

# Isolation of aspirin

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Once the aspirin is prepared, it is isolated from the reaction solution and then it is purified. The aspirin is insoluble in cold water, and it is isolated by filtering the chilled reaction solution. Purification is essential to remove any unreacted salicylic acid and acetic anhydride as well as the acetic acid product and phosphoric acid. Acetic anhydride is caused to decompose by the addition of water once the formation of aspirin is complete.  $C_4H_6O_3$  (Acetic anhydride) +  $H_2O$  (Water) ----->  $2C_2H_4O_2$  (Acetic Acid)

The acetic acid and phosphoric acid are water soluble and it is removed by washing the aspirin with chilled water. Salicylic acid is only slightly soluble in water and is not completely removed in the washing steps. Phosphoric acid can be used instead of sulphuric acid if desired to obtain the higher yield, as sulphuric acid reacts more readily with the organic molecules involved in the reaction than phosphoric acid. However, phosphoric acid does not absorb water in the reaction; therefore it may be a slower process. Final purification is completed by the process of Recrystallisation. By recrystallising the crude aspirin slowly, it was possible to obtain large crystals with an exact structure by allowing the aspirin molecules to join together in a precise way. The regular molecular crystal structure of the final product makes it more difficult for impurities to be included, eliminating impurities present in the formless crude product. The impure aspirin is dissolved in warm ethanol. The solution is then cooled slowly, and the aspirin crystallises out of solution leaving the salicylic acid and other impurities behind. In my experiment, pure aspirin was obtained after filtering out the impurities and excess reagents through the filter paper. A method to check a solid compound's purity after recrystallisation is to check its melting point. The melting point of a

compound can be used to identify it and also to estimate its purity. Normally an impure compound will show a melting point which is lower than that of a pure compound. Therefore, if the sample of aspirin melts at a temperature below the accepted melting point two possibilities can exist; either the sample is impure or it is not aspirin. A pure substance will melt sharply at 1-20C per minute when nearing the expected melting point in order to get a more accurate range. An impure compound will melt over a wider temperature range.