

## Synthesis of acetylsalicylic acid – Protocol K

(following protocol reference 1)



**Reaction.** Place 10.0 g (36,2 mmol) of dry salicylic acid and 7.0 mL of dry pyridine in a 100mL conical flask. Then, without delay (this solution tends to become a semi-solid mass), run in 7.5 mL of acetyl chloride, adding about 1 mL of the chloride at a time, and shaking the mixture continuously during the addition. The heat of the reaction causes the temperature of the mixture to rise rapidly: therefor maintain the latter between 50 °C and 60°C throughout the addition, cooling the flask occasionally in cold water, if necessary. Finally, heat the mixture on a boiling-water bath for five minutes.

**Isolation.** After cooling in cold water, pour it in a thin stream into about 300 mL of cold water, stirring the mixture vigorously meanwhile. The crude acetylsalicylic acid either solidifies at once or separates as an oil, which rapidly crystalizes as the stirring proceeds. Liter the solid product at the pump, wash thoroughly with water and drain.

**Purification.** Recrystallize from a mixture of equal volumes of water and acetic acid.

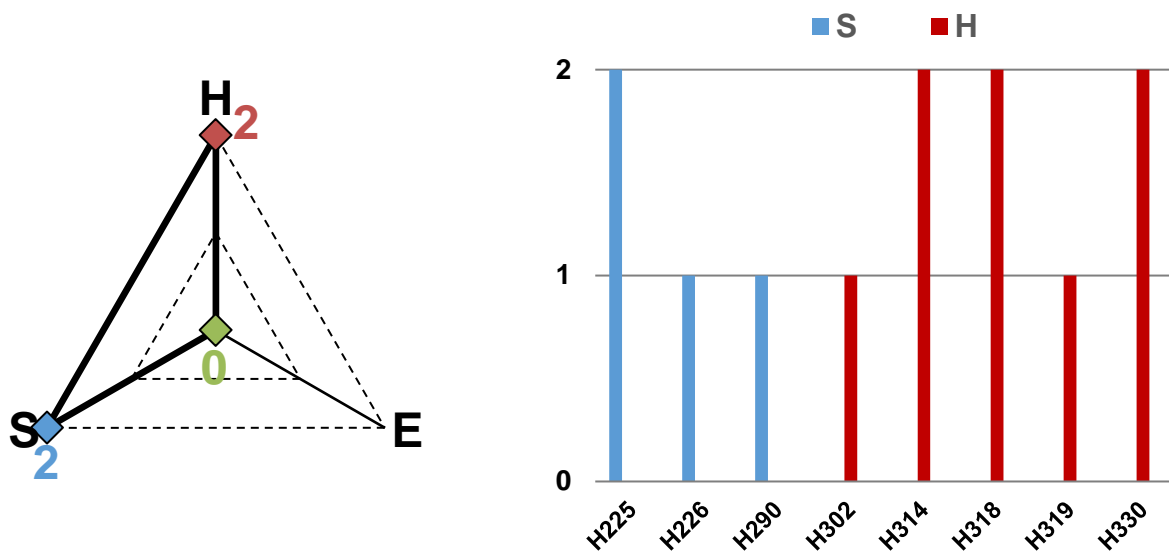
### Application of the SHE Tool

The application of SHE Tool for each substance involved is presented in Figure 1 (the SHE Triangle and the Spectra of Potential Hazards (SPH)) and Figure 2 (the global SHE Triangle and PHS).

For details about the construction of SHE tool see reference 2.

SHE Triangle	Hazard codes and hazard statements		Spectra of potential hazards (SPH)
<b>Salicylic acid</b>			
	H302	Harmful if swallowed.	
	H318	Causes serious eye damage.	
<b>Acetyl chloride</b>			
	H225	Flammable liquid and vapour.	
	H314	Causes severe skin burns and eye damage.	
<b>Pyridine</b>			
	H225	Flammable liquid and vapour.	
	H302	Harmful if swallowed	
	H312	Harmful in contact with skin	
	H315	Causes skin irritation	
	H332	Harmful if inhaled	
<b>Acetylsalicylic acid</b>			
	H302	Harmful if swallowed	
<b>Acetic acid</b>			
	H226	Flammable liquid and vapour.	
	H290	May be corrosive to metals	
	H314	Causes severe skin burns and eye damage.	
<b>Hydrochloric acid (diluted solution)</b>			
	H290	May be corrosive for metals	
<b>Water and acetic acid (aqueous solution)</b>			
		Without indication of hazards	

**Figure 1.** SHE analysis of the substances involved in the synthesis of acetylsalicylic acid: SHE triangles, hazard codes, hazard statements and SPH; ■ – physical hazards; ■ – health hazards; in red the hazard statements with the highest punctuation.



**Figure 2.** Global SHE Triangle and Spectra of Potential Hazards

### References

- (1) Vogel, A.I. Elemental Practical Organic Chemistry. Longmans Green and Co, Ltd: London, 1958, pp. 324-325.
- (2) Greenness digital catalog of laboratorial activities for teaching Green Chemistry.  
[http://educa.fc.up.pt/catalogo/en/construcao\\_she](http://educa.fc.up.pt/catalogo/en/construcao_she)