


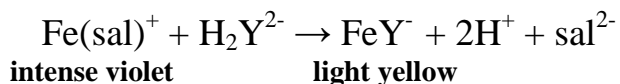
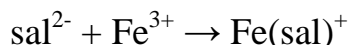
## Exercise no. 8

### Determination of Fe<sup>3+</sup> by EDTA titration using salicylic acid indicator

#### *Introduction*

The concentration of Fe(III) in sample solution will be determined by reaction with EDTA. The complexation is 1 to 1; each mole of EDTA complexes with a mole of Fe(III) forming a mole of Fe-EDTA complex. This reaction is carried out as a titration whereby the EDTA reagent is added slowly from a burette until an endpoint is reached, signalling that the moles of EDTA added equals the moles of Fe(III) in the solution being analyzed. In this experiment the indicator is

the **salicylic acid** (sal, C<sub>7</sub>H<sub>6</sub>O<sub>3</sub>, HOC<sub>6</sub>H<sub>4</sub>COOH, ) which forms in acid solution an **intensely violet** coloured complex with Fe(III). The Fe-EDTA (FeY<sup>-</sup>) complex on the other hand is **light yellow** (or colourless). So, the actual reactions are:



The formation of the EDTA complex is significantly favoured over the salicylate complex (it has a greater formation constant). The original solution is dark violet because of the Fe-salicylate complex. As the titration proceeds the solution retains a reddish colour until enough EDTA has been added to react with all the original Fe(III) salicylate complex. At that point (the endpoint) the final traces of red disappears and we see only the pale yellow of the Fe-EDTA. From the known concentration and volume of EDTA solution added at the endpoint, we calculate the concentration of iron in the sample solution.

### ***Experimental Procedure***

Dilute the analytical sample with distilled water in a measuring flask to the mark (100 mL) and mix completely. The solution in the measuring flask should be treated as 100 mL of the water sample. **Pipette** 10 mL of the solution into the conical flask. Add 50 mL of deionized water. Adjust pH of sample solution below pH 4, by adding 20 mL of 0.1 M HCl and 25 mL glycine (Gly, C<sub>2</sub>H<sub>5</sub>NO<sub>2</sub>). Swirl to mix. Add 0.5 mL of **salicylic acid indicator** (6% in acetone) to the solution. Swirl to mix. The solution should be a violet because of formation of (Fe(sal))<sup>+</sup> complex. Titrate the solution immediately with EDTA (0.005M) against a white background until the **violet** solution turns a **light yellow**. Read the final volume (save the solution for colour comparison). Repeat the titration to obtain three concordant results (not differing more than 0.2 mL). Calculate the amount of carbonates and bicarbonates present in the sample using formula:

$$X = 0.005 \cdot V \cdot 55.85 \cdot 10 \text{ [g]}$$

V – volume of EDTA [L]

55.85 – molar mass of Fe [g/mol]