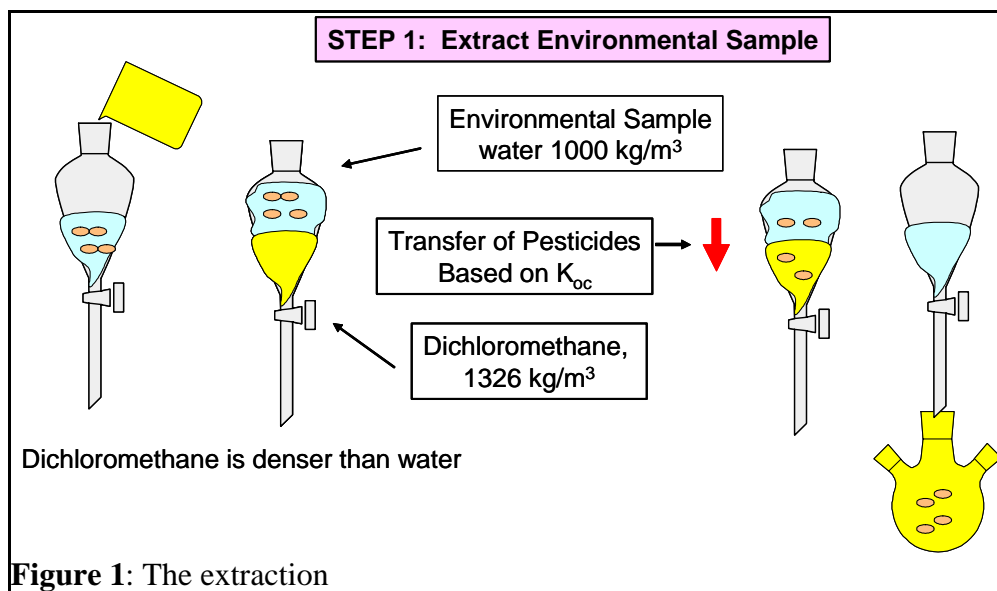
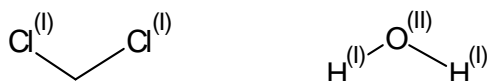


# The Chemistry of Extraction and Preparation

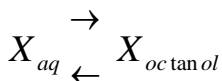
## Extraction



The aqueous environmental sample suspected of containing pesticides is placed in a separatory funnel and dichloromethane (yellow) added. The dichloromethane is denser (heavier) than water so it sinks to the bottom of the funnel.



Because pesticides high partition coefficients from water to octanol,  $K_{OW}$ , we predict that they will also move from water to dichloromethane.

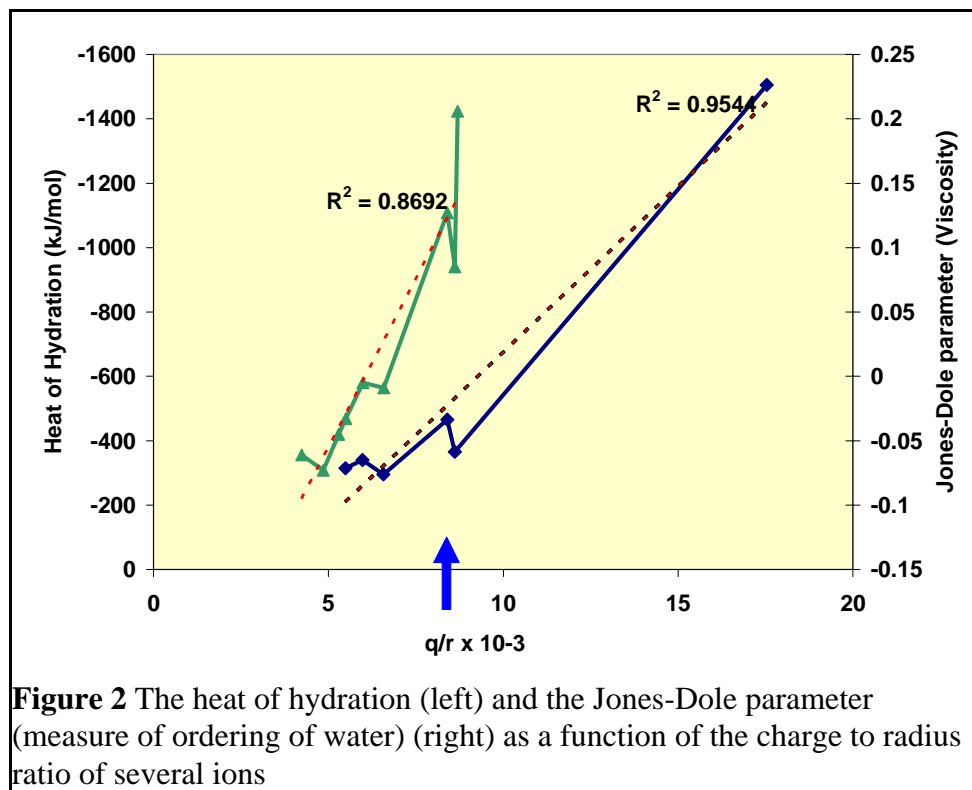


$$K_{OW} = \frac{[X]_{aqueous}}{[X]_{oc\ tan\ ol}}$$

After shaking to help move the pesticides into the dichloromethane fraction we pull off the dichloromethane containing the pesticides.

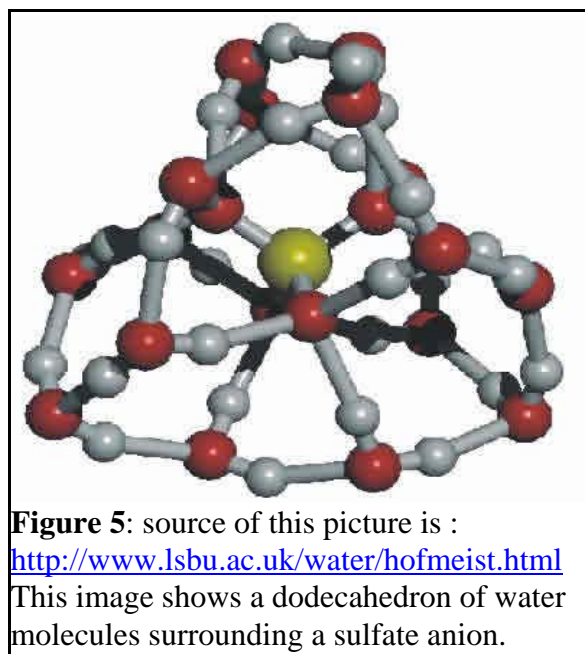
## Removal of Water

The sample is next concentrated. If the sample is concentrated while containing trace amounts of water the rate of hydrolysis of the pesticide may increase (increasing concentration



**Figure 2** The heat of hydration (left) and the Jones-Dole parameter (measure of ordering of water) (right) as a function of the charge to radius ratio of several ions

of water). (See Environmental Chemistry module). Therefore we must remove any trace amount of water within the sample prior to reducing the volume. This is accomplished by passing the dichloromethane containing the pesticides through a filter which contains anhydrous sodium sulfate. The reactive ingredient is the sulfate which strongly binds water. The reason for the strong binding of water is



**Figure 5:** source of this picture is : <http://www.lsbu.ac.uk/water/hofmeist.html>  
This image shows a dodecahedron of water molecules surrounding a sulfate anion.

related, in part, to the charge of the sulfate anion (2-) to the volume of the anion (as measured by the ionic radius). The larger the charge per volume the more likely the anion is to electrostatically attract water close enough to bind.

Figure 2 shows the heat of hydration and the Jones-Dole parameter (a measure of water organization) for a variety of anions with  $q/r$  (charge to radius) along the x axis. Sulfate causes a high degree of ordering of water molecules around it do to the number of oxygen and their arrangement which allows a cage of water to be formed. Figure 3 shows an image of the high degree of order of water around a sulfate anion.

## Concentration and Change of Solvent

In order to increase the sample concentration for analysis by GCMS we evaporate down the dichloromethane solvent containing the pesticide. The ease of evaporation is another reason that the dichloromethane was chosen for the extraction. Dichloromethane boils at 39.7 C, a temperature too low to be stable for storage of the sample. The temperature is low enough that injection of the solvent into the GC would require a cold injection in order to stabilize the sample and prevent rapid boiling off with loss of the sample. Hexane boils 30 C higher at 59 C. Acetone boils at 56.5. The mix of hexane and acetone is optimized to adjust the polarity of the solvent and its ability to separate compounds eluting on the GC column.

