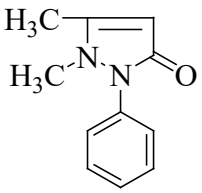
## PRACTICES

### Quantity Determination of Antipyrine (Phenazone)



M.W.=188

1-phenyl-2,3-dimethyl-5-pyrazolone

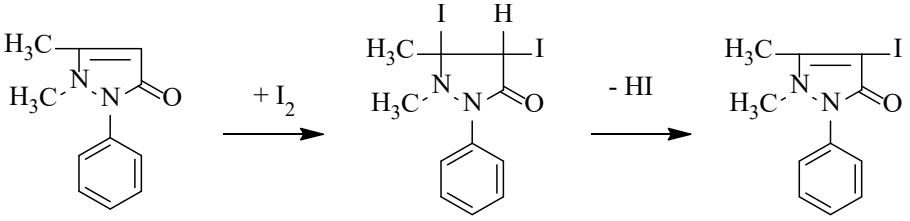
100 mg of substance is dissolved in 18 ml of 10% sodium acetate. Therefore, the reaction turn back resulting from the strong reducer effect of HI will be prevent. 15 ml of 0.1 N $I_2$ solution is added and waited for 20 minutes in dark place by often shaking. 5 ml of $CCl_4$ solution is added. It is shaken to dissolve the precipitate. Excess of iod is titrated by 0.1 N $Na_2S_2O_3$ solution. The end point of the reaction is determined by the disappearance of the pink color in the $CCl_4$ layer.

1 ml 0.1 N $I_2$ ……. is equivalent to 9.412 mg phenazone

(15 x f $I_2$) - (b x f $Na_2S_2O_3$)…… A mg

A x 100/tartım= % phenazone

**Reaction equation:**



$$HI + CH_3COONa \longrightarrow NaI + CH_3COOH$$

$$I_2 \text{ (excess)} + 2\ Na_2S_2O_3 \longrightarrow 2\ NaI + Na_2S_4O_6$$

**Standart solutions used in iodometric titration**

0.1N $I_2$, 0.1N $Na_2S_2O_3$

**Preperation of 0.1N $I_2$ Solution**

0.1 N iod solution contains 12.69 g of iod in 1000 ml. 13 g of iod and 30 g of KI is dissolved in 40 ml of water and complete volume to 1000 ml with water.

**Adjustment of 0.1N $I_2$ Solution to Arsentrioxide**

In the erlenmeyer, 0.2 g of dried $As_2O_3$ is dissolved in 2 ml of 20% NaOH by heating if necessary. 40 ml of water and 0.2 ml of methyl orange is added to the solution. Until the colour turn red from yellow, dilue HCl is added. Then, the solution is neutralized by $NaHCO_3$. In order to prevent the

formation of HI acid, 2 g of weak base $Na_2CO_3$ is added. The primary standart is titrated by 0.1 N $I_2$ solution in the presence of 10 drops of starch solution until forming blue colour.

1 ml of 0.1N $I_2$……is equivalent to 0.004946 g $As_2O_3$

**Reaction equation:**

$$As_2O_3 + 2\,I_2^{O} \longrightarrow As_2O_5 + 4\,HI$$

**Preperation and adjustment of 0.1N $Na_2S_2O_3$ Solution**

$Na_2S_2O_3$. $5H_2O$: 248.19

1000 ml of 0.1N $Na_2S_2O_3$ solution contains 24.82 g of $Na_2S_2O_3$.

26 g of $Na_2S_2O_3$ and 200 mg of $Na_2CO_3$ are dissolved in newly boiled and chilled distilled water and complete volume to 1000 ml.

In the adjustment, the reaction between thiosulfate ion and elemental iodine is utilized. Therefore, potassium iodate, potassium bromate, potassium bichromate, copper which release iodine or iodine from KI in acidic solution are used as standart.

Since iodine and thiosulphate solutions are used together in practice, firstly the factor of the iodine solution is determined. Then, the iodine solution is used as standart solution for determining the factor of thiosulfate solution.

10 ml of 0.1 N $I_2$ solution is titrated by 0.1 N $Na_2S_2O_3$ solution in the presence of 2 ml of starch solution until the blue colour dissepear.

$V_1 \text{ X } f_1 = V_2 \text{ X } f_2$ equation is used.