**Background:**

This experiment involves analysis of a hypothetical analgesic (pain-killing) drug mixture dubbed "Spartacetin." As with many over-the-counter painkillers, Spartacetin is a mixture of aspirin and another analgesic organic compound (either acetanilide or phenacetin) that you will identify by separation of the components and melting point analysis.

![Chemical structures of acetanilide, phenacetin, and acetaminophen.](attachment:chemical_structures.png)

You are already familiar with aspirin, the most widely used drug in the world. Acetanilide and phenacetin were discovered earlier (1886 and 1887, respectively) than aspirin (the remarkable properties of which were realized at the turn of the century). Phenacetin was Bayer's first pharmaceutical product, created from waste materials left over from its dye manufacturing. Acetanilide and phenacetin are both antipyretic and analgesic, and owe their activity to metabolic breakdown to paracetamol, better known as acetaminophen (the active ingredient in Tylenol). Note that acetaminophen, unlike aspirin, is not anti-inflammatory, but it has the benefit of not irritating the stomach lining. Acetaminophen has fewer side effects, and has largely replaced acetanilide and phenacetin. (Ironically, Bayer first prepared phenacetin from acetaminophen on the assumption that the phenolic OH group would irritate the stomach, so they wanted to "mask" the -OH as -OCH\(_2\)CH\(_3\).)

Bayer's next addition to the pharmaceutical industry was (rediscovered) diacetylmorphine, prepared by Felix Hoffmann of aspirin fame within just two weeks of the aspirin synthesis. They tested this new drug on their workers and found in made them
feel heroic, thus the name "heroin.". Believing they had a non-addictive alternative to morphine, heroin was marketed for a variety of ills, including baby's colic.

A common mixture of drugs, no longer marketed in the US because of side effects and possible carcinogenicity, is APC - aspirin, phenacetin, caffeine. Caffeine is often mixed with aspirin. It is interesting that caffeine intensifies the analgesic properties of aspirin - so if you take a plain aspirin tablet, chasing it with coffee, tea, or caffeinated soda should help.

![Tetraglyme Chemical Structure](image)

**tetraglyme**

In any case, our mixture, Spartacetin contains no caffeine (or heroin), but it does have, in addition to aspirin and the "unknown" analgesic (either acetanilide or phenacetin) two other components: starch and tetraglyme. Starch is representative of binder compounds used to keep the drug together in pill form, and tetraglyme (AKA tetraethyleneglycol dimethyl ether) is an organic impurity in the questionable batch of Spartacetin you are asked to analyze.

**The Experiment:**

The best way to understand this experiment is to look at the flow diagram while reading this section and also the procedure section. Just the basic design of the experiment will be discussed here.

The goal is to separate the components of Spartacetin and to analyze the unknown component (acetanilide or phenacetin) by melting point analysis. The amounts of components recovered will allow you to estimate the original composition of the Spartacetin you were given. The key techniques are liquid-liquid extraction, melting point analysis, and recrystallization (used to remove the tetraglyme impurity from the crude unknown). You may wish to view the technique videos.

The following table of solubilities will be useful to understanding the design of the experiment:
We will first remove the starch by gravity filtration. The dichloromethane filtrate contains the remaining components. Extracting with water would not help in separation since aspirin and the unknown are both sparingly soluble in water. We will therefore do employ the technique of "chemically active extraction," taking advantage of the fact that our unknown is not acidic whereas aspirin is. So when we extract the dichloromethane solution with aqueous sodium hydroxide, the aspirin is deprotonated and migrates to the aqueous layer, leaving the unknown (and most of the tertaglyme behind in the dichloromethane layer.

We isolate the aspirin as before, by acidifying, chilling, and vacuum filtration. The crude unknown is isolated by evaporation of the (low boiling) dichloromethane. We recrystallize the crude unknown from water to obtain the purified unknown.

Melting point and mixed melting point analysis is used to identify the unknown. We will also do a melting point on a set aside sample of crude unknown as a way of observing the efficiency of the purification process (the purified unknown would be expected to have a higher and narrower melting point range than the crude material).