MANGANOMETRY– Determination of the total iron concentration in iron ore.

Laboratory instruction

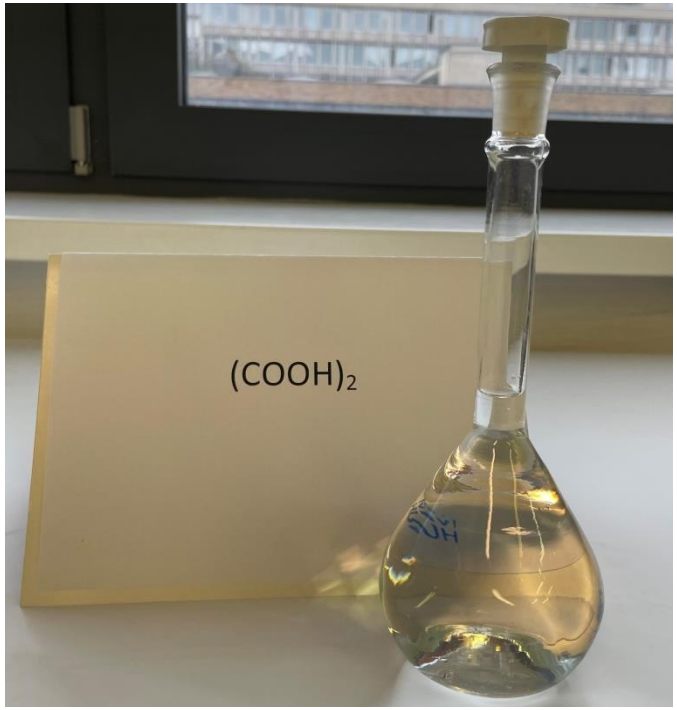
Required working solutions:

- Stock solution of standard compound - oxalic acid $(COOH)_2$.

The solution is prepared by differential weighing on analytical balances.

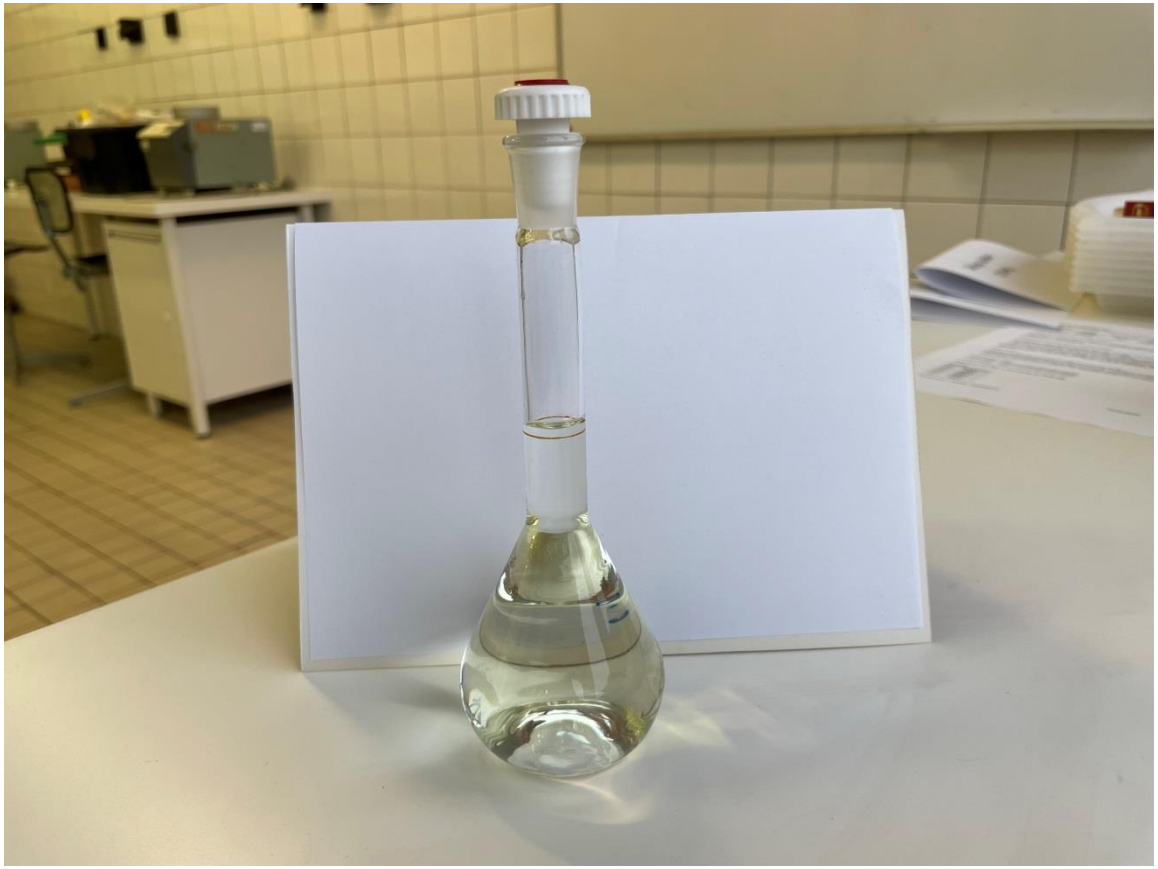
V=250 ml, c=0,015 mol/l.

Please note that the oxalic acid exists in solid form as dihydrate (COOH) 2.2H2O.

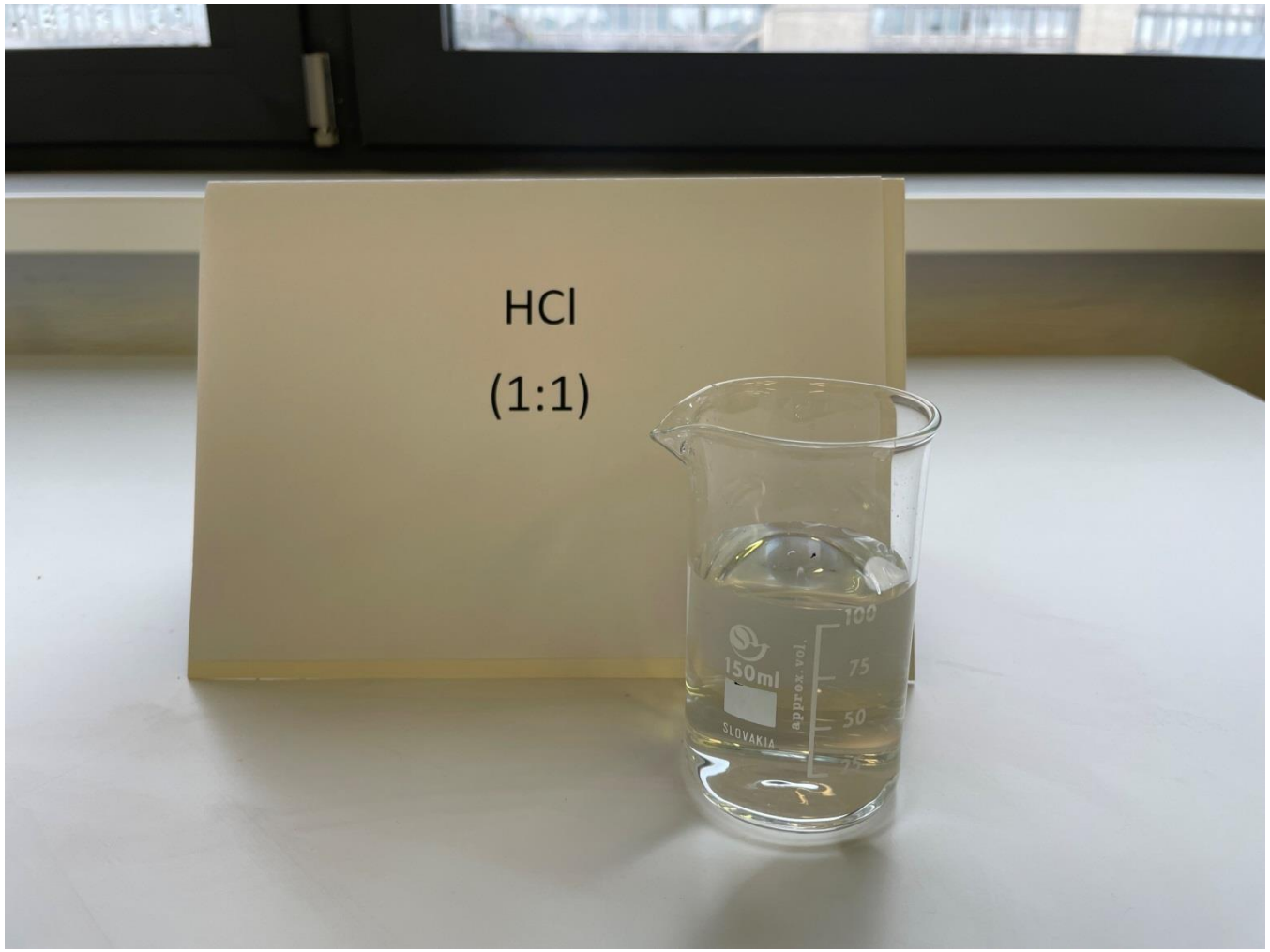


- (Volumentric) Solution of $KMnO_4$ with approximate concentration of 0,012 mol/l. The solution is prepared by dilluting of stock solution with approximate concentration of 0,2 mol/l in 250 ml volumetric flask

- Sample solution:



- Solution of dilluted HCl:



- Other required solutions:
  - 34 % sulfuric acid
  - 7 % mercuric (II) chloride
  - Zimmermann-Reinhardt's reagent
  - 12,5 % stannous(II) chloride

- Burette preparation:

Wash the burette twice with deionized water, after by (volumetric) solution and fill with a permanganate solution to the zero mark (in the case of permanganate solutions, observe the upper meniscus in the burette!).

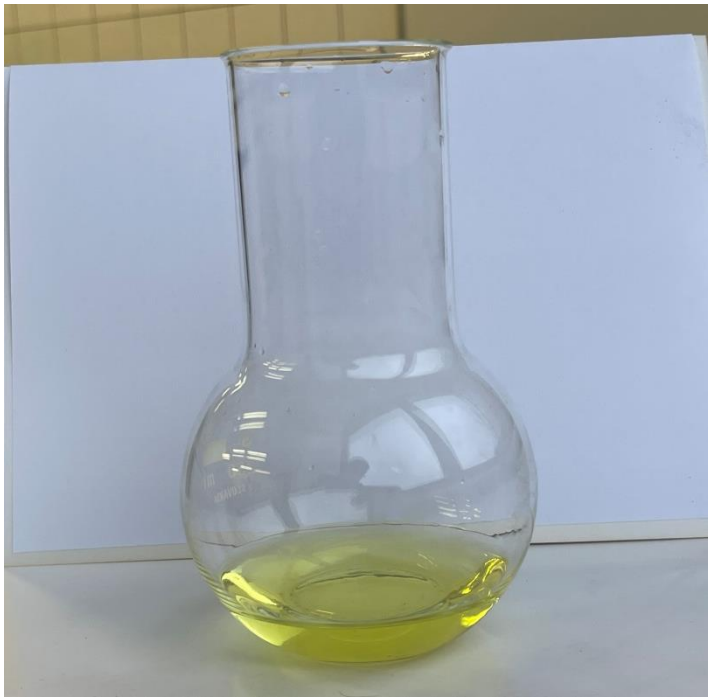
Standardization of permanganate solution:

Pipette 25 ml of oxalic acid stock solution into the titration flask, add 2 ml of H_2SO_4 (34%) and heat to boiling point. Titrate with potassium permanganate until the first permanent pink color. Whole standardization repeat 3 times . Obtained volumes record and average.

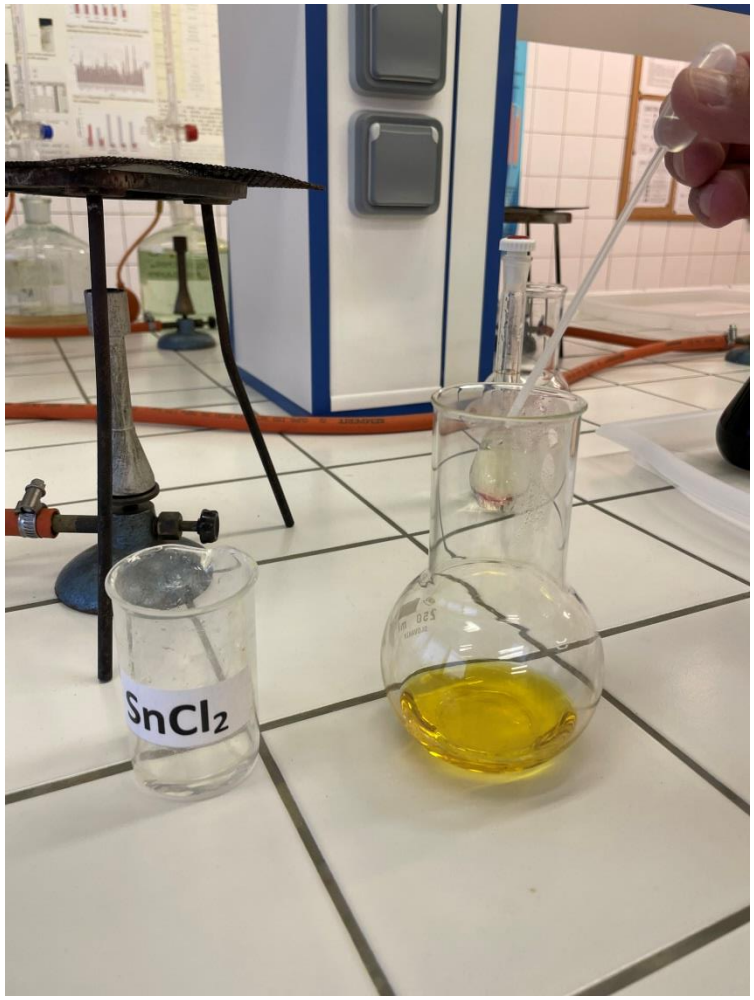
The permanganate concentration is calculated from solution consumed during titration.

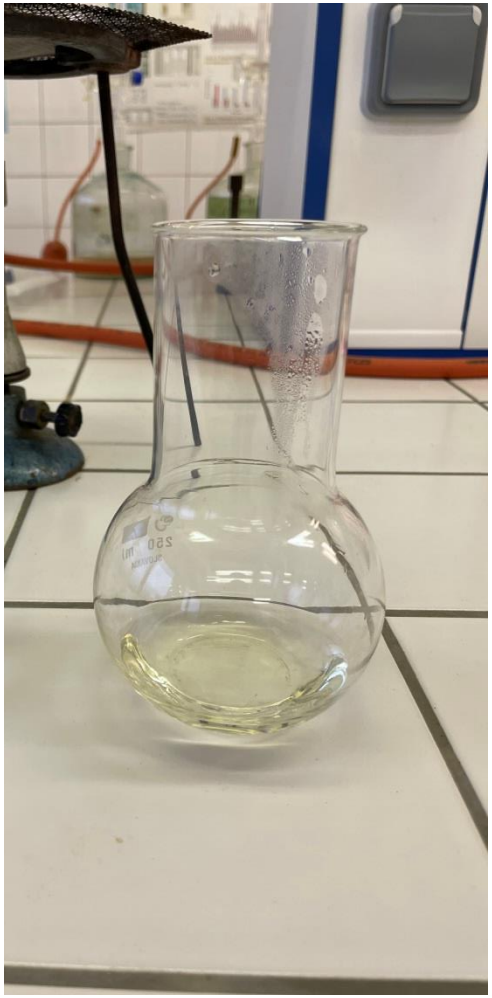
Determination of Total iron Fe^{2+} and Fe^{3+} :

1. Pipette 25 ml of the sample solution into the titration flask, add 10 ml of diluted HCl (1: 1) and heat the solution to boiling

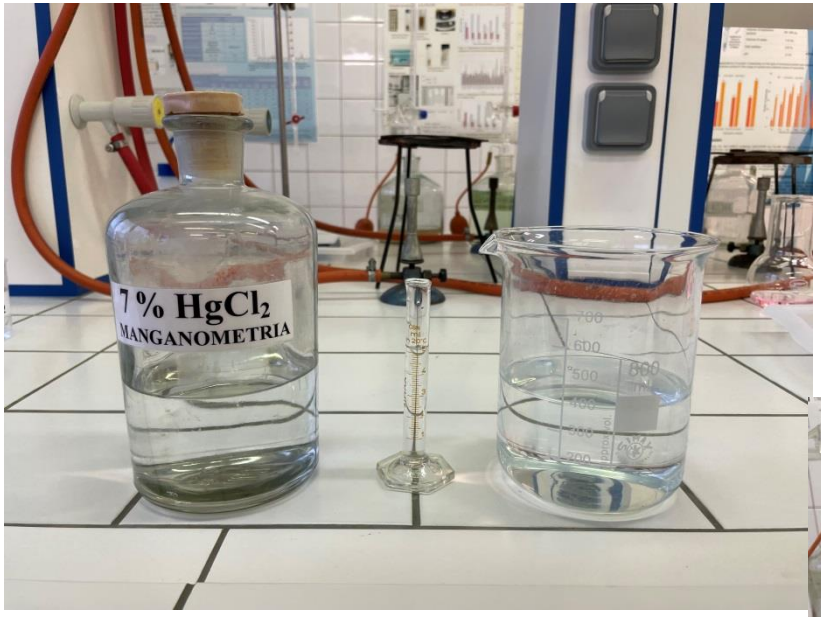


2. To the hot yellow sample solution, add $SnCl_2$ dropwise to the decolorization (+2 extra drops). This will reduce Fe^{3+} ions in solution to Fe^{2+}

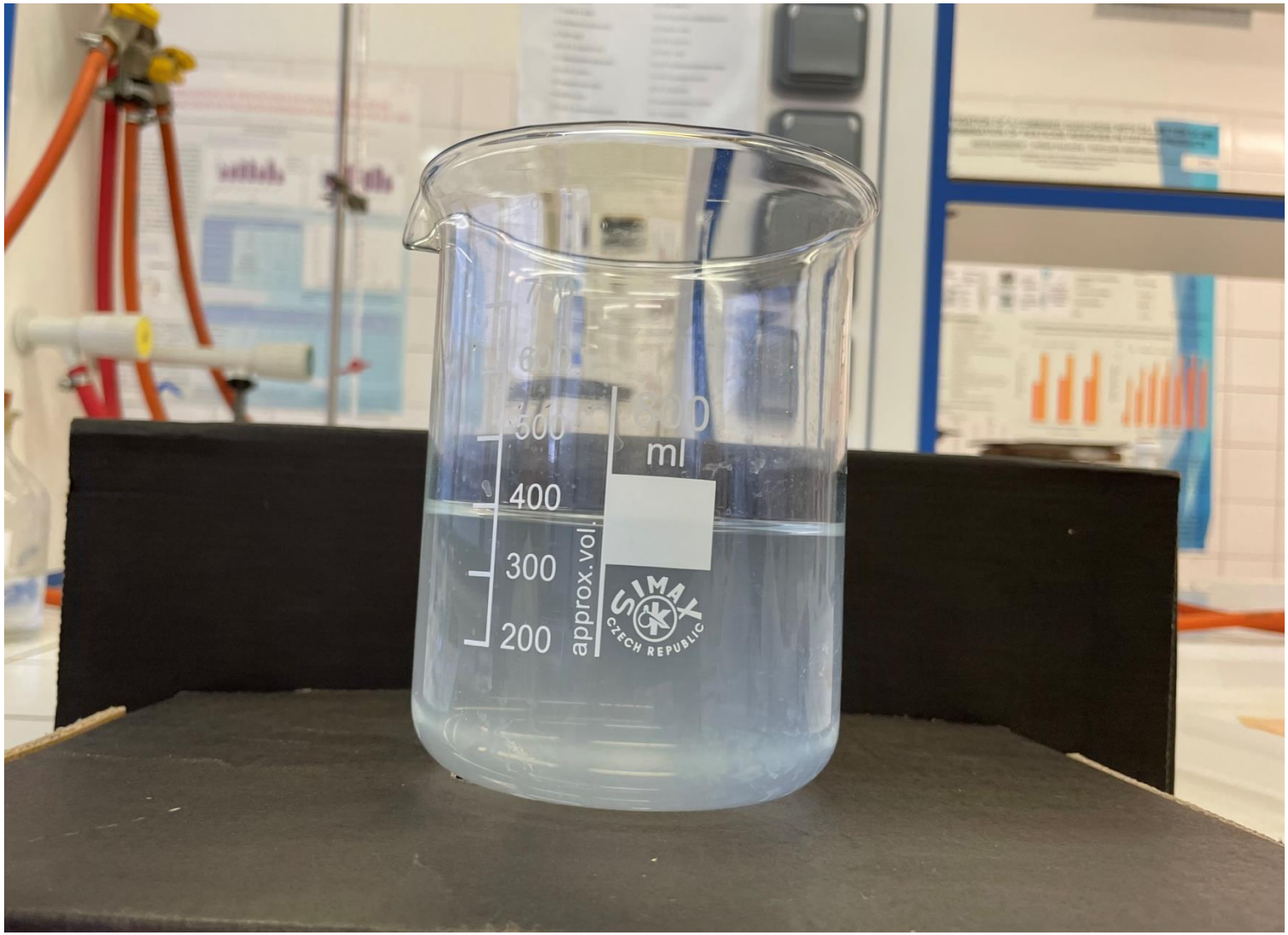




3. Transfer the solution quantitatively to an 800 ml beaker and dilute to 400 ml with water. After cooling, add 5 ml $HgCl_2$ all at once, with vigorous stirring.



A slight white haze develops after 2-5 minutes, .



4. Add 10 ml of Zimmermann-Reinhardt's reagent.

Zimmermann-Reinhardt's reagent contains:

$MnSO_4$: prevents oxidation of chlorides and thus increases the consumption of $KMnO_4$

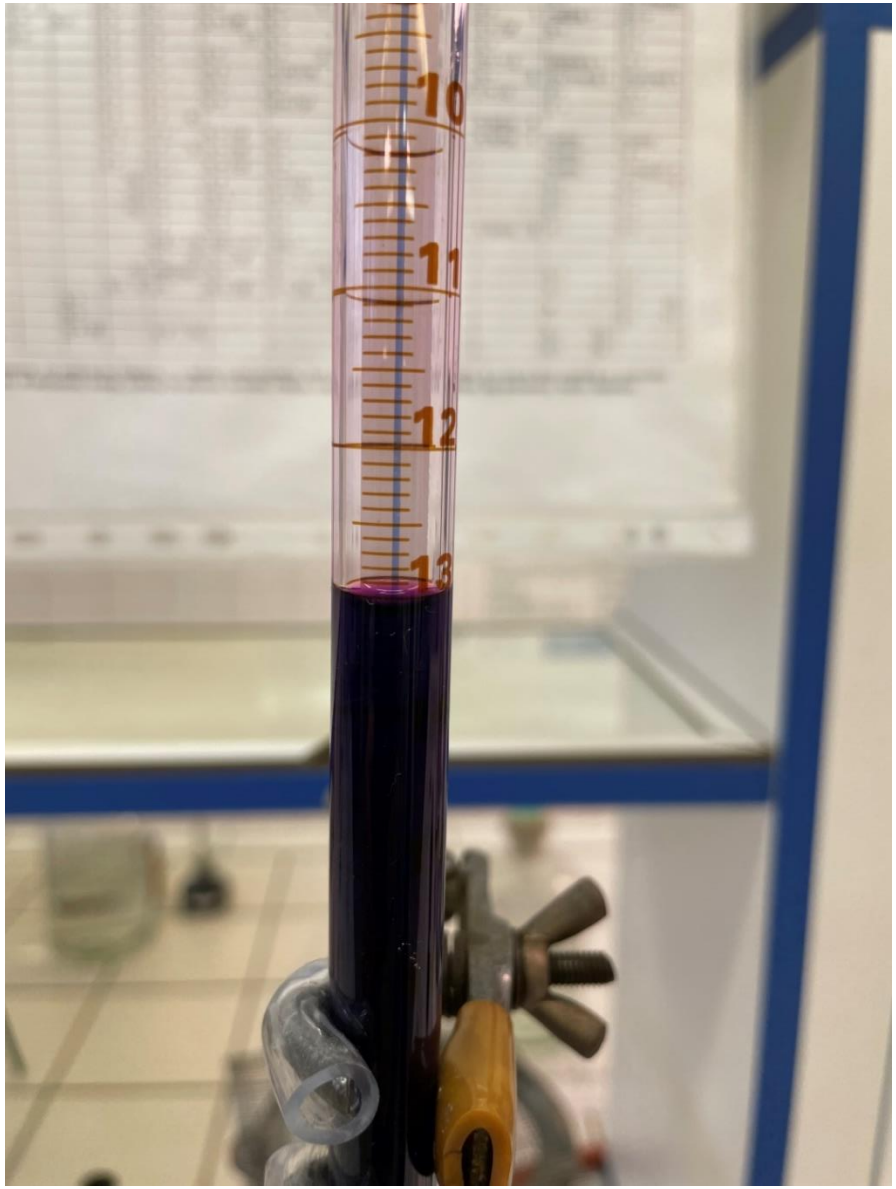
H_3PO_4: binds Fe^{3+} do colorless complex

H_2SO_4: form acidic environment

5. Titrate the entire contents of the beaker with permanganate solution until a pale pink color persists for at least 15 seconds. Mix the mixture with a glass rod.

Whole determination repeat 3 times . Obtained volumes record and average.

6. Calculated averaged consumption from the burette use for calculation of the weight of Fe in the supplied sample.



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