), and water (50 mL), and warmed to dissolve all the solids. The flask was then fitted with a reflux condenser and an addition funnel, and the hypochlorite solution added to the addition funnel. The hypochlorite solution was then added all at once, and the mixture allowed to stir at room temperature until the foam had disappeared. Butanone (77 g, mol) was then added through the condenser, then stirred strongly for 1.5 hours. After this time, the mixture was allowed to separate into two layers, and the upper organic layer separated. To the organic layer was added sulphuric acid (40 g, 0.75 mol eq.) dissolved in water (100 mL), then the methyl ethyl ketone-water azeotrope was distilled off at 73.5°C. After distillation, the residue was cooled in an ice bath to crystallise the product out, and the precipitate was filtered off. The final yield was 32.83 g (47.16%) of white crystals. A fresh hypochlorite solution gave a 53% yield. From the distillate of butanone-water azeotrope, 75% of the starting volume was recovered.

Synthesis of 3-nitrophthalic acid



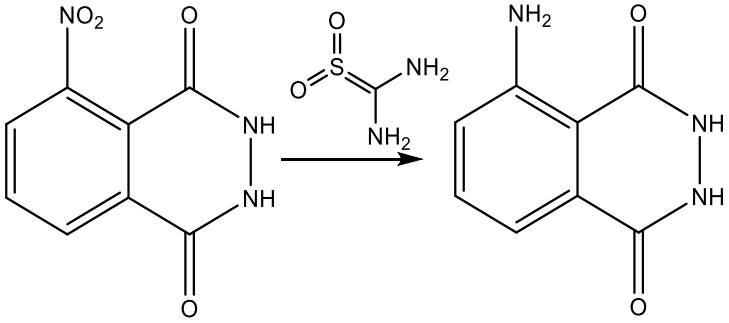
To a 500 mL round bottomed flask was added phthalic anhydride (30 g, mol), nitric acid (30.4 g, 90%), and water (10 g). Sulphuric acid (52 g, 18.4 mol dm^{-3}) was then added slowly to the stirred reaction mixture, after which the reaction mixture was heated to 90°C for 3 hours. The reaction mixture was allowed to cool to room temperature, at which point water (80 mL) was added and stirred to generate a suspension, and the precipitate filtered off. The dried solids were recrystallized from the minimum of hot water, and the crystals collected. The liquor was allowed to crystallise for 3 days in the freezer. The final yield was 14.93 g (35%) of white fluffy powder, melting at 205-213°C (lit. 218°C).

Synthesis of 3-nitrophthalhydrazide



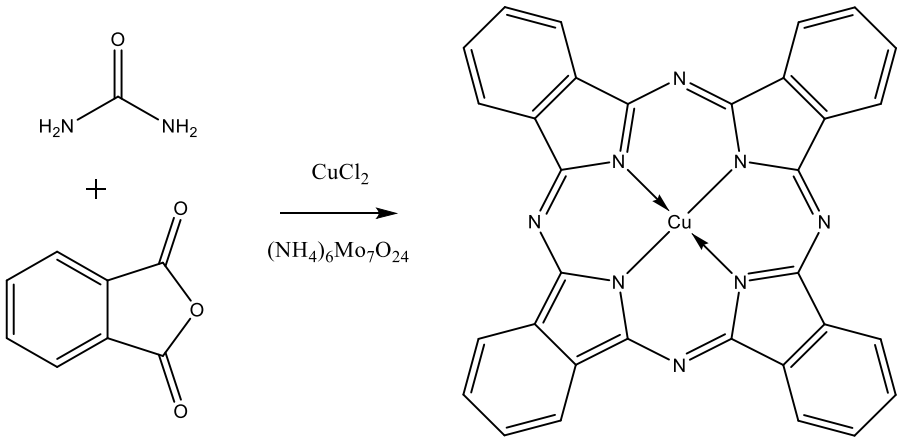
To a 125 mL round bottomed flask was added 3-nitrophthalic acid (10.00 g, 47.4 mmol), and potassium hydroxide solution (5.52 g, 94.8 mmol, in 30 mL water), and stirred until the reaction was complete. Hydrazine sulphate (6.16 g, mmol), and diethylene glycol (30 mL) were added to the reaction mixture with stirring, and then heated to 160-170°C for 3 hours. After cooling, water (60 mL) was added, and stirred to dissolve the solids, then filtered to remove the precipitate, then washed with water (30 mL). The solids were sucked dry, and then dried in a desiccator to constant weight. The final yield was 8.71 g (89%) of pale mustard-yellow powder.

Synthesis of luminol



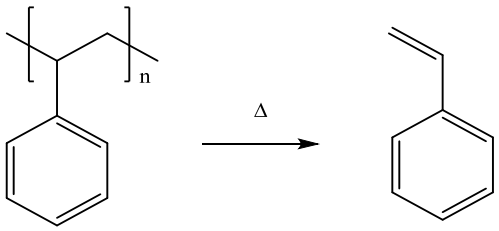
To a 500 mL round bottomed flask was added 3-nitrophthalhydrazide (6.67g, mmol), ammonia solution (75 mL, 4.5 mol dm^{-3}), and thiourea dioxide (13.94 g, mmol). The reaction mixture was then stirred at 90°C for 30 minutes after the exothermic reaction had died down. The reaction mixture was then allowed to cool to room temperature, and glacial acetic acid (21.28 g) added, and allowed to cool to room temperature. The precipitate was filtered off, and washed with hydrochloric acid (170 mL, 3 mol dm^{-3}), and water (2x 50 mL). The damp solids were then air dried to yield 3.76 g (66%) of tan-yellow powder.

Synthesis of copper phthalocyanine



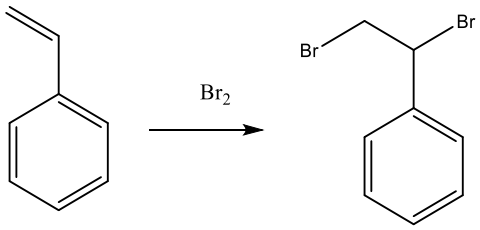
To a 150 mL beaker was added finely and intimately ground urea (5.5 g, 92 mmol), phthalic anhydride (2.96 g, 20 mmol), anhydrous cupric chloride (0.67 g, mmol), ammonium heptamolybdate tetrahydrate (60 mg, 1 mol%). The beaker was place in a crystallising dish containing silica gel for thermal insulation. The reaction mixture was then microwaved with a watch glass on top for 30 seconds in an 800 W microwave, and the cycle repeated. 6x 60 s cycles of microwaving were then conducted, with a 10 s rest period between each. After cooling, the reaction mixture was ground to a powder, and mixed with water (60 mL) with stirring. The precipitate was then filtered off, and washed with water (50 mL), sodium hydroxide solution (2x 50 mL, 10%), hydrochloric acid (2x 50 mL, 10%), water (4x 50 mL), and finally acetone (5x 10 mL). The solid was then dissolved with sulphuric acid (20 mL, 18.4 mol dm^{-3}), and filtered, then washed with sulphuric acid (10 mL, 18.4 mol dm^{-3}). The filtrate was then added dropwise to water (200 mL) in an Erlenmeyer flask. The precipitate was then filtered off, and washed with water (100 mL), then air dried to yield 0.92 g (32%) of intensely dark blue powder.

Synthesis of styrene



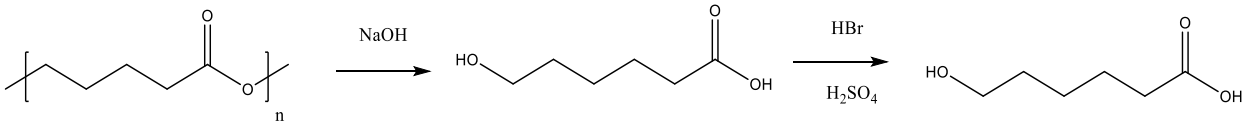
To a 50 mL round bottomed flask was added polystyrene (26.04 g, 250 mmol), and apparatus was set up for distillation. The polystyrene was then thermolyzed with a propane torch, and the vapours partially condensed with an air condenser, and the receiving flask was surrounded with ice-water. The crude styrene was vacuum distilled, and the fraction at 47-48°C / 17 mmHg to yield 34.96 g (67%) of clear colourless mobile liquid.

Synthesis of styrene dibromide



To a 250 mL round bottom flask was added styrene (34.96 g, 336 mmol), and dichloromethane (35 mL), then chilled in the freezer to below 0°C. A solution of bromine (53.86 g) in dichloromethane (35 mL) was added dropwise to the reaction flask cooled in an ice bath. The solvent was then removed, and the solid recrystallized from hot 67% isopropanol (600 g). The final yield was 73.17 g (82.6%) of fluffy opalescent white crystals with a pleasant smell melting at 69-71°C (lit. 65-73°C).

Synthesis of 6-bromohexanoic acid



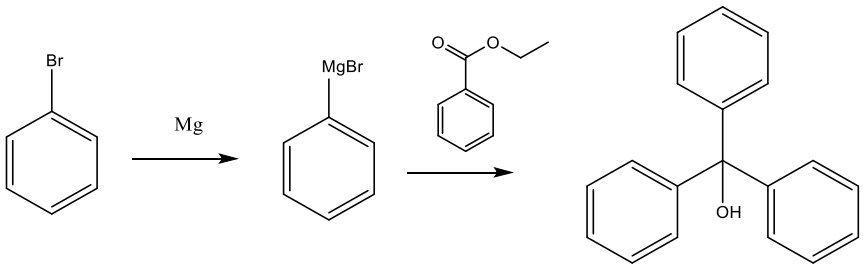
Synthesis of 6-hydroxyhexanoic acid

To a 500 mL Erlenmeyer flask was added caprolactone (0.5 mol), sodium hydroxide (25 g, 0.625 mol), and water (100 mL). The reaction mixture was stirred under gentle heat for 8 hours. After cooling, hydrochloric acid (0.625 mol, 20.2%) was added, and the reaction mixture filtered. The filtrate was reduced considerably in volume, and acetone (100 mL) added to precipitate the remaining sodium chloride. The solids were filtered off, and the solvent removed.

Synthesis of 6-bromohexanoic acid

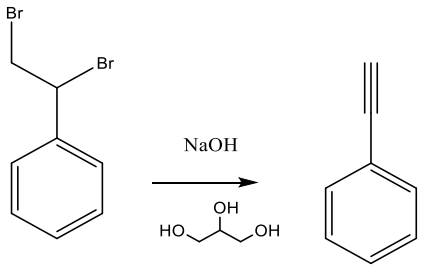
Hydrobromic acid (120ml, 48%, 2 mol eq.), and sulphuric acid (26 g, 18.4 mol dm^{-3}) were added to the residue, and refluxed for 6 hours. To the cooled reaction mixture, dichloromethane (50 mL), and water (50 mL) was added, and the lower organic phase separated off, and washed with water (100 mL, 60 mL). The solvent was removed, and dried over magnesium sulphate, then the crude product vacuum distilled, and the fraction at 180°C / 32 mmHg collected. The final yield was 53.09 g (54.4%) of colourless crystalline solid, melting at 20-32°C. Impurities due likely due to hydrogen bromide and 5-hexenoic acid.

Synthesis of triphenylmethanol



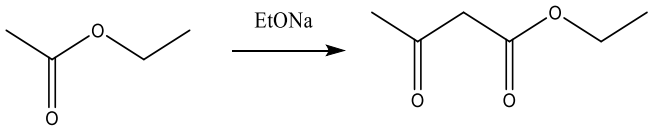
To a flame-dried 250 mL round bottomed flask was added freshly crushed magnesium turnings (2.43 g, 100 mmol), and a single crystal of iodine. The iodine was sublimed onto the magnesium, and sodium-dried diethyl ether (25 mL) added. To a flame-dried addition funnel was added dry bromobenzene (15.70 g, mmol) dissolved in sodium-dried diethyl ether (20 mL). The bromobenzene was added slowly to the reaction flask, and after the reaction mixture has stopped boiling, a warm water bath was applied for 15 minutes to reflux the ether. To the reaction mixture was dropwise added dry ethyl benzoate (7.51 g, 50 mmol) dissolved in sodium-dried diethyl ether (15 mL). After complete addition, the mixture was refluxed for 30 minutes. The reaction mixture was then poured into a mixture of sulphuric acid (100 mL, 0.5 mol dm^{-3}) and ice (25 g). The organic layer was separated off, and washed with sulphuric acid (50 mL, 1 mol dm^{-3}), and brine (50 mL). The organic layer was dried with anhydrous magnesium sulphate, and petroleum ether/hexanes (30 mL) added, and the ether removed then chilled in an ice bath. The precipitate was filtered off, and washed with cold hexanes (2x 7.5 mL). The crude yield was 2.47 g, and a second batch of 1.31 g. The first batch melted at 158-160°C (lit. 160-163°C), the second melted at 151-156°C. The second batch was recrystallized from boiling ether (12.5 mL) and hexanes (12.5 mL). Ether removed, and crystals boiled off. Final total yield was 5.84 g (45.0%) of fine white crystals.

Synthesis of phenylacetylene



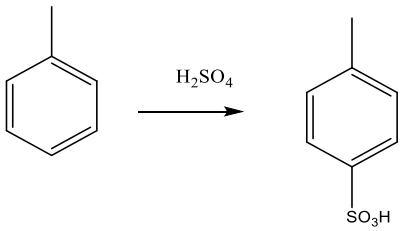
To a 500 mL two-neck round bottomed flask fitted with a thermometer and a calcium chloride drying tube was added sodium hydroxide (40 g, 1 mol), and dry glycerol (250 mL). The reaction mixture was heated and stirred to dissolve the solids, then styrene dibromide (65.99 g, 0.25 mol) was added. The reaction mixture was then heated to 200°C, and the apparatus set up for distillation. The reaction was run until no more distillate was collected at 200°C. To the distillate was added diethyl ether (20 mL) and the organic layer separated, then washed with distilled water (30 mL), then dried over magnesium sulphate. Diethylene glycol (20 g) was then added, and the solution vacuum fractionated, collecting the product at 79-83°C / 79 mmHg. The final yield was 15.30 g (59.9%) as a clear colourless liquid.

Synthesis of ethyl acetoacetate



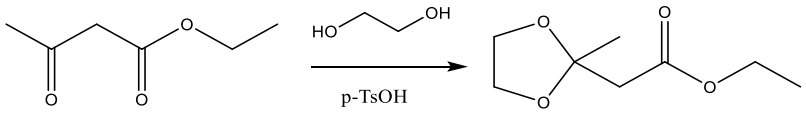
To a 1000 mL round bottomed flask was added ethyl acetate (196 g, 2.22 mol), anhydrous ethanol (4 g, mmol), and freshly sliced sodium (20 g, mol). The mixture was refluxed until the sodium had all dissolved. Acetic acid (110 mL, 50% in water) was then added and the organic layer separated off, and the solvent removed. A vacuum distillation was then set up, and the fraction at 85-91.5°C / 30 mmHg collected. The final yield was 73.8 g (25.5%) of clear slightly yellow liquid.

Synthesis of p-toluenesulphonic acid monohydrate



To a 200 mL round bottomed flask was added sulphuric acid (37 g, 18.4 mol dm^{-3}) and toluene (100 mL). The reaction mixture was refluxed with a Dean-Stark trap for 5 hours until 7.3 mL (100% - 7.4 mL) of water had collected. The reaction mixture was then allowed to cool to room temperature, and water (6.7 g) added. The precipitate was filtered off, and recrystallized from water (25 mL) by saturating the solution with hydrogen chloride. The crystals were filtered off, and dried over concentrated sulphuric acid and sodium hydroxide for 2 weeks when the mass had stopped dropping to yield 40.54 g (67.6%) of slightly off-white very hygroscopic powder melting at 99-103°C (lit. 103-106°C); impurity likely water.

Synthesis of fructone



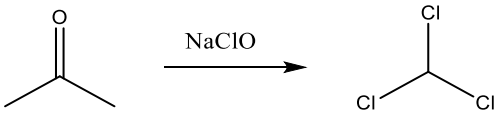
To a 100 mL round bottomed flask was added ethyl acetoacetate (13.01 g, 100 mmol), ethylene glycol (6.52 g, mmol), and p-toluenesulphonic acid monohydrate (60 mg). Benzene (60 mL) was added to a Dean Stark tap, and the reaction mixture was then refluxed for 1.5 hours until 1.7 mL of water had collected. The Dean Stark trap was drained, and the remaining benzene distilled off. Dichloromethane (30 mL) was added, and washed with sodium hydroxide (15 mL, 10%). The lower organic phase was separated, and washed with water (2x 25 mL), then dried over calcium chloride. The solvent was removed, and a short-path vacuum distillation apparatus set up, and the product collected at 115°C / 44 mmHg. The final yield was 10.96 g (62.3%) of clear colourless sweet-smelling liquid.

Preparation of bromine

$2\ KMnO_4 + 10\ NaBr + 14\ H_2SO_4 = 2\ KHSO_4 + 10\ NaHSO_4 + 8\ H_2O + 5\ Br_2 + 2\ MnSO_4$

To a 500 mL round bottomed flask set up for distillation fitted with a pressure equalising addition funnel was added anhydrous sodium bromide (308.68 g, 3 mol) and dissolved in water (350 mL). Finely powdered potassium permanganate (94.22 g) was then added with constant stirring, and sulphuric acid (500 g, 93%) added to the addition funnel. The sulphuric acid was added slowly, and the bromine collected, heating the reaction flask as necessary. After no more bromine was condensed, the distillation was stopped. The upper aqueous layer was removed from the distillate, and washed with sulphuric acid (25 mL, 98%) and stored in a polythene bottle in the freezer. The final yield was 228.14 g (95.2%).

Synthesis of chloroform



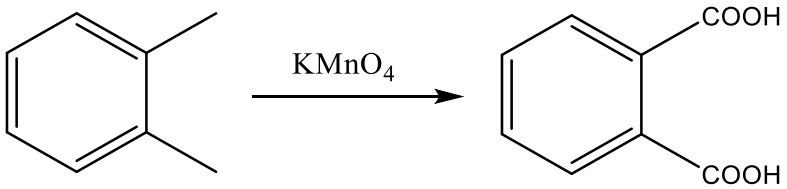
To a jug of bleach (4.5 L, 6.0%, 4.5 mol) cooled to -2°C and acetone (87.12 g, 1.5 mol) was added. This was a 2% excess of sodium hypochlorite. The jug was shaken and allowed to react in a cool place and separate. The upper aqueous layer was decanted, and the lower organic layer separated. The product was washed once with brine, then dried over calcium chloride and the process repeated, and the combined products distilled collecting the fraction at 60°C. The final yield was 238 g (66.5%).

Synthesis of fuming nitric acid

$NaNO_3 + H_2SO_4 \rightarrow HNO_3 + NaHSO_4$

To a 500 mL round bottomed flask set up for distillation was added ammonium nitrate (200 g), and cold sulphuric acid (280 g, 93%, -20°C). The reaction mixture was distilled under vacuum until no more distillate collected. The final yield was 175 g (93%) of 90% nitric acid.

Synthesis of phthalic acid



To a 500 mL three necked round bottomed flask fitted with a reflux condenser and a solid addition funnel was added o-xylene (10.63 g), water (200 mL), and potassium permanganate (63.27 g) to the solids addition funnel. The potassium permanganate was added gradually over the course of the reaction. After complete addition and the reaction judged complete, the reaction mixture was allowed to cool to room temperature and the precipitate filtered off. Hydrochloric acid (50 g, 36%) was added to the stirred filtrate, and the precipitate filtered off and washed with ice water. The product was then recrystallised from water. The final yield was 8.95 g (53.8%).